U.S. Army Corps of Engineers
Alaska District

SUPPLEMENT TO THE NORTHEAST CAPE HTRW REMEDIAL ACTIONS WORK PLAN

NORTHEAST CAPE
ST. LAWRENCE ISLAND, ALASKA

FUDS No. F10AK0969-03

FINAL
OCTOBER 2013

F10AK096903_07.11_0502_p
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APPENDICES

ACRONYMS AND ABBREVIATIONS

AAC	Alaska Administrative Code
ADEC	Alaska Department of Environmental Conservation
ALS	ALS Environmental
bgs	below ground surface
BTEX	benzene, toluene, ethylbenzene, and xylenes
CFR	Code of Federal Regulations
CoC	chain-of-custody
DL	detection limit
DoD	U.S. Department of Defense
DQO	data quality objective
DRO	diesel-range organics
EPA	U.S. Environmental Protection Agency
GPS	global positioning system
GRO	gasoline-range organics
HTRW	Hazardous, Toxic, or Radioactive Waste
IDW	investigation-derived waste
Jacobs	Jacobs Engineering Group Inc.
LCS	laboratory control sample
LCSD	laboratory control sample duplicates
LOD	limit of detection
LOQ	limit of quantitation
mg/kg	milligram per kilogram
µg/L	micrograms per liter
mg/L	milligrams per liter
MS	matrix spike
MSD	matrix spike duplicate
NE Cape	Northeast Cape
PAH	polycyclic aromatic hydrocarbons
PCB	polychlorinated biphenyls
POL	petroleum, oil, and lubricants
POP	project operating procedures
PPE	personal protective equipment
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
QSM	Quality Systems Manual

ACRONYMS AND ABBREVIATIONS (Continued)

RCRA	Resource Conservation and Recovery Act
RI	Remedial Investigation
RRO	residual-range organics
SDSFIE	spatial data standard for facilities, infrastructure, and environment
SOP	standard operating procedure
UFP-QAPP	Uniform Federal Policy-Quality Assurance Project Plan
USACE	U.S. Army Corps of Engineers

PROJECT OVERVIEW

The U.S. Army Corps of Engineers (USACE) has contracted Jacobs Engineering Group Inc. (Jacobs) to conduct sampling at three Northeast Cape (NE Cape) sites under the Hazardous, Toxic, or Radioactive Waste (HTRW) Contract No. W911KB-11-D-0005, Task Order 09. Site-specific sampling is intended to facilitate the first five-year review associated with those sites where the sampling will occur. This document is a supplement to the *Northeast Cape HTRW Remedial Actions Work Plan* (NE Cape Work Plan) (USACE 2013), and addresses additional sampling.

NE Cape is located on St. Lawrence Island, approximately 60 air miles from the Village of Savoonga, Alaska and 135 air miles from the city of Nome, Alaska (Figure A-1). Currently, fieldwork at NE Cape is managed according to the NE Cape Work Plan (USACE 2013) authored by Bristol Environmental Remediation Services, LLC (Bristol).

Jacobs will collect surface water samples at Kangukhsam Mountain Spring, ponds downgradient of Site 7 Landfill, and ponds and a stream downgradient of the Site 9 landfill. Additionally, two groundwater grab samples will be collected downgradient of the Site 7 and Site 9 landfills if sufficient groundwater is present.

This Supplement will obtain stakeholder approval and conduct fieldwork by following the procedures specified in the approved *2013 Northeast Cape HTRW Remedial Actions Work Plan*. Stakeholder approval and fieldwork will be completed prior to 2013 demobilization of the remote camp at NE Cape. Worksheet #9 of this Supplement outlines additional scoping meeting and consensus decisions reached regarding Jacobs fieldwork and technical approach.

This Supplement to the NE Cape Work Plan addresses the following:

- Adds a contractor conducting fieldwork (Jacobs)
- Identifies the locations of surface water samples and groundwater grab samples
- Provides the procedures for surface water and groundwater grab sample collection
- Adds information for the laboratory supporting the testing of samples (ALS Environmental [ALS])

ORGANIZATION OF THIS SUPPLEMENT

The NE Cape Work Plan will be the primary document under which fieldwork will be conducted (USACE 2013); only those worksheets that reflect changes necessary due to the addition of a contractor or procedure are included in this Supplement. For simplicity, worksheets that would require only a contractor name change were not updated. Table 1 summarizes the changes to the worksheets in the NE Cape Work Plan and indicates which worksheets are included in this supplement. Those worksheets that would only require a name change are applicable as originally written.

This Supplement contains the following sections:

- **Project Overview:** Provides the overall field sampling objectives.
- **Background:** Provides a brief description and reference to site information
- **Field Objectives:** Outlines the task planned for field sampling
- **Field Sampling:** Details the technical approach for sampling activities
- **Land Survey:** Details the technical approach for survey activities
- **Waste Management and Decontamination:** Details the handling and management of contaminated material and waste generated as part of this field sampling effort.
- **Worksheets 1 through 37, as applicable**: Only those worksheets that are revised are included in this supplement. The original style of UFP-QAPP worksheets used is consistent with those provided in the *Northeast Cape HTRW Remedial Actions Work Plan* (USACE 2013).
- **Appendix A – Figures:** Site figures identifying the general site location and proposed sampling locations.
- **Appendix B – Field Standard Operating Procedures (SOP):** Provides SOPs to be followed for the proper execution of field activities.
- **Appendix C – Laboratory Certificates and SOPs:**
  - Current Environmental Laboratory Accreditation Program and Alaska Department of Environmental Conservation (ADEC) certificates
  - Laboratory analytical SOPs specific to this task order.
- **Appendix D – Responses to Comments:** Presents comments on the draft version of the Work Plan and responses to the comments.

The Accident Prevention Plan and Site Safety and Health Plan included in the NE Cape Work Plan was reviewed and found to be in compliance with the Code of Federal Regulations, Title 40 (40 CFR) USACE EM 385-1-1. Bristol's Accident Prevention Plan and Site Safety and Health Plan will be followed while working at Bristol's remote camp.

Table 1
Summary of updated QAPP Worksheet Changes

Worksheet #	Worksheet Excluded (no change)	Worksheet Included	Summary of Change
1		X	Changed title for the supplement; added preparer's name.
2		X	Updated title, contractor name, and number.
3		X	Distribution list updated for project managers, clients, etc. Document Control Number updated.
4		X	Personnel sign-off sheet for Jacobs.
5		X	Project organization chart for Jacobs and new subcontractors.
6		X	Communication pathways updated with Jacobs' procedures. Added new analytical laboratory subcontractor.
7		X	Personnel responsibilities for Jacobs.
8		X	Personnel training requirements for Jacobs.
9		X	The scoping session held on 12 August 2013 was added.
10		X	The problem statement was modified to focus on surface water and groundwater grab sampling.
11		X	Tasks were modified to reflect focus on surface water and groundwater grab sampling.
12	X		No changes.
13	X		No changes.
14		X	Updated for Jacobs tasks
15		X	Updated for new analytical laboratory.
16		X	Updated for Jacobs' portion of the schedule.
17		X	Sampling rationale updated for focus on surface water and groundwater grab sampling.
18		X	Updated to include new sample locations, matrices, and rationale.
19		X	Updated for new analytical laboratory.
20		X	The number of quality control (QC) samples has been updated based on the new sampling approach.
21		X	Changed SOP references to current Jacobs SOPs. Added SOP for drilling/core logging and soil sampling.

Table 1
Summary of updated QAPP Worksheet Changes (Continued)

Worksheet #	Worksheet Excluded (no change)	Worksheet Included	Summary of Change
22		X	Modified YSI and survey equipment details to match Jacobs SOPs.
23		X	Updated for new analytical laboratory.
24		X	Updated for new analytical laboratory.
25		X	Updated for new analytical laboratory.
26	X		No changes.
27	X		No changes.
28	X		No changes.
29	X		No changes.
30		X	Updated for new analytical laboratory.
31	X		No changes.
32	X		No changes.
33	X		Jacobs chemist will conduct data validation and prepare the validation report for this effort.
34	X		No changes.
35	X		No changes.
36		X	The Verifications Summary was updated to be performed by Jacobs.
37		X	Data Usability and Chemical Data Assessment – Jacobs will self-perform.
Appendix A		X	Changed to include figures.
Appendix B		X	Includes Jacobs SOPs.
Appendix C		X	Includes ALS Kelso laboratory certificates and analytical SOPs.
Appendix D		X	Includes comments on the draft Work Plan, and responses to the comments.

Note:
For definitions, see the Acronyms and Abbreviations Section.

BACKGROUND

NE Cape is located at the northeast tip of St. Lawrence Island in the Bering Sea off the western coast of Alaska. Remedial activities at many sites are ongoing at the time of this Supplement. A detailed site description and a narrative of previous investigations are provided in the *Northeast Cape HTRW Remedial Actions Work Plan* (USACE 2013).

FIELD OBJECTIVES

The field objectives for Jacobs are outlined below:

- **Kangukhsam Mountain Spring:** Collect one surface water samples for analysis of GRO; benzene, toluene, ethylbenzene, and xylenes (BTEX); diesel-range organics (DRO), residual-range organics (RRO), polycyclic aromatic hydrocarbons (PAH), polychlorinated biphenyls (PCB); eight Resource Conservation and Recovery Act (RCRA) metals; and zinc.
- **Site 7 Landfill:** Collect surface water samples at three locations and one groundwater grab sample for analysis of GRO, BTEX, DRO, RRO, PAHs, PCBs, eight RCRA metals, nickel, and zinc.
- **Site 9 Landfill:** Collect surface water samples at three nearby locations and one groundwater grab sample for analysis of GRO, BTEX, DRO, RRO, PAHs, PCBs, eight RCRA metals, and zinc.
- Collect survey data for each sample location
- Transfer investigation-derived waste (IDW) to Bristol at the conclusion of fieldwork for transportation and disposal.

FIELD SAMPLING

This section details the technical approach and planned activities at NE Cape including surface water sampling and groundwater grab sampling. Samples will be collected to evaluate the condition of surface water and groundwater in the specific sample collection locations and to support the first Five Year Record of Decision Review at NE Cape.

Surface Water Sampling

Surface water sampling areas were reviewed with the USACE and ADEC and confirmed at a scoping meeting outlined on Worksheet #9 in this Supplement. Surface water samples will be collected from three sites; Kangukhsam Mountain Spring, near Site 7 Landfill, and near Site 9 Landfill. The Kangukhsam Mountain Spring is generally located south of the gravel borrow area along the east side of the road (Figure A-3). The spring's location will be verified with the seasonal residents of the Native Village of Northeast Cape and the onsite USACE Quality Assurance (QA) Representative prior to sample collection. The proposed location of the Site 7 Landfill samples (Figure A-4) and Site 9 Landfill samples (Figure A-5) will be confirmed with the onsite USACE QA Representative prior to sample collection.

Surface water samples will be collected directly from the surface water body slightly below the surface of the water directly adjacent to the shoreline in accordance with the procedures detailed in JE-SOP-4100 *Surface Water or Wastewater Sampling* (Appendix B). In general, an unused disposable 300-milliliter (mL) Teflon® dipper will be utilized to collect the surface water. Initially, a reading of the field parameters listed on Worksheet #14 will be recorded for the water body. Once field parameters are recorded, surface water will be collected and then transferred to the sample containers provided by the laboratory. A pin flag will be placed at the sampling location and photographs of the area will be taken. The surface water sample location will be surveyed prior to removing the pin flag. Observations made by field staff at the time of sampling and the field parameter readings will be recorded in the field logbook. Samples will be analyzed by the offsite laboratory for the test parameters listed on Worksheet #14.

Groundwater Grab Sampling

Groundwater grab samples will be collected at two downgradient locations near the Site 9 Landfill in accordance with the procedures detailed in JE-SOP-4010 *Groundwater Grab Sampling* (Appendix B). The groundwater in the 6-inch to 24-inch below ground surface (bgs) interval will be accessed using a screened drive point. The drive point will be advanced using hand tools. Once the drive point is in place, new ¼-inch inside diameter polyethylene tubing

will be inserted into the screen and attached to a peristaltic pump. Although no development or stabilization of the sampling point is planned, limited purging will be attempted to clear sediment if groundwater flow into the screen is at least 0.25 liters per minute without purging dry. If the groundwater grab sampling point does not produce water or support a flow rate of 0.25 liters per minute without purging dry, the USACE and ADEC project manager will be notified. Any groundwater present in low-producing well points will be added to sample containers in a one-time attempt to obtain samples prior to discontinuing groundwater grab sampling.

Assuming sufficient groundwater is produced by the screened drive point, field parameters listed on Worksheet #14 will be recorded prior to sample collection. Observations made by field staff at the time of sampling and the field parameter readings will be recorded in the field logbook and on the groundwater sampling form. Once field parameters are recorded, groundwater will be collected into the sample containers provided by the laboratory. Photographs of the area will be taken. The groundwater sample location will be surveyed prior to removing the screened drive point. Samples will be analyzed by the offsite laboratory for the test parameters listed on Worksheet #14.

LAND SURVEY

Sample locations will be surveyed in accordance with Jacobs JE-SOP-1000 *Global Positioning System (GPS) Land Surveying* (Appendix B) and the USACE Alaska District *Manual for Electronic Deliverables* (USACE 2011). Land survey equipment may include a real-time kinematic GPS (Leica Viva, Trimble® R8, or equivalent). Survey data will be reported with positional data in the Universal Transverse Mercator Zone 6 projection with the World Geodetic System 1984 datum. Vertical data will be reported as orthometric heights in North American Vertical Datum of 1988. Data will be reported in meters.

A control network will be established by obtaining monument and control point locations from National Geodetic Survey data sheets or a Continuously Operating Reference Station. If these are not available, a temporary control point will be set and referenced using a National

Oceanic and Atmospheric Administration Online Positioning User Service correction. Data collection procedures, data QA/quality control (QC) procedures, and reporting requirements are outlined in Jacobs JE-SOP-1000 *Global Positioning System Surveying* (Appendix B).

Features will be collected using a pre-loaded Spatial Data Standard for Facilities, Infrastructure, and Environment (SDSFIE) 3.0-compliant code list as specified in JE-SOP-1000 *Global Positioning System Surveying* (Appendix B). A key for shorthand names will be included in the survey logbook. Horizontal and vertical locations of control points and sample locations will be collected to sub 1.0-meter (3.28-foot) accuracy. Top of casing elevations for groundwater grab sampling screened drive points will not be measured. Temporary drive points will be removed at the conclusion of sampling activities and will be replaced with survey lathe to mark their locations.

A data summary table generated in accordance with the procedures described above will be included as an appendix to the sampling report.

WASTE MANAGEMENT AND DECONTAMINATION

This section addresses management of anticipated waste streams generated by the site investigation. Waste management will follow all appropriate procedures mandated by the state and federal governments. Waste generated by investigation activities is expected to include IDW (e.g., used personal protective equipment [PPE], disposable sampling equipment etc.), wastewater, and general refuse. Although all waste is expected to be non-hazardous in nature, known or suspected contaminants of concern at the site include petroleum, oil, and lubricants (POL), BTEX, PAHs, PCBs, and metals. This section discusses anticipated waste quantities, waste accumulation, and temporary storage.

Table 2 presents waste streams that may be generated during field activities. Similar waste streams will be consolidated with waste generated as part of remedial action activities and will be managed and disposed of at the designated disposal facility by Bristol in accordance with the NE Cape Work Plan (USACE 2013).

Table 2
Anticipated Waste Streams

Waste Type	Contaminant of Concern	Container	Estimated Quantity
Purge and Decontamination Wastewater (non-hazardous)	POL, BTEX, PAH, PCB, metals	Bucket	10 gallons
Solid IDW (non-hazardous)	POL, BTEX, PAH, PCB, metals	Trash bag	1 trash bag
General Refuse	None	Trash bag	1 trash bag

Notes
* IDW includes PPE, disposable sampling equipment, core sleeves, and other materials that were in contact with potentially contaminated soil or water.
For additional definitions, see the Acronyms and Abbreviations section.

Solid IDW generated during drive point installation and sampling activities will be comingled into a single waste stream. Waste will be containerized in a contractor trash bag and staged at the site temporarily until transported offsite by Bristol.

Reusable sampling equipment such as the meters used to measure water levels and field parameters will be decontaminated between sampling locations in accordance with JE-SOP-2000 *Decontamination* (Appendix B). Decontamination wastewater will be containerized in a bucket.

REFERENCES

ADEC (Alaska Department of Environmental Conservation. 2012 (April). *Oil and Other Hazardous Pollution Control Regulations—Discharge Reporting, Cleanup, and Disposal of Oil and Other Hazardous Substances*. Title 18 Alaska Administrative Code Section 75.

ADEC. 2010 (May). *Draft Field Sampling Guidance*. Division of Spill Prevention and Response. Contaminated Sites Program.

ADEC. 2009. *ADEC Environmental Laboratory Data and Quality Assurance Requirements Technical Memorandum*.

DoD (U.S. Department of Defense). 2010 (October). Q*uality Systems Manual for Environmental Laboratories*. DoD Environmental Quality Workgroup, Department of the Navy, Lead Service. Version 4.2, Final.

EPA (U.S. Environmental Protection Agency). 2010 (January). USEPA Contract Laboratory Program. *National Functional Guidelines for Inorganic Superfund Data Review*. http://www.epa.gov/superfund/programs/clp/download/ism/ism1nfg.pdf. Accessed 19 June 2013.

EPA. 2008 (June). USEPA Contract Laboratory Program. *National Functional Guidelines for Superfund Organic Methods Data Review*. http://www.epa.gov/superfund/programs/clp/download/somnfg.pdf. Accessed 19 June 2013.

USACE (U.S. Army Corps of Engineers). 2013 (June). *Northeast Cape HTRW Remedial Actions Work Plan*. Prepared by Bristol Environmental Remediation Services, LLC.

USACE. 2011 (October). *Manual for Electronic Deliverables*. USACE Alaska District.

QAPP WORKBOOK

WORKSHEET #1
TITLE AND APPROVAL PAGE

Site Name/Project Name:	Northeast Cape Formerly Used Defense Site/Five-Year Review Northeast Cape Formerly Used Defense Site
Site Location:	St. Lawrence Island, Alaska
Document Title:	Supplement to the Northeast Cape HTRW Remedial Actions Work Plan
Lead Organization:	U.S. Army Corps of Engineers (USACE)
Preparer's Name and Organizational Affiliation:	Kevin Maher, Jacobs Engineering Group Inc.
Preparer's Address, Telephone Number, and Email Address:	4300 B. St. #600 Anchorage, Alaska 99503 907-751-3429, Kevin.Maher@jacobs.com
Preparation Date (Day/Month/Year):	22 August 2013
Investigative Organization's Project Manager Signature:	
Printed Name/Organization:	Kevin Maher, Jacobs Engineering Group Inc.
Date:	
Lead Organization's Project Manager Signature:	
Printed Name/Organization:	Valerie Palmer, U.S. Army Corps of Engineers
Date:	
Approval Signature:	
Printed Name/Organization:	Curtis Dunkin, Alaska Department of Environmental Conservation (ADEC)
Date:	
Document Control Numbering System:	HTRW-J07-05F45902-J21-0002

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WORKSHEET #2
QAPP IDENTIFYING INFORMATION

Title:	Supplement to the Northeast Cape HTRW Remedial Actions Work Plan
Revision Number:	0
Revision Date:	August 2013
Site Name/Project Name:	Northeast Cape Formerly Used Defense Site/Five-Year Review Northeast Cape Formerly Used Defense Site
Site Location:	St. Lawrence Island, Alaska
Site Number/Code:	FUDS No. F10AK0969-03
Operable Unit:	Not Applicable
Contractor Name:	Jacobs Engineering Group Inc.
Contractor Number:	Not applicable
Contract Title:	Five-Year Review Northeast Cape Formerly Used Defense Site
Work Assignment Number:	W911KB-11-D-0005, Task Order 09

1. **Regulatory Program:** State of Alaska regulations (Alaska Oil and Hazardous Substance Pollution Control Act), Alaska Administrative Code, Title 18, Chapter 75 (18 AAC 75), EPA Code of Federal Regulations, Title 40 Part 761 (40 CFR 761), and Comprehensive Environmental Response, Compensation, and Liability Act Executive Order 12316 as amended by the Superfund Amendments and Reauthorization Act of 1986.

2. **Approval Entity:** USACE/ADEC

3. **The QAPP is (select one):** ☐**Generic** ☒**Project Specific**

4. **Dates of scoping sessions:** 13 August 2013

5. **Dates and titles of QAPP documents written for previous site work, if applicable:**

Title	Approval Date
Northeast Cape HTRW Remedial Actions Work Plan	June 2013

6. **Organizational partners (stakeholders) and connection with lead organization:**

Partners	Connection
USACE	Technical oversight organization
USACE	Lead organization
ADEC	State regulatory agency

7. **Data users:** Same as above under number 6.

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WORKSHEET #3
DISTRIBUTION LIST

This worksheet lists the recipients of the approved QAPP and subsequent QAPP revisions, addenda, and amendments.

QAPP Recipients	Title	Organization	Telephone Number	Email Address	Document Control Number
Valerie Palmer	Project Manager	USACE	907-753-2578	valerie.palmer@usace.army.mil	HTRW-J07-05F45902-J21-0002
Curtis Dunkin	Project Manager	ADEC	907-269-3053	curtis.dunkin@alaska.gov	HTRW-J07-05F45902-J21-0002
Kevin Maher	Project Manager	Jacobs	907-751-3429	kevin.maher@jacobs.com	HTRW-J07-05F45902-J21-0002

Note:
For definitions, refer to the Acronyms and Abbreviations section.

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WORKSHEET #4
PROJECT PERSONNEL SIGN-OFF SHEET

The following table will be signed and dated 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. Each organization will forward signed sheets to the central project file.

Project Personnel	Title	Telephone Number	Signature	Date
Kevin Maher	Jacobs Project Manager	907-751-3429		
Katie Bloom	Jacobs Project Engineer	907-751-3360		
David Summerville	Jacobs Project Chemist	907-751-3342		

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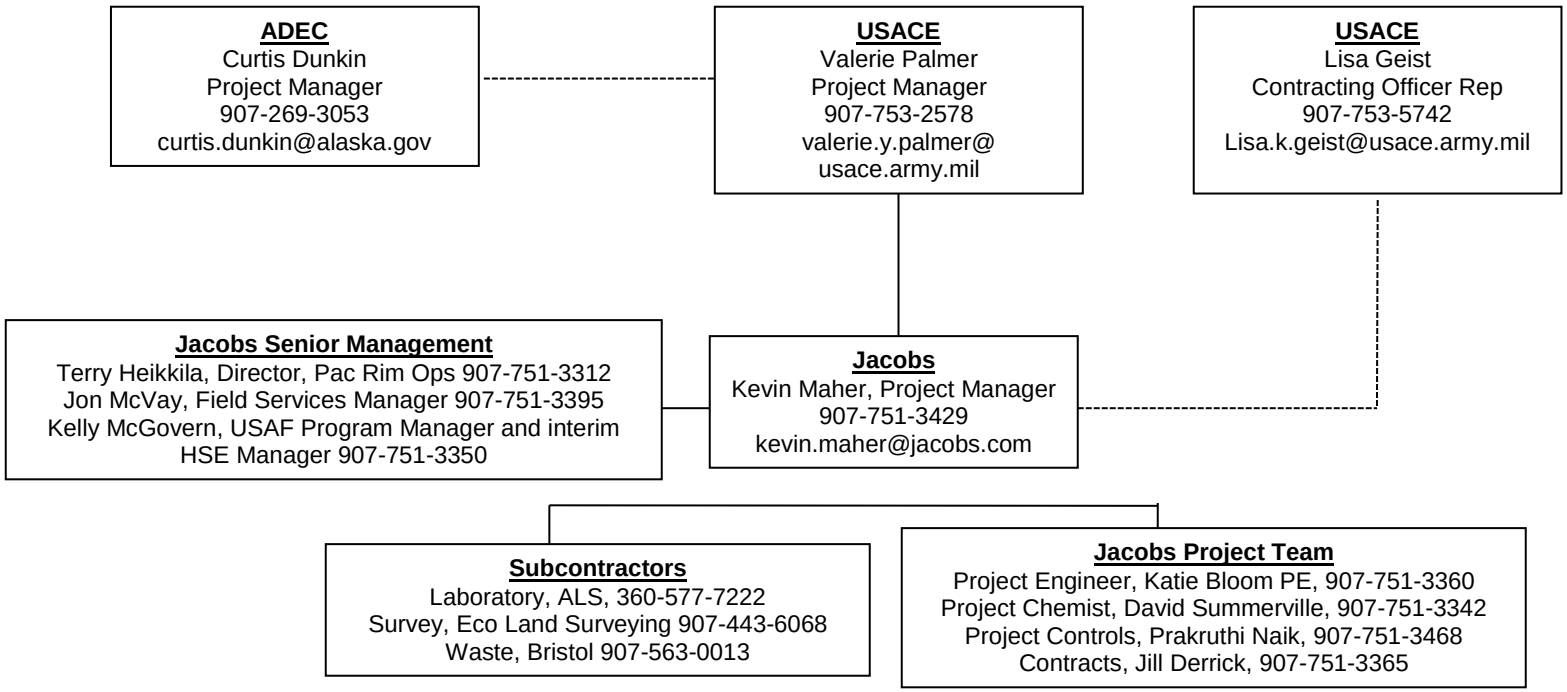
WORKSHEET #5
PROJECT ORGANIZATIONAL CHART

The following chart identifies relationships between all organizations involved in the project, including the lead organization and all contractor and subcontractor organizations. The names and phone numbers of all project managers, project team members, and/or project contacts are provided for each organization providing field sampling, on-site and off-site analysis, and data review services.



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WORKSHEET #6
COMMUNICATION PATHWAYS

The following table describes the communication pathways and modes of communication that will be used during the project after the QAPP supplement has been approved. The communications pathways presented below define the procedures for soliciting and/or obtaining approval between project personnel, between different contractors, and between samplers and laboratory staff. These procedures should be followed when any approved project activity requires real-time modifications to achieve project goals or a QAPP amendment must be obtained. Procedures for stopping work and responsible entities should also be identified.

Communication Drivers	Responsible Entity	Name	Phone Number	Email address	Procedure (Timing, Pathways, etc.)
Point of contact with ADEC Stakeholders during project execution	USACE Project Manager	Valerie Palmer	907-552-7893	valerie.y.palmer@usace.army.mil	All technical, QA, and decision-making matters concerning the execution of the project (verbal, written, or electronic).
Amendments to the QAPP	USACE Project Manager	Valerie Palmer	907-552-7893	valerie.y.palmer@usace.army.mil	Coordination of QAPP amendment dialogue. ADEC, and EPA will be notified of significant corrective actions by phone, email, or fax within one week.
Contracting issues	USACE Contracting Officer Representative	Lisa Geist	907-753-5742	lisa.k.geist@usace.army.mil	First point of contact for contract issues. Only the Contracting Officer can change contract terms and give stop work notice.
Planning and preparatory communication and meetings	Jacobs Project Manager	Kevin Maher	907-751-3429	kevin.maher@jacobs.com	Facilitate readiness review, pre-construction meeting, and status meetings with client and stakeholders as appropriate.
Change in field conditions	Jacobs Project Manager	Kevin Maher	907-751-3429	kevin.maher@jacobs.com	Coordination with USACE, and ADEC to adjust approach if necessary. Contact will generally be initiated by the end of the next business day.

Worksheet #6 – Communication Pathways (Continued)

Communication Drivers	Responsible Entity	Name	Phone Number	Email address	Procedure (Timing, Pathways, etc.)
Change to fieldwork	Jacobs Site Lead	Kevin Maher	907-751-3429	kevin.maher@jacobs.com	Notify Jacobs Project Manager of fieldwork issue by close of business the next business day.
	Jacobs Project Manager	Kevin Maher	907-751-3429	kevin.maher@jacobs.com	Notifies USACE Project Manager and USACE Contracting Officer Representative generally by the end of the next business day.
	USACE Contracting Officer	Christine Dale	907-753-5618	christine.a.dale@usace.army.mil	Evaluates the situation based on information provided by USACE Project Manager and USACE Contracting Officer Representative and selects the recommended solution or an alternate solution.
Analytical data quality issues	Jacobs Project Chemist	David Summerville	907-751-3342	david.summerville@jacobs.com	Identifies any data quality issues and works with the laboratory Project Manager to resolve issues, notifies Jacobs Project Manager of the issue and resolution. If there is a significant data quality issue, the Project Chemist may immediately notify the Jacobs Project Manager and the Field Team Leader.
	ALS Laboratory Project Manager	Greg Salata	360-501-3376	gregory.salata@alsglobal.com	Identifies data quality issues and provides solutions for the Jacobs Project Chemist to resolve the issue.
Analytical data quality issues (continued)	Jacobs Project Manager	Kevin Maher	907-751-3429	kevin.maher@jacobs.com	Notifies USACE Project Manager and USACE Chemist of data quality issues and resolution within one week.

Worksheet #6 – Communication Pathways (Continued)

Communication Drivers	Responsible Entity	Name	Phone Number	Email address	Procedure (Timing, Pathways, etc.)
	ADEC Project Manager	Curtis Dunkin	907-269-3053	curtis.dunkin@alaska.gov	Evaluates whether actions to resolve data quality issues are adequate, with input from the USACE Project Manager and Jacobs Project Manager, if necessary.
Accident or unplanned field event	Jacobs Site Lead	Kevin Maher	907-751-3386	kevin.maher@jacobs.com	Notify USACE by phone within 24 hours.
Stop work/initiate corrective action	Jacobs Site Lead/On-site Personnel	Kevin Maher	907-751-3386	kevin.maher@jacobs.com	Stops work and notifies Jacobs Project Manager by the end of the next business day.
	Jacobs Project Manager	Kevin Maher	907-751-3429	kevin.maher@jacobs.com	Notifies USACE Project Manager by the end of the next business day. Recommends corrective action. May consult with other Jacobs staff as appropriate.
	USACE Contracting Officer Representative	Lisa Geist	907-753-5742	lisa.k.geist@usace.army.mil	First point of contact for contract issues. She will advise whether to copy the contracting officer.
Change Orders	USACE Contracting Officer	Christine Dale	907-753-5618	christine.a.dale@usace.army.mil	Only the Contracting Officer can change the contractual elements of this task order

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WORKSHEET #7
KEY PERSONNEL RESPONSIBILITIES AND QUALIFICATIONS TABLE

The following table identifies Jacobs project personnel associated with the field effort. Resumes for key personnel are located at the Jacobs Anchorage office.

Name	Organizational Affiliation	Title	Responsibilities	Education and Experience Qualifications
Kevin Maher	Jacobs	Project Manager	Coordinate day-to-day project activities, oversee technical and logistic aspects of the project, resolve issues, development, and maintenance of detailed project schedule, review all reports before submittal to client/customer, and represent the project team at meetings.	B.S. Chemistry, 22 years of experience in the environmental field including chemistry, environmental sampling, and project management.
Kevin Maher	Jacobs	Site Lead and Site Safety and Health Officer	Responsible for implementation of the Work Plan Supplement and Site Health and Safety Plan. Also responsible for day-to-day field coordination, activities, procedures, and modifications.	B.S. Chemistry, 22 years of experience in chemistry, environmental projects, and project management.
Katie Bloom	Jacobs	Project Engineer	Responsible for engineering review of the Work Plan Supplement and Site Health and Safety plan to ensure preparation of the plans in accordance with applicable requirements.	B.S. Biological Systems Engineering, 9 years of experience in environmental projects, registered professional civil engineer, AK CE12455.
David Summerville	Jacobs	Project Chemist	Coordinate with the analytical laboratory, review data, and ensure that data quality objectives are met.	M.S. Chemistry, 7 years of environmental experience including chemistry and environmental sampling.

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WORKSHEET #8
SPECIAL PERSONNEL TRAINING REQUIREMENTS TABLE

This table provides an overview of specialized training required for this project and specifies where training records and certificates can be located.

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
Jacobs field personnel	40 Hour HAZWOPER (current 8 hour refresher) 29 CFR1910.120(e)	Environmental Management, Inc., Jacobs	Initial and annual refresher	All field personnel	All personnel, Jacobs Drilling Subcontractor	Project files
Jacobs Site Lead Site Safety and Health Officer	30 Hour OSHA Construction Safety and Health	Environmental Management, Inc. or Jacobs	Initial	Jacobs Site Lead Site Safety and Health Officer	Jacobs	Project files
Jacobs field personnel and Subcontractor Supervisors	First Aid certification	Red Cross of Alaska, Total Safety	Every 2 years	All field personnel	All personnel, Jacobs, Drilling Subcontractor	Project files
	Adult CPR/AED certification		Every 2 years			
	Bloodborne pathogens training		Annual			

Notes:
AED = automated external defibrillator
CPR = cardiopulmonary resuscitation
HAZWOPER = Hazardous Waste Operations and Emergency Response Standard
OSHA = Occupational Safety and Health Administration
For additional definitions, refer to the Acronyms and Abbreviations section.

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WORKSHEET #9
PROJECT SCOPING SESSION PARTICIPANTS SHEET

Each worksheet documents a scoping session held for the project and identifies team members responsible for planning the project.

Project Name:	Five-Year Review Northeast Cape Formerly Used Defense Site
Site Name:	Kangukhsam Mountain Spring, Site 7 Landfill, and Site 9 Landfill
Projected Date(s) of Sampling:	11 September 2013 – 17 September 2013
Site Location:	St. Lawrence Island, Alaska
Project Manager:	Kevin Maher
Date of Session:	13 August 2013
Scoping Session Purpose:	Introduce the intent to supplement the current QAPP to meet the accelerated schedule and confirm sampling locations and discuss the role the data will play in the Five Year Review.

Name	Title	Affiliation	Phone Number	Email Address
Valerie Palmer	Project Manager	USACE	907-753-2578	valerie.y.palmer@usace.army.mil
Lisa Geist	Contacting Officer Representative	USACE	907-753-5742	lisa.k.geist@usace.army.mil
Aaron Shewman	Project Engineer	USACE	907-753-5558	aaron.f.shewman@usace.army.mil
Curtis Dunkin	Project Manager	ADEC	907-269-3053	curtis.dunkin@alaska.gov
Kevin Maher	Project Manager	Jacobs	907-751-3429	kevin.maher@jacobs.com
Katie Bloom	Project Engineer	Jacobs	907-751-3360	katie.bloom@jacobs.com

Project Name:	Five-Year Review Northeast Cape Formerly Used Defense Site
Comments:	All agreed that a supplement to the Work Plan is an acceptable approach for the Jacobs sampling effort the support the five year review.
Action Items:	Kevin Maher will provide an electronic version of the draft Supplement to the area stakeholders as early as possible.
Consensus Decisions:	All agreed that a supplement to the Work Plan is an acceptable approach for the Jacobs sampling effort. An electronic distribution-only version of the draft Supplement to stakeholders is needed for review. If groundwater grab sampling locations do not produce groundwater or if they will not support a flow rate of 0.25 liters per minute, groundwater grab sampling will be discontinued.

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WORKSHEET #10
PROBLEM DEFINITION

This worksheet defines the problem and the environmental questions to be answered for the current project. Project decision "if…, then…" statements should be included that link data results with possible actions.

Prompt	Information
The problem to be addressed by the project:	Exposure to contaminants of potential concern is an ongoing concern to land owners and local residents. The USACE has received public comments requesting that an evaluation of surface water and groundwater occur for the presence of contaminants of potential concern. This sampling effort will determine if the contaminants of potential concern are present at the proposed sampling locations.
The environmental questions being asked:	Using ADEC 18 AAC 70 and 18 AAC 75 (Table C) criteria, are contaminants of potential concern present downgradient from landfills (Site 7 Landfill and Site 9 Landfill) in surface water and groundwater? Using ADEC 18 AAC 70 and 18 AAC 75 (Table C) criteria, are contaminants of potential concern present in Kangukhsam Mountain Spring?
Site history:	Multiple remedial investigations and removal activities have occurred at Northeast Cape sites. See *2013 Northeast Cape HTRW Remedial Actions Work Plan* for the discussion of site history.
The possible classes of contaminants and the affected matrices:	BTEX, GRO, DRO/RRO, PAHs, PCBs, and metals
The rationale for inclusion of chemical and nonchemical analyses:	BTEX, GRO, DRO/RRO, PAHs, PCBs, and metals are contaminants of potential concern at Northeast Cape sites based on historical sample data.
Project decision conditions ("If..., then..." statements):	If contaminants of potential concern are identified above the applicable criteria following this effort, then additional samples may need to be collected in the future to fully assess potential exposure.

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WORKSHEET #11
PROJECT QUALITY OBJECTIVES/SYSTEMATIC PLANNING PROCESS STATEMENTS

The following table summarizes project quality objectives (PQO) in terms of type, quantity, and quality of data determined using a systematic planning process. Project quality objectives are included in the form of qualitative and quantitative statements.

Who will use the data?	ADEC, USACE, and Jacobs
What will the data be used for?	The purpose of the surface water and groundwater grab sampling is to determine if the surface water and/or groundwater is affected by the contaminants of potential concern at Kangukhsam Mountain Spring, Site 7 Landfill, and Site 9 Landfill.
What types of data are needed? (target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques)	All samples will be analyzed by an offsite laboratory for GRO, BTEX, DRO, RRO, PAHs, PCBs, eight RCRA metals, and zinc. Site 7 Landfill samples will also be analyzed for nickel. Samples will be collected in accordance with the procedures identified in JE-SOP-4010 and JE-SOP-4100 (Appendix B).
How "good" do the data need to be in order to support the environmental decision?	Analytical data must meet the requirements established by the ADEC *Environmental Laboratory Data and Quality Assurance Requirements Technical Memorandum* (ADEC 2009). Laboratory analytical data must be of sufficient sensitivity and quality that results can be evaluated against the applicable regulatory criteria. Sample analysis will be performed in accordance with the U.S. Department of Defense (DoD) Quality Systems Manual (QSM), v.4.2 (DoD 2010). Data quality and detection limits must be adequate to evaluate the analytical results against ADEC action levels.
How much data are needed? (number of samples for each analytical group, matrix, and concentration)	Number of samples, analytical methods, matrices, and concentrations are summarized in Worksheets #18 and #20.
Where, when, and how should the data be collected/generated?	Field sampling is scheduled to occur in September 2013. Samples will be collected from the locations indicated on Figure A-3, Figure A-4, and Figure A-5. A project timeline is presented in Worksheet #16. Data will be collected according to the SOPs in Appendix B and generated according to laboratory SOPs in Appendix C.
Who will collect and generate the data?	Jacobs will collect all samples and generate field measured parameters. ALS Kelso will generate offsite analytical results. A licensed surveyor (Eco Land Surveying) will collect and generate land survey data.

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

How will the data be reported?	A summary of the data will be included in the draft and final versions of the sampling report. Reports will be provided electronically and in hard copy. Analytical data will be reported by the laboratory in Staged Electronic Data Deliverable Version 5.2 Stage 2a format and a Level IV laboratory data package in searchable portable document format.
How will the data be archived?	The USACE and ADEC will archive the data according to their procedures. Jacobs will maintain all project data until the end of the task order period of performance.

WORKSHEET #14
SUMMARY OF PROJECT TASKS

This worksheet provides a brief overview of the project activities, including sampling, analysis, and data management tasks.

Sampling Tasks:	Collecting surface water and groundwater grab samples.
Analysis Tasks:	All samples will be field measured for the following: Temperature pH Dissolved oxygen Conductivity Oxidation-reduction potential Turbidity All samples will be analyzed by an offsite laboratory for the following: GRO by AK101 BTEX by SW8260 DRO/RRO by AK102/103 PAHs by SW8270 SIM PCBs by SW8082 Metals by SW6020/SW7470
Quality Control Tasks:	Field quality control (QC) samples will include trip blanks, temperature blanks, field duplicates, and matrix spike (MS) samples. Field QC samples will be submitted at the following frequencies: • Trip blanks: one per cooler containing volatile samples. • Temperature blanks: one per cooler. • Field duplicates: one field duplicate per ten samples. • Matrix spikes: one MS/MS duplicate (MSD) sample set per 20 samples. • Laboratory QC samples will include method blanks, laboratory control samples, surrogate spikes, and laboratory replicate samples. Laboratory QC samples will be prepared and analyzed at the frequency specified by the DoD *QSM* v.4.2 (QSM) and the analytical methods. All field QC samples will be handled, documented, packaged, and shipped according to the procedures in Worksheet #27 of the *Northeast Cape HTRW Remedial Actions Work Plan* (USACE 2013).
Waste Handling Tasks:	IDW will be containerized and transferred to Bristol for offsite transportation to an appropriate disposal facility.
Secondary Data:	See Worksheet #13.

Worksheet #14 – Summary of Project Tasks (Continued)

Data Management Tasks:	Analytical laboratory data will be reviewed for quality using ADEC checklists per sample delivery group, and presented in a Remedial Investigation (RI) report. Analytical laboratory, survey, and geospatial data will be managed utilizing *Manual for Electronic Deliverables* (USACE 2011), and SDSFIE 3.0 specifications.
Documentation and Records:	The possession and handling of individual samples must be traceable from the time of sample collection until the time the analytical laboratory reports the results of the analysis to the appropriate parties. Sample documentation and record keeping for all samples collected as part of this project include the following: • Sample collection documentation (field logbook and field sampling forms) • Photographs • Sample Identification • Chain-of-custody (CoC) records and sample summary tables • Airway bills • Sample packaging (cooler custody seals) and shipping • Surveying field notes and spatial data • Records to be produced include: • Sampling Report and Chemical Data Quality Assessment
Assessment/Audit Tasks:	Iterative field sampling technical evaluations will be conducted throughout the sampling event.
Data Review Tasks:	The analytical data will be reviewed by the Jacobs Project Chemist. The evaluation will include a review of the following: • CoC and sample receipt records • Laboratory case narratives • Laboratory data including analytical methodology; sample holding times; laboratory blanks; detection limits (DL) and limits of quantitation (LOQ); surrogate recoveries; MS/MSD recoveries; and precision. The analytical data will be evaluated for compliance with project-specific data quality objectives (DQO). The most current version of DoD QSM v. 4.2 (DoD 2010), method criteria, laboratory criteria, and best professional judgment will apply, in that order. The Data Quality Assessment will identify any data requiring qualification and identify effects on data usability. Data Qualifiers identified in the Statement of Work will be utilized for the chemical data quality assessment and are presented in Worksheet #37.

WORKSHEET #15
REFERENCE LIMITS AND EVALUATION TABLE

The following tables identify the target analytes/contaminants of concern and project-required action limits for each matrix, analytical group, and concentration level. The tables also specify the achievable detection limits, limits of detection (LOD), and limits of quantitation (LOQ) for each analyte.

Matrix: Groundwater
Analytical Group: AK101, AK102/3
Concentration Level: Low – High

Analyte	CAS Number	Project Evaluation Level (mg/L)[1]	Project Screening Limit (mg/L)	Achievable Laboratory Limits (mg/L)[2]		
				DL	LOD	LOQ
Gasoline-range organics	8006-61-9	1.3	0.1	0.013	0.025	0.05
Diesel-range organics	68334-30-5	1.5	0.1	0.011	0.02	0.8
Residual-range organics	Not applicable	1.1	0.1	0.019	0.05	0.5

Notes:
[1] Project Evaluation Level derived from Table 15-3 of the 2013 QAPP (USACE 2013) using the cleanup levels from "Cleanup levels from 2009 Decision Document" column.
[2] Achievable DLs, LODs, and LOQs are provided by ALS.
Project screening level set to 1/10 of the project evaluation level
CAS = Chemical Abstract Service
DL = Detection limit
LOD = Limit of detection
LOQ = Limit of quantitation
For additional definitions, see the Acronyms and Abbreviations section.

Worksheet #15 – Reference Limits and Evaluation Table (Continued)

Matrix: Groundwater
Analytical Group: Polychlorinated Biphenyls
Concentration Level: Low – High

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Project Screening Limit (µg/L)	Achievable Laboratory Limits (µg/L)[2]		
				DL	LOD	LOQ
PCB-1016 (Aroclor 1016)	12674-11-2	0.5	0.005	0. 0021	0.005	0.005
PCB-1221 (Aroclor 1221)	11104-28-2	0.5	0.005	0. 0021	0.005	0.01
PCB-1232 (Aroclor 1232)	11141-16-5	0.5	0.005	0. 0021	0.005	0.005
PCB-1242 (Aroclor 1242)	53469-21-9	0.5	0.005	0. 0021	0.005	0.005
PCB-1248 (Aroclor 1248)	12672-29-6	0.5	0.005	0. 0021	0.005	0.005
PCB-1254 (Aroclor 1254)	11097-69-1	0.5	0.005	0. 0021	0.005	0.005
PCB-1260 (Aroclor 1260)	11096-82-5	0.5	0.005	0. 0021	0.005	0.005

Notes:
[1] Project Evaluation Level derived from Table 15-3 of the 2013 QAPP (USACE 2013) using the "Evaluation Criteria from 18 AAC 75 table C" column
[2] Achievable DLs, LODs, and LOQs are provided by ALS.
Project screening level set to 1/10 of the project evaluation level
CAS = Chemical Abstract Service
DL = Detection limit
LOD = Limit of detection
LOQ = Limit of quantitation
For additional definitions, see the Acronyms and Abbreviations section.

Worksheet #15 – Reference Limits and Evaluation Table (Continued)

Matrix: Groundwater
Analytical Group: BTEX
Concentration Level: Low – High

Analyte	CAS Number	Project Action Limit (mg/L)[1]	Project Screening Limit (mg/L)	Achievable Laboratory Limits (mg/L)[2]		
				DL	LOD	LOQ
Benzene	71-43-2	0.005	0.0005	0.000062	0.0001	0.0005
Ethylbenzene	100-41-4	0.7	0.07	0.00005	0.0001	0.0005
m,p-Xylenes	179601-23-1	10	1	0.00011	0.0002	0.0005
o-Xylene	95-47-6	10	1	0.000074	0.0002	0.0005
Toluene	108-88-3	1.0	0.1	0.000054	0.0001	0.0005

Notes:
[1] Project Evaluation Level derived from Table 15-3 of the 2013 QAPP (USACE 2013) using the most stringent of the "Cleanup Level from 2009 decision document" or the "Evaluation Criteria from 18 AAC 75 table C" columns
[2] Achievable DLs, LODs, and LOQs are provided by ALS.
Project screening level set to 1/10 of the project evaluation level
CAS = Chemical Abstract Service CAS = Chemical Abstract Service
DL = Detection limit
LOD = Limit of detection
LOQ = Limit of quantitation
For additional definitions, see the Acronyms and Abbreviations section.

Worksheet #15 – Reference Limits and Evaluation Table (Continued)

Matrix: Groundwater
Analytical Group: PAHs
Concentration Level: Low – High

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Project Screening Limit (µg/L)	Achievable Laboratory Limits (µg/L)[2]		
				MDL	LOD	LOQ
1-Methylnaphthalene	90-12-0	150	15	0.0035	0.005	0.02
2-Methylnaphthalene	91-57-6	150	15	0.0023	0.005	0.02
Acenaphthene	83-32-9	2,200	220	0.0044	0.005	0.02
Acenaphthylene	208-96-8	2,200	220	0.0034	0.005	0.02
Anthracene	120-12-7	11,000	1,100	0.0036	0.005	0.02
Benz(a)anthracene	56-55-3	1.2	0.12	0.0026	0.005	0.02
Benzo(a)pyrene	50-32-8	0.2	0.02	0.0043	0.005	0.02
Benzo(b)fluoranthene	205-99-2	1.2	0.1	0.0041	0.005	0.02
Benzo(g,h,i)perylene	191-24-2	1,100	110	0.0029	0.005	0.02
Benzo(k)fluoranthene	207-08-9	12	1.2	0.003	0.005	0.02
Chrysene	218-01-9	120	12	0.0034	0.005	0.02
Dibenzo(a,h)anthracene	53-70-3	0.12	0.06	0.0025	0.005	0.02
Fluoranthene	206-44-0	1,500	150	0.01	0.005	0.02
Fluorene	86-73-7	1,500	150	0.0038	0.005	0.02
Indeno(1,2,3-cd)pyrene	193-39-5	1.2	0.12	0.0026	0.005	0.02
Naphthalene	91-20-3	730	73	0.0038	0.005	0.02
Phenanthrene	85-01-8	11,000	1,100	0.0050	0.005	0.02
Pyrene	129-00-0	1,100	110	0.0053	0.005	0.02

Notes:
[1] Project Evaluation Level derived from Table 15-3 of the 2013 QAPP (USACE 2013) using the "Evaluation Criteria from 18 AAC 75 table C" column
[2] Achievable DLs, LODs, and LOQs are provided by ALS.
Project screening level set to 1/10 of the project evaluation level
CAS = Chemical Abstract Service
DL = Detection limit
LOD = Limit of detection
LOQ = Limit of quantitation
MDL = method detection limit
For definitions, see the Acronyms and Abbreviations section.

Worksheet #15 – Reference Limits and Evaluation Table (Continued)

Matrix: Groundwater
Analytical Group: RCRA Metal, Nickel, Zinc, and Mercury
Concentration Level: Low – High

Analyte	CAS Number	Project Action Limit (mg/L)[1]	Project Screening Limit (mg/L)	Achievable Laboratory Limits (mg/kg)[2]		
				DL	LOD	LOQ
Arsenic (total)	7440-38-2	0.01	0.001	0.0002	0.0002	0.0005
Arsenic (dissolved)	7440-38-2	0.01	0.001	0.0002	0.0002	0.0005
Barium (total)	7440-39-3	2	0.2	0.00002	0.00002	0.00005
Barium (dissolved)	7440-39-3	2	0.2	0.00002	0.00002	0.00005
Cadmium (total)	7440-43-9	0.005	0.0005	0.000007	0.000007	0.00002
Cadmium (dissolved)	7440-43-9	0.005	0.0005	0.000007	0.000007	0.00002
Total Chromium (total)	7440-47-3	0.1	0.01	0.00004	0.00004	0.0002
Total Chromium (dissolved)	7440-47-3	0.1	0.01	0.00004	0.00004	0.0002
Lead (total)	7439-92-1	0.015	0.0015	0.000005	0.000005	0.00002
Lead (dissolved)	7439-92-1	0.015	0.0015	0.000005	0.000005	0.00002
Selenium (total)	7782-49-2	0.05	0.005	0.0004	0.0004	0.001
Selenium (dissolved)	7782-49-2	0.05	0.005	0.0004	0.0004	0.001
Silver (total)	7440-22-4	0.1	0.01	0.000005	0.000005	0.00002
Silver (dissolved)	7440-22-4	0.1	0.01	0.000005	0.000005	0.00002
Mercury (total)	7439-97-6	0.002	0.0002	0.00002	0.00005	0.0002
Mercury (dissolved)	7439-97-6	0.002	0.0002	0.00002	0.00005	0.0002
Nickel (total)	7440-02-0	0.1	0.01	0.00002	0.00005	0.0002
Nickel (dissolved)	7440-02-0	0.1	0.01	0.00002	0.00005	0.0002
Zinc (total)	7440-66-6	5.0	0.5	0.0002	0.00025	0.00075
Zinc (dissolved)	7440-66-6	5.0	0.5	0.0002	0.00025	0.00075

Notes:
[1] Project Evaluation Level derived from Table 15-3 of the 2013 QAPP (USACE 2013) using the most stringent of the "Cleanup Level from 2009 decision document" or the "Evaluation Criteria from 18 AAC 75 table C" columns
[2] Achievable DLs, LODs, and LOQs are provided by ALS.
CAS = Chemical Abstract Service
DL = Detection limit
LOD = Limit of detection
LOQ = Limit of quantitation
For definitions, see the Acronyms and Abbreviations section

Worksheet #15 – Reference Limits and Evaluation Table (Continued)

Matrix: Surface Water
Analytical Group: AK101, AK102/3
Concentration Level: Low – High

Analyte	CAS Number	Project Evaluation Level (mg/L)[1]	Project Screening Limit (mg/L)	Achievable Laboratory Limits (mg/L)[2]		
				DL	LOD	LOQ
Gasoline-range organics	8006-61-9	No Sheen	0.1	0.013	0.025	0.05
Diesel-range organics	68334-30-5	No Sheen	0.1	0.011	0.02	0.8
Residual-range organics	Not applicable	No Sheen	0.1	0.019	0.05	0.5

Notes:
[1] Project Evaluation Level derived from Table 15-4 of the 2013 QAPP (USACE 2013) using the "Cleanup Levels from 2009 Decision Document" column.
[2] Achievable DLs, LODs, and LOQs are provided by ALS.
CAS = Chemical Abstract Service
DL = Detection limit
LOD = Limit of detection
LOQ = Limit of quantitation
For additional definitions, see the Acronyms and Abbreviations section.

Worksheet #15 – Reference Limits and Evaluation Table (Continued)

Matrix: Surface Water
Analytical Group: Polychlorinated Biphenyls
Concentration Level: Low – High

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Project Screening Limit (µg/L)	Achievable Laboratory Limits (µg/L)[2]		
				DL	LOD	LOQ
PCB-1016 (Aroclor 1016)	12674-11-2	0.5	0.005	0. 0021	0.005	0.005
PCB-1221 (Aroclor 1221)	11104-28-2	0.5	0.005	0. 0021	0.005	0.01
PCB-1232 (Aroclor 1232)	11141-16-5	0.5	0.005	0. 0021	0.005	0.005
PCB-1242 (Aroclor 1242)	53469-21-9	0.5	0.005	0. 0021	0.005	0.005
PCB-1248 (Aroclor 1248)	12672-29-6	0.5	0.005	0. 0021	0.005	0.005
PCB-1254 (Aroclor 1254)	11097-69-1	0.5	0.005	0. 0021	0.005	0.005
PCB-1260 (Aroclor 1260)	11096-82-5	0.5	0.005	0. 0021	0.005	0.005

Notes:
[1] Project Evaluation Level derived from Table 15-4 of the 2013 QAPP (USACE 2013) using the "Evaluation Criteria from 18 AAC 70" column
[2] Achievable DLs, LODs, and LOQs are provided by ALS.
Project screening level set to 1/10 of the project evaluation level
CAS = Chemical Abstract Service
DL = Detection limit
LOD = Limit of detection
LOQ = Limit of quantitation
For additional definitions, see the Acronyms and Abbreviations section.

Worksheet #15 – Reference Limits and Evaluation Table (Continued)

Matrix: Surface Water
Analytical Group: BTEX
Concentration Level: Low – High

Analyte	CAS Number	Project Action Limit (mg/L)[1]	Project Screening Limit (mg/L)	Achievable Laboratory Limits (mg/L)[2]		
				DL	LOD	LOQ
Benzene	71-43-2	0.005	0.0005	0.000062	0.0001	0.0005
Ethylbenzene	100-41-4	0.7	0.07	0.00005	0.0001	0.0005
m,p-Xylenes	179601-23-1	10	1	0.00011	0.0002	0.0005
o-Xylene	95-47-6	10	1	0.000074	0.0002	0.0005
Toluene	108-88-3	1.0	0.1	0.000054	0.0001	0.0005
Total Aromatic Hydrocarbons (sum of BTEX)	Not applicable	0.010	Not applicable	Not applicable	Not applicable	Not applicable

Notes:
[1] Project Evaluation Level derived from Table 15-3 of the 2013 QAPP (USACE 2013) using the ""Evaluation Criteria from 18 AAC 70" column
[2] Achievable DLs, LODs, and LOQs are provided by ALS.
Project screening level set to 1/10 of the project evaluation level
CAS = Chemical Abstract Service CAS = Chemical Abstract Service
DL = Detection limit
LOD = Limit of detection
LOQ = Limit of quantitation
For additional definitions, see the Acronyms and Abbreviations section.

Worksheet #15 – Reference Limits and Evaluation Table (Continued)

Matrix: Surface Water
Analytical Group: PAHs
Concentration Level: Low – High

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Project Screening Limit (µg/L)	Achievable Laboratory Limits (µg/L)[2]		
				MDL	LOD	LOQ
1-Methylnaphthalene	90-12-0	NA	0.02	0.0035	0.005	0.02
2-Methylnaphthalene	91-57-6	NA	0.02	0.0023	0.005	0.02
Acenaphthene	83-32-9	NA	0.02	0.0044	0.005	0.02
Acenaphthylene	208-96-8	NA	0.02	0.0034	0.005	0.02
Anthracene	120-12-7	NA	0.02	0.0036	0.005	0.02
Benz(a)anthracene	56-55-3	NA	0.02	0.0026	0.005	0.02
Benzo(a)pyrene	50-32-8	0.2	0.02	0.0043	0.005	0.02
Benzo(b)fluoranthene	205-99-2	NA	0.02	0.0041	0.005	0.02
Benzo(g,h,i)perylene	191-24-2	NA	0.02	0.0029	0.005	0.02
Benzo(k)fluoranthene	207-08-9	NA	0.02	0.003	0.005	0.02
Chrysene	218-01-9	NA	0.02	0.0034	0.005	0.02
Dibenzo(a,h)anthracene	53-70-3	NA	0.02	0.0025	0.005	0.02
Fluoranthene	206-44-0	NA	0.02	0.01	0.005	0.02
Fluorene	86-73-7	NA	0.02	0.0038	0.005	0.02
Indeno(1,2,3-cd)pyrene	193-39-5	NA	0.02	0.0026	0.005	0.02
Naphthalene	91-20-3	NA	0.02	0.0038	0.005	0.02
Phenanthrene	85-01-8	NA	0.02	0.0050	0.005	0.02
Pyrene	129-00-0	NA	0.02	0.0053	0.005	0.02
Total Aqueous Hydrocarbons (sum of PAHs)	Not applicable	15	Not applicable	Not applicable	Not applicable	Not applicable

Notes:
[1] Project Evaluation Level derived from Table 15-3 of the 2013 QAPP (USACE 2013) using the "Evaluation Criteria from 18 AAC 70" column
[2] Achievable DLs, LODs, and LOQs are provided by ALS.
Project screening level set to the limit of quantitation
CAS = Chemical Abstract Service
DL = Detection limit
LOD = Limit of detection
LOQ = Limit of quantitation
MDL = method detection limit
For definitions, see the Acronyms and Abbreviations section.

Worksheet #15 – Reference Limits and Evaluation Table (Continued)

Matrix: Surface Water
Analytical Group: RCRA Metal, Nickel, Zinc, and Mercury
Concentration Level: Low – High

Analyte	CAS Number	Project Action Limit (mg/L)[1]	Project Screening Limit (mg/L)	Achievable Laboratory Limits (mg/kg)[2]		
				DL	LOD	LOQ
Arsenic (total)	7440-38-2	0.01	0.001	0.0002	0.0002	0.0005
Arsenic (dissolved)	7440-38-2	0.01	0.001	0.0002	0.0002	0.0005
Barium (total)	7440-39-3	2	0.2	0.00002	0.00002	0.00005
Barium (dissolved)	7440-39-3	2	0.2	0.00002	0.00002	0.00005
Cadmium (total)	7440-43-9	0.005	0.0005	0.000007	0.000007	0.00002
Cadmium (dissolved)	7440-43-9	0.005	0.0005	0.000007	0.000007	0.00002
Total Chromium (total)	7440-47-3	0.1	0.01	0.00004	0.00004	0.0002
Total Chromium (dissolved)	7440-47-3	0.1	0.01	0.00004	0.00004	0.0002
Lead (total)	7439-92-1	Not applicable	0.00002	0.000005	0.000005	0.00002
Lead (dissolved)	7439-92-1	Not applicable	0.00002	0.000005	0.000005	0.00002
Selenium (total)	7782-49-2	0.05	0.005	0.0004	0.0004	0.001
Selenium (dissolved)	7782-49-2	0.05	0.005	0.0004	0.0004	0.001
Silver (total)	7440-22-4	NA	0.00002	0.000005	0.000005	0.00002
Silver (dissolved)	7440-22-4	NA	0.00002	0.000005	0.000005	0.00002
Mercury (total)	7439-97-6	0.002	0.0002	0.00002	0.00005	0.0002
Mercury (dissolved)	7439-97-6	0.002	0.0002	0.00002	0.00005	0.0002
Nickel (total)	7440-02-0	Not applicable	0.0002	0.00002	0.00005	0.0002
Nickel (dissolved)	7440-02-0	Not applicable	0.0002	0.00002	0.00005	0.0002
Zinc (total)	7440-66-6	Not applicable	0.00075	0.0002	0.00025	0.00075
Zinc (dissolved)	7440-66-6	Not applicable	0.00075	0.0002	0.00025	0.00075

Notes:
[1] Project Evaluation Level derived from Table 15-3 of the 2013 QAPP (USACE 2013) using the ""Evaluation Criteria from 18 AAC 70" column
[2] Achievable DLs, LODs, and LOQs are provided by ALS.
CAS = Chemical Abstract Service
DL = Detection limit
LOD = Limit of detection
LOQ = Limit of quantitation
For definitions, see the Acronyms and Abbreviations section

WORKSHEET #16
PROJECT SCHEDULE/TIMELINE TABLE

This worksheet lists all project activities as well as QA assessments that will be performed during the course of the project, including the anticipated start and completion dates.

Activities	Organization	Dates		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Develop Draft Work Plan Supplement	Jacobs	8 August 2013	28 August 2013	Draft Supplement to QAPP	28 August 2013
Respond to comments on Work Plan Supplement	Jacobs	4 September 2013	5 September 2013	Response to comments	5 September 2013
Finalize Draft Work Plan Supplement	Jacobs	10 September 2013	10 September 2013	Final Supplement to QAPP	12 September 2013
Field Activities	Jacobs	11 September 2013	18 September 2013	Fieldwork	None
Draft Sampling Report	Jacobs	18 October 2013	18 November 2013	Sampling Report	19 November 2013

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WORKSHEET #17
SAMPLING DESIGN AND RATIONALE

This worksheet describes the project sampling approach, including the rationale for selecting sample locations and matrices for each analytical group and concentration level.

PHYSICAL BOUNDARIES

The physical boundaries of the site are described on Worksheet #11.

TIME PERIOD

Sampling is scheduled to occur in September 2013.

SAMPLING DESIGN AND RATIONALE

During a scoping session on 13 August 2013 (Worksheet #9), the field objectives and locations for the 2013 sampling was reviewed. As a result, the general scoping items were incorporated into this supplement. The resulting data obtained from the Jacobs sampling effort at three sites will provide information for incorporation into the first NE Cape five-year review.

The field objectives will be met by:

- Collecting one surface water sample at Kangukhsam Mountain Spring. Sample results will be used to determine if contaminants of potential concern are present and support the first NE Cape five-year review
- Collecting three surface water samples and one groundwater grab sample near the Site 7 Landfill. Groundwater from the 6-inch to 24-inch bgs interval will be accessed via a screened drive point. The screened drive point will be advanced using hand tools
- Collecting three surface water samples and one groundwater grab sample downgradient of the Site 7 Landfill. Groundwater from the 6-inch to 24-inch bgs interval will be accessed via a screened drive point. The screened drive point will be advanced using had tools.
- Proposed sample locations will be field-located using maps of the area. Following sample collection, each location will be recorded using GPS and photographs. Sampling frequency, location, and the analytical suite are included in Table 17-1. Sample results will be used to determine if contaminants of potential concern are present and support the

first NE Cape five-year review. Sample results will be compared to the project action limits listed in Worksheet #15.

Table 17-1
Sample Locations, Description, and Analytical Suite

Sample Type	Sampling Location	Collection Description	Analytical Suite
Surface Water	Kangukhsam Mountain Spring (Figure A-5)	Collect from slightly below the surface using a dipper	GRO (AK101) BTEX(SW8260) DRO/RRO (AK102/3) PAHs (SW8270 SIM) PCBs (SW8082) Metals–8 RCRA, Zinc (SW6020/SW7471)
Surface Water	Site 7 Landfill (Figure A-3)	Collect from slightly below the surface using a dipper	GRO (AK101) BTEX(SW8260) DRO/RRO (AK102/3) PAHs (SW8270 SIM) PCBs (SW8082) Metals–8 RCRA, Nickel, Zinc (SW6020/SW7471)
Groundwater (Grab)	Site 7 Landfill (Figure A-3)	Collect from the 6-inch to 24-inch bgs interval using a peristaltic pump inserted into a screened drive point	GRO (AK101) BTEX(SW8260) DRO/RRO (AK102/3) PAHs (SW8270 SIM) PCBs (SW8082) Metals–8 RCRA, Nickel, Zinc (SW6020/SW7471)
Surface Water	Site 9 Landfill (Figure A-4)	Collect from slightly below the surface using a dipper	GRO (AK101) BTEX(SW8260) DRO/RRO (AK102/3) PAHs (SW8270 SIM) PCBs (SW8082) Metals–8 RCRA, Zinc (SW6020/SW7471)
Groundwater (Grab)	Site 9 Landfill (Figure A-4)	Collect form the 6-inch to 24-inch bgs interval using a peristaltic pump inserted into a screened drive point	GRO (AK101) BTEX(SW8260) DRO/RRO (AK102/3) PAHs (SW8270 SIM) PCBs (SW8082) Metals–8 RCRA, Zinc (SW6020/SW7471)

Note:
For definitions, see the Acronyms and Abbreviations section.

Worksheet #17 – Sampling Design and Rationale (Continued)

SAMPLE DOCUMENTATION AND NAMING

Accurate and thorough recordkeeping is critical to documenting sample custody and includes CoC records. The field logbook and sampling forms will be the primary tools for capturing information collected during the field effort. Details of daily activities, including collection of every sample, will be recorded in a bound, sequentially paginated field logbook. All field notes will be entered in permanent ink. If any changes are made to the field record, the original notation will be crossed out with a single line, initialed, and dated by the person making the correction. The field logbook will contain all information required to recreate the sampling event.

At a minimum, field logbooks will contain the following information:

- Project name and number
- Date and time of work
- Name and location of site
- Names of field personnel
- Summary of equipment preparation procedures
- Description/sketch of work area
- Weather conditions
- Field observations (stressed biota, staining, etc.)
- Sample locations
- Date and time of each sample collection event
- Field screening locations and results
- Sample identification number, location, matrix, and depth
- Explanation of any deviations from the field sampling SOP(s), including rationale for deviation

SAMPLE IDENTIFICATION PROCEDURES

Samples will be given a unique identifier reflecting the sample location, sample medium or type, and sample year. Sample containers will be labeled to match the CoC records. At the time of sampling, appropriate sample numbers will be recorded in the field logbook. An alphanumeric sample identification system will be used and will consist of the following elements:

- Year: Two digit code for the sampling year (13 for 2013)
- Site Location: KMS, 7LF, or 9LF
- Two digit code for the sample type
  - WS for surface water
  - WG for groundwater
- Sample Number: Two digit code for the boring or surface sample location
- Depth: The approximate depth in feet that the sample was collected. Surface samples will be recorded at a depth of 0 feet bgs.
- Duplicate marker for samples will not be applied to the sample ID. Sample duplicates will be tracked in the field notebook and appear as any other sample on the CoC.

Table 17-2 describes the general format for sample identification.

Table 17-2
Example Sample Identification

Year	Site Location	Sample Type and Number	Depth
13	KMS	SW01	0

Example: 13-KMS-SW01-0 (primary sample)
Example: 13-KMS-SW02-0 (duplicate sample)

CHAIN-OF-CUSTODY PROCEDURES

The CoC provides the ability to trace the possession and handling of samples from the time of collection through analysis and final disposition. Samples are collected by, and will remain in

the custody of, the field team until they are shipped to the analytical laboratory. Each CoC will follow these procedures and contain the information listed in Table 17-3 and below.

Table 17-3
Chain-of-Custody Information

Sample Specific	Container Specific	Project Specific
Sample ID Location ID Collection Date and Time Sampler Initials Sample Matrix	Container type Preservative (if any) Quantity Analysis requested	CoC ID Number Cooler ID NPDL Number

- Each CoC submitted to the laboratory will be assigned an identification number
  - The first two digits define the sampling year (e.g. 13).
  - The next characters define the project (NECape).
  - The next two digits are incremented sequentially per CoC (01, 02, etc.).

Example: 13NECAPE-01

- The cooler will be given a unique identification name or number, which will be recorded on the CoC form.
- The CoC form will be signed by the person transferring custody at any stage of shipment.
- A separate CoC form will be completed for each cooler.
- The CoC form will be placed in a plastic bag and taped to the inside lid of the cooler.
- The sample information from the CoC will be entered into a sample summary spreadsheet that is maintained in the field to track all analytical samples collected during the field effort.

PACKAGING AND SHIPPING COORDINATION

In preparation for sample shipment, the following steps will be followed:

- Store primary samples, QC samples, temperature blanks, and trip blanks together in a temperature-controlled refrigerator or cooler with gel ice until ready for packaging.
- Check that all sample container lids are secure.
- Match printed labels to sample containers, wipe off condensation, apply labels, and secure labels with clear packing tape.

- Individually wrap each jar in bubble wrap. Secure the bubble wrap with either self-adhesive strip, or with rubber bands.
- Place absorbent material or vermiculite in bottom of cooler.
- Line cooler with a heavy-duty trash bag or other durable waterproof bag.
- Stack samples, temperature blank, trip blank, and six to eight frozen gel packs inside, taking care to pack samples upright and securely. Fill excess space between the cooler and garbage bag with bubble wrap. Generally eight 1-liter containers will fit within a standard cooler.
- Tape the signed CoC form to the inside or the cooler lid. Apply two custody seals to the lid of the cooler (front right and back left) with clear packing tape and strapping tape.
- Label the cooler with the sender's address, the recipient's address, the cooler name, and an "Excepted Quantities" sticker if the cooler contains methanol-preserved samples.

Once shipped by the field team to the analytical laboratory, the CoC and shipment tracking information will be communicated to the Jacobs Project Chemist and the analytical laboratory Project Manager.

SAMPLE RECEIPT, CUSTODY, AND DISPOSAL

A sample custodian at the laboratory will accept custody of the shipped samples shortly after arrival. The laboratory will complete the Jacobs cooler receipt form when samples are received to document sample condition, discrepancies from the CoC, and any other items of interest. Cooler receipt information, including signed CoCs, custody seals, and completed cooler receipt form will be provided to the Jacobs Project Chemist and the USACE Chemist (receipt.cooler@usace.army.mil) within 24 hours of cooler receipt. Discrepancies or other data quality issues identified by the laboratory will be reviewed with the USACE Chemist and corrective action measures (if any) will be documented and reviewed with the laboratory.

The sample custodian will maintain proper procedures as the appropriate departments complete analysis. Disposal of the samples will occur only after analyses and QA/QC checks are completed, and the final data report has been submitted. Following completion of chemical analysis, the samples will continue to be maintained according to preservation

methods indicated on Worksheets #19 and #30 for at least two weeks after expiration of the appropriate holding time. After the two weeks have expired, the samples may be moved to an un-refrigerated storage location, but shall be kept for a minimum of 60 days after Jacobs has received the hardcopy deliverables unless other arrangements have been authorized by Jacobs. After expiration of the storage time identified above or other arrangements authorized by Jacobs, samples will be disposed of in accordance with federal, state, and local requirements.

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WORKSHEET #18
SAMPLING LOCATIONS AND METHODS/SOP REQUIREMENTS TABLE

The following table lists site locations that will be sampled, including the sample ID number (if available) and the matrix and depth (if applicable) at which samples will be taken. The table also provides the analytical group, number of samples to be collected, sampling standard operating procedure, and a short reference for the sampling location rationale.

Sampling Location/ID No.[1]	Matrix	Depth (units)	No. of Primary Samples	Analyte/ Analytical Group	Sampling SOP Reference	Rationale for Sampling Location
Groundwater Grab Sample	Water	Groundwater table or refusal	2	Field Parameters GRO (AK101) DRO/RRO (AK102, AK103) BTEX (SW8260) PAHs (SW8270 SIM) PCBs (SW8082) Metals (SW6020) Mercury (SW7470)	NE-POP-4010	The USACE requested that the groundwater directly downgradient of the Site 7 and Site 9 Landfills receive additional evaluation for inclusion into the first five-year review.
Surface Water Sample	Water	Slightly below the water surface	7	Field Parameters GRO (AK101) DRO/RRO (AK102, AK103) BTEX (SW8260) PAHs (SW8270 SIM) PCBs (SW8082) Metals (SW6020) Mercury (SW7470)	NE-POP-4100	The USACE requested that six surface water bodies receive additional evaluation for inclusion into the first five-year review.

Notes:
[1] See Worksheet #17.
For definitions, see the Acronyms and Abbreviations section

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WORKSHEET #19
Analytical SOP Requirements Table

The following table provides the analytical and preparation method/SOP and associated sample volume, container specifications, preservation requirements, and maximum holding time for each matrix, analytical group, and concentration level.

Matrix	Analytical Group	Concentration Level	Analytical/Preparation Method Laboratory SOP Reference	Sample Volume	Containers (number, size, and type)[1]	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
Groundwater Surface Water	GRO	Med -High	PET-GRO Rev. 9	40 mL	(3) 40 mL glass w/Teflon® lined lid	Cool to 4 ± 2°C, HCl	14 days
	DRO\RRO	Med-High	PET-SVF Rev. 12	1 L	(2) 1 L amber glass w/ Teflon®-lined lid	Cool to 4 ± 2°C, HCl	14 days / 40 days from preparation to analysis
	BTEX	Low-Med	VOC-5035 Rev. 9 VOC-8260 Rev. 17	40 mL	(3) 40 mL glass w/ Teflon®-lined lid	Cool to 4 ± 2°C, HCl	14 days
	PAHs	Low	EXT-3520 Rev. 14 SVM-8270P Rev.8	1 L	(2) 1 L amber glass w/ Teflon®-lined lid	Cool to 4 ± 2°C	7 days / 40 days from preparation to analysis
	PCB	Low-Med	EXT-3520 Rev. 14 SOC-8082Ar Rev.16	1 L	(2) 1 L amber glass w/ Teflon®-lined lid	Cool to 4 ± 2°C	none / 40 days from preparation to analysis
	Metals	Low	MET-DIG Rev.14 MET-3020A Rev.15 MET-3050 Rev. 13 MET-6020 Rev. 15	250 mL	(1) 250 mL poly	Cool to 4 ± 2°C, HNO_3	28 days
	Mercury	Low	MET-7470A Rev.15	250 mL	(1) 250 mL poly	Cool to 4 ± 2°C, HNO_3	28 days

Notes:

[1]Containers for certain analytical parameters may be combined as per instructions from the laboratory
HCl = hydrochloric acid
$HN0_3$ = hydrogen nitrate
L = liter
ml - milliliters
For additional definitions, see the Acronyms and Abbreviations section.

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WORKSHEET #20
FIELD QUALITY CONTROL SAMPLE SUMMARY TABLE

The following table summarizes the number of field QC samples that will be collected and sent to the laboratory for each matrix, analytical group, and concentration level.

Matrix	Analytical Group	Concentration Level	Primary Samples	Field Duplicates	Matrix Spike/ Matrix Spike Duplicates	Total Samples[1]
Groundwater	GRO (AK101), DRO (AK102), RRO (AK103)	High	2	1	1/1	5
	BTEX (SW8260)	Low	2	1	1/1	5
	PAHs (SW8270 SIM)	Low	2	1	1/1	5
	PCB (SW8082)	Med	2	1	1/1	5
	Metals (SW6020)	Low	2	1	1/1	5
	Mercury (SW7470)	Low	2	1	1/1	5
Surface Water	AK101, AK102, AK103	High	7	1	1/1	10
	BTEX (SW8260)	Low	7	1	1/1	10
	PAHs (SW8270 SIM)	Low	7	1	1/1	10
	PCB (SW8082)	Med	7	1	1/1	10
	Metals (SW6020)	Low	7	1	1/1	10
	Mercury (SW7470)	Low	7	1	1/1	10

Notes:
[1] Total Samples does not include trip blanks.
Duplicates and MS/MSD samples will be collected from each of the sampling location per USACE request.
For definitions, see the Acronyms and Abbreviations section.

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WORKSHEET #21
PROJECT SAMPLING SOP REFERENCES TABLE

The following table lists all SOPs associated with project sampling including, but not limited to, sample collection, sample preservation, equipment cleaning and decontamination, supply inspection and acceptance, and sample handling and custody. References or copies of the SOPs are provided.

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
JE-SOP-1000	Global Positioning System (GPS) Surveying, April 2012	Jacobs	PPE, real-time kinematic GPS or mapping-grade GPS equipment	No	
JE-SOP-2000	Decontamination, January 2011	Jacobs	PPE, brushes, detergent, rinse water	No	
JE-SOP-2100	Waste Management, April 2013	Jacobs	PPE	No	
NE-POP-4010	Groundwater Grab Sample Collection, August 2013	Jacobs	PPE, peristaltic pump, oil/water interface probe, and YSI multi-meter	Yes	This SOP was updated for project specific requirements
NE-POP-4100	Surface Water Sample Collection, August 2013	Jacobs	PPE, hand tools and YSI multi-meter	Yes	This SOP was updated for project specific requirements
NE-POP-7000	Field Documentation, August 2013	Jacobs	Logbook	Yes	This SOP was updated for project specific requirements

Notes:
Jacobs SOPs and POPs associated with this supplement are provided in Appendix B.
For definitions, see the Acronyms and Abbreviations section.

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WORKSHEET #22
FIELD EQUIPMENT CALIBRATION, MAINTENANCE, TESTING, AND INSPECTION TABLE

The following table identifies all field equipment and instruments (other than analytical instrumentation) that require calibration, maintenance, testing, or inspection and provides the SOP reference number for each type of equipment. In addition, this table documents the frequency, acceptance criteria, and corrective action requirements for calibrating each type of equipment.

Field Equipment	Activity	SOP Reference	Title or Position of Responsible Person	Frequency	Acceptance Criteria	Corrective Action
YSI Multi-meter	Measuring field parameters	NE-POP-4010 NE-POP-4100	Jacobs Sampler	Every sample location	+/- 15% of accepted value	Re-calibrate as needed. If acceptable calibration cannot be obtained, remove equipment from service and return to vendor.
Land survey and positioning equipment (real-time kinematic GPS)	Collecting Sample Locations	JE-SOP-1000	Surveyor	Successful equipment self-diagnostics during power up.	Table 5.2 – "Survey Quality"; (USACE 2011).	If acceptable accuracy cannot be obtained and self-diagnostic does not reveal a problem, remove equipment from service and return to vendor.

Note:
For definitions, see the Acronyms and Abbreviations section.

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WORKSHEET #23
ANALYTICAL SOP REFERENCES TABLE

The following table lists all SOPs that will be used to perform onsite or offsite analysis, and indicates whether the procedure produces screening or definitive data. References or copies of the SOPs are provided in Appendix C.

Laboratory SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	ADEC Approved Method	SOP Option or Equipment Type	Modified for Project Work? (Y/N)
VOC-8260.r17	VOCs by GC/MS, Rev. 17	Definitive	BTEX	SW8260	GC/MS	N
PET-GRO.r9	GRO by GC, Rev. 9	Definitive	GRO (AK101)	AK101	GC/FID	N
PET-SVF.r12	Analysis of Water, Solids and Soluble Waste Samples for Semi-Volatile Fuel Hydrocarbons, Rev. 12	Definitive	DRO/RRO (AK102/103)	AK102/AK103	GC/FID	N
MET-DIG r.14	Metals Digestion of Aqueous Samples	Definitive	Metals	Not applicable	Preparation	N
MET-6020.r15	Determination of Metals and Trace Elements by ICP-MS, Rev. 15	Definitive	Metals	SW6020	ICP/MS	N
MET-7470A.r15	Mercury in Liquid Waste, Rev. 15	Definitive	Mercury	SW7470	CVAA	N
SMV-8270S.r6	SVOCs by GC/MS SIM	Definitive	PAHs	SW8270	GC/MS	N
SOC-8082Ar.r16	PCBs as Aroclors, Rev. 16	Definitive	PCBs	SW8082	GC/ECD	N

Notes:
CVAA = cold vapor atomic absorption spectrophotometry
ECD = electron capture detection
FID = flame ionization detection
GC = gas chromatograph
ICP = inductively coupled plasma
MS = mass spectrometry
SIM = Selected Ion Monitoring
For additional definitions, see the Acronyms and Abbreviations section.

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WORKSHEET #24
ANALYTICAL INSTRUMENT CALIBRATION TABLE

The following table identifies all analytical instruments that require calibration and provides the SOP reference number for each. In addition, the table documents the frequency, acceptance criteria, and corrective action requirements for each calibration procedure.

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
GC-MS (8260C 8720D SIM)	Tuning	Prior to ICAL and at the beginning of each 12-hour period	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Lab Manager/Analyst or certified instrument technician	VOA-8260 SVM-8270S
	Breakdown check (DDT-Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples	Degradation ≤20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	Correct problem, then repeat breakdown checks.	Lab Manager/Analyst or certified instrument technician	VOA-8260 SVM-8270S
	Five-point ICAL for linear calibration	Prior to sample analysis	RSD for each analyte +/- 20% or least square regression ≥0.995.	Correct problem then repeat ICAL.	Lab Manager/Analyst or certified instrument technician	VOA-8260 SVM-8270S
	Second source calibration verification	After ICAL	All analytes within ±30% of expected value.	Correct problem and verify second source standard; rerun second source verification. If fails, correct problem and repeat initial calibration.	Lab Manager/Analyst or certified instrument technician	VOA-8260 SVM-8270S

Worksheet # 24 – Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
GC-MS (8260C 8720D SIM) (continued)	RT window position for each analyte and surrogate	Once per ICAL	Position shall be set using the midpoint standard for the ICAL.	Not applicable	Lab Manager/Analyst or certified instrument technician	VOA-8260 SVM-8270S
	RRT	With each sample	RRT of each target analyte in each calibration standard within ±0.06 RRT units of ICAL.	Correct problem, then reanalyze all samples analyzed since the last RT check. If fails, then rerun ICAL and samples.	Lab Manager/Analyst or certified instrument technician	VOA-8260 SVM-8270S
	CCV	Daily, before sample analysis, unless ICAL performed same day and after every 12 hours	All analytes within ±20% of expected value (% difference) or not more than 20% of comparable difference by 40%	Correct problem and rerun CCV. Reanalyze all samples since last successful calibration verification. If fails, repeat initial calibration.	Lab Manager/Analyst or certified instrument technician	VOA-8260 SVM-8270S
	IS	Each CCV and sample	RT ± 30 seconds from RT of the IS in the ICAL mid-point standard. EICP area within -50% to +200% of area from IS in ICAL mid-point standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed during failure is mandatory.	Lab Manager/Analyst or certified instrument technician	VOA-8260 SVM-8270S
	Breakdown check (DDT-Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples	Degradation ≤20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	Correct problem, then repeat breakdown checks.	Lab Manager/Analyst or certified instrument technician	VOA-8260 SVM-8270S

Worksheet # 24 – Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
AK 101, 102, 103	Five-point ICAL for linear calibration	Prior to sample analysis	RSD for each analyte +/- 20% or least square regression ≥0.995.	Correct problem then repeat ICAL.	Lab Manager/Analyst or certified instrument technician	PET-SVF PET-GRO
	Second source calibration verification	After ICAL	±25% of expected value	Correct problem and verify second source standard; rerun second source verification. If fails, correct problem and repeat initial calibration.	Lab Manager/Analyst or certified instrument technician	PET-SVF PET-GRO
	RT window position for each analyte and surrogate	Once per ICAL	Position shall be set using the midpoint standard for the ICAL.	Not applicable	Lab Manager/Analyst or certified instrument technician	PET-SVF PET-GRO
	RRT	With each sample	RRT of each target analyte in each calibration standard within ±0.06 RRT units of ICAL.	Correct problem, then reanalyze all samples analyzed since the last RT check. If fails, then rerun ICAL and samples.	Lab Manager/Analyst or certified instrument technician	PET-SVF PET-GRO
	CCV	Daily, before sample analysis, unless ICAL performed same day and after every 12 hours	±25% of expected value	Correct problem and rerun CCV. Reanalyze all samples since last successful calibration verification. If fails, repeat initial calibration.	Lab Manager/Analyst or certified instrument technician	PET-SVF PET-GRO

Worksheet # 24 – Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
PCBs	5-point ICAL for linear calibration	Prior to sample analysis	RSD for each analyte +/- 20% or least square regression ≥0.995.	Correct problem then repeat initial calibration.	Lab Manager/Analyst or certified instrument technician	SOC-8082Ar
	Second source calibration verification (ICV)	Once after each initial calibration	Analytes within ±20% of expected value (initial source).	Correct problem and verify second source standard. Rerun second source verification. If fails, correct problem and repeat initial calibration.	Lab Manager/Analyst or certified instrument technician	SOC-8082Ar
	Establishment and verification of the RT window for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift for establishment of RT; and with each CCV for verification of RT	Using the midpoint standard or the CCV at the beginning of the analytical shift for RT establishment; and analyte must fall within established window during RT verification.	Not applicable	Lab Manager/Analyst or certified instrument technician	SOC-8082Ar
	CCV	Daily, before sample analysis, unless ICAL performed same day, every 10 samples, and at the end of the analysis sequence	All analytes within ±20% of expected value (% difference).	CCV: Correct problem and rerun CCV. If fails, repeat initial calibration. Reanalyze all samples since last successful calibration verification.	Lab Manager/Analyst or certified instrument technician	SOC-8082Ar

Worksheet # 24 – Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
Metals	IDLs	Every 3 months	In accordance with manufacturer's recommendation or laboratory SOP.	Notify the manufacturer if problem occurs.	Lab Manager/Analyst or certified instrument technician	MET-ICPMS
	Tuning	Prior to initial calibration	Mass calibration ≤0.1 amu from true value; Resolution <0.9 amu full width at 10% peak height; For stability, RSD ≤5% for at least four replicate analyses.	Correct problem, then repeat tuning.	Lab Manager/Analyst or certified instrument technician	MET-ICPMS
	IC using either single or multi-point standard calibration	Daily prior to analysis of sample	Correlation coefficient ≥0.998.	Correct problem, then repeat initial calibration.	Lab Manager/Analyst or certified instrument technician	MET-ICPMS
	Linear dynamic range or high level check standard	Once every 6 months or when the system is repaired	The calculated value should be within ±10% of the true values.	Correct problem, then repeat the calibration process.	Lab Manager/Analyst or certified instrument technician	MET-ICPMS
	Interference check solution	At the beginning of each analytical run	ICS-A: Absolute value of concentration for all non-spiked analytes <2 × MDL (unless they are a verified trace impurity from one of the spiked analytes). ICS-AB: Within ±20% of its true value.	Correct problem, then repeat the calibration process or use internal standards to eliminate the problem.	Lab Manager/Analyst or certified instrument technician	MET-ICPMS

Worksheet # 24 – Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
Metals (continued)	Second-source ICV	Once after standard calibration	Within ±10% of its true value.	Correct problem, then repeat the calibration process.	Lab Manager/Analyst or certified instrument technician	MET-ICPMS
	Run lower limit of quantitation limit	Once after ICV	Within ±20% of its true value.	Qualify the data as estimated values.	Lab Manager/Analyst or certified instrument technician	MET-ICPMS
	Internal standards	Every analysis	Internal standard intensity within 30 to 120% of intensity of the internal standard in the initial calibration.	Terminate the analysis, correct the problem, recalibrate and reanalyze the samples.	Lab Manager/Analyst or certified instrument technician	MET-ICPMS
	CCV	Following IC, after every 10 samples and the end of the sequence	±10% of its true value.	Terminate analysis; recalibrate and reanalyze the samples.	Lab Manager/Analyst or certified instrument technician	MET-ICPMS
	CCB	After IC, after CCV calibration, after every 10 samples and the end of the sequence	Less than 1/2 reporting limit.	Terminate analysis; recalibrate and reanalyze the samples.	Lab Manager/Analyst or certified instrument technician	MET-ICPMS

Worksheet # 24 – Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
Mercury	Establish IDLs	Every 3 months	In accordance with manufacturer's recommendation or lab SOP.	Notify the manufacturer if problem occurs.	Certified instrument technician	MET-7471
	Calibrate using the multi-point standard calibration	Daily prior to analysis of sample	Correlation coefficient ≥0.995.	Correct problem then repeat initial calibration.	Lab Manager/Analyst or certified instrument technician	MET-7471
	Establish linear dynamic range	Once every 6 months or when the system is repaired	The calculated value should be within ±10% of the true value.	Correct problem then repeat the calibration process.	Lab Manager/Analyst or certified instrument technician	MET-7471
	Run second source calibration verification (ICV)	Once after standard calibration	±10% of its true value.	Correct problem then repeat the calibration process.	Lab Manager/Analyst or certified instrument technician	MET-7471
	Run CCV	Once every 10 samples	±10% of its true value.	Terminate analysis; recalibrate and reanalyze the samples.	Lab Manager/Analyst or certified instrument technician	MET-7471
	Run CCB	Once every 10 samples	Less than the established lower limit of quantitation for any desired target analyte.	Terminate analysis; recalibrate and reanalyze the samples.	Lab Manager/Analyst or certified instrument technician	MET-7471

Worksheet # 24 – Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
Mercury (continued)	Instrument blank	Daily at beginning and end of day and every 20 field measurements	Less that instrument detection limit	Correct problem, repeat measurement. If acceptance criteria still cannot be met, re-zero instrument.	Field analyst	MET-7471
	Duplicate (two consecutive readings)	10% of field measurements	RPD < 50%	Correct problem, repeat measurement.	Field analyst	MET-7471
	Establish IDLs	Every 3 months	In accordance with manufacturer's recommendation or lab SOP.	Notify the manufacturer if problem occurs.	Certified instrument technician	MET-7471
Water Bath	Measure water temperature against a calibrated thermometer	Annually	In accordance with unit model and manufacturer's recommendation or laboratory SOP.	Terminate analysis, recalibrate, and verify before sample analysis.	Lab Manager/Analyst or certified instrument technician	ADM-SEMC
Drying Oven	Measure oven temperature against a calibrated thermometer	Annually	In accordance with unit model and manufacturer's recommendation or laboratory SOP.	Terminate analysis, recalibrate, and verify before sample analysis.	Lab Manager/Analyst or certified instrument technician	ADM-SEMC
Analytical Balance	Calibrate against verified (National Institute of Standards and Technology) mass	Daily or prior to analyzing samples	In accordance with unit model and manufacturer's recommendation or laboratory SOP.	Terminate analysis, recalibrate, and verify before sample analysis.	Lab Manager/Analyst or certified instrument technician	ADM-BAL

Worksheet # 24 – Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
pH Meter	Run a minimum 3 point calibration; run CCV	Daily or prior to analyzing samples; one CCV for every 10 samples	±0.05 unit.	Terminate analysis, recalibrate, and verify before sample analysis.	Lab Manager/Analyst or certified instrument technician	QAM App E

Note:
CCB = continuing calibration blank
CCV =continuing calibration verification
CVAA = cold vapor atomic absorption spectrophotometry
DDT = dichlorodiphenyltrichloroethane
EICP = extracted ion current profile
GC = gas chromatograph
HPLC = high-performance liquid chromatography
ICAL = initial calibration
ICV = initial calibration verification
IDL = instrument detection level
IS = internal standard
MDL = method detection limit
MS = mass spectroscopy
ppm = parts per million
RRT = relative retention time
RSD = relative standard deviation
RT = retention time
For additional definitions, see the Acronyms and Abbreviations section

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WORKSHEET #25
ANALYTICAL INSTRUMENT AND EQUIPMENT MAINTENANCE, TESTING, AND INSPECTION TABLE

The following table identifies all analytical instruments that require maintenance, testing, or inspection, and provides the SOP reference number for each. The table documents the frequency, acceptance criteria, and corrective action requirements for each maintenance activity.

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC-ECD GC-FID GC-MS	Change gas purifier	Not applicable	Visually inspect if traps are changing color	Every 6 to 12 months	No moisture	Replace indicating traps	Analyst or certified instrument technician	VOA-8260 SVM-8270S PET-SVF PET-GRO SOC-8082Ar
	Change syringes/ syringe needles	Not applicable	Visually inspect for wear or damage	Every 3 months	Not applicable	Replace syringe if dirt is noticeable in the syringe	Analyst or certified instrument technician	
	Change inlet liner, liner O-rings, and inlet septum	Not applicable	Visually inspect for dirt or deterioration	Weekly for liner, monthly for O-rings, and daily for septum	Not applicable	Replace and check often	Analyst or certified instrument technician	
	Change front-end column	Not applicable	Check peak tailing, decreased sensitivity, retention time changes, etc.	Weekly, monthly, or when needed	Not applicable	Remove 1/2 to 1 meter from the front of the column when experiencing problems	Analyst or certified instrument technician	
	Replace/refill carrier gas line oxygen and moisture traps	Not applicable	Not applicable	Yearly or as needed	Not applicable	Not applicable	Analyst	

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

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC-ECD	Perform 'Wipe Test' and clean up the baseline	Not applicable	Baseline is noisy	Every 6 month or as needed	In accordance with manufacturer's recommendation or lab SOP	Thermally clean by "baking-out" the instrument overnight	Analyst or certified instrument technician	SOC-8082Ar
GC-MS	Change tune MSD, check the calibration vial, and replace the foreline pump oil	Not applicable	Visually inspect and monitor the fluid becoming discolored	As needed or every 6 months	In accordance with manufacturer's recommendation or lab SOP	Keep plenty of PFTBA; refill the vial and check the fluid; change when the fluid becomes discolored	Analyst or certified instrument technician	VOA-8260 SVM-8270S
	Run tuning program to determine if source is functioning properly	Not applicable	Not applicable	Daily	Not applicable	Cool system, vent, disassemble and clean	Analyst	VOA-8260 SVM-8270S
	Replace columns	Not applicable	Not applicable	If chromatograms indicate possible contamination	Not applicable	Not applicable	Analyst	VOA-8260 SVM-8270S
	Vacuum rough pump oil level is checked	Not applicable	Not applicable	Every 4 to 6 weeks	Not applicable	Oil added if needed	Analyst	VOA-8260 SVM-8270S

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

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
ICP-MS	Clean the instrument; Change gas, gas purifier and tubing, and dispose of wastes	Check instrument connections, gas flow, pressure, ion mass, and detector sensitivity	Visually inspect for wear of damage and check indicators from computer controls	Daily or when needed	Intensity of spectrum is within manufacturer's recommendation and mass calibration difference is < 0.1 amu from the true value	Call for maintenance service	Analyst or certified instrument technician	MET-ICPMS
Water Bath (precision micro-processor controlled)	Check instrument connections, water level, and thermometer	Measure water temperature against a calibrated thermometer	Visually inspect for wear or damage and indicator from computer controls	Daily and annual maintenance from manufacturer	Refer to manufacturer's recommendation	Return to manufacturer for recalibration or call for maintenance service	Analyst or certified instrument technician	ADM-SEMC
Drying Oven	Thermometer indicator	Measure oven temperature against a calibrated thermometer	Visually inspect for wear or damage and indicator from computer controls	Daily and annual maintenance from manufacturer	Refer to manufacturer's recommendation	Return to manufacturer for recalibration or call for maintenance service	Analyst or certified instrument technician	ADM-SEMC
Analytical Balance	Check digital LCD display and ensure a flat base for the Instrument	Calibrate against verified (NIST) mass	Visually inspect for wear or damage and indicator from computer controls	Daily and annual maintenance from manufacturer	Refer to manufacturer's recommendation	Return to manufacturer for recalibration or call for maintenance service	Analyst or certified instrument technician	ADM-BAL

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

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
pH Meter	Check LCD display and pH probe	Three point calibration using known standards	Visually inspect for wear or damage and indicator from computer controls	Daily and annual maintenance from manufacturer	± 0.05 unit	Return to manufacturer for recalibration or call for maintenance service	Analyst or certified manufacture instrument technician	QAM App E

Notes:
GC = gas chromatograph
ECD = electron capture detection
ICP = inductively coupled plasma
LCD = liquid crystal display
MS = mass spectroscopy
NIST = National Institute of Standards and Testing
PFTBA = perfluorotributylamine

WORKSHEET #30
ANALYTICAL SERVICES TABLE

The following table identifies the laboratories or organizations that will provide analytical services for the project, including onsite screening, onsite definitive, and offsite laboratory analytical work. If applicable, subcontractor laboratories and backup laboratory or organizations are identified in the event that the primary laboratory or organization cannot be used.

Matrix	Analytical Group	Concentration Level	Sample Location/ID Numbers	Analytical SOP	Data Package Turnaround Time	Laboratory/ Organization (Name and Address, Contact Person and Telephone Number)	Backup Laboratory
Surface Water Groundwater	VOCs GRO, DRO, RRO PAHs PCB Metals	Low to High	See Worksheets #17, #18, #20	See Worksheet #23	30 days	ALS Laboratories, 1317 S. 13th Ave. Kelso, WA 98626 USA 360-577-7222	None

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WORKSHEET #36
VALIDATION (STEPS IIA AND IIB) SUMMARY TABLE

This table identifies 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.

Step IIa/IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa and IIb	Surface Water	All	Low/med/high	DoD QSM 4.2 and EPA National Functional Guidelines (EPA 2008, 2010).	Jacobs
IIa and IIb	Groundwater	All	Low/med/high	DoD QSM 4.2 and EPA National Functional Guidelines (EPA 2008, 2010).	Jacobs

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WORKSHEET #37
USABILITY ASSESSMENT

This worksheet describes the procedures, methods, and activities that will be used to determine whether data are of the right type, quality, and quantity to support environmental decision-making for the project. The section also describes how data quality issues will be addressed and how limitations on the use of the data will be handled.

The analytical data will be reviewed by the Jacobs Project Chemist. This evaluation will consist of a review of CoC and sample receipt records, laboratory case narratives, laboratory data including analytical methodology, sample holding times, laboratory blanks, method detection limits and reporting limits, surrogate recoveries, laboratory control sample (LCS)/laboratory control sample duplicate (LCSD) recoveries, and MS/MSD recoveries. The analytical data will be evaluated for compliance with project-specific DQOs. In the absence of guidance, the most current version of the DoD QSM, ADEC Technical Memorandum, method criteria, laboratory criteria, and best professional judgment will apply, in that order. The Data Quality Assessment will identify any data requiring qualifications and identify effects on data usability.

Analytical data will be tabulated in the completed report. All analytical results, including duplicate results, will be reported with any appropriate qualifiers in accompanying data tables. This UFP-QAPP and the methods described herein, along with laboratory acceptance criteria, may be referenced by the Project Chemist to assess data usability and to apply qualifiers to the analytical results tables, as appropriate. A summary report of the data quality and usability, including summary tables of the analytical results, case narrative, corrective actions, and CoC records, will be provided in the sampling report. The report will provide a detailed description of any corrective actions and/or systematic problems that were identified during the data review process. Complete data deliverables will be provided to Jacobs Project Chemist by the subcontracted laboratory and submitted to the client.

Analytical DQOs will be evaluated by reviewing the following QA parameters and criteria presented below.

Precision. Precision will be evaluated by comparing the following:

- LCS and LCSD (if prepared and analyzed) to determine the precision of the laboratory procedures and verify matrix interference
- MS and MSD samples to determine the effect of the sample matrix on the precision of the results generated using the selected analytical method
- Primary and field duplicate sample results

Relative percent difference will be calculated for LCS/LCSD, field duplicates, and MS/MSD by using the following formula:

$$RPD = \frac{2|(D_1 - D_2)|}{D_1 + D_2} x\,100$$

Where: $D1$ = first sample value
$D2$ = second sample value (replicate)

Worksheet #37 – Usability Assessment (Continued)

Accuracy. Accuracy is evaluated by reviewing the following:

- Calibrations – initial and continuing; acceptance, and frequency (deviations are assumed to be noted in case narratives)
- Surrogates – recovery and frequency
- LCS and LCSD recoveries
- MS and MSD recoveries
- Relative response factors and relative standard deviation (appropriate calibration procedures improve accuracy of measurement results; deviations are assumed to be noted in case narratives)
- Method blanks (detections in the method blank may indicate potential high bias in associated samples)

Accuracy formula for surrogates, laboratory control samples, and matrix spikes will be calculated by using the following formula:

$$\%\ Recovery = \frac{(0 - X)}{T}\ x\ 100$$

Where: 0 = measured quantity of analyte in sample plus spiked solution
X = measured sample prior to spiking
T = quantity of analyte spiked

Representativeness. Representativeness is evaluated by reviewing the following:

- Sample quantities and locations
- Sampling procedures and equipment
- Sample CoCs and field logbooks
- Holding times and preservation

Completeness. Completeness is a quantitative evaluation (expressed as a percentage) of the number of valid results and the number of results generated.
Completeness will be calculated by using the following formula:

$$\%\ C = 100\ x\ (\frac{V}{n})$$

Where: %C = percent completeness
V = number of measurements judged valid
n = total number of measurements planned

Worksheet #37 – Usability Assessment (Continued)

Comparability. Comparability is a qualitative indicator of the confidence with which one data set can be compared to another. To ensure data-set comparability, the following steps will be taken:

- SOPs and established analytical methods will be followed.
- Instruments will be operated within their calibrated range according to established procedures that are based on approved methodology.
- Data will be reported in conventional and standard units.

Sensitivity. Sensitivity is evaluated by comparing the following against project action limits:

- Limits of quantitation
- Limits of detection
- Method detection limits

Data Qualifiers

The data review will identify any data requiring qualifications and identify effects on data usability. Qualifiers to be applied to the analytical data set, as appropriate, include the following:

ND (LOD)	The analyte result is less than the limit of detection (value in parenthesis).
J	The analyte result is considered an estimated value because the reported result is below the limit of quantitation but above the detection limit (formerly the method detection limit.
B	The analyte is detected in the method blank and/or the trip blank above the limit of detection, and the concentration in the sample did not exceed the blank concentration by a factor of 5 (factor of 10 for common laboratory contaminants acetone and methylene chloride).
M(L,H,N)	The result is was an estimated value and biased (low, high, or uncertain) because the analyte failed recovery criteria in the MS or MSD sample, or both.
Q(L,H,N)	The result was an estimated value and biased (low, high, or uncertain) due to a laboratory quality control failure.
JH	The numerical result was considered an estimate because the analysis was performed at greater than the method holding time and less than twice the method holding time.
R	The analyte result is rejected. A rejected result is not usable and therefore the "R" qualifier will replace any reported value.

Qualification will not be required in the following circumstances:

- Surrogate or MS recoveries were outside QC limits, and the sample was diluted by a factor of 5 or greater.
- MS recoveries were outside QC limits, and the parent sample concentration was greater than the spiked concentration.
- An analyte was detected in the method blank, but there was no detection in the sample.
- CCV, MS, or LCS recoveries exceeded upper control limits, and there was no detection of the analyte in the sample(s).

Data may be rejected on the following grounds:

- Initial calibration (per compound) criteria not met
- Less than 10% analyte recovery in the associated laboratory control sample or continuing calibration verification sample
- Continuing calibration (per compound) not verified at the frequency specified in the laboratory SOP.
- Any compound with the associated LCS or surrogate recovery less than 10 percent

Note:
For definitions, refer to the Acronyms and Abbreviations section.

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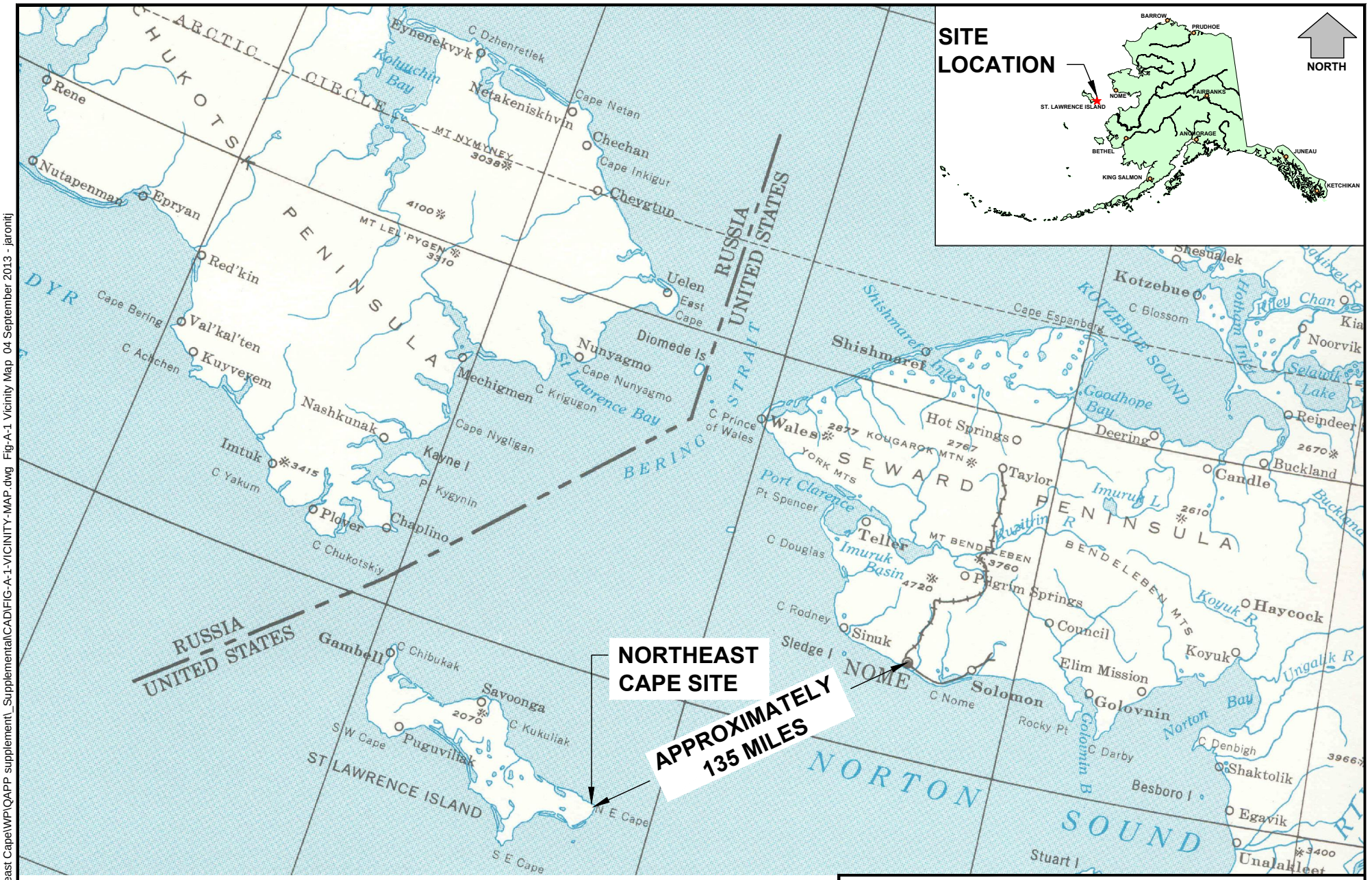
APPENDIX A

Figures

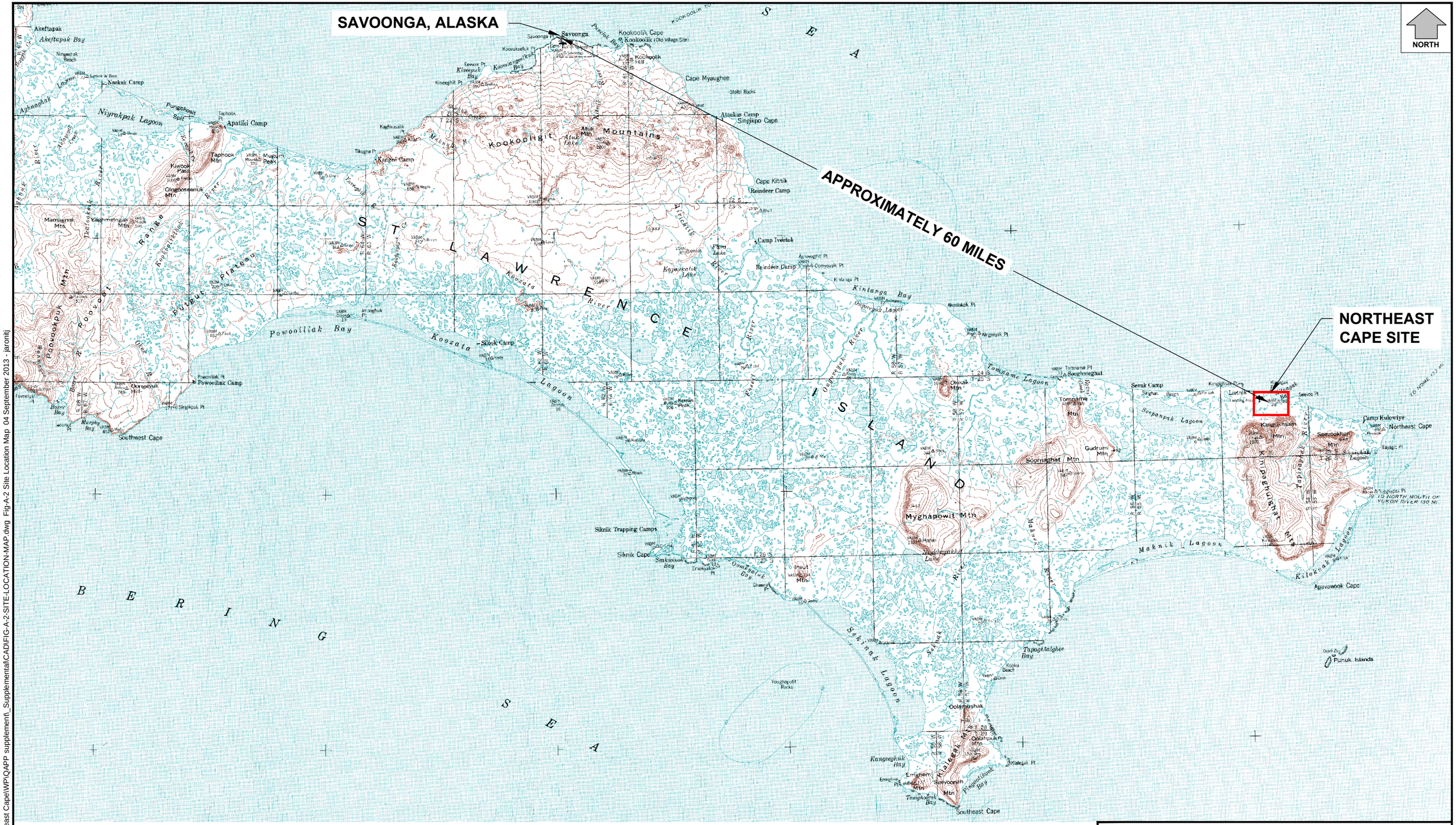


VICINITY MAP
NORTHEAST CAPE
NORTHEAST CAPE REMEDIAL ACTIONS

ST. LAWRENCE ISLAND, ALASKA

JACOBS®	DATE:	PROJECT MANAGER:	FIGURE NO. :
	09/04/2013	K. MAHER	A-1

I:\AE-HTRW\TO09-Northeast Cape\WP\QAPP supplement_Supplemental\CAD\FIG-A-1-VICINITY-MAP.dwg Fig-A-1 Vicinity Map 04 September 2013 - jaronitj

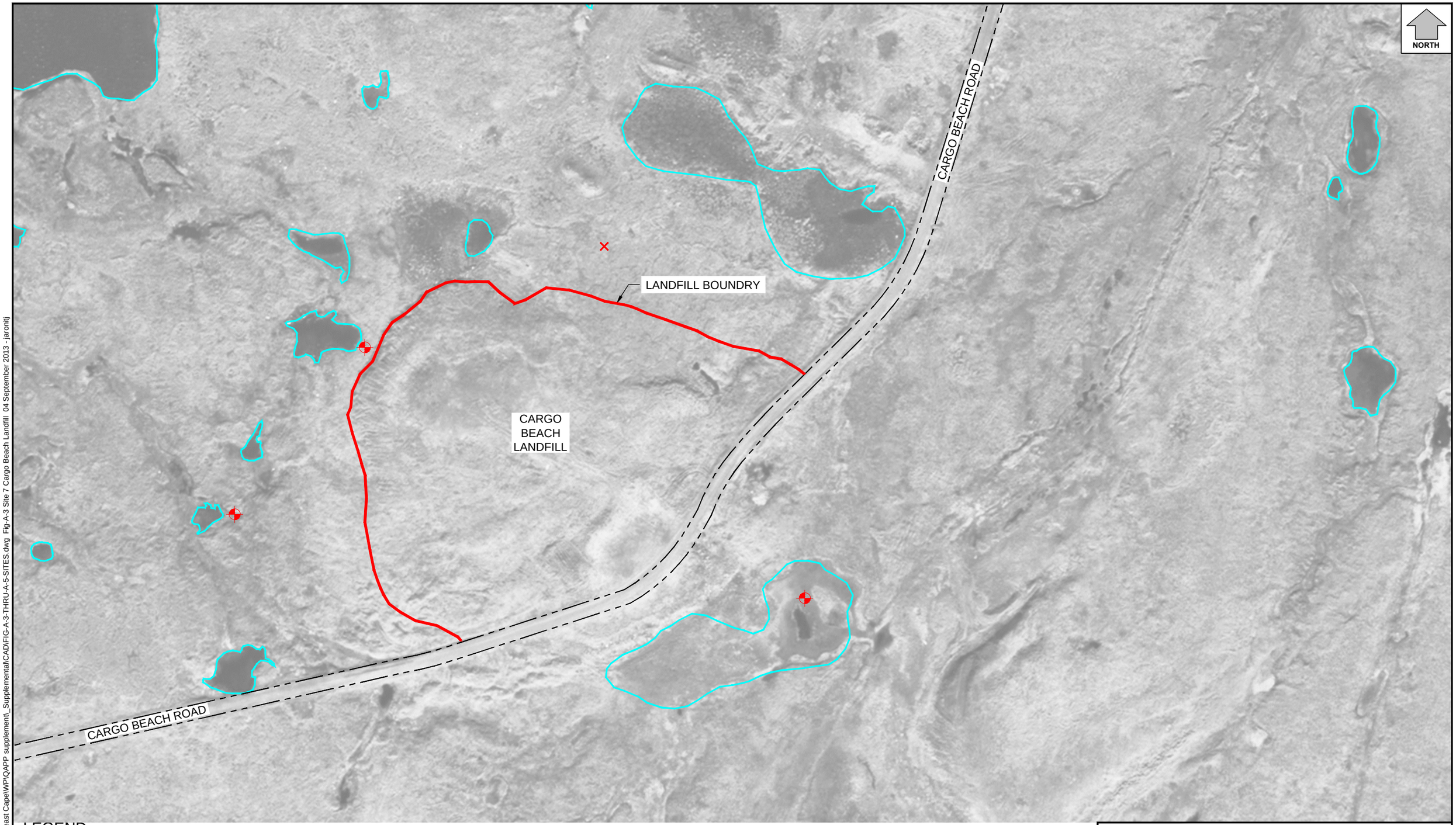


0 1 2 3 5
MILES

SITE LOCATION MAP
NORTHEAST CAPE
NORTHEAST CAPE REMEDIAL ACTIONS
ST. LAWRENCE ISLAND, ALASKA

JACOBS®	DATE: 09/04/2013	PROJECT MANAGER: K. MAHER	FIGURE NO. : A-2

I:\AE-HTRWT009-Northeast Cape\WP\QAPP supplement_Supplemental\CAD\FIG-A-2-SITE-LOCATION-MAP.dwg Fig-A-2 Site Location Map 04 September 2013 - jaronij



LEGEND

- SURFACE WATER SAMPLE (2013 PROPOSED)
- GROUNDWATER SAMPLE (2013 PROPOSED)
- ROAD
- HYDROLOGICAL FEATURE
- LANDFILL BOUNDRY

0 50 100 150
FEET

SITE 7 - CARGO BEACH LANDFILL
PROPOSED SAMPLE LOCATIONS
NORTHEAST CAPE REMEDIAL ACTIONS

ST. LAWRENCE ISLAND, ALASKA

JACOBS	DATE: 09/04/2013	PROJECT MANAGER: K. MAHER	FIGURE NO. : A-3

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NORTH

CARGO BEACH ROAD

CULVERT

POND

ACCESS ROAD

LANDFILL CAP

DIVERSION TRENCH

POND

FORMER AST's

FORMER MOC AREA

CONCRETE PAD 98

LEGEND

- SURFACE WATER SAMPLE (2013 PROPOSED)
- GROUNDWATER SAMPLE (2013 PROPOSED)
- ROAD
- HYDROLOGICAL FEATURE
- CULVERT
- FORMER STRUCTURE

0 50 100 150
FEET

SITE 9 - HOUSING AND OPERATIONS LANDFILL
PROPOSED SAMPLE LOCATIONS
NORTHEAST CAPE REMEDIAL ACTIONS
ST. LAWRENCE ISLAND, ALASKA

JACOBS®

DATE:	PROJECT MANAGER:	FIGURE NO. :
09/04/2013	K. MAHER	A-4

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NORTH

KANGUKSHAM MOUNTAIN SPRING
DIRECTION OF FLOW

ROAD TO WHITE ALICE SITE

BORROW
SOURCE

CULVERT

LOWER
TRAM
FOOTPRINT

KANGUKSHAM MOUNTAIN SPRING
DIRECTION OF FLOW

LEGEND

- SURFACE WATER SAMPLE (2013 PROPOSED)
- ROAD
- HYDROLOGICAL FEATURE
- CULVERT
- FORMER STRUCTURE

0 50 100 150
FEET

KANGUKSHAM MOUNTAIN SPRING
PROPOSED SAMPLE LOCATIONS
NORTHEAST CAPE REMEDIAL ACTIONS

ST. LAWRENCE ISLAND, ALASKA

JACOBS	DATE: 09/04/2013	PROJECT MANAGER: K. MAHER	FIGURE NO. : A-5

I:\AE-HTRWTO09-Northeast Cape\WP\QAPP supplement_Supplemental\CAD\FIG-A-3-THRU-A-5-SITES.dwg Fig-A-5 Kanguksham Mountain Spring 04 September 2013 - jaronitj

APPENDIX B

Jacobs Standard Operating Procedures,

Project Operating Procedures,

JE-SOP-1000 Global Position System Surveying

JE-SOP-2000 Decontamination

NE-POP-4010 Groundwater Grab Sample Collection

NE-POP-4100 Surface Water Sampling

NE-POP-7000 Field Documentation

JACOBS™

Standard Operating Procedure

Document No:	Page:
JE-SOP-1000	**1 of 8**
Effective Date:	Rev.
Feb 21 2013	**2**

Global Positioning System Surveying

Table of Contents

ATTACHMENTS

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) describes the procedures for global positioning system (GPS) land surveying, including those for real-time kinematic (RTK) and mapping-grade land surveys. RTK-GPS surveying utilizes a local correction signal, either publicly broadcast or provided by a base reference station set over a monument or control point, to derive positions and elevations with centimeter-level accuracy. Mapping-grade GPS surveying does not require a correction signal but may include post-processing to obtain meter-level accuracy.

This SOP defines a standard set of procedures that will result in defensible survey data meeting data quality objectives. These procedures may be varied or changed as required by site conditions, equipment limitations, or other factors, but any variances need to be documented in the field notes in accordance with the quality assurance/quality control (QA/QC) requirements (Section 3.4). Project contract documents define the specific data quality objectives that must be met, and project work plans should describe the general nature of the surveying to be performed. Other potentially useful guidance documents are listed in the references section.

2.0 EQUIPMENT

Level D personal protective equipment or project requirements (if more stringent) will apply. GPS surveying involves the following equipment:

- RTK-GPS system: Leica System 1200, Leica Viva, and Trimble R6/R7 are common equipment systems
- CORS signal repeater station (needed when line of sight to the CORS is obstructed)
- Mapping-grade survey system: Magellan® Mobile Mapper™, Trimble® GeoXH6000, Leica Viva Uno are common equipment systems

3.0 PROCEDURES

3.1. Monuments and Control Points

For surveying within range of a Continuously Operating Reference Station (CORS) or similar service, no base station is needed. The CORS broadcasts a correction signal along with its known coordinates. The rover will be configured to receive the correction signal, and the survey session (Section 3.3) may proceed.

In the absence of a CORS signal, the base station will be erected over a known monument or control point within 5 miles of the survey area. Monuments and control points are also needed for check shots. Monument and control point coordinates will meet the following criteria:

- Preferably provide accuracy better than 0.5 meters horizontally and 0.08 meters vertically (half of the values listed by USACE [USACE 2011] for better accuracy)
- Preferably updated in the last 5 years
- Preferably horizontal coordinates will be referenced to World Geodetic System 1984 (WGS 84) or North American Datum 1983 (NAD 83) (avoiding the complications of North American Datum 1927 [NAD 27])
- Preferably provide height (elevation) referenced to WGS 84 (ellipsoid height) or North American Vertical Datum 1988 (NAVD 88) (orthometric height)
- No detectable disturbance to the monument or surrounding ground

Monument and control point locations may be obtained from National Geodetic Survey (NGS) data sheets, existing reports, government-furnished material, or previous surveys. If these are unavailable or doubtful, new coordinates will be determined using static-mode GPS processed by the Online Positioning User Service (OPUS) operated by the National Oceanic and Atmospheric Administration (NOAA). In static mode, readings will be recorded once per epoch (every 30 seconds). Recent improvements in OPUS provide accurate coordinates with as little as 15 minutes of static data, although 2 to 4 hours of static data are still recommended.

If necessary, temporary control points will be placed in stable locations and will consist of one of the following:

- Rebar with cap (in soil)
- Hub (2-inch-by-2-inch-by-4-inch wooden stake) with tack (in soil)
- Magnetic nail (in asphalt)
- Etching on a permanent surface (i.e. rock outcrop or concrete slab)

3.2. Setting Up the Base Reference Station

In the absence of a CORS signal, a base reference station will be erected over a known monument or control point. Condition and description of the monument will be documented in the logbook.

A GPS antenna will be mounted on a level tribrach attached to a tripod located directly over the control point. The coordinates will be entered into the GPS base station, which will be allowed to initialize, acquire satellites, and begin broadcasting a correction signal. The following details will be recorded in the field logbook:

- X, Y, and Z coordinates entered into the base station controller along with the projection system and vertical datum
- The antenna height above the monument or control point
- The approximate time the base station was initialized

3.3. Initializing a Survey Session

A new survey job will be created in the GPS rover for each project, preferably for each day of surveying. The name of the job will include site, project, and time-specific information enabling subsequent survey data users to identify data contents and applicable dates. Survey quality control check shots and other QA/QC tasks will be collected in accordance with Section 3.4.

All points, lines, or areas collected will be named in a consistent fashion, using a pre-loaded code list (preferably SDSFIE 2.6 or 3.0 compliant) as shown in Table 1, a group of shorthand names for features from an industry standard data set, or a key for the shorthand will be included in the survey team logbook (e.g., Point ID equals location ID) (USACE 2011).

Table 1
Survey Code List

Code	Code Name	Survey Feature Attributes
Structure Codes		
BBC	BuildingCorner	Actual, Offset, Other
BF	Fence	Metal, Wood, Plastic, Other
BUL	UtilityLocation	Proposed Excavation, Temporary Survey Marks, Power, Natural Gas, Communication, Water, Reclaimed Water, Sewer and Drains, Other
BW	Wall	Stone, Brick, Wood, Concrete, Soldier Pile, Soil Nail, Unknown
Observation Codes		
OL	Observation	Photo, Sketch, Wildlife, Notes, Other
OSL	SampleLocation	Pre-Construction, Post-Construction, Waste Characterization, Soil Characterization, Water Characterization, Work Exposure, Field Screening, Other
Survey Codes		
SCP	ControlPoint	NGS Monument, Other Monument, Temp Control Point, Check Shot
SG	Grid	Sampling, Excavation, Archaeological, Other
SM	MiscPoint	OPEN TEXT
STS	Transect	Geophysical Survey, Excavation Sidewall, Observation Traverse
Topographic Codes		
TBL	BreakLine	OPEN TEXT
TGS	GroundShot	OPEN TEXT
TSL	StationLine	Base Line, Sub-Line, Centerline, Other
TS	Shoreline	Lake, Pond, River, Stream, Ocean, Sea, Former, Other
TTR	TransRoute	Highway, Primary Road, Gravel Road, Dirt Road, Railroad, Trail, Other
TWF	WaterFeature	Lake, Pond, River, Stream, Ocean, Sea, Former, Other
Exploration		
XBH	Borehole	OPEN TEXT
XMW	Well	Monitoring Well, Lysimeter, Water Well
XMZ	LandManageZone	Excavation, Stockpile, Storage Area, Staging Area, Area of Interest, Other
XPW	PiezoWell	OPEN TEXT
Environmental Codes		
AST	AST	Gasoline, AvGas, Diesel, Arctic Diesel, Lube Oil, Used Oil, Bunker C, Sewage, Water, Chemicals, Other
UST	UST	Gasoline, AvGas, Diesel, Arctic Diesel, Lube Oil, Used Oil, Bunker C, Sewage, Water, Chemicals, Other
UXO	UXO	OPEN TEXT

3.4. Quality Assurance/Quality Control

Quality of survey data will be assessed based on the horizontal and vertical quality reported by the GPS system for each point collected and comparison of survey check shots. Quality assurance measures will include the collection of check shots on known monuments or other control points during each survey session (NOAA 2011, USACE 2003).

The surveyor will document setup and operations in the field logbook, preferably as follows:

- The CORS station being used as control
  - Include coordinates and approximate distance from survey site
- Sketch of each monument or control point set or occupied (non CORS setups)
  - Include distance and direction to a fixed object
- Sketch of the survey with point numbers in areas with high point density
- Coordinates and coordinate system entered into the base station each time it is set up
- Any change in rover height and the time of the change
- Variance from the SOP
  - Description of why the change was made
  - An assessment of the impacts to survey quality.

The following procedures will be conducted and noted in the field logbook for each survey session (if applicable):

- Visual inspection of the base station prior to data collection to verify that the tribrach sight (optical or laser) is centered over the monument or control point (non-CORS setups)
- Take check shots on a known control point or monument:
  - Prior to data collection (1 location)
  - At each separate survey site (1 location)
  - Each 4 hours of continuous survey (1 location)
  - Post data collection (1 location, same as initial locations)
- Visual inspection of the base station post data collection to verify that the tribrach sight (optical or laser) is centered over the monument or control point (non-CORS)

Survey accuracy will be estimated as described below:

Two Standard Deviation Estimated Accuracy

$$Easting(E) Accuracy: Acc_E = 2 * \sqrt{|E_a - E_m|^2_1 + |E_a - E_m|^2_2 + \cdots + |E_a - E_m|^2_n}$$

$$Northing(N)\ Accuracy: Acc_N = 2 * \sqrt{|N_a - N_m|^2_1 + |N_a - N_m|^2_2 + \cdots + |N_a - N_m|^2_n}$$

$$Elevation(V)\ Accuracy: Acc_V = 2 * \sqrt{|V_a - V_m|^2_1 + |V_a - V_m|^2_2 + \cdots + |V_a - V_m|^2_n}$$

Combined Positional Estimated Accuracy

$$Positional(P)\ Accuracy: Acc_P = \sqrt{Acc_E{}^2 + Acc_N{}^2}$$

a = actual/published value
m = measured value
Acc = Accuracy

The following additional features will be collected as needed to assist quality assurance evaluation:

- Permanent site features (i.e. slab corners, fire hydrants, rock outcrops)
- Aerially recognizable features (i.e. intersections, bends in roads/rivers, field corners)

4.0 SURVEY DATA REPORT

Northing, easting, and vertical (if applicable) coordinates will be reported in the systems outlined below unless a different system is required by the project statement of work:

- Projection: Universal Transverse Mercator (UTM) in the applicable zone (Attachment 1)
- Horizontal Datum: WGS 84
- Vertical Datum: NAVD 88 (orthometric heights from Geoid 09)

A summary survey table will report survey data with the at least the following fields:

- Point ID
- Description
- Northing Coordinate (Y)
- Easting Coordinate (X)
- Orthometric Elevation (Z)
- Ellipsoid Height
- Date and Time Taken
- Survey Duration
- Survey Method
- Survey Equipment Used
- Spatial Reference
- Positional Precision
- Height Precision
- Positional Accuracy
- Height Accuracy

Attachment 2 is provided for Alaska State Plane zone reference as this coordinate system was often used for previous surveys. X, Y, and Z coordinates will be reported in the survey table using the units specified in the project statement of work. A template survey table is included as Attachment 3.

5.0 REFERENCES

NOAA (National Oceanic and Atmospheric Administration). 2011 (August). NGS (National Geodetic Survey). *User Guidelines for Single Base Real Time GNSS Positioning*

USACE. (U.S. Army Corp of Engineers). 2012 (March). *Survey Markers and Monumentation.* Engineering Manual (EM) 1110-1-1002.

USACE. 2011 (October). Alaska District. *Manual for Electronic Deliverables*.

USACE. 2009 (October). Alaska District. *Manual for Electronic Deliverables*.

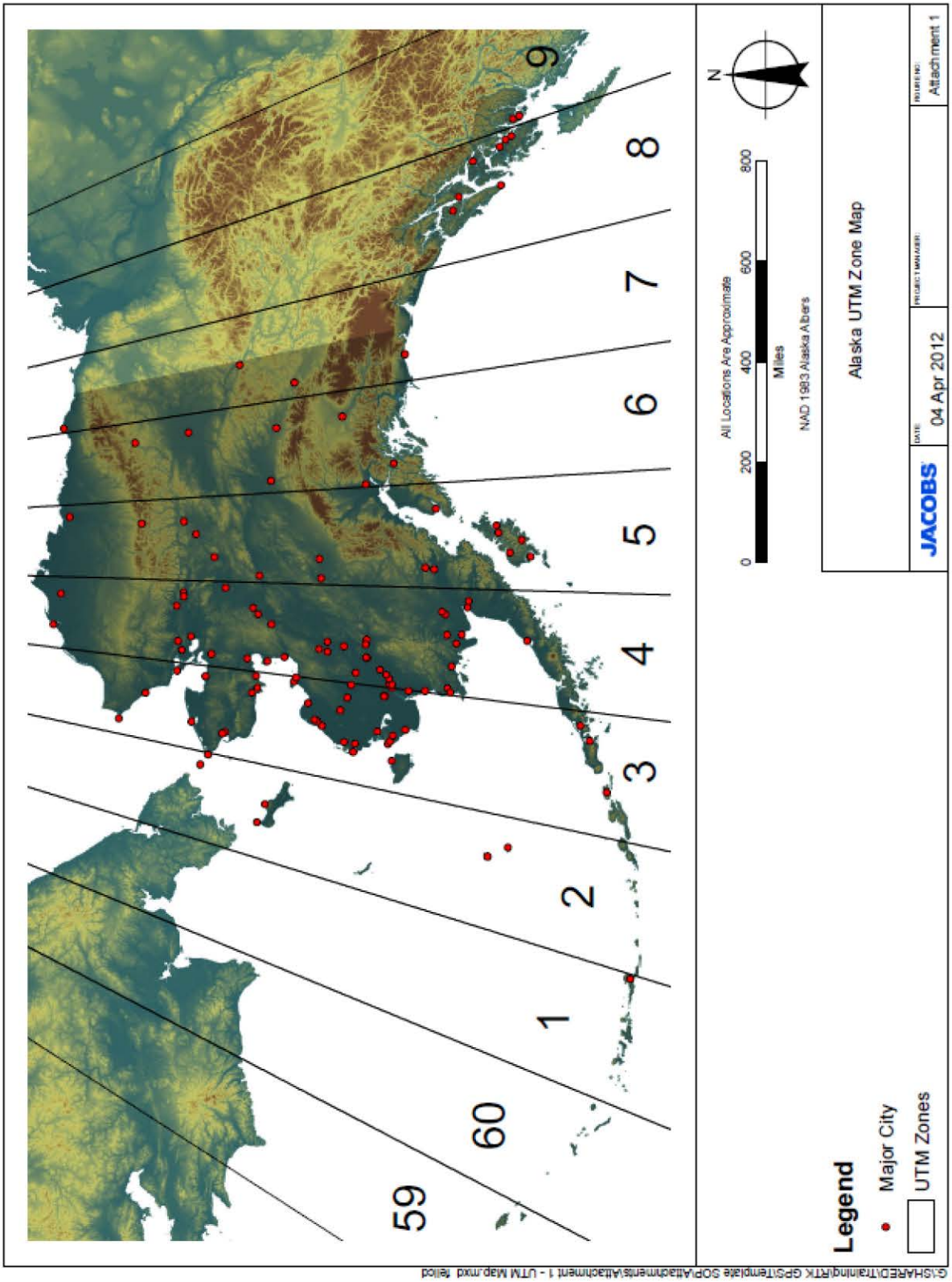
USACE. 2007 (January). *Control and Topographic Surveying*. EM 1110-1-1005.

USACE. 2005 (September). *Geospatial Data and Systems*. EM 1110-1-2909.

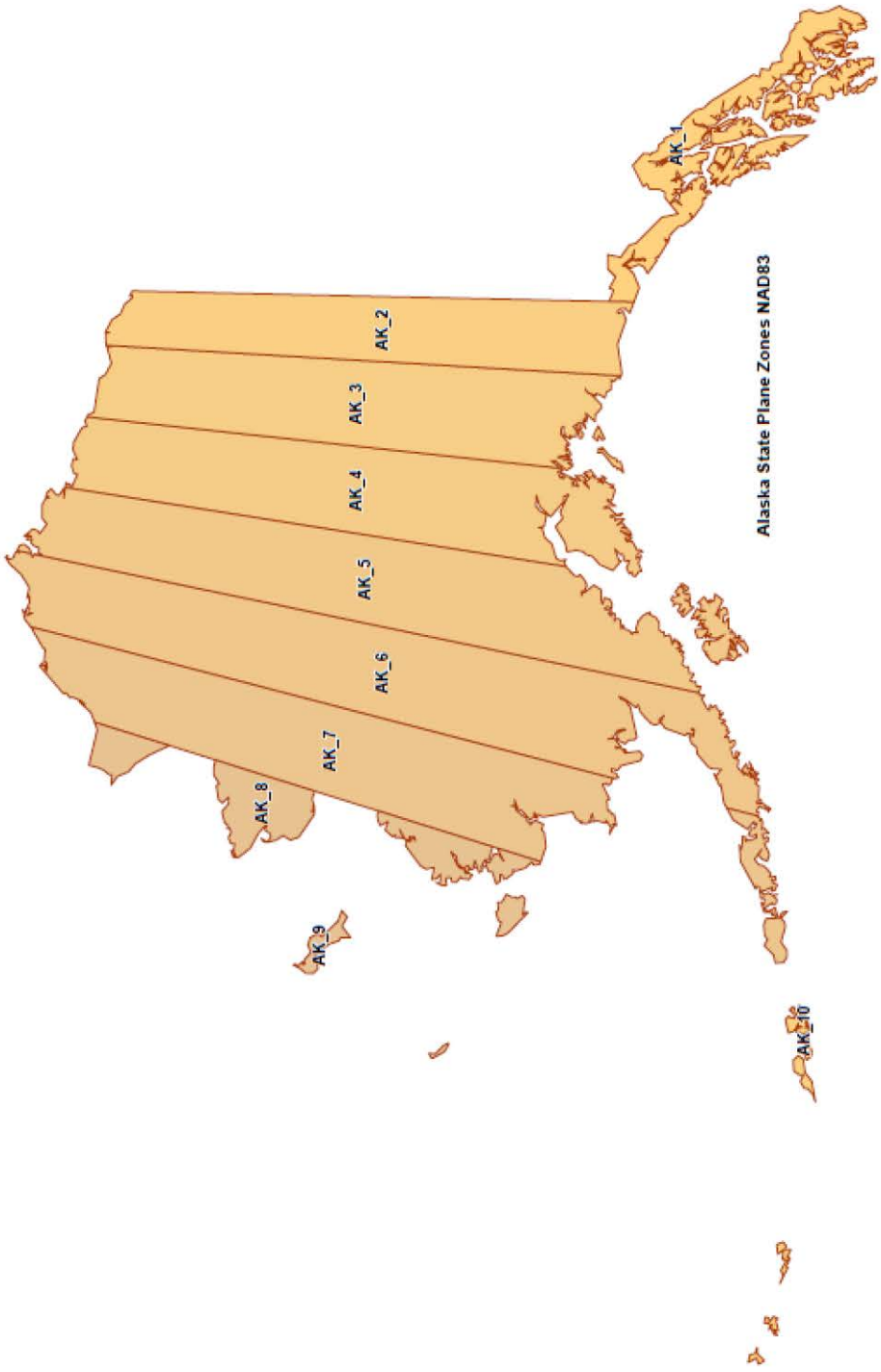
USACE. 2003 (July). *NAVSTAR Global Positioning System Surveying*. EM 1110-1-1003.

USAF (U.S. Air Force). 2005 (December). *Contractors Guide*.

Attachment 1 Alaska Universal Transverse Mercator Zone Map



Attachment 2 Alaska State Plane Zones NAD83



Attachment 3 Template Survey Table

PoingID	Northing	Easting	Orthometric Height	Ellipsoid Height	Description	Date Time Taken	Survey Duration	Survey Method	Survey Equipment	Projection	Geographic Datum	Position Accuracy	Height Accuracy	Position Precision	Height Precision	Position UOM	Elevation UOM	Quality UOM
Example1	1111000.00	256897.00	59.50	50.50	Test data point	3/2/2012 10:15	3/2/2012 to 8/2/2012	RTK-GPS	Leica Viva	UTM Zone 2 North	WGS 1984	0.05000	0.11000	.0023	.0056	Meters	Meters	Meters

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Standard Operating Procedure

Document No: JE-SOP-2000	Page: 1 of 2
Effective Date: 18 January 2011	Rev. 0

Decontamination

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures for decontamination of re-usable equipment used during sampling activities. All non-disposable equipment shall be decontaminated between sampling locations and at the end of the work shift.

This SOP defines a standard set of procedures that may be varied or changed as required by site conditions, equipment limitations, or other factors. Actual decontamination procedures and variances from this SOP will be documented in the field logbooks.

2.0 INTERFERENCES

Improper decontamination may cause cross-contamination of field screening and analytical samples. To prevent cross-contamination of samples, sampling equipment will be disposable and used only once, or reusable but decontaminated before each use. Manufacturer and/or laboratory-certified clean glassware will be used to contain analytical samples.

3.0 EQUIPMENT

Decontamination equipment may include, but is not limited to, the following:

- Appropriate personal protective equipment (PPE) (at minimum, safety glasses and nitrile gloves)
- 5-gallon buckets
- Potable water
- Organic free deionized (DI) water
- LiquiNox® or similar detergent
- Stiff bristle brushes
- Other hand tools for gross decontamination (e.g. shovels and brooms)
- Logbook

4.0 PROCEDURES

4.1. Gross Decontamination

Heavy equipment used onsite will undergo decontamination prior to leaving the site to eliminate contaminant migration from the site as well as the potential for cross-contamination of sites. Gross decontamination includes the removal of potentially contaminated materials

with a shovel or other hand tools and stiff bristle brushes for equipment contaminated by soil, and wiping visible contamination from equipment contaminated by water or free product. All materials removed during gross decontamination will be accumulated and managed with similar waste streams according to the Waste Management section of the Work Plan.

4.2. Decontamination of Sampling Equipment

Contaminant-free disposable sampling equipment will be used whenever possible. All non-disposable equipment that may directly or indirectly contact samples (e.g., shovels, buckets, sampling devices, and instruments) shall be decontaminated prior to sampling. Decontamination will take place over catch basins (e.g., wash tubs and buckets) to minimize the spread of contaminants. Re-usable sampling equipment that may be exposed to the samples will be decontaminated with the following steps:

- Scrub/rinse the equipment using a solution of potable water and laboratory-grade detergent (LiquiNox® or similar product).
- Triple-rinse the equipment with potable water.
- Rinse the equipment with deionized water.

Decontamination water will be accumulated and managed according to the Waste Management section of the Work Plan.

4.3. Quality Control

The following quality control (QC) sample may be collected:

- Equipment rinsate blank

5.0 HEALTH AND SAFETY

Procedures for working with potentially hazardous materials as well as the relevant Material Safety Data Sheets (MSDS) for each chemical that will be used at the site are included in the Site Safety and Health Plan. Personnel using this procedure must be trained on the information contained in the MSDSs, engineering controls, and the PPE used for this procedure.

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Project Operating Procedure

Groundwater Grab Sample Collection

Document No: NE-POP-4010	Page: 1 of 5
Effective Date: 1 August 2013	Rev. 0

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

This Project Operating Procedure (POP) describes the collection of representative water grab samples from soil borings using a submersible pump during drilling. This POP defines a standard set of procedures applicable under typical site conditions and equipment; they may be varied as dictated by actual site conditions and equipment characteristics. Field notes shall be sufficiently specific to document adherence to these procedures or to record relevant details of variances. For specific sampling locations and analytes, refer to the Supplement to the NE Cape Work Plan.

1.1. Sample Containers, Preservation, Handling, and Storage

Samples will be collected into appropriate sample containers as specified and provided by the analytical laboratory subcontractor. Upon collection, containers will be labeled with the sample identification string and time of collection as specified in the work plan. Samples will be stored in pre-chilled coolers at 4 ± 2 °C (or as required by the analytical method).Samples will then be packaged and shipped to the analytical laboratory for analysis.

1.2. Potential Interferences

A potential issue associated with groundwater grab sampling is cross-contamination. To prevent cross-contamination between locations, new unused tubing will be used at each location and disposable equipment that may be in contact samples (e.g., multi-meter and oil/water interface probe) will be decontaminated prior to use. See the Decontamination Standard Operating Procedure for decontamination procedures.

2.0 EQUIPMENT

Groundwater grab sampling equipment may include, but is not limited to the equipment listed in Table 1.

Table 1
Sampling Equipment

Activity	Type/Name	Quantity	Notes
All	Modified Level D personal protective equipment	Per person	
All	Logbook, ballpoint pen/felt tip marker	As needed	
All	Nylon twine	As needed	
Sampling	Groundwater sampling form	1 per well	
Sampling	Sample labels	As needed	Information specified in the Work Plan shall be included.
Sampling	Sampling containers and packing materials	As needed	Preservatives in sample containers, as specified by the analytical method
Sampling	Tape measure	1 per sampling team	Engineer scale (hundredths of a foot), preferable
Sampling	Oil/Water interface probe	1	Product/water level measurements
Sampling	Multi-meter with flow-thru cell (YSI or equivalent)	1	Field parameter measurements
Sampling	Peristaltic pump or bailer	1	Collecting water from sampling point
Sampling	5-gallon bucket	As needed	Storage of purged wastewater or decontamination water
Sampling	Graduated cylinder or beaker	1	To measure pumping rate of water from well
Sampling	deionized water	As needed	Used for decontamination of development equipment
Sampling	Tubing	As needed	Appropriately sized to match selected pumps

3.0 PROCEDURES AND EQUIPMENT

3.1. Recording Field Observations

Grab-sampling information will be recorded in the logbook and on field sampling forms.

3.2. Field Instrument Calibration

Field instrument calibration will be verified in accordance with the manufacturer's recommended procedures for each instrument prior to use. Operation and maintenance manuals will be available in the field for reference. Calibrations will be evaluated at the beginning of each day prior to use. If any reading is outside +/- 15% from the expected calibration standard, the equipment will be re-calibrated.

3.3. Free Product and Water Level Measurement

The depth to free product (if present) and the depth to groundwater will be measured with an interface probe. Interface probes provide distinct responses when immersed in nonconductive product or conductive water. The type and order of measurement activities include determining the reference level and taking water level measurements:

- Reference Level
  - Measure and record sampling point stickup above the ground (Measured to the nearest 0.1 foot)
  - Use this measurement as a correction to report "below ground surface" depths.
- Product and Water Level Measurements (Measured to the nearest 0.01 foot)
  - Measure the depth to free product (if present)
  - Measure the thickness of free product (if present)
  - Measure the depth to groundwater
  - Monitor the depth to groundwater during purging and sample collection

3.4. Groundwater Grab Sample Collection Using Temporary Well Points (SP16 or similar screened tooling)

Temporary well points (e.g., Geoprobe® SP16 tooling) are small-diameter screens for one-time sampling of groundwater. They are grab-sampled rather than developed and purged in according to the procedures associated with permanent monitoring wells.

Grab sampling will utilize a peristaltic pump attached to the sampling port (for SP16 tooling) or connected to a length of new polyethylene tubing inserted down the riser to the screen. At shallow depths, pumping may induce drawdown until the resultant increase in hydraulic head increases the recharge rate to match the pumping rate. Purging commences after the water level stabilizes. Purging of temporary groundwater grab sample locations will be attempted to clear out sediment from the sampling point as follows:

- A length of new silicone tubing will be threaded through the pump.
- Set pumping speed so that a flow rate of 0.25 liters per minute (as measured with a graduated bucket) is established.
- Purging will remove three tubing volumes of water; then field parameter measurement and sampling will commence

Table 2
Tubing Purge Volumes

Tubing Inside Diameter (inches)	Volume (milliliters per foot)
1/8	2.4
¼	9.7
3/8	21.7
½	38.6

Note:
The minimum purge volume assumes 5 tubing volumes

If a flow rate of 0.25 liters per minute induces drawdown or purges the sampling point dry, determine the recharge rate of the sampling point. Grab sampling may be discontinued at a location that will not produce sufficient water.

3.5. Field Parameter Measurements

Verify the calibration of the multi-meter using the appropriate solutions. Recalibrate the meter according to the manufactures instructions if needed. Using the metal cup provided with the YSI, complete the following:

- Fill the cup to ¾ full from the outlet end of the tubing attached to the peristaltic pump
- Insert the probe assembly into the cup and power on the meter.
- Allow the meter to equilibrate for 2 minutes.
- Record readings in the field logbook [temperature, pH, oxidation reduction potential (orp), conductivity, dissolved oxygen, and turbidity)], then note any other items of interest about the measurement process.

After readings are recorded in the logbook and/or field form, drain the cup into the IDW collection bucket and decontaminate the probe using an alcanox/water solution wash followed by a DI water rinse. Repeat the wash and rinse two additional times.

Power off the meter and return it to the storage case.

3.6. Sample Collection

Begin filling sample containers according to volatility of the analytes of interest using the containers provided by the laboratory. The container for the highest volatility analysis (VOCs) will be collected first followed by PAHs. The order of the remaining containers for other analytes will not need to be specified. In general the following will apply to sample collection:

- Fill VOC containers first and make sure no headspace in present in the containers.
- Do not overfill container as preservatives could be lost affecting sample quality
- Label sample containers immediately following collection.
- Place samples upright in a pre-chilled cooler immediately after sample collection. Upon return to the field office, samples will be refrigerated until packaged for shipping.
- Record in the field logbook the sample identification, the sample collection location (sketch), the depth from which the sample was collected, and if a duplicate sample was collected, any discoloration or odor, and other pertinent details

Specific sample containers and preservatives are outlined on Worksheet #19.

4.0 QUALITY CONTROL SAMPLES

The following quality control (QC) samples will be collected at each location:

- Matrix spike and matrix spike duplicate (MS/MSD)
- Field duplicate

The frequency of QC samples is stated in Worksheet #20.

5.0 HEALTH AND SAFETY

Procedures for working with potentially hazardous materials as well as the relevant Material Safety Data Sheets (MSDS) for each chemical that will be used at the site are included in the SSHP. Personnel using this POP must be trained on the information contained in the MSDSs, engineering controls, and the personal protective equipment (PPE) outlined in the SSHP.

Water samples will be treated as potentially containing contaminants of concern. Care must be used when handling water samples to prevent the possible spreading of contaminants in the work area. At a minimum, Level D PPE, including nitrile gloves and safety glasses, will be worn while collecting water samples. Purged groundwater will be handled and disposed of as described in the waste management section of the Work Plan.

6.0 REFERENCES

ADEC. 2010 (May). *Draft Field Sampling Guidance*.

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Project Operating Procedure

Document No: NE-POP-4100	Page: 1 of 2
Effective Date: 1 August 2013	Rev. 0

Surface Water or Wastewater Sampling

Table of Contents

1.0 SCOPE AND APPLICATION

This Project Operating Procedure (POP) describes the procedures for collection of analytical samples from surface water. Samples are obtained from the water surface using hand tools.

This POP defines a standard set of procedures that may be varied or changed as required by site conditions, equipment limitations, or other factors. Actual collection procedures and variances from this POP will be documented in the field logbooks.

Analytical surface water samples will be collected in accordance with Alaska Administrative Code (AAC) Title 18 Chapter 75 protocol.

1.1. Sample Preservation, Containers, Handling, and Storage

Samples will be collected using the appropriate unused sample containers (with preservative, if required by the analytical method) provided by the analytical laboratory. Sample containers will be labeled with the sample identification number, date and time of collection, and sampler initials. Samples will be maintained at 4 degrees Celsius (°C) ± 2 °C while in storage (if required by the analytical method). Samples will then be packaged and transported to the subcontracted laboratory for analysis.

1.2. Potential Interferences

One potential issue associated with grab sampling surface water is cross-contamination between samples. To prevent cross-contamination of samples, sampling equipment will be disposable and used only once.

2.0 EQUIPMENT

Surface water sampling equipment may include, but is not limited to, the following:

- Appropriate personal protective equipment (PPE) (at a minimum safety glasses and nitrile gloves)
- Appropriate size and quantity of sample containers (with preservative, if required by the analytical method) and sample packing materials
- Disposable Teflon® dipper
- Sample labels
- Survey stakes, pin flags, or similar to mark sample locations
- Tape measure
- Resealable plastic bags
- Chain-of-custody
- Camera
- Logbook

3.0 PROCEDURES

3.1. Surface Water Sampling

Surface water samples will be collected using hand tools. Typically, surface samples are collected from just below the water surface using a Teflon dipper. Sampling from depths greater than 6 inches below water surface is considered outside the scope of the procedure.

3.2. Collecting Samples

Samples will be collected at the locations specified in the Work Plan. Samples will be collected as follows:

- Don new PPE (gloves, etc.) before starting sample collection.
- Verify that all needed equipment is readily available and that the sample containers are new and have been properly prepared.
- Label container and sample-specific data sheet, if applicable.
- Using a dipper, collect water from the sampling location by slightly submerging the dipper just below the water surface at a slight angle. Allow the container to fill with minimal agitation of the water.
- Begin filling sample containers according to volatility of the analytes of interest using the containers provided by the laboratory. The container for the highest volatility analysis (VOCs) will be collected first followed by PAHs. The order of the remaining containers for other analytes will not need to be specified. In general the following will apply to sample collection:
- Fill VOC containers first and make sure no headspace in present in the containers.
- Do not overfill container as preservatives could be lost affecting sample quality
- Label sample containers immediately following collection.
- Place samples upright in a pre-chilled cooler immediately after sample collection. Upon return to the field office, samples will be refrigerated until packaged for shipping.
- Record in the field logbook the sample identification, the sample collection location (sketch), the depth from which the sample was collected, and if a duplicate sample was collected, any discoloration or odor, and other pertinent details
- For field filtered samples, a peristaltic pump will be used to transfer water from the dipper through a disposable in-line 0.45 µm filter prior to the sample container.

Specific sample containers and preservatives are outlined on Worksheet #19.

4.0 QUALITY CONTROL SAMPLES

The following quality control (QC) samples will be collected at each location:

- Matrix spike and matrix spike duplicate (MS/MSD)
- Field duplicate

The frequency of QC samples is stated in Worksheet #20.

5.0 HEALTH AND SAFETY

Procedures for working with potentially hazardous materials, as well as the relevant Material Safety Data Sheets (MSDS) for each chemical that will be used at the site, are included in the Health and Safety Plan, provided separately from the Supplement to the NE Cape Work Plan. Personnel using this procedure must be trained on the information contained in the MSDSs, engineering controls, and the personal PPE outlined in this procedure.

All samples will be treated as potentially containing contaminants of concern. Care must be used when handling samples to prevent the possible spreading of contaminants in the work area. At a minimum, Level D PPE, including nitrile gloves and safety glasses, will be worn while collecting soil samples.

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Project Operating Procedure

Document No: NE-POP-7000	Page: 1 of 3
Effective Date: 01 August 2013	Rev. 0

Field Documentation

TABLE OF CONTENTS

ATTACHMENTS

1.0 SCOPE AND APPLICATION

This Project Operating Procedure (POP) describes procedures for documenting field activities and guidance on types and specificity of data to be recorded. Procedures are included for documentation on logbooks, field forms, and/or photographic record. This POP provides a standard set of procedures applicable under typical site conditions and equipment; they may be varied as dictated by actual site conditions and equipment characteristics. Field notes shall be sufficiently specific to document adherence to these procedures or to record all relevant details of any variances.

This POP is consistent with *Field Sampling Guidance* issued by the Alaska Department of Environmental Conservation (ADEC 2010). That document may be referred to for an overview of considerations, equipment, and procedures. For specific sampling goals, refer to the project Work Plan.

2.0 EQUIPMENT

Activity	Type/Name	Quantity	Notes
All	Modified Level D PPE	Per person	See Site Safety Health Plan (SSHP)
All	Camera	1	
All	Logbook	As needed	
All	Waterproof pen	As needed	
All	Field Forms	As needed	

3.0 PROCEDURES

Field documentation procedure includes documentation format, list of entries, entry changes, and management

3.1. Documentation Format

Field activities shall be recorded using a logbook. Logbooks shall be bound books that are permanently assigned to a specific project. A copy of the daily logbook checklist (Attachment 1) shall be inserted on the backside of logbook front cover to be used as a reference tool for field documentation.

Field forms and camera may be used for field documentation in a variety of activities. Field forms include borehole logs, well construction, well sampling, site safety and health plan forms, etc. It is not necessary to duplicate information recorded on a field form into the logbook.

3.2. List of Entries

All logbooks and filed form entries shall be printed legibly using a waterproof pen. All form fields shall be completed in full on a daily basis. At the beginning of each daily entry, the following information shall be recorded:

- Project name / Site ID/ Client
- Date
- Weather, site conditions, and other salient observations
- Level of PPE used
- Full name of onsite personnel, affiliations and project title e.g., team leader (including all visitors)
- Daily objectives
- Time and location of activity
- Filed observations and comments

For investigation activities, the entry for each day shall contain a complete record of daily activities including, but not limited to, the following information, unless the data is recorded on a field form:

- Deviations from the Work Plan
- Photographic log
  - Photographer's name
  - Roll and frame number, or digital photograph number
  - Date and time
  - Description of photograph including sampling point, sample name, depth and other relevant identifying information such as direction faced (e.g., facing north) and relationship of photograph to site features
- Site sketches with reference (i.e. "N" arrow)
- Survey and location i.e., samples or debris (PGS coordinates when possible)
- Field measurements
- Field measurements
- Equipment calibrations and maintenance
- For each sample record:
  - Sample identification numbers
  - Date, time, sampler
  - Media container(s), preservations including Lot number
  - QC samples
  - Analysis
  - Chain-of-custody form numbers
  - Sample shipments (when, what, destination, shipment air bill numbers)
  - Decontamination procedures used
  - Waste tracking (when, how much, destination)
  - Daily summary of activities (i.e., number of samples collected)
  - Time of departure from the site

Logbooks are also used as a daily record for remediation activities. General entries similar to the ones listed above are used in remediation activity logbooks. Additional information shall be documented of remediation activities include but not limited to the following:

- Daily excavation activities
- Waste disposal quantities and methods of transport
- System performance data from any remediation systems (e.g., soil vapor extraction, high vacuum extraction, etc.)
- System or equipment calibration or maintenance performed
- Other pertinent information in regards to remediation daily activities

3.3. Entry Changes

Entry changes should be avoided by carefully entering data in the logbook. Data or other information that is entered incorrectly shall be corrected by drawing a single line through the incorrect entry such that the incorrect entry is not obscured and the correct entry/information is placed next to the incorrect entry in the logbook or field form. The lined through entry shall be initialed and dated.

3.4. Management

Logbooks and field forms will be kept in the project file when complete or when not in use. If forms or logbooks are used in the field for an extended period of time, copies of used pages will be made, delivered to the office, and filed in the project file on a periodic basis.

Photograph prints and negatives will be stored in the project file. Digital photographs will be stored in the electronic project file. Digital photographs shall be downloaded from the camera on a regular basis.

4.0 HEALTH AND SAFETY

Procedures for working with potentially hazardous materials as well as the relevant Material Safety Data Sheets (MSDS) for each chemical that will be used at the site are included in the Site Safety and Health Plan (SSHP). Personnel using this POP must be trained on the information contained in the MSDSs, engineering controls, and the PPE outlined in the SSHP.

Before site activities begin, all personnel onsite including subcontractors and visitors shall either read or be briefed on the information provided in the SSHP and commit to follow applicable procedures by signing the SSHP training record presented in the SSHP.

5.0 REFERENCES

ADEC. 2010 (May). *Draft Field Sampling Guidance*.

USEPA, 2013 (May). Field Branches Quality System and Technical Procedures-Quality System Procedures: Logbooks (SESDPROC-010-R5), Region 4, Science and Ecosystem Support Division (SESD), Athens , Georgia, http://www.epa.gov/region4/sesd/fbqstp/Logbooks.pdf

ATTACHMENT 1
Daily Logbook Checklist

Did you remember … ???

Daily Logbook Checklist

- ☐ Project name / Site ID / Client
- ☐ Date
- ☐ Weather, site conditions, and other salient observations
- ☐ Level of PPE used
- ☐ Full names of onsite personnel and affiliations (including all visitors)
- ☐ Daily objectives
- ☐ Field measurements and calibrations
- ☐ Time and location of activity
- ☐ Field observations and comments
- ☐ Deviations from the Work Plan
- ☐ Site photographs
- ☐ Site sketches (with reference i.e. “N” arrow)
- ☐ Survey and location i.e. samples or debris (GPS coordinates when possible)
- ☐ For each sample record:
  - – Date, time, sampler(s)
  - – Sample ID
  - – Media, container(s), preservatives
  - – QC (dup/MS/MSD)
  - – Analysis
  - – MeOH lot #
  - – Tare weight

- ☐ Sample shipments (when, what, destination)
- ☐ Waste tracking (when, how much, destination)
- ☐ Daily summary of activities (i.e. # of samples collected)

Did you remember … ???

Daily Logbook Checklist

- ☐ Project name / Site ID / Client
- ☐ Date
- ☐ Weather, site conditions, and other salient observations
- ☐ Level of PPE used
- ☐ Full names of onsite personnel and affiliations (including all visitors)
- ☐ Daily objectives
- ☐ Field measurements and calibrations
- ☐ Time and location of activity
- ☐ Field observations and comments
- ☐ Deviations from the Work Plan
- ☐ Site photographs
- ☐ Site sketches (with reference i.e. “N” arrow)
- ☐ Survey and location i.e. samples or debris (GPS coordinates when possible)
- ☐ For each sample record:
  - – Date, time, sampler(s)
  - – Sample ID
  - – Media, container(s), preservatives
  - – QC (dup/MS/MSD)
  - – Analysis
  - – MeOH lot #
  - – Tare weight

- ☐ Sample shipments (when, what, destination)
- ☐ Waste tracking (when, how much, destination)
- ☐ Daily summary of activities (i.e. # of samples collected)

APPENDIX C

Laboratory Standard Operating Procedures

ADEC Certification	
DoD ELAP Certification	
EXT-3510.r10	Separatory Funnel Liquid-Liquid Extraction
EXT-3520.r14	Continuous Liquid-Liquid Extraction
MET-6020.r15	Determination of Metals and Trace Elements by ICP-MS
MET-7470A.r15	Mercury in Liquid Waste
MET-DIG.r14	Metals Digestion of Aqueous Samples
PET-GRO.r9	GRO by GC
PET-SVF.r12	Analysis of Water, Solids, and Soluble Waste Samples for Semivolatile Fuel Hydrocarbons
SOC-8082Ar.r16	PCBs as Aroclors
SVM-8270S.r6	SVOCs by GC-MS SIM
VOC-8260.r17	VOCs by GC-MS

THE STATE OF ALASKA
Department of Environmental Conservation
Laboratory Approval Program

Scope of Approval

Expiration: 06/12/2014

ALS Environmental-Kelso, WA UST-040
1317 S 13th Avenue
Kelso, WA 98626

is approved by the State of Alaska Department of Environmental Conservation , pursuant to 18 AAC 78, to perform analysis for the parameters listed below using the analytical methods indicated. Approval for all parameters is final. Approval is for the latest version of a method unless specified otherwise in a note. EPA refers to the U.S. Environmental Protection Agency. AK refers to Alaska Methods 101, 102 and 103 for the determination of gasoline, diesel and residual range organics in soil and water. ASTM refers to the American Society for Testing and Materials.

Contaminated Sites

Method/Test Name	Reference	Analyte	Matrix	Status
6010C	EPA	Total Arsenic	Soil	Approved
6010C	EPA	Total Barium	Soil	Approved
6010C	EPA	Total Cadmium	Soil	Approved
6010C	EPA	Total Chromium	Soil	Approved
6010C	EPA	Total Lead	Soil	Approved
6010C	EPA	Total Nickel	Soil	Approved
6010C	EPA	Total Vanadium	Soil	Approved
6010C	EPA	Total Arsenic	Water	Approved
6010C	EPA	Total Barium	Water	Approved
6010C	EPA	Total Cadmium	Water	Approved
6010C	EPA	Total Chromium	Water	Approved
6010C	EPA	Total Lead	Water	Approved
6010C	EPA	Total Nickel	Water	Approved
6010C	EPA	Total Vanadium	Water	Approved
6020A	EPA	Total Arsenic	Soil	Approved
6020A	EPA	Total Barium	Soil	Approved
6020A	EPA	Total Cadmium	Soil	Approved
6020A	EPA	Total Chromium	Soil	Approved
6020A	EPA	Total Lead	Soil	Approved

Contaminated Sites

Method/Test Name	Reference	Analyte	Matrix	Status
6020A	EPA	Total Nickel	Soil	Approved
6020A	EPA	Total Vanadium	Soil	Approved
6020A	EPA	Total Arsenic	Water	Approved
6020A	EPA	Total Barium	Water	Approved
6020A	EPA	Total Cadmium	Water	Approved
6020A	EPA	Total Chromium	Water	Approved
6020A	EPA	Total Lead	Water	Approved
6020A	EPA	Total Nickel	Water	Approved
6020A	EPA	Total Vanadium	Water	Approved
7060A	EPA	Total Arsenic	Water	Approved
8021B	EPA	BTEX	Water	Approved
8082A	EPA	Polychlorinated Biphenyls-PCB	Soil	Approved
8082A	EPA	Polychlorinated Biphenyls-PCB	Water	Approved
8260C	EPA	BTEX	Soil	Approved
8260C	EPA	Total Volatile Chlorinated Solvents	Soil	Approved
8260C	EPA	BTEX	Water	Approved
8260C	EPA	Total Volatile Chlorinated Solvents	Water	Approved
8270D	EPA	PAH	Soil	Approved
8270D	EPA	PAH	Water	Approved
AK101	AK	Gasoline Range Organics	Soil	Approved
AK101	AK	Gasoline Range Organics	Water	Approved
AK101/8021B	EPA	BTEX-methanol preserved	Soil	Approved
AK102	AK	Diesel Range Organics	Soil	Approved
AK102	AK	Diesel Range Organics	Water	Approved
AK103	AK	Residual Range Organics	Soil	Approved

PERRY JOHNSON LABORATORY ACCREDITATION, INC.

Certificate of Accreditation

Perry Johnson Laboratory Accreditation, Inc. has assessed the Laboratory of:

Columbia Analytical Services, Inc.

1317 South 13th Avenue, Kelso, WA 98626

(Hereinafter called the Organization) and hereby declares that Organization has met the requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the DoD Quality Systems Manual for Environmental Laboratories Version 4.2 4/22/2009 and is accredited is accordance with the:

United States Department of Defense
Environmental Laboratory Accreditation Program
(DoD-ELAP)

This accreditation demonstrates technical competence for the defined scope:

Environmental Testing

(As detailed in the supplement)

Accreditation claims for such testing and/or calibration services shall only be made from addresses referenced within this certificate. This Accreditation is granted subject to the system rules governing the Accreditation referred to above, and the Organization hereby covenants with the Accreditation body's duty to observe and comply with the said rules.

For PJLA:

Initial Accreditation Date:	*Issue Date:*	*Accreditation No.:*	*Certificate No.:*
July 19, 2011	March 1, 2012	65188	L12-28

Tracy Szerszen
President/Operations Manager

Perry Johnson Laboratory
Accreditation, Inc. (PJLA)
755 W. Big Beaver, Suite 1325
Troy, Michigan 48084

The validity of this certificate is maintained through ongoing assessments based on a continuous accreditation cycle. The validity of this certificate should be confirmed through the PJLA website: www.pjlabs.com

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

Columbia Analytical Services, Inc.

1317 South 13th Avenue, Kelso, WA 98626

Julie Gish Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard / Method	Technology	Analyte
Aqueous	EPA 1631E	CVAFS	Mercury (Low level)
Aqueous	EPA 1664A	Gravimetry	Hexane Extractable Material (HEM)
Aqueous	EPA 1664A	Gravimetry	Total Petroleum Hydrocarbons (TPH)
Aqueous	EPA 180.1	Nephelometer	Turbidity
Aqueous	EPA 2340B	Calculation by 6010	Hardness as $CaCO_3$)
Aqueous	EPA 245.1	CVAA	Mercury
Aqueous	EPA 300.0	IC	Bromide
Aqueous	EPA 300.0	IC	Chloride
Aqueous	EPA 300.0	IC	Fluoride
Aqueous	EPA 300.0	IC	Nitrate + Nitrite as N
Aqueous	EPA 300.0	IC	Nitrate as N
Aqueous	EPA 300.0	IC	Nitrite as N
Aqueous	EPA 300.0	IC	Sulfate
Aqueous	EPA 353.2	Automated Colorimetry	Nitrate + Nitrite as N
Aqueous	EPA 7196A	Colorimetry	Chromium VI
Aqueous	EPA 7470A	CVAA	Mercury
Aqueous	EPA 8260C SIM	GC-MS	1,1,2,2-Tetrachloroethane
Aqueous	EPA 8260C SIM	GC-MS	1,1,2-Trichloroethane
Aqueous	EPA 8260C SIM	GC-MS	1,1-Dichloroethene
Aqueous	EPA 8260C SIM	GC-MS	1,2-Dibromoethane (EDB)
Aqueous	EPA 8260C SIM	GC-MS	1,2-Dichloroethane
Aqueous	EPA 8260C SIM	GC-MS	1,3 Butadine
Aqueous	EPA 8260C SIM	GC-MS	1,4-Dichlorobenzene
Aqueous	EPA 8260C SIM	GC-MS	Bromodichloromethane
Aqueous	EPA 8260C SIM	GC-MS	Carbon Tetrachloride
Aqueous	EPA 8260C SIM	GC-MS	Chlorodibromomethane
Aqueous	EPA 8260C SIM	GC-MS	Chloroform
Aqueous	EPA 8260C SIM	GC-MS	Chloromethane
Aqueous	EPA 8260C SIM	GC-MS	cis-1,2-Dichloroethene
Aqueous	EPA 8260C SIM	GC-MS	Dichloromethane (Methylene Chloride)
Aqueous	EPA 8260C SIM	GC-MS	Tetrachloroethene
Aqueous	EPA 8260C SIM	GC-MS	trans-1,2-Dichloroethene
Aqueous	EPA 8260C SIM	GC-MS	Trichloroethene

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Julie Gish Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard / Method	Technology	Analyte
Aqueous	EPA 8260C SIM	GC-MS	Vinyl chloride
Aqueous	EPA 9020B	Microcoulometric-titration detector	Total Organic Halides (TOX)
Aqueous	EPA 9040C	pH Meter	pH
Aqueous	EPA 9060A	TOC Meter	Total Organic Carbons (TOC)
Aqueous	SM 2130B	Nephelometer	Turbidity
Aqueous	SM 4500 CN- G	Colorimetry	Cyanide, Amenable
Aqueous	SM 4500 P-E	Colorimetry	ortho-phosphorous
Aqueous	SM 4500 S2 D	Distillation Unit	Sulfide
Aqueous	SM2320B	Titrimetry	Total Alkalinity (as $CaCO_3$)
Aqueous	SM2510B	Conductivity Meter	Specific Conductance
Aqueous	SM2540B	Balance	Solids, Total
Aqueous	SM2540C	Balance	Solids, Total Dissolved
Aqueous	SM2540D	Balance	Solids, Total Suspended
Aqueous	SM4500CN E	Colorimetry	Total Cyanide
Aqueous	SM4500CN-G	Colorimetry	Cyanide, Amenable
Aqueous	SM4500NH3 G	Colorimetry	Ammonia
Aqueous	SM5220C	Titrimetry	Chemical Oxygen Demand (COD)
Aqueous	SM5310C	TOC Meter	Total Organic Carbons (TOC)
Aqueous	SOP-LCP-PFC	HPLC/MS/MS	Perfluor-n butanoic acid (PFBA)
Aqueous	SOP-LCP-PFC	HPLC/MS/MS	Perfluor-n octanesulfonate (PFOS)
Aqueous	SOP-LCP-PFC	HPLC/MS/MS	Perfluor-n octanoic acid (PFOA)
Aqueous/Drinking Water	EPA 200.9	GFAA	Antimony
Aqueous/Drinking Water	EPA 200.9	GFAA	Selenium
Aqueous/Drinking Water	EPA 200.9	GFAA	Thallium
Aqueous/Drinking Water	EPA 200.9	GFAA	Arsenic
Aqueous/Drinking Water	EPA 200.9	GFAA	Lead
Aqueous/Solid	ASTM D 1426-93B	ISE	Nitrogen, Total Kjeldahl (TKN)
Aqueous/Solid	EPA 1630	CVAFS	Methyl Mercury
Aqueous/Solid	EPA 1020A	Closed Cup Flashpoint	Ignitability
Aqueous/Solid	EPA 314.0	IC	Perchlorate
Aqueous/Solid	EPA 350.1	Colorimetry	Ammonia
Aqueous/Solid	EPA 365.3	Colorimetry	Total Phosphorus
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Aluminum

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Accreditation is granted to the facility to perform the following testing:

Matrix	Standard / Method	Technology	Analyte
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Antimony
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Arsenic
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Barium
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Beryllium
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Boron
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Cadmium
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Calcium
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Chromium, total
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Cobalt
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Copper
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Iron
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Lead
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Magnesium
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Manganese
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Molybdenum
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Nickel
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Potassium
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Selenium
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Silver
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Sodium
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Strontium
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Thallium
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Tin
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Titanium
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Vanadium
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Zinc
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Aluminum
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Antimony
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Arsenic
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Barium
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Beryllium
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Boron
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Cadmium
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Chromium, total

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

Columbia Analytical Services, Inc.

1317 South 13th Avenue, Kelso, WA 98626

Julie Gish Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard / Method	Technology	Analyte
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Cobalt
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Copper
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Iron
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Lead
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Manganese
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Molybdenum
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Nickel
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Selenium
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Silver
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Strontium
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Thallium
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Tin
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Titanium
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Vanadium
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Zinc
Aqueous/Solid	EPA 7010	GFAA	Antimony
Aqueous/Solid	EPA 7010	GFAA	Arsenic
Aqueous/Solid	EPA 7010	GFAA	Chromium, total
Aqueous/Solid	EPA 7010	GFAA	Lead
Aqueous/Solid	EPA 7010	GFAA	Selenium
Aqueous/Solid	EPA 7010	GFAA	Thallium
Aqueous/Solid	EPA 7742	AA, Borohydride Reduction; GFAA	Selenium
Aqueous/Solid	EPA 8015C/AK103-RRO	GC-FID	Residual Range Organics (RRO)
Aqueous/Solid	EPA 8015C; AK101-GRO; NWTPH-Gx	GC-FID	Gasoline Range Organics (GRO)
Aqueous/Solid	EPA 8015C; AK102-DRO; NWTPH-Dx	GC-FID	Diesel Range Organics (DRO)
Aqueous/Solid	EPA 8021B	GC-FID	Benzene
Aqueous/Solid	EPA 8021B	GC-FID	Ethyl Benzene
Aqueous/Solid	EPA 8021B	GC-FID	Toluene
Aqueous/Solid	EPA 8021B	GC-FID	Xylene, total
Aqueous/Solid	EPA 8081A, B	GC-ECD	Aldrin
Aqueous/Solid	EPA 8081A, B	GC-ECD	Alpha-BHC

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

Columbia Analytical Services, Inc.

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Julie Gish Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard / Method	Technology	Analyte
Aqueous/Solid	EPA 8081A, B	GC-ECD	DDD (4,4)
Aqueous/Solid	EPA 8081A, B	GC-ECD	DDE (4,4)
Aqueous/Solid	EPA 8081A, B	GC-ECD	DDT (4,4)
Aqueous/Solid	EPA 8081A, B	GC-ECD	delta-BHC
Aqueous/Solid	EPA 8081A, B	GC-ECD	Dieldrin
Aqueous/Solid	EPA 8081A, B	GC-ECD	Endosulfan I
Aqueous/Solid	EPA 8081A, B	GC-ECD	Endosulfan II
Aqueous/Solid	EPA 8081A, B	GC-ECD	Endosulfan sulfate
Aqueous/Solid	EPA 8081A, B	GC-ECD	Endrin
Aqueous/Solid	EPA 8081A, B	GC-ECD	Endrin aldehyde
Aqueous/Solid	EPA 8081A, B	GC-ECD	Endrin ketone
Aqueous/Solid	EPA 8081A, B	GC-ECD	gamma-BHC
Aqueous/Solid	EPA 8081A, B	GC-ECD	gamma-Chlordane
Aqueous/Solid	EPA 8081A, B	GC-ECD	Heptachlor
Aqueous/Solid	EPA 8081A, B	GC-ECD	Heptachlor Epoxide (beta)
Aqueous/Solid	EPA 8081A, B	GC-ECD	Methoxychlor
Aqueous/Solid	EPA 8081A, B	GC-ECD	Toxaphene (total)
Aqueous/Solid	EPA 8081B	GC-ECD	2,4-DDD
Aqueous/Solid	EPA 8081B	GC-ECD	2,4-DDE
Aqueous/Solid	EPA 8081B	GC-ECD	2,4-DDT
Aqueous/Solid	EPA 8081B	GC-ECD	Chlorpyrifos
Aqueous/Solid	EPA 8081B	GC-ECD	cis-Nonachlor
Aqueous/Solid	EPA 8081B	GC-ECD	Hexachlorobenzene
Aqueous/Solid	EPA 8081B	GC-ECD	Hexachlorobutadiene
Aqueous/Solid	EPA 8081B	GC-ECD	Hexachloroethane
Aqueous/Solid	EPA 8081B	GC-ECD	Isodrin
Aqueous/Solid	EPA 8081B	GC-ECD	Mirex
Aqueous/Solid	EPA 8081B	GC-ECD	Oxychlordane
Aqueous/Solid	EPA 8081B	GC-ECD	trans-Nonachlor
Aqueous/Solid	EPA 8082, A	GC-ECD	2,2,3,3,4,4,5,5,6-Nonachlorobiphenyl (PCB 206)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,2,3,3,4,4,5,6-Octachlorobiphenyl (PCB 195)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,2,3,3,4,4,5-Heptachlorobiphenyl (PCB 170)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,2,3,3,4,4-Hexachlorobiphenyl (PCB 128)

Columbia Analytical Services, Inc.

1317 South 13th Avenue, Kelso, WA 98626

Julie Gish Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard / Method	Technology	Analyte
Aqueous/Solid	EPA 8082, A	GC-ECD	2,2,3,4,4,5,5-Heptachlorobiphenyl (PCB180)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,2,3,4,4,5,6-Heptachlorobiphenyl (PCB 183)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,2,3,4,4,5-Hexachlorobiphenyl (PCB 138)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,2,3,4,4,6,6-Heptachlorobiphenyl (PCB 184)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,2,3,4,5,5,6-Heptachlorobiphenyl (PCB 187)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,2,3,4,5-Pentachlorobiphenyl (PCB87)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,2,3,4,5-Pentachlorobiphenyl (PCB90)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,2,3,5-Tetrachlorobiphenyl (PCB44)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,2,4,4,5,5-Hexachlorobiphenyl (PCB153)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,2,4,5,5-Pentachlorobiphenyl (PCB 101)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,2,5,5-Tetrachlorbiphenyl (PCB 53)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,2,5-Trichlorobiphenyl (PCB18)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,3,3,4,4,5,5-Heptachlorobiphenyl (PCB 189)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,3,3,4,4,5-Hexachlorobiphenyl (PCB 156)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,3,3,4,4,5-Hexachlorobiphenyl (PCB 157)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,3,3,4,4,6-Hexachlorobiphenyl (PCB 158)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,3,3,4,4-Pentachlorobiphenyl (PCB 105)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,3,4,4,5,5 Hexachlorobiphenyl (PCB 167)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,3,4,4,5,6-Hexachlorobiphenyl (PCB 168)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,3,4,4,5-Pentachlorobiphenyl (PCB 114)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,3,4,4,5-Pentachlorobiphenyl (PCB 118)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,3,4,4,5-Pentachlorobiphenyl (PCB 123)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,3,4,4-Tetrachlorobiphenyl (PCB60)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,3,4,4-Tetrachlorobiphenyl (PCB66)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,4,4-Trichlorobiphenyl (PCB 28)
Aqueous/Solid	EPA 8082, A	GC-ECD	2,4-Dichlorobiphenyl (PCB8)
Aqueous/Solid	EPA 8082, A	GC-ECD	3,3,4,4,5,5-Hexachlorobiphenyl (PCB 169)
Aqueous/Solid	EPA 8082, A	GC-ECD	3,3,4,4,5-Pentachlorobiphenyl (PCB 126)
Aqueous/Solid	EPA 8082, A	GC-ECD	3,3,4,4-Tetrachlorobiphenyl (PCB 77)
Aqueous/Solid	EPA 8082, A	GC-ECD	3,4,4,5-Tetrachlorobiphenyl (PCB 81)
Aqueous/Solid	EPA 8082, A	GC-ECD	Aroclor 1016
Aqueous/Solid	EPA 8082, A	GC-ECD	Aroclor 1221
Aqueous/Solid	EPA 8082, A	GC-ECD	Aroclor 1232

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

Columbia Analytical Services, Inc.

1317 South 13th Avenue, Kelso, WA 98626

Julie Gish Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard / Method	Technology	Analyte
Aqueous/Solid	EPA 8082, A	GC-ECD	Aroclor 1242
Aqueous/Solid	EPA 8082, A	GC-ECD	Aroclor 1248
Aqueous/Solid	EPA 8082, A	GC-ECD	Aroclor 1254
Aqueous/Solid	EPA 8082, A	GC-ECD	Aroclor 1260
Aqueous/Solid	EPA 8082, A	GC-ECD	Aroclor 1262
Aqueous/Solid	EPA 8082, A	GC-ECD	Aroclor 1268
Aqueous/Solid	EPA 8082, A	GC-ECD	Decachlorobiphenyl (PC B209)
Aqueous/Solid	EPA 8151A	GC-ECD	2,4,5-T
Aqueous/Solid	EPA 8151A	GC-ECD	2,4,5-TP (Silvex)
Aqueous/Solid	EPA 8151A	GC-ECD	2,4-D
Aqueous/Solid	EPA 8151A	GC-ECD	2,4-DB
Aqueous/Solid	EPA 8151A	GC-ECD	Dalapon
Aqueous/Solid	EPA 8151A	GC-ECD	Dicamba
Aqueous/Solid	EPA 8151A	GC-ECD	Dichloroprop
Aqueous/Solid	EPA 8151A	GC-ECD	Dinoseb
Aqueous/Solid	EPA 8151A	GC-ECD	MCPA
Aqueous/Solid	EPA 8151A	GC-ECD	MCPP
Aqueous/Solid	EPA 8260B, C	GC-MS	1-phenylpropane
Aqueous/Solid	EPA 8260B, C	GC-MS	Benzene
Aqueous/Solid	EPA 8260B, C	GC-MS	DIPE
Aqueous/Solid	EPA 8260B, C	GC-MS	ETBE
Aqueous/Solid	EPA 8260B, C	GC-MS	Ethyl Benzene
Aqueous/Solid	EPA 8260B, C	GC-MS	Freon 11
Aqueous/Solid	EPA 8260B, C	GC-MS	Freon 113
Aqueous/Solid	EPA 8260B, C	GC-MS	MTBE
Aqueous/Solid	EPA 8260B, C	GC-MS	TAME
Aqueous/Solid	EPA 8260B, C	GC-MS	tert-Butyl alcohol
Aqueous/Solid	EPA 8260B, C	GC-MS	Toluene
Aqueous/Solid	EPA 8260B, C	GC-MS	Xylene, total
Aqueous/Solid	EPA 8260B, C	GC-MS	1,1,1,2-Tetrachloroethane
Aqueous/Solid	EPA 8260B, C	GC-MS	1,1,1-Trichloroethane
Aqueous/Solid	EPA 8260B, C	GC-MS	1,1,2,2-Tetrachloroethane
Aqueous/Solid	EPA 8260B, C	GC-MS	1,1,2-Trichloroethane

Columbia Analytical Services, Inc.
1317 South 13th Avenue, Kelso, WA 98626
Julie Gish Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard / Method	Technology	Analyte
Aqueous/Solid	EPA 8260B, C	GC-MS	1,1-Dichloroethane
Aqueous/Solid	EPA 8260B,C	GC-MS	1,1-Dichloroethene
Aqueous/Solid	EPA 8260B,C	GC-MS	1,1-Dichloropropene
Aqueous/Solid	EPA 8260B,C	GC-MS	1,2,3-Trichlorobenzene
Aqueous/Solid	EPA 8260B,C	GC-MS	1,2,3-Trichloropropane
Aqueous/Solid	EPA 8260B,C	GC-MS	1,2,4-Trichlorobenzene
Aqueous/Solid	EPA 8260B,C	GC-MS	1,2,4-Trimethylbenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	1,2-Dibromoethane (EDB)
Aqueous/Solid	EPA 8260B, C	GC-MS	1,2-Dichlorobenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	1,2-Dichloroethane
Aqueous/Solid	EPA 8260B, C	GC-MS	1,2-Dichloropropane
Aqueous/Solid	EPA 8260B, C	GC-MS	1,3,5-Trimethylbenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	1,3-Dichlorobenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	1,3-Dichloropropane
Aqueous/Solid	EPA 8260B, C	GC-MS	1,4-Dichlorobenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	2,2-Dichloropropane
Aqueous/Solid	EPA 8260B, C	GC-MS	2-Butanone (MEK)
Aqueous/Solid	EPA 8260B, C	GC-MS	2-Chloroethylvinlether
Aqueous/Solid	EPA 8260B, C	GC-MS	2-Chlorotoluene
Aqueous/Solid	EPA 8260B, C	GC-MS	2-Hexanone
Aqueous/Solid	EPA 8260B, C	GC-MS	4-Chlorotoluene
Aqueous/Solid	EPA 8260B, C	GC-MS	4-Isopropyltoluene
Aqueous/Solid	EPA 8260B, C	GC-MS	4-Methyl-2-pentanone (MIBK)
Aqueous/Solid	EPA 8260B, C	GC-MS	Acetone
Aqueous/Solid	EPA 8260B, C	GC-MS	Acetonitrile
Aqueous/Solid	EPA 8260B, C	GC-MS	Acrolein
Aqueous/Solid	EPA 8260B, C	GC-MS	Acrylonitrile
Aqueous/Solid	EPA 8260B, C	GC-MS	Benzene
Aqueous/Solid	EPA 8260B, C	GC-MS	Bromobenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	Bromochloromethane
Aqueous/Solid	EPA 8260B, C	GC-MS	Bromodichloromethane
Aqueous/Solid	EPA 8260B, C	GC-MS	Bromoform
Aqueous/Solid	EPA 8260B, C	GC-MS	Bromomethane

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

Columbia Analytical Services, Inc.

1317 South 13th Avenue, Kelso, WA 98626

Julie Gish Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard / Method	Technology	Analyte
Aqueous/Solid	EPA 8260B, C	GC-MS	Carbon disulfide
Aqueous/Solid	EPA 8260B, C	GC-MS	Carbon Tetrachloride
Aqueous/Solid	EPA 8260B, C	GC-MS	Chlorobenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	Chlorodibromomethane
Aqueous/Solid	EPA 8260B, C	GC-MS	Chloroethane
Aqueous/Solid	EPA 8260B, C	GC-MS	Chloroform
Aqueous/Solid	EPA 8260B, C	GC-MS	Chloromethane
Aqueous/Solid	EPA 8260B, C	GC-MS	cis-1,2-Dichloroethene
Aqueous/Solid	EPA 8260B, C	GC-MS	cis-1,3-Dichloropropene
Aqueous/Solid	EPA 8260B, C	GC-MS	Dibromomethane
Aqueous/Solid	EPA 8260B, C	GC-MS	Dichlorodifluoromethane
Aqueous/Solid	EPA 8260B, C	GC-MS	Dichloromethane (Methylene Chloride)
Aqueous/Solid	EPA 8260B, C	GC-MS	Di-isopropylether (DIPE)
Aqueous/Solid	EPA 8260B, C	GC-MS	Ethylbenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	Hexachlorobutadiene
Aqueous/Solid	EPA 8260B, C	GC-MS	Isopropylbenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	Methyl-tert-butylether (MTBE)
Aqueous/Solid	EPA 8260B, C	GC-MS	Naphthalene
Aqueous/Solid	EPA 8260B, C	GC-MS	n-Butylbenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	n-Propylbenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	sec-Butylbenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	Styrene
Aqueous/Solid	EPA 8260B, C	GC-MS	tert-amylmethylether (TAME)
Aqueous/Solid	EPA 8260B, C	GC-MS	tert-butylbenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	Tetrachloroethene
Aqueous/Solid	EPA 8260B, C	GC-MS	Toluene
Aqueous/Solid	EPA 8260B, C	GC-MS	trans-1,2-Dichloroethene
Aqueous/Solid	EPA 8260B, C	GC-MS	trans-1,3-Dichloropropene
Aqueous/Solid	EPA 8260B, C	GC-MS	Trichloroethene
Aqueous/Solid	EPA 8260B, C	GC-MS	Trichlorofluoromethane (Freon 11)
Aqueous/Solid	EPA 8260B, C	GC-MS	Vinyl acetate
Aqueous/Solid	EPA 8260B, C	GC-MS	Vinyl chloride
Aqueous/solid	EPA 8260B, C	GC-MS	Xylenes, total

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

Columbia Analytical Services, Inc.

1317 South 13th Avenue, Kelso, WA 98626

Julie Gish Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard / Method	Technology	Analyte
Aqueous/Solid	EPA 8270C, D	GC-MS	1,2,4-Trichlorobenzene
Aqueous/Solid	EPA 8270C, D	GC-MS	1,2-Dichlorobenzene
Aqueous/Solid	EPA 8270C, D	GC-MS	1,3-Dichlorobenzene
Aqueous/Solid	EPA 8270C, D	GC-MS	1,4-Dichlorobenzene
Aqueous/Solid	EPA 8270C, D	GC-MS	2,4,5-Trichlorophenol
Aqueous/Solid	EPA 8270C, D	GC-MS	2,4,6-Trichlorophenol
Aqueous/Solid	EPA 8270C, D	GC-MS	2,4-Dichlorophenol
Aqueous/Solid	EPA 8270C, D	GC-MS	2,4-Dimethylphenol
Aqueous/Solid	EPA 8270C, D	GC-MS	2,4-Dinitrophenol
Aqueous/Solid	EPA 8270C, D	GC-MS	2,4-Dinitrotoluene
Aqueous/Solid	EPA 8270C, D	GC-MS	2,6-Dichlorophenol
Aqueous/Solid	EPA 8270C, D	GC-MS	2,6-Dinitrotoluene
Aqueous/Solid	EPA 8270C, D	GC-MS	2-Chloronaphthalene
Aqueous/Solid	EPA 8270C, D	GC-MS	2-Chlorophenol
Aqueous/Solid	EPA 8270C, D	GC-MS	2-Methyl-4,6-Dinitrophenol
Aqueous/Solid	EPA 8270C, D	GC-MS	2-Methylnaphthalene
Aqueous/Solid	EPA 8270C, D	GC-MS	2-Methylphenol
Aqueous/Solid	EPA 8270C, D	GC-MS	2-Nitroaniline
Aqueous/Solid	EPA 8270C, D	GC-MS	2-Nitrophenol
Aqueous/Solid	EPA 8270C, D	GC-MS	3,3-Dichlorobenzidine
Aqueous/Solid	EPA 8270C, D	GC-MS	3-Nitroaniline
Aqueous/Solid	EPA 8270C, D	GC-MS	4-Bromophenyl-phenylether
Aqueous/Solid	EPA 8270C, D	GC-MS	4-Chloro-3-methylphenol
Aqueous/Solid	EPA 8270C, D	GC-MS	4-Chloroaniline
Aqueous/Solid	EPA 8270C, D	GC-MS	4-Chlorophenyl-phenylether
Aqueous/Solid	EPA 8270C, D	GC-MS	4-Methylphenol (and/or 3-Methylphenol)
Aqueous/Solid	EPA 8270C, D	GC-MS	4-Nitroaniline
Aqueous/Solid	EPA 8270C, D	GC-MS	4-Nitrophenol
Aqueous/Solid	EPA 8270C, D	GC-MS	Acenaphthene
Aqueous/Solid	EPA 8270C, D	GC-MS	Acenaphthylene
Aqueous/Solid	EPA 8270C, D	GC-MS	Aniline
Aqueous/Solid	EPA 8270C, D	GC-MS	Anthracene
Aqueous/Solid	EPA 8270C, D	GC-MS	Azinphos-methyl (Guthion)

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

Columbia Analytical Services, Inc.

1317 South 13th Avenue, Kelso, WA 98626

Julie Gish Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard / Method	Technology	Analyte
Aqueous/Solid	EPA 8270C, D	GC-MS	Benzidine
Aqueous/Solid	EPA 8270C, D	GC-MS	Benzo(a)anthracene
Aqueous/Solid	EPA 8270C, D	GC-MS	Benzo(a)pyrene
Aqueous/Solid	EPA 8270C, D	GC-MS	Benzo(b)fluoranthene
Aqueous/Solid	EPA 8270C, D	GC-MS	Benzo(g,h,i)perylene
Aqueous/Solid	EPA 8270C, D	GC-MS	Benzo(k)fluoranthene
Aqueous/Solid	EPA 8270C, D	GC-MS	Benzoic acid
Aqueous/Solid	EPA 8270C, D	GC-MS	Benzyl alcohol
Aqueous/Solid	EPA 8270C, D	GC-MS	bis(2-Chloroethoxy)methane
Aqueous/Solid	EPA 8270C, D	GC-MS	bis(2-Chloroethyl)ether
Aqueous/Solid	EPA 8270C, D	GC-MS	bis(2-Chloroisopropyl)ether
Aqueous/Solid	EPA 8270C, D	GC-MS	bis(2-ethylhexy)phthalate
Aqueous/Solid	EPA 8270C, D	GC-MS	Butyl benzyl phthalate
Aqueous/Solid	EPA 8270C, D	GC-MS	Carbazole
Aqueous/Solid	EPA 8270C, D	GC-MS	Chlorpyrifos
Aqueous/Solid	EPA 8270C, D	GC-MS	Chrysene
Aqueous/Solid	EPA 8270C, D	GC-MS	Demeton O & S
Aqueous/Solid	EPA 8270C, D	GC-MS	Diazinon
Aqueous/Solid	EPA 8270C, D	GC-MS	Dibenzo(a,h)anthracene
Aqueous/Solid	EPA 8270C, D	GC-MS	Dibenzofuran
Aqueous/Solid	EPA 8270C, D	GC-MS	Dichlorvos
Aqueous/Solid	EPA 8270C, D	GC-MS	Diethyl phthalate
Aqueous/Solid	EPA 8270C, D	GC-MS	dimethoate
Aqueous/Solid	EPA 8270C, D	GC-MS	Dimethylphthalate
Aqueous/Solid	EPA 8270C, D	GC-MS	di-n-butylphthalate
Aqueous/Solid	EPA 8270C, D	GC-MS	Di-n-octylphthalate
Aqueous/Solid	EPA 8270C, D	GC-MS	Disulfoton
Aqueous/Solid	EPA 8270C, D	GC-MS	Ethoprop
Aqueous/Solid	EPA 8270C, D	GC-MS	Fluoranthene
Aqueous/Solid	EPA 8270C, D	GC-MS	Fluorene
Aqueous/Solid	EPA 8270C, D	GC-MS	Hexachlorobenzene
Aqueous/Solid	EPA 8270C, D	GC-MS	Hexachlorobutadiene
Aqueous/Solid	EPA 8270C, D	GC-MS	Hexachlorocyclopentadiene

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Matrix	Standard / Method	Technology	Analyte
Aqueous/Solid	EPA 8270C, D	GC-MS	Hexachloroethane
Aqueous/Solid	EPA 8270C, D	GC-MS	Indeno(1,2,3, cd)pyrene
Aqueous/Solid	EPA 8270C, D	GC-MS	Isophorone
Aqueous/Solid	EPA 8270C, D	GC-MS	Naphthalene
Aqueous/Solid	EPA 8270C, D	GC-MS	Nitrobenzene
Aqueous/Solid	EPA 8270C, D	GC-MS	N-Nitrosodiethylamine
Aqueous/Solid	EPA 8270C, D	GC-MS	N-Nitrosodimethylamine
Aqueous/Solid	EPA 8270C, D	GC-MS	N-Nitroso-di-n-propylamine
Aqueous/Solid	EPA 8270C, ~~D~~	GC-MS	N-Nitrosodiphenylamine
Aqueous/Solid	EPA 8270C, D	GC-MS	o-Toluidine
Aqueous/Solid	EPA 8270C, D	GC-MS	Parathion, ethyl
Aqueous/Solid	EPA 8270C, D	GC-MS	Parathion, methyl
Aqueous/Solid	EPA 8270C, D	GC-MS	Pentachlorobenzene
Aqueous/Solid	EPA 8270C, D	GC-MS	Pentachlorophenol
Aqueous/Solid	EPA 8270C, D	GC-MS	Phenanthrene
Aqueous/Solid	EPA 8270C, D	GC-MS	Phenol
Aqueous/Solid	EPA 8270C, D	GC-MS	Phorate
Aqueous/Solid	EPA 8270C, D	GC-MS	Pyrene
Aqueous/Solid	EPA 8270C, D	GC-MS	Pyridine
Aqueous/Solid	EPA 8270C, D	GC-MS	Ronnel
Aqueous/Solid	EPA 8270C, D	GC-MS	Stirophos
Aqueous/Solid	EPA 8270C, D	GC-MS	Sulfotepp
Aqueous/Solid	EPA 8270C, D	GC-MS	2,3,4,6-Tetrachlorophenol
Aqueous/Solid	EPA 8270C,D	GC-MS	1,2,4,5-Tetrachlorobenzene
Aqueous/Solid	EPA 8270SIM	GC-MS	2-Methylnaphthalene
Aqueous/Solid	EPA 8270SIM	GC-MS	Acenaphthene
Aqueous/Solid	EPA 8270SIM	GC-MS	Acenaphthylene
Aqueous/Solid	EPA 8270SIM	GC-MS	Anthracene
Aqueous/Solid	EPA 8270SIM	GC-MS	Benzo(a)anthracene
Aqueous/Solid	EPA 8270SIM	GC-MS	Benzo(a)pyrene
Aqueous/Solid	EPA 8270SIM	GC-MS	Benzo(b)fluoranthene
Aqueous/Solid	EPA 8270SIM	GC-MS	Benzo(g,h,i)perylene
Aqueous/Solid	EPA 8270SIM	GC-MS	Benzo(k)fluoranthene

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

Columbia Analytical Services, Inc.

1317 South 13th Avenue, Kelso, WA 98626

Julie Gish Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard / Method	Technology	Analyte
Aqueous/Solid	EPA 8270SIM	GC-MS	Chrysene
Aqueous/Solid	EPA 8270SIM	GC-MS	Dibenzo(a,h)anthracene
Aqueous/Solid	EPA 8270SIM	GC-MS	Fluoranthene
Aqueous/Solid	EPA 8270SIM	GC-MS	Fluorene
Aqueous/Solid	EPA 8270SIM	GC-MS	Indeno(1,2,3, cd)pyrene
Aqueous/Solid	EPA 8270SIM	GC-MS	Naphthalene
Aqueous/Solid	EPA 8270SIM	GC-MS	p-Dioxane
Aqueous/Solid	EPA 8270SIM	GC-MS	Phenanthrene
Aqueous/Solid	EPA 8270SIM	GC-MS	Pyrene
Aqueous/Solid	EPA 8330B	HPLC	1,3,5-Trinitrobenzene
Aqueous/Solid	EPA 8330B	HPLC	1,3-Dinitrobenzene
Aqueous/Solid	EPA 8330B	HPLC	2,4,6-Trinitrotoluene
Aqueous/Solid	EPA 8330B	HPLC	2,4-Dinitrotoluene
Aqueous/Solid	EPA 8330B	HPLC	2,6-Dinitrotoluene
Aqueous/Solid	EPA 8330B	HPLC	2-Amino-4,6-dinitrtoluene
Aqueous/Solid	EPA 8330B	HPLC	2-Nitrotoluene
Aqueous/Solid	EPA 8330B	HPLC	3,5-Dinitroaniline
Aqueous/Solid	EPA 8330B	HPLC	3-Nitrotoluene
Aqueous/Solid	EPA 8330B	HPLC	4-Amino-2,6-dinitrotoluene
Aqueous/Solid	EPA 8330B	HPLC	4-Nitrotoluene
Aqueous/Solid	EPA 8330B	HPLC	HMX (Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine)
Aqueous/Solid	EPA 8330B	HPLC	Nitrobenzene
Aqueous/Solid	EPA 8330B	HPLC	Nitroglycerin
Aqueous/Solid	EPA 8330B	HPLC	Pentachloronitrobenzene
Aqueous/Solid	EPA 8330B	HPLC	Pentaerythritoltetranitrate
Aqueous/Solid	EPA 8330B	HPLC	RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)
Aqueous/Solid	EPA 8330B	HPLC	Tetryl (methyl-2,4,6-trinitrophenylnitramine)
Aqueous/Solid	EPA 9012B,	Colorimetry	Total Cyanide
Aqueous/Solid	EPA 9030B	Distillation Unit	Sulfide
Aqueous/Solid	EPA 9056A	IC	Bromide
Aqueous/Solid	EPA 9056A	IC	Chloride
Aqueous/Solid	EPA 9056A	IC	Fluoride

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Matrix	Standard / Method	Technology	Analyte
Aqueous/Solid	EPA 9056A	IC	Sulfate
Aqueous/Solid	EPA 9065	Spectrophotometer	Total Phenolics
Aqueous/Solid	LCP-NITG	HPLC/UV	Nitroguanidine
Aqueous/Solid	SM4500 NH3 G	Colorimetry	Ammonia
Aqueous/Solid	SOC-OTTO	GC-ECD	Otto Fuel
Aqueous/Solid	SOC-Butyl	GC-FPD	Di-n-butyltin
Aqueous/Solid	SOC-Butyl	GC-FPD	n-Butyltin
Aqueous/Solid	SOC-Butyl	GC-FPD	Tetra-n-butyltin
Aqueous/Solid	SOC-Butyl	GC-FPD	Tri-n-butyltin
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Aldrin
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Alpha-BHC
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	beta-BHC
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	DDD (4,4)
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	DDE (4,4)
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	DDT (4,4)
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	delta-BHC
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Dieldrin
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Endosulfan I
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Endosulfan II
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Endosulfan sulfate
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Endrin
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Endrin aldehyde
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Endrin ketone
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	gamma-BHC
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Heptachlor
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Heptachlor Epoxide (beta)
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Methoxychlor
Drinking Water	EPA 504	GC-ECD	1,2-Dibromo-3-chloropropane (DBCP)
Drinking Water	EPA 504	GC-ECD	1,2-Dibromoethane (EDB)
Drinking Water	EPA 524.2	GC-MS	1,1,1,2-Tetrachloroethane
Drinking Water	EPA 524.2	GC-MS	1,1,1-Trichloroethane
Drinking Water	EPA 524.2	GC-MS	1,1,2,2-Tetrachloroethane
Drinking Water	EPA 524.2	GC-MS	1,1-Dichloroethane
Drinking Water	EPA 524.2	GC-MS	1,1-Dichloroethene

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

Columbia Analytical Services, Inc.

1317 South 13th Avenue, Kelso, WA 98626

Julie Gish Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard / Method	Technology	Analyte
Drinking Water	EPA 524.2	GC-MS	1,1-Dichloropropene
Drinking Water	EPA 524.2	GC-MS	1,2,3-Trichlorobenzene
Drinking Water	EPA 524.2	GC-MS	1,2,3-Trichloropropane
Drinking Water	EPA 524.2	GC-MS	1,2,4-Trichlorobenzene
Drinking Water	EPA 524.2	GC-MS	1,2,4-Trimethylbenzene
Drinking Water	EPA 524.2	GC-MS	1,2-Dibromoethane (EDB)
Drinking Water	EPA 524.2	GC-MS	1,2-Dichlorobenzene
Drinking Water	EPA 524.2	GC-MS	1,2-Dichloroethane
Drinking Water	EPA 524.2	GC-MS	1,2-Dichloropropane
Drinking Water	EPA 524.2	GC-MS	1,3,5-Trimethylbenzene
Drinking Water	EPA 524.2	GC-MS	1,3-Dichlorobenzene
Drinking Water	EPA 524.2	GC-MS	1,3-Dichloropropane
Drinking Water	EPA 524.2	GC-MS	1,4-Dichlorobenzene
Drinking Water	EPA 524.2	GC-MS	2,2-Dichloropropane
Drinking Water	EPA 524.2	GC-MS	2-Chlorotoluene
Drinking Water	EPA 524.2	GC-MS	4-Chlorotoluene
Drinking Water	EPA 524.2	GC-MS	4-Isopropyltoluene
Drinking Water	EPA 524.2	GC-MS	Benzene
Drinking Water	EPA 524.2	GC-MS	Bromobenzene
Drinking Water	EPA 524.2	GC-MS	Bromochloromethane
Drinking Water	EPA 524.2	GC-MS	Bromodichloromethane
Drinking Water	EPA 524.2	GC-MS	Bromoform
Drinking Water	EPA 524.2	GC-MS	Bromomethane
Drinking Water	EPA 524.2	GC-MS	Carbon Tetrachloride
Drinking Water	EPA 524.2	GC-MS	Chlorobenzene
Drinking Water	EPA 524.2	GC-MS	Chlorodibromomethane
Drinking Water	EPA 524.2	GC-MS	Chloroethane
Drinking Water	EPA 524.2	GC-MS	Chloroform
Drinking Water	EPA 524.2	GC-MS	Chloromethane
Drinking Water	EPA 524.2	GC-MS	cis-1,2-Dichloroethene
Drinking Water	EPA 524.2	GC-MS	cis-1,3-Dichloropropene
Drinking Water	EPA 524.2	GC-MS	Dibromomethane
Drinking Water	EPA 524.2	GC-MS	Dichlorodifluoromethane
Drinking Water	EPA 524.2	GC-MS	Dichloromethane (Methylene Chloride)

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Julie Gish Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard / Method	Technology	Analyte
Drinking Water	EPA 524.2	GC-MS	Ethylbenzene
Drinking Water	EPA 524.2	GC-MS	Hexachlorobutadiene
Drinking Water	EPA 524.2	GC-MS	Isopropylbenzene
Drinking Water	EPA 524.2	GC-MS	m+p-Xylene
Drinking Water	EPA 524.2	GC-MS	Naphthalene
Drinking Water	EPA 524.2	GC-MS	n-Butylbenzene
Drinking Water	EPA 524.2	GC-MS	n-Propylbenzene
Drinking Water	EPA 524.2	GC-MS	o-Xylene
Drinking Water	EPA 524.2	GC-MS	sec-Butylbenzene
Drinking Water	EPA 524.2	GC-MS	Styrene
Drinking Water	EPA 524.2	GC-MS	tert-butylbenzene
Drinking Water	EPA 524.2	GC-MS	Tetrachloroethene
Drinking Water	EPA 524.2	GC-MS	Toluene
Drinking Water	EPA 524.2	GC-MS	trans-1,2-Dichloroethene
Drinking Water	EPA 524.2	GC-MS	trans-1,3-Dichloropropene
Drinking Water	EPA 524.2	GC-MS	Trichlorofluoromethane (Freon 11)
Drinking Water	EPA 524.2	GC-MS	Vinyl chloride
Drinking Water	EPA 524.2	GC-MS	Xylenes, total
Solid	ASTMD4129-92M, Lloyd Kahn	TOC Meter	Total Organic Carbons (TOC)
Solid	EPA 160.3M	Gravimetry	Solids, Total
Solid	EPA 7471A, B	CVAA	Mercury
Solid	EPA 9045D	pH Meter	pH
Solid	EPA 9056A	IC	Nitrate as N
Solid	EPA 9056A	IC	Nitrite as N
Solid	EPA 9071B	Gravimetry	Hexane Extractable Material (HEM)
Solid	GEN-AVS	Colorimetry	Acid Volatile Sulfides
Solid	GEN-NCEL	Colorimetry	Nitrocellulose
Solid	LCP-LCMS4	HPLC/MS/MS	1,3,5-Trinitrobenzene
Solid	LCP-LCMS4	HPLC/MS/MS	1,3-Dinitrobenzene
Solid	LCP-LCMS4	HPLC/MS/MS	2,4,6-Trinitrotoluene
Solid	LCP-LCMS4	HPLC/MS/MS	2,4-Dinitrotoluene
Solid	LCP-LCMS4	HPLC/MS/MS	2,6-Dinitrotoluene
Solid	LCP-LCMS4	HPLC/MS/MS	2-Amino-4,6-dinitrotoluene
Solid	LCP-LCMS4	HPLC/MS/MS	3,5-Dinitroaniline

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

Columbia Analytical Services, Inc.

1317 South 13th Avenue, Kelso, WA 98626

Julie Gish Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard / Method	Technology	Analyte
Solid	LCP-LCMS4	HPLC/MS/MS	4-Amino-2,6-dinitrotoluene
Solid	LCP-LCMS4	HPLC/MS/MS	HMX (Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine)
Solid	LCP-LCMS4	HPLC/MS/MS	Pentaerythritoltetranitrate
Solid	LCP-LCMS4	HPLC/MS/MS	RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)
Solid	LCP-LCMS4	HPLC/MS/MS	Tetryl (methyl-2,4,6-trinitrophenylnitramine)
Solid	LCP-Nitro	HPLC/MS/MS	2,4-Dinitrophenol
Solid	LCP-Nitro	HPLC/MS/MS	Picramic Acid
Solid	LCP-Nitro	HPLC/MS/MS	Picric Acid
Solid	PSEP	Gravimetry	Particle Size

Matrix	Standard / Method	Technology	Analyte
Aqueous	EPA 1640	Reductive Metals Precipitation	Prep Method
Aqueous	EPA 3010A	Acid Digestion	Metals Digestion
Aqueous	EPA 3020A	Acid Digestion	Metals Digestion
Aqueous	EPA 3520C	Continuous Liquid-Liquid Extraction	Extractable Prep
Aqueous	EPA 3535A	Solid Phase Extraction	Prep Method
Aqueous	EPA 5030B	Purge and Trap for Volatiles	Volatile Prep
Aqueous	SOP-MET-DIG	Acid Digestion	Metals Digestion
Aqueous/Solids	EPA 1311	TCLP Extraction	Physical Extraction
Aqueous/Solids	EPA 3620C	Florisil clean up	Extractable Cleanup
Aqueous/Solids	EPA 3630C	Silica gel clean up	Extractable Prep
Aqueous/Solids	EPA 3640A	Gel-Permeation Clean-up	Extractable Cleanup
Aqueous/Solids	EPA 3660	Sulfur Clean-up	Extractable Prep
Aqueous/Solids	EPA 3665A	Acid clean up	Extractable Cleanup
Aqueous/Solids	ASTM D3590-89	Digestion	TKN
Solid	EPA 3050B	Acid Digestion	Metals Digestion
Solid	EPA 3060	Alkaline Digestion for Cr(VI)	Alkaline Digestion for Cr(VI) only
Solid	EPA 3541	Automated Soxhlet Extraction	Extractable Prep
Solid	EPA 3550B	Ultrasonic Extraction	Extractable Prep
Solid	EPA 5035A	Purge and Trap for Volatiles	Voc Organics
Solid	EPA 5050	Bomb Digestion	Prep Method
Solids	EPA 9013	Midi-Distillation	Cyanides

Perry Johnson Laboratory Accreditation, Inc.

January 5, 2012

Julie Gish
Columbia Analytical Services
1317 South 13th Avenue
Kelso, WA 98626

Dear Ms. Gish:

This letter is to confirm that you have successfully completed your reaccreditation assessment. A certificate has now been granted and posted on our website. As you are aware, PJLA will no longer be issuing expiration dates on our certificates. Your certificate # **L12-27 & L12-28** will remain valid as long as you continue to maintain your annual assessments and reaccreditation assessments as stated in your customer agreement with PJLA. At this time, we have confirmed that your annual assessments will be conducted during the month of **Aug** each calendar year. This will include an interim surveillance assessment and a full system reassessment to be completed by **Aug 2014**. Once your reassessment is conducted and approved by our accreditation committee a revised status letter will be provided to you. Please allow PJLA at least 120 days from your assessment due date to issue this letter.

Please feel free to release this letter to any interested parties as confirmation of your certificate validity. Also, please remind them that your certificate is posted on our website at all times. Any changes in regards to your accreditation status will be reflected on our website.

We would like to thank you for your patronage over the past years and look forward to continuously serving your accreditation needs in the future. If we can assist you any further, please feel free to contact us at any time.

Sincerely,

Tracy Szerszen
President/Operations Manager

755 W. Big Beaver Rd. · Suite 1325 · Troy, MI 48084 · Phone: 1-877-369-LABS or 248-519-2603 · Fax: 248-213-0737

ALS Environmental

DOCUMENT TITLE:	***SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTION***
REFERENCED METHOD:	***EPA METHOD 3510C***
SOP ID:	***EXT-3510***
REV. NUMBER:	***10***
EFFECTIVE DATE:	***07/05/2013***

AMMONIA BY FLOW INJECTION ANALYSIS

EPA 350.1

SM 4500-NH3 B-1997, 4500-NH3 G-1997

ALS-KELSO

SOPID:	GEN-350.1	Rev. Number:	10	Effective Date:	07/19/2013

Approved By: _____ Date: 6/24/13
Department Supervisor – Harvey Jacky

Approved By: _____ Date: 6/24/13
QA Manager – Suzanne LeMay

Approved By: _____ Date: 6/25/13
Laboratory Director – Jeff Grindstaff

Issue Date:	_____	Doc Control ID#:	_____	Issued To:	_____

Standard Operating Procedure

For

SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTION

1. SCOPE AND APPLICATION

1.1. This procedure uses techniques described in EPA Method 3510C for extracting nonvolatile and semi-volatile organic compounds from aqueous samples. The procedure also describes concentration techniques suitable for preparing the extract for the appropriate determinative methods.

1.2. This method is applicable to the isolation and concentration of water insoluble and slightly water soluble organics in preparation for a variety of determinative methods which use chromatographic procedures.

2. SUMMARY OF METHOD

2.1. A measured volume of sample, usually 1 liter, is serially extracted at a specified pH with Dichloromethane using a separatory funnel.

2.2. The extract is dried, concentrated, and (if necessary) exchanged to an appropriate solvent for the determinative procedure. The extract may undergo additional cleanup steps defined in other procedures.

3. DEFINITIONS

3.1. **Batch** – A batch of samples is a group of environmental samples that are prepared and/or analyzed together as a unit with the same process and personnel using the same lot(s) of reagents. It is the basic unit for analytical quality control.

3.1.1. Preparation Batch – A preparation batch is composed of one to twenty field samples, all of the same matrix, meeting the criteria in Section 3.3 and with a maximum time between the start of processing of the first and last samples in the batch to be 24 hours.

3.2. **Sample**

3.2.1. Field Sample – An environmental sample collected and delivered to the laboratory for analysis; a.k.a., client's sample.

3.2.2. Laboratory Sample – A representative portion, aliquot, or subsample of a field sample upon which laboratory analyses are made and results generated.

3.3. **Quality System Matrix** – The *matrix* of an environmental sample is distinguished by its physical and/or chemical state and by the program for which the results are

intended. The following sections describe the matrix distinctions. These matrices shall be used for purpose of batch and quality control requirements.

3.3.1. Aqueous – Any groundwater sample, surface water sample, effluent sample, and TCLP or other extract. Specifically excluded are samples of the drinking water matrix and the saline/estuarine water matrix.

3.3.2. Drinking water – Any aqueous sample that has been designated a potable or potential potable water source.

3.3.3. Saline/Estuarine water – Any aqueous sample from an ocean or estuary or other salt-water source.

3.3.4. Non-aqueous Liquid – Any organic liquid with <15% settleable solids.

3.4. **Matrix Spike/Duplicate Matrix Spike (MS/DMS)**– In the matrix spike analysis, predetermined quantities of target analytes are added to a sample matrix prior to sample preparation and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the method used for the analysis. Duplicate samples are spiked, and analyzed as a MS/DMS pair. Percent recoveries are calculated for each of the analytes detected. The relative percent difference (RPD) between the duplicate spikes (or samples) is calculated and used to assess analytical precision.

3.5. **Laboratory Duplicates (DUP)** – Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. The relative percent difference (RPD) between the sample and its duplicate is calculated and used to assess analytical precision.

3.6. **Surrogate** – Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to extraction and analysis. Percent recoveries are calculated for each surrogate.

3.7. **Method Blank (MB)** – The method blank is an artificial sample composed of analyte-free water or solid matrix and is designed to monitor the introduction of artifacts into the analytical process. The method blank is carried through the entire analytical procedure.

3.8. **Laboratory Control Samples (LCS)** – The LCS is an aliquot of analyte free water or analyte free solid to which known amounts target analytes are added. The LCS is prepared and analyzed in exactly the same manner as the samples. The percent recovery is compared to established limits and assists in determining whether the batch is in control.

3.9. **Liquid – Liquid Extraction**: A solute is transferred from one solvent into another via partitioning between the aqueous and solvent phases. The solutes have a higher solubility in the solvent than in the aqueous solution being extracted.

4. SAFETY

4.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

4.2. Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in MSDSs where available. Refer to the ALS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

4.3. This method uses Dichloromethane, a known human carcinogen. Viton brand gloves should be used while rinsing, pouring or transferring the solvent

5. INTERFERENCES

5.1. Phthalate esters can pose difficulties when performing sample extractions for Organochlorine pesticides, PCBs, and other semi-volatile organics. Phthalates are easily extracted or leached from materials containing plastics during laboratory operations. Interferences from phthalates can best be minimized by avoiding contact with any plastic materials.

5.2. Routine cleaning of the extraction glassware is necessary. Refer to the SOP for Organic Extractions Glassware Cleaning.

6. RESPONSIBILITIES

It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

7. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

7.1. Refer to the applicable section in the determinative SOP for sample collection, preservation, and holding times.

7.2. The extract holding time is 40 days from sample preparation to analysis for most methods; however the determinative SOP must be consulted.

8. APPARATUS AND MATERIALS

8.1. Separatory funnel – Appropriate size, with Teflon stopcock.

8.2. Drying column – modified funnel with ground glass bottom. Glass wool is at bottom covered by sulfate.

8.3. Kuderna-Danish (K-D) apparatus (Kontes K-570025-0500).

8.3.1. Concentrator tube - 10 ml, graduated (Kontes K-570050-1025 or equivalent). A ground-glass stopper is used to prevent evaporation of extracts.

8.3.2. Evaporation flask - 500 ml (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs, clamps, or equivalent.

8.3.3. Snyder column - Three ball macro (Kontes K-503000-0121 or equivalent).

8.3.4. Springs - 1/2 inch (Kontes K-662750 or equivalent).

8.4. Boiling chips - Pre-cleaned via DCM rinse, approximately 10/40 mesh (silicon carbide or equivalent).

8.5. Water bath - Heated, with concentric ring cover, capable of temperature control (± 5°C). The bath should be used in a hood.

8.6. Vials - 2 ml, glass with Teflon lined screw-caps or crimp tops.

8.7. pH indicator paper - pH range including the desired extraction pH.

8.8. Erlenmeyer fleaker - 250 ml.

8.9. Syringe - appropriate size syringe or Eppendorf.

8.10. Graduated cylinder - Appropriate size, Class A or validated general lab grade

8.11. Graduated pipettes, appropriate size. Pipettes are pre-tested by lot for accuracy.

9. REAGENTS

9.1. Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination. The preparation for all laboratory prepared reagents and solutions must be documented in a laboratory logbook. Refer to ADM-RTL, *Reagent/Standards Login and Tracking* for the complete procedure and documentation requirements.

9.2. All prepared reagents should be stored in glass to prevent the leaching of contaminants from plastic containers.

9.3. Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One of SW-846.

9.4. Sodium hydroxide solution (l0N), NaOH. Dissolve 40 g NaOH in organic-free reagent water and dilute to 100 ml.

9.5. Sodium Chloride (granular), NaCl.

9.6. Sodium sulfate (granular, anhydrous), Na_2SO_4. Purify by heating at 400°C for 4 hours in a shallow tray.

9.7. Sulfuric acid solution (1:1 v/v), H_2SO_4, purchased. Specific projects may require the use of concentrated HCl.

9.8. Extraction/exchange solvents

9.8.1. Dichloromethane, CH_2Cl_2 – Pesticide quality or equivalent.

9.8.2. Hexane – Pesticide quality or equivalent.

9.8.3. 2-Propanol, $CH_3CH(OH)CH_3$ – Pesticide quality or equivalent.

9.8.4. Acetonitrile, CH_3CN – Pesticide quality or equivalent.

9.8.5. Methyl t-butyl ether (MTBE), Pesticide quality or equivalent.

10. PREVENTIVE MAINTENANCE

Routine cleaning of the extraction glassware is necessary. Refer to EXT-GC, *Organic Extractions Glassware Cleaning*.

11. PROCEDURE

11.1. Test-specific benchsheets are attached. These benchsheets list such information as solvents, solvent exchanges, weights, and volumes specified for the determinative method. Use the correct benchsheet and record all extraction and sample information. To assist the analyst, a brief description of the procedure is given on the backside of the benchsheet.

11.2. Transfer 1L (nominal) of sample to the separatory funnel. When the sample to be extracted is contained in a 1-liter (or smaller) bottle, mark the sample level on the bottle for later volume measurement. The contents of the bottle are poured directly into the separatory funnel. The sample bottle is then rinsed with a portion of the extraction solvent and added to the separatory funnel. Measure and record the sample volume by filling the bottle to the mark then measuring with a graduated cylinder. If high concentrations are anticipated or if sample volume is limited, a smaller volume may be used and then diluted with organic-free reagent water to 1 liter.

11.3. Samples with settled solid material or sediment.

11.3.1. If the sample contains a small amount of material, shake the sample to mix the material into the sample and analyze the entire sample.

11.3.2. If the amount of material is enough to interfere with sample extraction, the Project Chemist should be notified to determine the procedure to be used. The default procedure is to ***completely*** decant the liquid portion of the sample (without shaking the sample) into a graduated cylinder, measure the volume, and transfer to the extraction apparatus. Rinse the graduated cylinder with the extraction solvent. The remaining solid material may be analyzed separately (using an appropriate extraction procedure) depending on the Project Chemist's instructions. It should be documented on the benchsheet when decanting is performed.

11.4. Refer to the determinative SOP (see Table 1 for a list of applicable SOPs) for the preparation, concentration, storage, and expiration for the surrogate, LCS, and MS spiking solutions. These SOPs also list the resulting final spike concentrations. Add the amount indicated on bottle of the surrogate standard solution to all samples, spikes, and blanks (refer to the appropriate standard logbook for details on the surrogate standard solution and the matrix spike solution). For the LCS and sample(s) in each analytical batch selected for matrix spiking, add amount indicated on the bottle of the matrix spiking standard. One set of QC containing matrix spike, duplicate matrix spike, lab control sample and method blank is done for every 20 samples.

11.5. Check the pH of the sample by spotting a wide-range pH strip with the sample using a Pasteur pipette. If necessary, adjust the pH for the specific determinative method that will be used to analyze the extract. Adjustments in pH are made by using sodium hydroxide solution and/or sulfuric acid solution. Specific projects may require the use of concentrated HCl.

DETERMINATIVE PROCEDURE	INITIAL EXTRACTION PH	SECONDARY EXTRACTION PH
ORGANOCHLORINE PESTICIDES (8081)	5-9	none
PCBS AS AROCLORS (8082)	5-9	none
PAHS (8310)	As received	none
PAHs BY GC/MS (8270-SIM)	>11	none
SEMIVOLATILE ORGANICS BY GC/MS (8270)	<2	>11
ALKYLATED PAHs BY GC/MS (8270P)	< 2	None
1,4-Dioxanes	<2	>11

11.6. Add 60 ml of Dichloromethane to the separatory funnel, or 30mL if a 250mL sample volume is used..

11.7. Seal and shake the separatory funnel vigorously for 1-2 minutes with periodic venting to release excess pressure.

NOTE: Dichloromethane creates excessive pressure very rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and inverted once. Venting of the separatory funnel should be into a hood to avoid needless exposure of the analyst to solvent vapors.

11.8. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the solvent extract in a fleaker. If the emulsion cannot be broken (recovery of < 80% of the Dichloromethane, corrected for the water solubility of Dichloromethane), transfer the

sample, solvent, and emulsion into the extraction chamber of a continuous extractor and proceed as described in Method 3520, Continuous Liquid – Liquid Extraction.

11.9. Repeat the extraction two more times using fresh portions of solvent (Sections 11.5 through 11.6). Combine the three solvent extracts.

11.10. If further pH adjustment and extraction is required, adjust the pH of the aqueous phase to the desired pH. Serially extract three times with 60 ml of Dichloromethane, as outlined in Sections 11.5 through 11.6. Collect and combine the extracts and label the combined extract appropriately.

11.11. Perform the concentration using the Kuderna-Danish (K-D) Technique.

11.11.1. Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10 ml concentrator tube to a 500 ml funnel evaporation flask and rinsing 3 times with DCM. Dry the extract by passing it through a drying column containing about 10 cm of anhydrous sodium sulfate. Collect the dried extract in a K-D concentrator. Rinse the Erlenmeyer fleaker, which contained the solvent extract, with 20-30 ml of Dichloromethane and add it to the column to complete the quantitative transfer. Rinse the funnel 3 times with DCM.

11.11.2. Add one or two clean boiling chips to the flask and attach a three ball Snyder column. Pre-wet the Snyder column by adding about 1 ml of Dichloromethane to the top of the column. Place the K-D apparatus on a hot water bath (15-20-C above the boiling point of the solvent) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10-20 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 10 ml, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

11.11.3. If a solvent exchange is required, momentarily remove the Snyder column, add 15 ml of the exchange solvent, a new boiling chip, and reattach the Snyder column. Concentrate the extract, raising the temperature of the water bath, if necessary, to maintain proper distillation.

11.11.4. Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2 ml of Dichloromethane or exchange solvent. If sulfur crystals are a problem, proceed to Method 3660 for cleanup. The extract may be further concentrated by using the technique outlined in the next section or adjusted to 10.0 ml with the solvent last used. Measure the final extract volume using a 10mL graduated pipet.

11.12. If further concentration is needed, nitrogen blow-down technique is used to adjust the extract to the final volume required.

11.12.1. Place the concentrator tube in a warm water bath (approximately 35ºC) and evaporate the solvent volume to the required level using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon). **Do not let the sample go dry**.

CAUTION: Do not use plasticized tubing between the carbon trap and the sample.

11.12.2. The internal wall of the tube must be rinsed down several times with the appropriate solvent during the operation. During evaporation, the solvent level in the tube must be positioned to prevent water from condensing into the sample (i.e., the solvent level should be below the level of the water bath). Under normal operating conditions, the extract should not be allowed to become dry.

CAUTION: When the volume of solvent is reduced below 1 ml, semivolatile analytes may be lost.

11.13. Take to final volume and transfer the concentrated extract to a labeled autosampler vial (with a Teflon lined screw-cap or crimp top) of storage vial. Measure the final extract volume using a 1mL graduated pipet. The extracts obtained may now be analyzed for the target analytes using the appropriate determinative technique. The extract holding time is 40 days from sample preparation to analysis.

12. DATA REVIEW and REPORTING

12.1. Bench sheets are completed and a batch lot number is assigned. The Manufacturer's lot numbers or ID's for the reagents are added to bench sheets (see Attachments).

12.2. Following primary data review, all data is reviewed by a secondary analyst. Refer to ADM-DREV, *Laboratory Data Review Process* for details. The person responsible for final review of the bench sheet should assess the overall validity and quality of the results.

13. QUALITY CONTROL

13.1. Initial Precision and Recovery Validation

13.1.1. The accuracy and precision of the procedure must be validated before analyses of samples begin, or whenever significant changes to the procedures have been made. To do this, four water samples are spiked with the LCS spike solution, then prepared and analyzed. Refer to the determinative method and SOP for acceptance limits.

13.2. Ongoing QC Samples required are described in the ALS-Kelso Quality Assurance Manual and in the SOP for Sample Batches. Additional QC Samples may be required in project specific quality assurance plans (QAPP). Refer to the SOP for the determinative method for minimum QC requirements.

13.3. Any reagent blanks, laboratory control samples, or matrix spike samples should be subjected to exactly the same analytical procedures as those used on actual samples.

14. CORRECTIVE ACTION

14.1. Refer to CE-QA008, *Non Conformity and Corrective Action* for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2. Handling out-of-control or unacceptable data

14.2.1. On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, runlogs, for example.

14.2.2. Some examples when documentation of a nonconformity is required using a Nonconformity and Corrective Action Report (NCAR):

- Quality control results outside acceptance limits for accuracy and precision
- Method blanks or continuing calibration blanks (CCBs) with target analytes above acceptable levels
- Sample holding time missed due to laboratory error or operations
- Deviations from SOPs or project requirements
- Laboratory analysis errors impacting sample or QC results
- Miscellaneous laboratory errors (spilled sample, incorrect spiking, etc)
- Sample preservation or handling discrepancies due to laboratory or operations error

15. METHOD PERFORMANCE

Available method performance data is given in the reference method. In addition, this procedure was validated through single laboratory studies of accuracy and precision as specified in the determinative procedures.

16. POLLUTION PREVENTION AND WASTE MANAGEMENT

16.1. It is the laboratory's practice to minimize the amount of solvents, acids, and reagents used to perform this method wherever feasibly possible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvents and/or reagents used in this method can be minimized when recycled or disposed of properly.

16.2. The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Environmental Health and Safety Manual.

16.3. This method uses Dichloromethane and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and recycled off site.

16.4. This method uses non-halogenated solvents and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and disposed of in accordance with Federal and State regulations.

16.5. This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 5-9 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the ALS EH&S Manual for details.

16.6. This method uses a base. Waste base is hazardous to the sewer system and to the environment. All waste must be neutralized to a pH of 5-9 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the ALS EH&S Manual for details

17. TRAINING

17.1. Training outline

17.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

17.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

17.1.3. Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

17.2. Training is documented following the *Corporate Training Policy* (CE-QA003) and the *ALS, Kelso Training Procedure (ADM-TRAIN).*

NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst reads and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

18. METHOD MODIFICATIONS

18.1. There are no known modifications in this laboratory standard operating procedure from the reference method.

19. CHANGES SINCE THE LAST REVISION

19.1. Re-formatted SOP to ALS style.
19.2. Changed references from CAS to ALS throughout the document.
19.3. Updated former CAS Corporate SOPs to ALS Corporate SOPs.
19.4. Updated Table 1 to reflect current practice.

20. REFERENCES

20.1. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," March 12, 2007.

20.2. EPASW846, Test Methods For Evaluating Solid Waste, Third Edition, Update III, December 1996, Method 3510C, Revision 3

TABLE 1

APPLICABLE DETERMINATIVE SOPs

ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY: CAPILLARY COLUMN TECHNIQUE	SOC-8081
PCBS AS AROCLORS	SOC-8082Ar
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS – method 8270D	SVM-8270D
PAHS by GC/MS SIM (8270-SIM PAH)	SVM-8270P
CONGENER-SPECIFIC DETERMINATION OF PCBS BY METHOD 8082A	SOC-8082C
1,4-DIOXANES	SVO_SIM
FUEL HYDROCARBONS	PET-SVF
ALIPHATIC HYDROCARBONS	PET-ALIPHAT
TOTAL PETROLEUM HYDROCARBONS	PET-TPH

ATTACHMENTS
Test-Specific Benchsheets
(14 pages)

QuikChem® Method 12-107-06-1-B

DETERMINATION OF AMMONIA BY FLOW INJECTION ANALYSIS

Written by Helen Jonland

Applications Group

Revision Date:

14 November 2001

LACHAT INSTRUMENTS
6645 WEST MILL ROAD
MILWAUKEE, WI 53218-1239 USA

LACHAT INSTRUMENTS

QuikChem® Method 12-107-06-1-B

Ammonia (Phenolate) in 2 M KCl Soil Extracts

1 to 20 mg N/L

– Principle –

Exchangeable ammonium is extracted from soil with 2 M KCl. The KCl extract is filtered and the filtrate is analyzed for ammonia by the phenolate method. This method is based on the Berthelot reaction. Ammonia reacts with alkaline phenol, then with sodium hypochlorite to form indophenol blue. Sodium nitroprusside (nitroferricyanide) is added to enhance sensitivity. The absorbance of the reaction product is measured at 630 nm, and is directly proportional to the original ammonia concentration.

– Interferences –

1. Calcium and magnesium ions may precipitate if present in sufficient concentration. Tartrate or EDTA is added to the sample in-line in order to prevent this problem.
2. Color, turbidity and certain organic species may interfere. Turbidity is removed by manual filtration. Sample color may be corrected for by running the samples through the manifold without color formation. See System Note 5 for specific instructions.

– Special Apparatus –

Please see Parts and Price list for Ordering Information

1. Heating Unit
2. PVC PUMP TUBES MUST BE USED FOR THIS METHOD

Revised by A. Vaclav 14 November 2001.Written and copyrighted © by H. Jonland 16 August 1988 by **Lachat Instruments, 6645 West Mill Road, Milwaukee, WI 53218-1239 USA**. Phone: 414-358-4200 FAX: 414-358-4206.

CONTENTS

QuikChem® Method 12-107-06-1-B

DETERMINATION OF AMMONIA BY FLOW INJECTION ANALYSIS

1. SCOPE AND APPLICATION

1.1. This method covers the determination of ammonia in 2 M KCl soil extracts.

1.2. The applicable range is 1 to 20 mg N/L. The method throughput is 90 injections per hour.

2. INTERFERENCES

2.1. Calcium and magnesium ions may precipitate if present in sufficient concentration. EDTA is added to the sample in-line in order to prevent this problem.

2.2. Color, turbidity and certain organic species may interfere. Turbidity is removed by manual filtration. Sample color may be corrected for by running the samples through the manifold without color formation. See System Note 5 for specific instructions.

3. SAFETY

3.1. The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.

3.2. Each laboratory is responsible for maintaining a current awareness file of the Occupational Health and Safety Act (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data sheets (MSDS) should be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.

3.3. The following chemicals have the potential to be highly toxic or hazardous, for detailed explanation consult the MSDS.

3.3.1. Phenol

3.3.2. Sodium Hydroxide

3.3.3. Sodium Nitroprusside

4. EQUIPMENT AND SUPPLIES

4.1. Balance -- analytical, capable of accurately weighing to the nearest 0.0001 g.

4.2. Glassware -- Class A volumetric flasks and pipettes or plastic containers as required. Samples may be stored in plastic or glass.

4.3. Flow injection analysis equipment designed to deliver and react sample and reagents in the required order and ratios.

4.3.1. Sampler

4.3.2. Multichannel proportioning pump

4.3.3. Reaction unit or manifold

4.3.4. Colorimetric detector

4.3.5. Data system

4.4. Special Apparatus

4.4.1. Heating unit

4.4.2. PVC PUMP TUBES MUST BE USED FOR THIS METHOD

5. REAGENTS AND STANDARDS

5.1. PREPARATION OF REAGENTS

Use deionized water (10 megohm) for all solutions.

Degassing with helium:

To prevent bubble formation, degas all solutions except the standards with helium. Use He at 140kPa (20 lb/in^2) through a helium degassing tube (Lachat Part No. 50100.) Bubble He through the solution for one minute.

Reagent 1. Potassium Chloride Carrier and Standards Diluent, 2 M

By Volume: In a **1 L** volumetric flask, dissolve **150 g potassium chloride** (KCl) in about **900 mL DI water**. Dilute to the mark with **DI water** and invert three times. Degas with helium.

By Weight: To a tared **1 L** container add **150 g potassium chloride** (KCl) and **941 g DI water.** Shake until dissolved. Degas with helium.

Reagent 2. Sodium Phenolate

CAUTION: Wear gloves. Phenol causes severe burns and is rapidly absorbed into the body through the skin.

By Volume: In a **1 L** volumetric flask, dissolve **88 mL 88% liquified phenol** or **83 g crystalline phenol** (C_6H_5OH) in approximately **600 mL DI water**. While stirring, slowly add **32 g sodium hydroxide** (NaOH). Cool, dilute to the mark with **DI water**, and invert three times. Prepare fresh every 3-5 days or when solution turns brown.

By Weight: To a tared **1 L** container, add **888 g DI water**. Add **94.2 g 88% liquified phenol** or **83 g crystalline phenol** (C_6H_5OH). While stirring, slowly add **32 g sodium hydroxide** (NaOH). Cool and invert three times to mix thoroughly. Prepare fresh every 3-5 days or when solution turns brown.

Reagent 3. Sodium Hypochlorite

Dilute **250 mL or 250 g 5.25% sodium hypochlorite** (NaOCl) to **500 mL or 500 g DI water**. Invert to mix. Prepare fresh daily.

Reagent 4. Buffer

In a **1 L** volumetric flask, dissolve **50.0 g disodium ethylenediamine tetraacetate** (Na_2EDTA) and **5.5 g sodium hydroxide** (NaOH) in about **900 mL DI water**. Dilute to the mark and invert three times. Degas with helium. Prepare fresh monthly.

Reagent 5. Sodium Nitroprusside

Dissolve **3.50 g sodium nitroprusside** in **1000 g** or **1 L DI water**. Prepare fresh every 1-2 weeks.

5.2. PREPARATION OF STANDARDS

To prepare the stock and working standards, the following containers will be requires:

By Volume: Two 1 L and six 250 mL volumetric flasks.

By Weight: Two 1 L, and six 250 mL containers.

Standard 1. Stock Standard 1000 mg N/L as NH_3

In a **1 L** volumetric flask dissolve **3.819 g ammonium chloride** (NH_4Cl) that has been dried for two hours at 110°C in about **800 mL KCl diluent** (Reagent 1). Dilute to the mark with **KCl diluent** (Reagent 1) and invert three times.

Standard 2. Working Stock Standard Solution 100.0 mg N/L as NH_3

By Volume: In a **1 L** volumetric flask, dilute **100 mL Stock Standard** (Standard 1) to the mark with **KCl diluent** (Reagent 1). Invert three times.

By Weight: To a tared **1 L** container add about **100 g Stock Standard** (Standard 1). Divide the actual weight of the solution by 0.1 and make up to this resulting total weight with **KCl diluent** (Reagent 1), using a wash bottle or disposable pipet to add the last 10 g or so.

Working Standards (Prepare Daily)	**A**	**B**	**C**	**D**	**E**
Concentration mg N/L	20	10	5	1	0

By Volume

Volume (mL) of **stock standard 2** diluted to 250 mL with Reagent 1	50.0	25.0	12.5	2.5	0.0

By Weight

Weight (g) of **stock standard 2** diluted to final weight (~250 g) divided by **factor** below with Reagent 1	50.0	25.0	12.5	2.5	0.0
Division Factor Divide exact weight of the standard by this **factor** to give the final weight	0.2	0.1	0.05	0.01	---

6. SAMPLE COLLECTION, PRESERVATION AND STORAGE

6.1. Ammonia is volatile and will leave the sample slowly, even through polyethylene bottles. The samples should be run within 24 hours. If this cannot be done, the samples should be adjusted to a pH of 3-5 with dilute phosphoric or sulfuric acid.

7. PROCEDURE

7.1. CALIBRATION PROCEDURE

7.1.1. Inspect all modules for proper connections.

7.1.2. Turn on power and all modules.

7.1.3. Place reagent feedlines into proper containers. Raise tension levers on pump tube cassettes.

7.1.4. Establish a stable baseline. Set zero on colorimeter. If necessary, manually inject a high standard to check gain.

7.1.5. Program data system to initial parameters or to those empirically determined.

7.1.6. Place calibration standards and blank in sample tray in descending order of concentration followed by unknowns and check standards.

7.1.7. At end of run, place all feedlines in deionized water. Flush system and pump dry.

7.1.8. Turn off pump, all modules, and release pump tube cassettes.

7.2. SYSTEM NOTES

7.2.1. If baseline drifts, peaks are too wide, or other problems with precision arise, clean the manifold by the following procedure:

7.2.2. Place all reagent lines in deionized water and pump to clear reagents (2-5 minutes).

7.2.3. Place reagent lines and carrier in 1 M hydrochloric acid (1 volume conc HCl added to 11volumes of deionized water) and pump for several minutes.

7.2.4. Place all reagent lines in deionized water and pump until the HCl is thoroughly washed out.

7.2.5. Resume pumping reagents.

7.2.6. If samples are colored or are suspected to show a background absorbance, this interference should be subtracted. This can be done by the following procedure:

7.2.6.1. Calibrate the system in the normal manner. (If using a 4000 series instrument, the baseline subtraction must be used.)

7.2.6.2. Disable the check standard or DQM features and analyze the samples.

7.2.6.3. Place reagent and carrier lines in DI water and allow the baseline to stabilize.

7.2.6.4. Inject samples again without recalibrating

7.2.6.5. Subtract the "background" concentration from the original concentration to give the corrected concentration.

Corrected Concentration = Original Concentration - Background Concentration

7.2.7. Use consumer bleaches with caution. Proprietary additives may contribute to staining of tubing and data quality.

7.2.8. Add reagents in the order that they appear on the manifold to reduce staining.

8. DATA ANALYSIS AND CALCULATIONS

8.1. Calibration is done by injecting standards. The data system will then prepare a calibration curve by plotting response versus standard concentration. Sample concentration is calculated from the regression equation.

8.2. Report only those values that fall between the lowest and highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.

8.3. Report results in mg N/L.

9. METHOD PERFORMANCE

9.1. The method support data are presented in section 11. This data was generated according to a Lachat Work Instruction during development of the method.

9.2. Although Lachat Instrument publishes method performance data, including MDL, precision, accuracy and carryover studies, we cannot guarantee that each laboratory will be capable of meeting such performance. Individual laboratory and instrument conditions, as well as laboratory technique, play a major role in determining method performance. The support data serves as a guide of the potential method performance. Some labs may not be able to reach this level of performance for various reasons, while other labs may exceed it.

10. REFERENCES

10.1. U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79--020, Revised March 1983, Method 350.1

11. TABLE, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

11.1. DATA SYSTEM PARAMETERS FOR THE QUIKCHEM IV

Pump Speed: 35
Cycle Period: 40 s
Sample Loop Length: microloop
Load Period: 10 s
Inject Period: 30 s
Inject to start of peak period: 25 s
Inject to end of peak period: 60 s

Gain:

Gain: 260 x 1

11.2. SUPPORT DATA FOR THE QUIKCHEM IV

QuikCalc II Sample Report
8/16/1998 9: 4

Tray ID: Reference: [illegible]
Method: ammonia Operator:

Tray Comments:

Analyte of ammonia OverRange
Base Subtraction Yes Yes
Technique Area Area

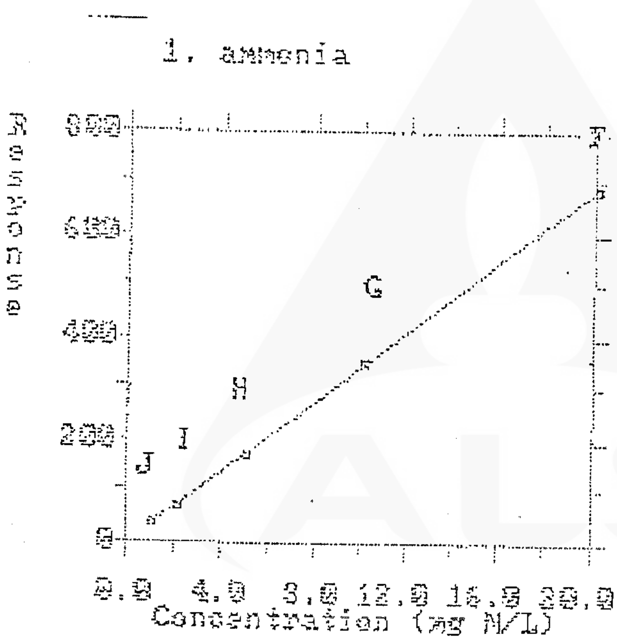
---------- Calibration ----------
Seg 1 C.C. [illegible]

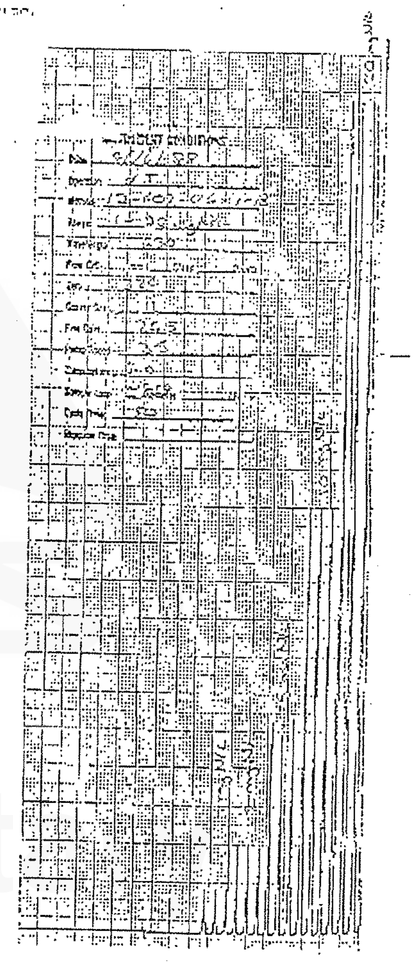
Calibration Statistics For Channel 1: ammonia

Seg: 1 Slope: 34.26416, Intercept: 1.006, CC: 0.9999 (Rspns Line: [illegible])

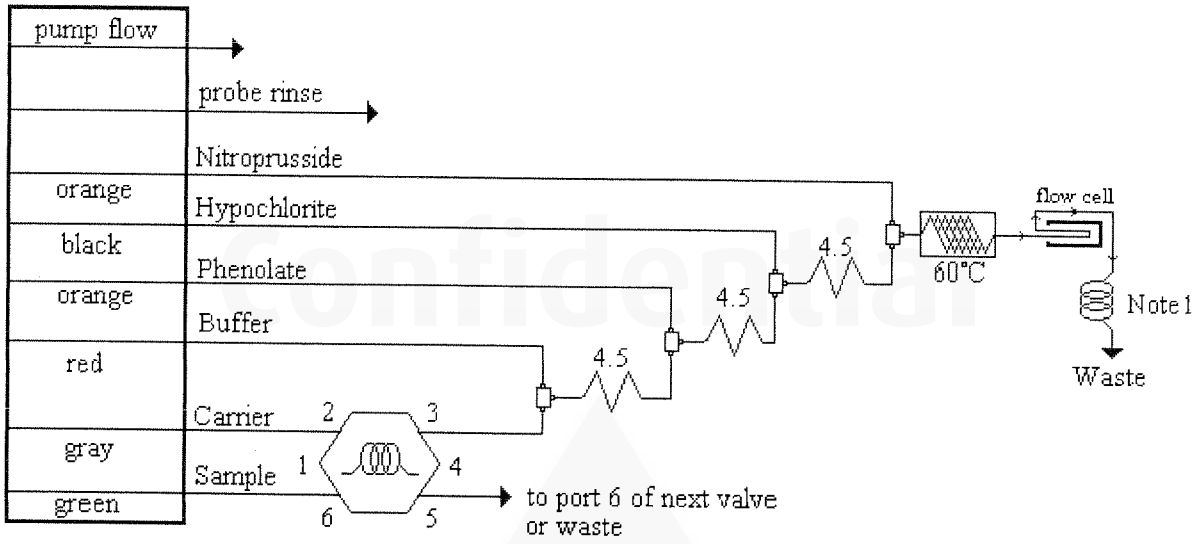
Std	Ave Rspns	Conc	Derived	%Res	Rspns 1	Rspns 2	Rspns 3
6 F	606	20.000	19.947	0.3	606	606	601
7 G	348	10.000	10.1[illegible]	-1.2	340	340	345
8 H	171	5.000	4.974	0.5	175	170	169
9 I	69	2.000	1.976	1.1	68	70	69
10 J	35	1.000	0.978	2.2	34	35	35

Press [D] if you want to delete [undelete] a data point, any key otherwise.

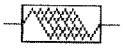




11.3. AMMONIA MANIFOLD DIAGRAM



Carrier: **Reagent 1**
Manifold Tubing: **0.8 mm (0.032 in) i.d.** This is **5.2 µL/cm**.
AE Sample Loop: Microloop
QC8000 Sample Loop: Microloop
Interference Filter: 630 nm

Apparatus: An injection valve, a 10 mm path length flow cell, and a colorimetric detector module is required. The  shows 650 cm of tubing wrapped around the heater block at the specified temperature.

4.5: **70** cm of tubing on a 4.5 cm coil support

Note 1: PVC PUMP TUBES MUST BE USED FOR THIS METHOD.

QuikChem® Method 10-107-06-1-K

DETERMINATION OF AMMONIA (PHENOLATE) BY FLOW INJECTION ANALYSIS

(LOW FLOW METHOD)

Written by Dave Diamond

Applications Group

Revision Date:

15 March 2001

LACHAT INSTRUMENTS
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LACHAT INSTRUMENTS
A Hach Company Brand

QuikChem® Method 10-107-06-1-K

Ammonia (Phenolate) in Potable and Surface Waters

0.2 to 20 mg N/L

– Principle –

This method is based on the Berthelot reaction. Ammonia reacts with alkaline phenol, then with sodium hypochlorite to form indophenol blue. Sodium nitroprusside (nitroferricyanide) is added to enhance sensitivity. The absorbance of the reaction product is measured at 630 nm, and is directly proportional to the original ammonia concentration in the sample.

If distillation is required the sample is buffered at a pH of 9.5 with a borate buffer to decrease hydrolysis of cyanates and organic nitrogen compounds, and is distilled into a solution of boric acid.

– Interferences –

1. Calcium and magnesium ions may precipitate if present in sufficient concentration. EDTA is added to the sample in-line in order to prevent this problem.
2. Color, turbidity and certain organic species may interfere. Turbidity is removed by manual filtration. Sample color may be corrected for by running the samples through the manifold without color formation.
3. Cyanate, which may be encountered in certain industrial effluents, will hydrolyze to some extent even at the pH of 9.5 at which distillation is carried out.
4. Residual chlorine must be removed by pretreatment of the sample with sodium thiosulfate or other reagents before distillation.
5. Method interference may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that bias analyte response.
6. Eliminate any marked variation in acidity or alkalinity among samples because intensity of measured color is pH dependent. Likewise, ensure that pH of standard ammonia solutions approximates that of samples.

– Special Apparatus –

Please see Parts and Price list for Ordering Information

1. Heating Unit Lachat Part No. A85X00 (X=1 for 110V, X=2 for 220V)
2. PVC PUMP TUBES MUST BE USED FOR THIS METHOD.

Revised by D. Lynch on 15 March 2001.

ATTACHMENT 2 - Bran & Luebbe Method
(6 pages)

BRAN+LUEBBE
1025 Busch Parkway Buffalo Grove, IL 60089
(847) 520-0700
FAX: (847) 520-0855

AutoAnalyzer Applications

Method No. US- 696D-82X

Ammonia in Water and Wastewater

Range: 0.010-3 mg/l N

Description

The automated procedure for the determination of ammonia utilizes the Berthelot Reaction, in whi the formation of a blue colored compound believed to be closely related to indophenol occurs whe the solution of an ammonium salt is added to sodium phenoxide, followed by the addition of sodiu hypochlorite. A solution of EDTA is added to the sample stream to eliminate the precipitation of th hydroxides of calcium and magnesium.[1,2,3,4,5,6] Sodium nitroprusside is added to intensify the blue color.

Hardware:	Pump tubes:
37°C heating bath (MT12)	7+1air+1 sampler wash

Performance data using aqueous standards

Test range 0010-3 mg/L N

	AA3	AAII
Sampling rate	40/hr	40/hr
Sample: wash ratio	4:1	4/1
Sensitivity at 3mg/L	0.3576	0.22
Reagent Absorbance	0.062	0.4
Coefficient of Variation 30 replicates at 1.5mg/L	0.429	1.2
Pooled standard deviation 25 at 5 levels	0.013mg/L	NA
Correlation coefficient (Linear)	0.9993	NA
Detection limit (determined according to EPA procedure pt.135, app. B)	0.016	NA
Carryover	0.2%	NA
Lag time	9 min	NA
Base	-16465	NA
Gain	24	NA
Lamp Intensity	58	NA

Note: the above performance specifications were developed with the exclusive use of genuine Bran+Luebbe parts.
*Trademark of Atlas Chemical

1. Van Slyke, O.D. and Hillern, A.J., BioChem., 102, p.499, 1933
2. Kallman, S., Presentation at Div. 1 Meeting of ASTM Committee E-3, April,1967, San Diego, CA
3. Bolleter, W.T., Bushman, C.J. and Tidwell, P.N., Anal. Chem., 33, P.592, 1961.
4. Tellow, J.A. and Wilson, A.L., Analyst, 89, p.453, 1954.
5. Tarugi, A. and Lenci, F., Boll Chim. Farm., 50, p.9070. 1912.
6. VWPCA Methods of Chem. Anal. of Water & Wastewater, November 1969, p.137.

Rev. X 1/15/99 Bran+Luebbe USA

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FAX: (847) 520-0855

Reagents

Unless otherwise specified, all chemicals should be of ACS grade or equivalent.

List of Raw Materials

Ammonium Sulfate $(NH_3)_2SO_4$) Primary Standard Grade
Brij-35, 30% Solution*
Disodium, ethylenediamine-tetracetate
Phenol, loose crystals (C_6H_5OH)
Sodium Hydroxide (NaOH)
Sodium Hypochlorite, 5% Solution (NaOCl) Commercial Grade; e.g. Clorox
Sodium Nitroprusside ($Na_2Fe(CN)_5NO.2H_20$)
Sulfuric Acid, conc. (H_2SO_4)

Reagent Make-Up

All water used in reagent and standard preparation must be ammonia free.

Alkaline Phenol

Phenol, loose crystals	83 g
Sodium Hydroxide, solid	48 g
Distilled Water, qs	1000 ml

Preparation:
Add 83 g of loose crystal phenol to about 800 ml of distilled water. While cooling under tap water or in an ice bath, slowly with swirling add 48 g of sodium hydroxide. Cool to room temperature, dilute to 1000 ml with distilled water and mix thoroughly. Store in an amber glass container. This material is corrosive. Stability: Two weeks.

Sodium Hypochlorite Solution

Sodium Hypochlorite Solution, 5%	210 ml
Distilled Water, qs	500 ml

Preparation:
Dilute 210 ml of sodium hypochlorite solution 5% to 500 ml with distilled water and mix thoroughly. Prepare fresh weekly.

Sodium Nitroprusside Solution

Sodium Nitroprusside	3.5 g
Distilled Water, qs	1000 ml

Preparation:
Dissolve 3.5 g of sodium nitroprusside in about 600 ml of distilled water. Dilute to one liter with distilled water and mix thoroughly. Store in an amber container. Stability: One month.

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Disodium EDTA

Disodium EDTA	33.6 g
Sodium Hydroxide, 50% w/w	0.8 g
Distilled Water, qs	1000 ml
Brij-35	2 ml

Preparation:
Dissolve approximately 0.8 g of 50% w/w sodium hydroxide and 33.6 g of disodiurn EDTA in about 800 ml of distilled water. Dilute to one liter. Add 2 ml of Brij-35 and mix well.

Standard Preparation

Stock standard A, 100 mg/L N

Ammonium Sulfate	0.4717 g
Chloroform	1 ml
Distilled Water, qs	1000 ml

Preparation:
In a one liter volumetric flask containing about 800 ml of distilled water dissolve 1 ml of chloroform. Add 0.4717 g of ammonium sulfate and swirl to dissolve. Dilute to one liter with distilled water and mix thoroughly.

Stock Standard B. 20 mg/L N

Stock Standard A	20.0 ml
Distilled Water, qs	100 ml

Preparation:
Dilute 20.0 ml of stock standard A to 100 ml with distilled water and mix thoroughly. Prepare fresh daily.

Working Standard Solutions

ml Stock B	mg/l N
2.0	0.4
4.0	0.8
6.0	1.2
8.0	1.6
10.0	2.0
15.0	3.0

Preparation:
Transfer aliquots of Stock Standard 6 as noted above, to individual 100 ml volumetric flasks. Dilute to volume with distilled water and mix thoroughly. Prepare fresh daily.

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Sample Preparation

1. Preserve by addition of 2 ml/L H_2SO_4 and refrigerate at 4° C.
2. Sample turbidity should be removed by filtration prior to analysis. It may interfere with this chemistry.
3. Sample cups can be rinsed with 1:1 hydrochloric acid, followed by distilled water and finally by an aliquot of the sample itself.

System Cleansing Procedure

Pump 5N H_2SO_4 as the cleaning solution.

System Performance

Maximum reagent/water baseline	0.40 AU
Reagent Equilibration time	5 min
Sensitivity of high standard at a STD CAL of 1.0	0.22 AU
Coefficient of Variation	1.2%

Flow Diagram

1. If analysis is to be performed in a contaminated environment, scrub the air with 5N H2SO4
2. Check that the appropriate harness and optical filters are secured in their proper positions.

CONSUMMABLES

The following annual consumption rates are based on system operation 8 hours/day, 250 days/year.

Description	Part Number	Est. Annual Usage
ORN/BLU, 0.05mL/min	116-0549-03	5pkg./12
ORN/YEL, 0.16mL/min	116-0549-05	3pkg./12
BLK/BLK, 0.32mL/min	116-0549-07	5pkg./12
ORN/ORN, 0.42mL/min	116-0549-08	3pkg./12
WHT/WHT, 0.60mL/min	116-0549-09	3pkg./12
GRY/GRY, 1.00mL/min	116-0549-11	3pkg./12
GRN/GRN, 2.00mL/min	116-0549-14	3pkg./12
Tubing air bar silicone	116-0543-01	8 ft.
Tubing Kel-F, 0.050" ID	171-0745-01	3 ft.
Tubing Polyeth., 0.015" ID	562-2002-01	3 ft.
Tubing Polyeth, 0.030" ID	562-2003-01	3 ft.

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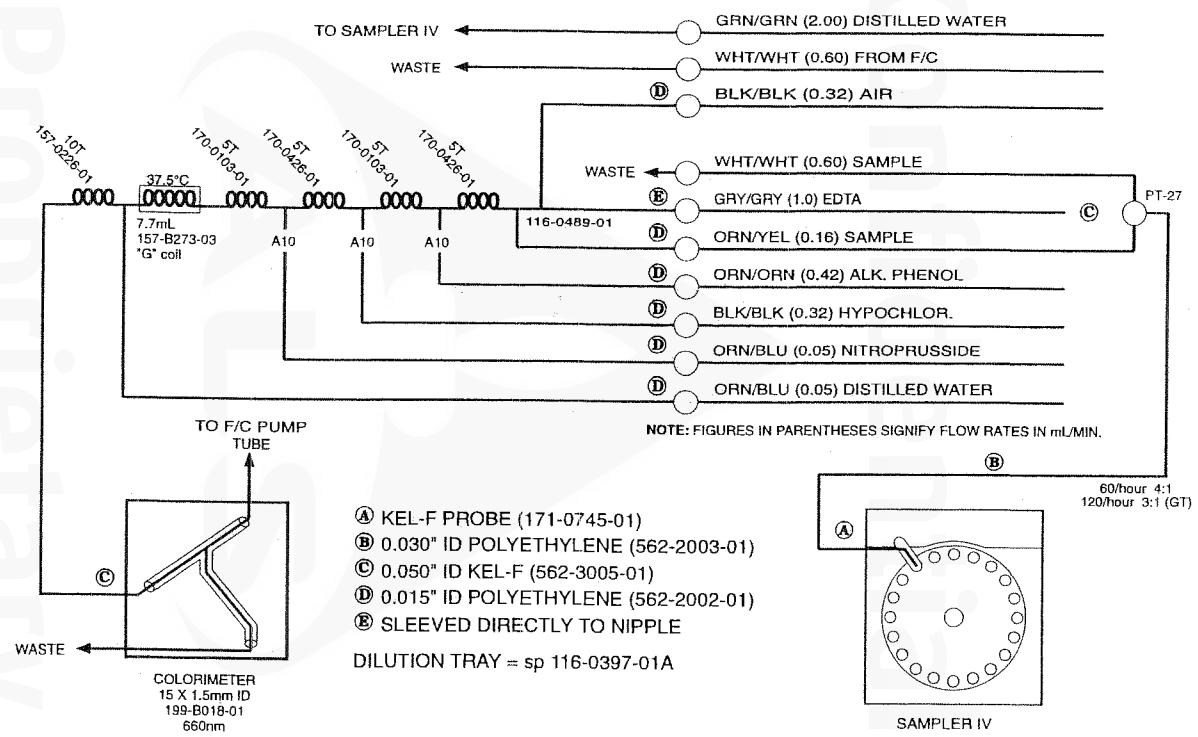
SPARES	Part Number	Recommended
AA3 Flowcell, 1.5 x 10mm	169+B040-01	1pc.
AAII Flowcell, 1.5 x15mm	199-B018-01	1pc.
AA3 Filter, 660nm	165-B044-66	1pc.
AAII Filter, 660nm	170-B070-26	1pc.
AAII Heating Bath, 7.7mL, 37°C	157-B273-03	1pc.
AA3 Heating Bath Coil, 5.37mL	169+B440-03	1pc.
5-turn mixing coil, right	170-0103-01	1pc.
5-turn mixing coil, left	170-0426-01	1pc.
10-turn mixing coil	157-0226-01	1pc.
Glass fitting, A10	116-B034-01	1pc.
Injection Fitting	116-0489-01	1pc.
Straight tube	116-G004-01	1pc.
Glass U	172-9038-01	1pc.
Glass Z	170-0204-01	1pc.
Glass tube	170-G022-01	1pc.
Glass tube with injection	170-G071-01	1pc.

Method 696D-82W

BRAN+LUEBBE

Ammonia in water and wastewater
Manifold 116-D857-01
Harness number: 116-B346-01
Range: 0.02-3mg/L NH_3-N



4/8/94

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ALS Environmental

DOCUMENT TITLE:	***CONTINUOUS LIQUID - LIQUID EXTRACTION***
REFERENCED METHOD:	***EPA METHOD 3520C***
SOP ID:	***EXT-3520***
REV. NUMBER:	***14***
EFFECTIVE DATE:	***07/12/2013***

CONTINUOUS LIQUID - LIQUID EXTRACTION

EPA METHOD 3520C

ALS-KELSO

SOPID:	EXT-3520	Rev. Number:	14	Effective Date:	07/12/2013

Approved By: A Bailey Date: 6/28/13
Department Supervisor - Heather Bailey

Approved By: Suzanne L. May Date: 6/28/13
QA Manager - Suzanne LeMay

Approved By: Date: 6/28/13
Laboratory Director - Jeff Grindstaff

Issue Date:	_____	Doc Control ID#:	_____	Issued To:	_____

Standard Operating Procedure

for

CONTINUOUS LIQUID - LIQUID EXTRACTION

1. SCOPE AND APPLICATION

1.1. This procedure uses techniques described in EPA Method 3520C for extracting nonvolatile and semi-volatile organic compounds from aqueous samples.

1.2. This method is applicable to the isolation and concentration of water insoluble and slightly water soluble organics in preparation for a variety of determinative methods which use chromatographic procedures. Refer to the determinative procedure to determine if this procedure is suitable for the analysis being performed.

1.3. Continuous Liquid - Liquid Extraction may be used when a solute is to be transferred from one solvent into another. This procedure involves multiple extractions using immiscible solvents. The solvent is reused as the condensate from a total reflux.

2. METHOD SUMMARY

2.1. A measured volume of sample is placed in a continuous liquid-liquid extractor, pH adjusted (if necessary), and extracted with an organic solvent for a determined period of time. In this procedure, the extracting solvent has a higher density than that of the aqueous solution being extracted, allowing the reflux of the heavier extracting solvent to be diverted through the sample, extracting the solute, and then siphoned back into the boiling flask.

2.2. The extract is dried, concentrated, and (if necessary) exchanged to an appropriate solvent for the determinative procedure. The extract may undergo additional clean-up steps defined in other procedures.

3. DEFINITIONS

3.1. **Batch** - A batch of samples is a group of environmental samples that are prepared and/or analyzed together as a unit with the same process and personnel using the same lot(s) of reagents. It is the basic unit for analytical quality control.

3.1.1. Preparation Batch - A preparation batch is composed of one to twenty field samples, all of the same matrix, and with a maximum time between the start of processing of the first and last samples in the batch to be 24 hours.

3.1.2. Analysis Batch - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration (initial or continuing verification) followed by sample extracts interspersed with calibration standards (CCBs, CCVs, etc.) The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria indicate an out-of-control situation.

3.2. **Sample**

3.2.1. Field Sample - An environmental sample collected and delivered to the laboratory for analysis; a.k.a., client's sample.

3.2.2. Laboratory Sample - A representative portion, aliquot, or subsample of a field sample upon which laboratory analyses are made and results generated.

3.3. **Quality System Matrix** - The *matrix* of an environmental sample is distinguished by its physical and/or chemical state and by the program for which the results are intended. The following sections describe the matrix distinctions. These matrices shall be used for purpose of batch and quality control requirements.

3.3.1. Aqueous - Any groundwater sample, surface water sample, effluent sample, and TCLP or other extract. Specifically excluded are samples of the drinking water matrix and the saline/estuarine water matrix.

3.3.2. Drinking water - Any aqueous sample that has been designated a potable or potential potable water source.

3.3.3. Saline/Estuarine water - Any aqueous sample from an ocean or estuary or other salt-water source.

3.3.4. Non-aqueous Liquid - Any organic liquid with <15% settleable solids.

3.3.5. Animal tissue - Any tissue sample of an animal, invertebrate, marine organism, or other origin; such as fish tissue/organs, shellfish, worms, or animal material.

3.3.6. Solids - Any solid sample such as soil, sediment, sludge, and other materials with >15% settleable solids.

3.3.7. Chemical waste - Any sample of a product or by-product of an industrial process that results in a matrix not described in one of the matrices in Sections 3.3.1 through 3.3.6. These can be such matrices as non-aqueous liquids, solvents, oil, etc.

3.3.8. Miscellaneous matrices – Samples of any composition not listed in 3.3.1 – 3.3.7. These can be such matrices as plant material, paper/paperboard, wood, auto fluff, mechanical parts, filters, wipes, etc. Such samples shall be batched/grouped according to their specific matrix.

3.4. Matrix Spike/Duplicate Matrix Spike (MS/DMS) Analysis - In the matrix spike analysis, predetermined quantities of target analytes are added to a sample matrix prior to sample preparation and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the method used for the analysis. Duplicate samples are spiked, and analyzed as a MS/DMS pair. Percent recoveries are calculated for each of the analytes detected. The relative percent difference (RPD) between the duplicate spikes (or samples) is calculated and used to assess analytical precision. The concentration of the spike should be at the mid point of the calibration range or at levels specified by a project analysis plan.

3.5. Laboratory Duplicates (DUP) – Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. The

relative percent difference (RPD) between the sample and its duplicate is calculated and used to assess analytical precision.

3.6. Surrogate - Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to extraction and analysis. Percent recoveries are calculated for each surrogate.

3.7. Method Blank (MB) - The method blank is an artificial sample composed of analyte-free water or solid matrix and is designed to monitor the introduction of artifacts into the analytical process. The method blank is carried through the entire analytical procedure.

3.8. Laboratory Control Samples (LCS) – The LCS is an aliquot of analyte free water or analyte free solid to which known amounts target analytes are added. The LCS is prepared and analyzed in exactly the same manner as the samples. The percent recovery is compared to established limits and assists in determining whether the batch is in control.

3.9. Independent Verification Standard (ICV) - A mid-level standard injected into the instrument after the calibration curve and prepared from a different source than the initial calibration standards. This is used to verify the validity of the initial calibration standards

3.10. Continuing Calibration Verification Standard (CCV) - A mid-level standard analyzed at specified intervals. Used to verify that the initial calibration curve is still valid for quantitative purposes.

3.11. Instrument Blank (CCB) - The instrument blank (also called continuing calibration blank) is a volume of clean solvent analyzed on each column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrumental analysis itself, particularly with regard to the carry-over of analytes from standards or highly contaminated samples into subsequent sample analyses.

3.12. Duplicates and Duplicate Matrix Spikes are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. Depending on the method of analysis, either a duplicate analysis (and/or a matrix spiked sample) or a matrix spiked sample and duplicate matrix spiked sample (MS/DMS) are analyzed.

3.13. Standard Reference Material (SRM) – A material with specific certification criteria and is issued with a certificate or certificate of analysis that reports the results of its characterizations and provides information regarding the appropriate use(s) of the material. An SRM is prepared and used for three main purposes: (1) to help develop accurate methods of analysis; (2) to calibrate measurement systems used to facilitate exchange of goods, institute quality control, determine performance characteristics, or measure a property at the state-of-the-art limit; and (3) to ensure the long-term adequacy and integrity of measurement quality assurance programs.

3.14. Reflux Condenser: An auxiliary vessel for a distillation column that condenses vapors and returns liquid to the column.

3.15. Total Reflux: A distillation column is said to be operating under total reflux when all of the vapor leaving the column is condensed and returned. No products are withdrawn from the system. The reflux ratio is infinity.

3.16. Reflux Ratio: The quantity of liquid reflux per unit quantity of product removed from the process unit, such as a distillation tower or extraction column.

4. SAFETY

4.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personal protective equipment, such as safety glasses, lab coat and the correct gloves.

4.2. Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in MSDSs where available. Refer to the ALS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

4.3. This method uses Dichloromethane, a known human carcinogen. Viton brand gloves should be used while rinsing, pouring or transferring the solvent

5. INTERFERENCES

5.1. Rinse the bottom of the reflux condenser with CH_2Cl_2 and/or Acetone to eliminate any possible contaminants from entering the extractor.

5.2. Rinse all glass surfaces involved in the extraction process thoroughly with CH_2Cl_2 (reagent grade or re-distilled). Three rinses is usually adequate.

6. RESPONSIBILITIES

It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

7. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

7.1. Refer to the applicable section in the determinative SOP for sample collection, preservation, and holding times.

7.2. The extract holding time is 40 days from sample preparation to analysis.

8. APPARATUS AND MATERIALS

8.1. Continuous liquid/liquid extraction body

8.2. 500 ml round bottom flask, with green Keck clip

8.3. Graduated cylinder, 1 liter, Class A, TC.

8.4. Stir rod and pH paper

8.5. Allihn condensor

8.6. Boiling chips - Pre-cleaned via Soxhlet extraction, approximately 10/40 mesh (silicon carbide or equivalent).

8.7. Graduated pipets, 1, 2 and 5mL. Pipets are pre-tested by lot for bias and precision.

9. REAGENTS

9.1. Dichloromethane - Pesticide grade or redistilled if batch testing determines that the solvent is suitable for use.

9.2. Hexane, C_6H_{14}. Pesticide quality or equivalent.

9.3. 2-Propanol, $(CH_3)_2CHOH$. Pesticide quality or equivalent.

9.4. Acetonitrile, CH_3CN. Pesticide quality or equivalent.

9.5. Iso-octane $(CH_3)_3CCH_2CH(CH_3)_2$. Pesticide quality or equivalent.

9.6. Acetone CH_3COCH_3 - Pesticide grade or equivalent.

9.7. Sodium hydroxide solution (l0N), NaOH. Dissolve 40 g NaOH in organic-free reagent water and dilute to 100 ml.

9.8. Sodium sulfate (granular, anhydrous), Na_2SO_4. Purify by heating at 400°C for 4 hours in a shallow tray.

9.9. Sulfuric acid solution (1:1 v/v), H_2SO_4.

9.10. Organic free DI Water prepared for each batch by adding 50 mL DCM to 4 liters of water and shaking for 1 minute.

10. PREVENTIVE MAINTENANCE

Routine cleaning of the extraction glassware is necessary. Refer to the SOP for *Organic Extractions Glassware Cleaning.*

11. PROCEDURE

11.1. Test-specific benchsheets are attached. These benchsheets list such information as solvents, solvent exchanges, weights, and volumes specified for the determinative method. Use the correct benchsheet and record all extraction and sample information. To assist the analyst, a brief description of the procedure is given on the backside of the benchsheet.

11.2. Rinse the unit thoroughly, in the hood, with CH_2Cl_2 (wearing solvent resistant gloves only) three times, discarding the CH_2Cl_2 rinse into a DCM rinse container. It is very important to use sufficient volumes of CH_2Cl_2 when rinsing. Place the rinsed unit on the grid clamping device and tighten retaining chain when the desired height is attained. Attach a support ring under the unit to support additional weight when the sample volume is added. Rinse a 500 ml flask 3 times with CH_2Cl_2, and add 3 or 4 boiling chips. Attach the flask to the

bottom of the drying apparatus with a snap ring, making a final height adjustment so that the unit contacts the heating mantel correctly. Add ~500 ml CH_2Cl_2 to the body of the extractor in preparation for the addition of the sample. Rinse a 1 liter graduated cylinder with CH_2Cl_2 which will be used for the addition of the water or sample to the body of the extractor.

11.3. Procedure For Sample Extraction

11.4. Transfer 1 L (nominal) to the pre-rinsed graduated cylinder and carefully pour into the body of the extractor. When the sample to be extracted is contained in a l-liter (or smaller) bottle, mark the sample level on the bottle for later volume measurement. The contents of the bottle are poured directly into the extractor. The sample bottle is then rinsed with a portion of the extraction solvent and added to the extractor. Measure and record the sample volume by filling the bottle to the mark then measuring with a Class A TC graduated cylinder. Record the sample volume on the benchsheet.

NOTE: When adding organic free DI water or sample to the mouth of the extractor body, be careful not to pour the volume down the side arm siphon. When measuring out the correct volume to add to the units, be sure to measure the volume of organic free DI water for the LCS and the MB prior to the sample. In this manner less glassware will be generated for the glasswasher.

11.4.1. Samples with settled solid material or sediment.

11.4.1.1. If the sample contains a small amount of material, shake the sample to mix the material into the sample and analyze the entire sample.

11.4.1.2. If the amount of material is enough to interfere with sample extraction, the Project Chemist should be notified to determine the procedure to be used. The default procedure is to *completely* decant the liquid portion of the sample (without shaking the sample) into a graduated cylinder, measure the volume, and transfer to the extraction apparatus. Rinse the graduated cylinder with the extraction solvent. The remaining solid material may be analyzed separately (using an appropriate extraction procedure) depending on the Project Chemist's instructions. It should be documented on the benchsheet when decanting is performed.

11.4.2. Refer to the determinative SOP (see Table 1 for a list of applicable SOPs) for the preparation, concentration, storage, and expiration for the surrogate, LCS, and MS spiking solutions. These SOPs also list the resulting final spike concentrations. Add the surrogate spiking solution into the sample. For the LCS and sample(s) in each analytical batch selected for matrix spiking, add the prescribed volume matrix spiking standard. Each standard should be brought to room temperature before using. Addition of surrogate and spike is routinely witnessed by a second analyst to assure completeness.

11.4.3. Check the pH of the sample with wide-range pH paper and adjust the pH, if necessary, to the pH indicated below, using 1:1 (v/v) sulfuric acid or 10 N sodium hydroxide. Attach the reflux condenser to the unit, turn on and raise temperature setting until unit is cycling properly (2 to 3 drips of solvent per second).

DETERMINATIVE PROCEDURE	INITIAL EXTRACTION PH	SECONDARY EXTRACTION PH
ORGANOCHLORINE PESTICIDES (8081)	5-9	none
PCBS AS AROCLORS (8082)	5-9	none
SOCs BY GC/MS (8270)	<2	>11
SIM PAH	5-9	none

11.4.4. Adjust the volume in the extractor body to reach the cycling volume point, with extracting solvent (There should be a minimum of 300 ml of CH_2Cl_2 in the boiling flask. If not, add additional DI water).

11.4.5. Make sure that each continuous liquid - liquid extractor is cycling properly. Check the chiller for proper temperature and operation. For proper operation, the water flow must be continuous from the top outlet of the condensers and the condenser must be cold to the touch before extraction begins.

11.4.6. Cycle the unit for an 18-24 hour time period. Method 8270 and 625 analyses are light-sensitive, cover the extraction flasks with aluminum foil.

11.4.7. After the cycling period is completed, shut off all of the temperature controls and allow the unit to cool completely. Remove the reflux condenser from the extractor body and clamp securely to the grid apparatus. Remove the extractor body from the grid apparatus by removing the retaining chain and upper snap ring from the drying flask, being careful not to spill the extract in the flask.

11.4.8. Method 8270 and 625 analyses only: Cap and store the initial extract. Repeat sections 11.3.4 through 11.3.7 using a new aliquot of extraction solvent and making the secondary pH adjustment (pH >11) as specified in the method (see Table 1 of EPA Method 3520C for 8270).

11.4.9. Decant off the remaining extraction solvent from the body of the extractor into the correct waste disposal container, and dump the remaining sample into a collection bucket/sink for neutralization.

11.4.10. Rinse the body of the extractor thoroughly in hot tap water before returning to the glasswasher.

11.4.11. Pour the remaining extract from the 500 ml flask into a prerinsed K-D apparatus through a funnel containing glass wool and anhydrous sodium sulfate. Add one boiling chip to the K-D apparatus and attach a CH_2Cl_2 rinsed Snyder column to the top of the K-D apparatus. Place the K-D apparatus on the S-evap. Set to the proper temperature.

Method 8270 and 625 Analyses Only: Pour (combine) the initial extraction (acid side) and secondary extract (base side) through a funnel containing glass wool and anhydrous sodium sulfate into a K-D apparatus.

11.5. Concentrate the extract to approximately 5-10 mls. Remove the K-D apparatus from the S-evap and allow to cool. Disassemble the K-D apparatus. Depending on the determinative

method, the extract may be further concentrated, solvent exchanged, or adjusted to the necessary volume with the solvent last used. For solvent exchange, continue to section 11.5, if further concentration is required; nitrogen blowdown technique (11.6) is used to adjust the extract to the final volume. Return all glassware to the glasswasher as soon as possible.

11.6. Solvent exchange is required for certain analyses in order to obtain a final extract in a solvent compatible with the analytical system used. Exchange solvents are used as listed below.

Determinative Method	Exchange Solvent Required for Analysis	Exchange Solvent Required for Cleanup
8081	Hexane	Hexane
608	Hexane	Hexane
8082	Hexane	Hexane
8270	none	-

11.7. Nitrogen Blowdown Technique

11.7.1. Place the concentrator tube in a warm water bath at < 35°C and evaporate the solvent volume to the required level using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon). **Do not let the sample go dry**.

CAUTION: Do not use plasticized tubing between the carbon trap and the sample.

11.7.2. The internal wall of the tube must be rinsed down several times with the appropriate solvent during the operation. During evaporation, the solvent level in the tube must be positioned to prevent water from condensing into the sample (i.e., the solvent level should be below the level of the water bath). Under normal operating conditions, the extract should not be allowed to become dry.

CAUTION: When the volume of solvent is reduced below 1 ml, semi-volatile analytes may be lost.

11.8. Measure the final extract volume using an appropriate graduated pipet (see 8.7). Transfer the concentrated extract to the appropriate labeled autosampler vial or storage vial. The extracts obtained may now be analyzed for the target analytes using the appropriate determinative technique. The extract holding time is 40 days from sample preparation to analysis.

12. DATA REVIEW and REPORTING

12.1. Bench sheets are completed and a batch lot number is assigned. The Manufacturer's lot numbers or ID's for the reagents are added to bench sheets (see Attachments).

12.2. Following primary data review, all data is reviewed by a secondary analyst. Refer to the *SOP for Laboratory Data Review Process (ADM-DREV)* for details. The person responsible for final review of the bench sheet should assess the overall validity and quality of the results.

13. QUALITY CONTROL

13.1. Refer to the SOP for the determinative method and the SOP for Analytical Batches and Analytical Sequences for minimum QC requirements.

13.2. Any reagent blanks, laboratory control samples, or matrix spike samples should be subjected to exactly the same analytical procedures as those used on actual samples.

14. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

14.1. Refer to the SOP for *Non Conformance and Corrective Action (CE-QA008)* for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2. Handling out-of-control or unacceptable data

14.2.1. On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, runlogs, for example.

14.2.2. Some examples when documentation of a nonconformity is required using a Nonconformity and Corrective Action Report (NCAR):

- Quality control results outside acceptance limits for accuracy and precision
- Method blanks or continuing calibration blanks (CCBs) with target analytes above acceptable levels
- Sample holding time missed due to laboratory error or operations
- Deviations from SOPs or project requirements
- Laboratory analysis errors impacting sample or QC results
- Miscellaneous laboratory errors (spilled sample, incorrect spiking, etc)
- Sample preservation or handling discrepancies due to laboratory or operations error

15. POLLUTION PREVENTION AND WASTE MANAGEMENT

15.1. It is the laboratory's practice to minimize the amount of solvents, acids, and reagents used to perform this method wherever feasibly possible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvents and/or reagents used in this method can be minimized when recycled or disposed of properly.

15.2. The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Environmental Health and Safety Manual.

15.3. This method uses Dichloromethane and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and recycled off site.

15.4. This method uses non-halogenated solvents and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the

hazardous waste storage area and disposed of in accordance with Federal and State regulations.

15.5. This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 5-9 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the ALS EH&S Manual for details.

15.6. This method uses a base. Waste base is hazardous to the sewer system and to the environment. All waste must be neutralized to a pH of 5-9 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the ALS EH&S Manual for details

16. METHOD PERFORMANCE

Available method performance data is given in the reference method. In addition, this procedure was validated through single laboratory studies of accuracy and precision as specified in the determinative procedures.

17. TRAINING

17.1. Training outline

17.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

17.1.2. Assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

17.1.3. Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

17.2. Training is documented following the Corporate *Training Policy* SOP and the *ALS, Kelso Training Procedure.*

NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

18. REFERENCES

18.1. EPA SW-846, Test Methods For Evaluating Solid Waste, Third Edition, Update III, December 1996, Method 3520C, Revision 3

19. METHODS MODIFICATIONS

19.1. There are no known modifications in this laboratory standard operating procedure from the reference method.

20. CHANGES SINCE THE LAST REVISION

20.1. Re-formatted SOP to ALS style.
20.2. Changed references from CAS to ALS throughout the document.
20.3. Updated former CAS Corporate SOPs to ALS Corporate SOPs.
20.4. Improved the Definitions Section.
20.5. Sec. 9.9: Removed prep instruction – solution is purchased.
20.6. Sec. 11.4.3: Removed 8141 and added SIM PAH.
20.7. Sec. 11.6: Removed 8141.
20.8. Added a Method Modifications Section.
20.9. Added Section 12 Data Review and Reporting.
20.10. Table 1: Removed 8141.

TABLE 1

APPLICABLE DETERMINATIVE SOPs

CONGENER-SPECIFIC DETERMINATION OF PCBS BY GG/ECD	SOC-8082C
ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY: CAPILLARY COLUMN TECHNIQUE	SOC-8081
PCBS AS AROCLORS	SOC-8082A
POLYNUCLEAR AROMATIC HYDROCARBONS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY SIM	SOC-8270P
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS	SOC-8270C
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS – LOW LEVEL PROCEDURE	SOC-8270L
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS SELECTED ION MONITORING	SOC-8270S
ORGANOCHLORINE PESTICIDES AND PCBs	SOC-608

Attachments
Test-Specific Benchsheets
(10 pages)

Additional Prep Information For EPA 3520C for waters by EXT-3520

Service Request _____ **Workgroup** _____

DCM (GC^2) Lot#: _____ Sulfuric Acid Lot#: _____ NaOH Lot#: _____

pH<2 (Time/Date/Initial):_____

pH>11 (Time/Date/Initial):_____

Continuous $Start_1$ (Time/Date/Initial): _____

Continuous $Stop_1$ (Time/Date/Initial): _____

Continuous $Start_2$ (Time/Date/Initial): _____

Continuous $Stop_2$ (Time/Date/Initial): _____

Sulfate Lot # _____ Glass Wool Lot#:_____

S-Evap (Time/Date/Initial): _____ S-Evap Therm. ID: _____

Temp as measured: _____°C Correction factor:_____ °C Adjusted temp:_____ °C

N-Evap (Time/Date/Initial): _____ N-Evap Therm. ID:_____

Temp as measured: _____°C Correction factor: _____ °C Adjusted temp: _____ °C

Clean-up #1: _____ Time/Date/Initial: _____

Clean-up #2: _____ Time/Date/Initial: _____

Archive Storage: _____

Date Completed (Time/Date/Initial): _____

Comments/Observations:

_____

_____

_____

Bench Sheet Review Check List
❑ Hold Times Met (if no, Reason: _____)
❑ Prep date, dept, method, product code correct in stealth
❑ Spike Information correct
❑ Weights/Volumes and units correct on raw and final bench sheets
❑ Sample IDs have been checked—Bottle numbers appended if required
❑ Names present for: Started by, Completed by, relinquished by, and witnessed by.
❑ Training has been circled
❑ Extract Storage recorded
❑ Additional Prep Sheet completely filled out (NA or line out Blanks)
❑ All clean-ups have been noted on additional prep sheet
❑ Signed service request with Form V, if applicable, has been attached

Note: **a) Any Acids or Bases used in this procedure must be checked for contamination prior to use. (See ALS SOP)**

b) All anhydrous sodium sulfate (Na_2SO_4) used in this procedure must be thoroughly rinsed with Methylene chloride (DCM).

Steps 1 - 5 MUST be done in a fume hood.

1. Thoroughly rinse continuous extractors and condensers with DCM.
2. Add approximately 500mL of DCM to the extractor body.
3. Using a graduated cylinder, measure out *volume* of sample. If the entire contents of the bottle are to be extracted, mark the level of the sample on the outside of the bottle prior to adding contents to the extractor body.
4. Slowly add the contents of the sample to the extractor body, using caution not to allow water to enter into the boiling flask. (This can be done by tilting the extractor back, adding the water slowly down the back of the extractor, and slowly tilting the extractor forward to allow the DCM layer to flow into the boiling flask.) If necessary, add additional DI water to the extractor to ensure proper operation. Use 1 liter of DCM rinsed DI water for the MB and LCS.
5. Rinse graduated cylinder or sample bottle with 60mL of DCM and add to the extractor.
6. Add surrogate and matrix spikes. See chart. **(Witnessed by a trained analyst)**
7. Check the pH of the sample with narrow range pH paper and adjust to a pH of *primary pH* with 1:1 H_2SO_4 or 1:1 NaOH.
8. With heating mantles set on 4, cycle for 18-24 hours. Record start and end times.
9. If secondary pH extraction is required proceed, otherwise skip to step 12.
10. When extracts are cooled sufficiently, attach a clean boiling flask with 300mL of DCM to the extractor, then adjust the pH of the sample with narrow range pH paper to *Secondary pH* with 1:1 H_2SO_4 or 1:1 NaOH.
11. With heating mantles set on 4, cycle for an additional 18-24 hours. Record start and end times.
12. When extracts are cooled sufficiently, decant extracts into pre-rinsed KD apparatus assembled with a TRS modified funnel (funnel set up with a small plug of DCM rinsed glass wool, then anhydrous Na_2SO_4[a]). **Always pour the primary extract first**. Rinse through the Na_2SO_4 and apply vacuum to complete the transfer.
13. Concentrate the extracts on an S-Evap with the temperature set between 70-75°C. Remove the KD when the solvent level is low enough to remove the collector. Extreme caution must be used to ensure that the sample does not concentrate too low or to dryness.
14. Based on sample analysis and compound list, determine cleanup direction to take. See chart.
15. If GPC cleanup is performed, only half the original extract is retained. Therefore the extract must be concentrated to 1/2 the final volume to achieve the correct "calculated" final volume.
16. Concentrate the extracts to < *calculated final volume* on the N-Evap under a **gentle** stream of nitrogen with the temperature not exceeding 35°C. Extreme caution must be used to ensure that the sample does not concentrate too low or to dryness.
17. Bring extracts to *calculated final volume* in DCM and place in a labeled, *color*, 2mL autosampler vial.
18. Complete all necessary paperwork. Turn extracts and paperwork over to MS analysts.

Test	Volume	Surrogate/Amount	Spike/Amount	Primary pH	Secondary pH	Calculated Final Volume	Color	Cleanup
8270/625/ 8270 PAH	1 Liter	AP/1ml.	8270/1mL Benzidine/50µL (1mg/mL)	<2	>11	1 ml.	clear	GPC if needed
8270 Appendix IX	1 Liter	AP/1ml.	8270/1mL Benzidine/50µL +App IX Spikes	<2	>11	1 ml.	clear	GPC if needed
8270-LL	1 Liter	AP/50µl	8270/50µL Benzoic/50µL	<2	N/A *** >11 for Aniline	2 ml.	amber	GPC if needed
SIM-PAH/ SIM ALK	1 Liter	AP/20µl	PAH/100µL	neutral	N/A	5 ml.	green	If needed Silica Gel*
SIM-PAH-PCP	1 Liter	AP/20µl	PAH/100 µL Penta/400µL	<2	N/A	5 ml.	green	GPC if needed
SIM-PAH-ULL	1 Liter	ULL AP/100µL(10ppm)	BARR/500µL** or ULL PAH/500µL(1ppm)	neutral	N/A	1mL	green	Silica gel if needed*
SIM-PAH-PCP-ULL	1 Liter	ULL AP/100µL(10ppm)	BARR/500µL** or ULLPAH/500µL(1ppm) Penta/100µL(25ppm)	<2	N/A	1mL	green	GPC if needed
Thermo Retec	1 Liter	AP/50µL	TR/50µL	<2	N/A	5mL	clear	none
NDMA	1 Liter	NDMA-D6/100µL	NDMA-d6/100µL	>11	N/A	2mL	amber	none
1,2,3 TCP	1 Liter	TCP/20µL(5ppm)	1,2,3 TCP/200µL(0.5ppm)	neutral	N/A	2mL	amber	none

***If aniline is present, needs a secondary pH change of >11 and a 2 day extraction.

Additional Prep Information for 625-LL waters by EXT-3520

Service Request:_____ **Workgroup:**_____

DCM (GC^2) Lot#: _____ Sulfuric Acid Lot#:_____ NaOH Lot#:_____

pH<2 (Time/Date/Initial):_____

pH>11 (Time/Date/Initial):_____

Continuous Start 1 (Time/Date/Initial):_____

Continuous Stop 1 (Time/Date/Initial):_____

Continuous Start 2 (Time/Date/Initial):_____

Continuous Stop 2 (Time/Date/Initial):_____

Sulfate Lot#:_____ Glass Wool Lot#:_____

S-Evap (Time/Date/Initial):_____ S-Evap Therm. ID: _____

Temp as measured: _____°C Correction factor:_____ °C Adjusted temp:_____ °C

N-Evap (Time/Date/Initial):_____ N-Evap Therm. ID:_____

Temp as measured:_____°C Correction factor:_____ °C Adjusted temp:_____ °C

Date Completed (Time/Date/Initial):_____

Comments/Observations:_____

_____

_____

_____

Bench Sheet Review Check List
❑ Hold Times Met (if no, Reason: _____)
❑ Prep date, dept, method, product code correct in stealth
❑ Spike Information correct
❑ Weights/Volumes and units correct on raw and final bench sheets
❑ Sample IDs have been checked—Bottle numbers appended if required
❑ Names present for: Started by, Completed by, relinquished by, and witnessed by.
❑ Training has been circled
❑ Extract Storage recorded
❑ Additional Prep Sheet completely filled out (NA or line out Blanks)
❑ All clean-ups have been noted on additional prep sheet
❑ Signed service request with Form V, if applicable, has been attached

ALS Environmental
Appendix from EXT-3520 For Extracting 625-LL in water by EPA Method 3520

Steps 1-5 MUST be done in a fume hood

1. All lab equipment that comes into contact with sample must be thoroughly rinsed with DCM prior to use.
2. Add approximately 500 mL of DCM to the extractor body.
3. Using a graduated cylinder, measure out 1L of sample. If the entire contents of the bottle are to be extracted, mark the level of the sample on the outside of the bottle prior to adding contents to the extractor.
4. Slowly add the contents of the sample to the extractor body, using caution not to allow water to enter into the boiling flask. (This can be done by tilting the extractor back, adding the water slowly down the back of the extractor, and slowly tilting the extractor forward to allow the DCM layer to flow into the boiling flask. If necessary, add additional DI water to the extractor to ensure proper operation. Use 1L of DCM rinsed DI water for the MB and LCS's.
5. Rinse graduated cylinder or sample bottle with 60 mL of DCM and add to the extractor.
6. Add 50uL of AP surrogate to all samples and QC, and matrix spikes (see chart). **(Witnessed by a trained analyst)**
7. Check the pH of the sample with narrow range pH paper and adjust to a pH of <2 with 1:1 H_2SO_4 or 1:1 NaOH.
8. With the heating mantles set on 4, cycle for 18-24 hours. Record start and stop times.
9. When extracts are cooled sufficiently, attach a clean boiling flask with 300mL of DCM to the extractor, then adjust the pH of the sample with narrow range pH paper to >11 with 1:1 H_2SO_4 or 1:1 NaOH.
10. With the heating mantles set on 4, cycle for an additional 18-24 hours. Record start and stop times.
11. When extracts are cooled sufficiently, decant extracts into pre-rinsed KD apparatus assembled with a TRS modified funnel (funnel set up with a small plug of DCM rinsed glass wool, then DCM rinsed granular anhydrous Na_2SO_4). **Always pour the primary extract first**. Rinse through Na_2SO_4 with DCM and apply vacuum to complete the transfer.
12. Concentrate the extracts on an S-Evap with the temperature set between 70-75°C. Remove the KD when the solvent level is low enough to remove the collector. Extreme caution must be used to ensure that the sample does not concentrate too low or to dryness.
13. Concentrate the extracts to <2mL on the N-Evap under a gentle stream of nitrogen with the temperature not exceeding 35°C. Extreme caution must be used to ensure that the sample does not concentrate too low or to dryness.
14. Bring extracts to 2mL in DCM and place 1mL in an amber vial for the 625-LL portion and 1mL into a clear vial for the benzidine portion.
15. Complete all necessary paperwork.

QC	SPIKE	AMOUNT
LCS/DLCS/MS/DMS	8270 Benzidine Pentachlorobenzene	50uL 100uL 50uL

Additional Prep Information For 608 Waters

Service Request #_____ **Work Group #:**_____

Solvents/Reagents used:

DCM Lot #_____

Continuous Start (Time/Date/Initial):_____

Continuous Stop (Time/Date/Initial):_____

Cleanups:

Solvent Exchanged To Hexane (Time/Date/Initial):_____ **Hexane Lot #**_____
S-Evap Time/Date/Intial:_____ **Thermometer ID:**_____
Temp as measured:_____°C **Correction factor:**_____°C **Adjusted temp:**_____°C

N-Evap Time/Date/Initial:_____ **Thermometer ID:**_____
Temp as measured:_____°C **Correction factor:**_____°C **Adjusted temp:**_____°C

Florisil Clean-up (3620)(Time/Date/Initial): _____ **Florisil Lot #** _____
9:1 Hexane:Acetone Lot #:_____ **1:1 Hexane:Acetone Lot #:**_____
N-Evap Time/Date/Initial:_____ **Thermometer ID:**_____
Temp as measured:_____°C **Correction factor:**_____°C **Adjusted temp:**_____°C

Archive:

Vial:_____ **Vial Storage:**_____

Archived Extract Storage:_____

Comments/Observations:_____

_____

_____

_____

Bench Sheet Review Check List
❑ Hold Times Met (if no, Reason: _____)
❑ Prep date, dept, method, product code correct in stealth
❑ Spike Information correct
❑ Weights/Volumes and units correct on raw and final bench sheets
❑ Sample IDs have been checked—Bottle numbers appended if required
❑ Names present for: Started by, Completed by, relinquished by, and witnessed by.
❑ Training has been circled
❑ Extract Storage recorded
❑ Additional Prep Sheet completely filled out (NA or line out Blanks)
❑ All clean-ups have been noted on additional prep sheet
❑ Signed service request with Form V, if applicable, has been attached

ALS Environmental
STANDARD OPERATING PROCEDURE FOR EXTRACTION OF

Organochlorine Pesticides and PCBs in Water

EPA 608

Method EPA 3520

1. Thoroughly rinse all glassware with DCM
2. Mark the meniscus on the side of the sample container for later determination of sample volume. Note any sediment layer in the sample bottle.
3. Add 500mL of Methylene Chloride (DCM) to the continuous extractor. Add several boiling chips to the flask.
4. Pour entire sample into the continuous extractor. Rinse sample bottle with 5-10mL DCM. Add DCM rinse to the continuous extractor. Use 1L of DI water for the MB and LCS.
5. Add surrogate to all samples and QC. Add matrix spike to MS/DMS and/or LCS/DLCS.
6. Check the pH of the sample with wide-range pH paper and adjust (if necessary) with 1:1 H_2SO_4 or 10N NaOH.
7. Add sufficient **DI water** (if necessary) to the extractor to ensure proper operation. Extract for 18-24 hours. Record the start and stop times on the bench sheet.
8. Decant the extract into a K-D apparatus. Rinse flask three times with 3-5mL DCM pouring each rinse into the K-D. If water is present in the extract, pour the extract through a modified funnel containing a small amount of anhydrous sodium sulfate. Rinse the sulfate with DCM.
9. Evaporate extracts on the S-Evap, keeping the temperature between 70-75°C. When the extracts are at 5-10mLs, squirt 5mL of Hexane down into the Snyder column, keeping the collector immersed in the hot water bath. Let the extracts solvent exchange into Hexane on the S-Evap (70-75°C) until solvent stops dripping (about 20 min.). **Do not let extracts go dry**.
10. Concentrate extracts to 1-2mL on the N-Evap (30-35°C) under a gentle stream of nitrogen. Use extreme caution to ensure that the extracts do not concentrate too low or to dryness.
11. Concentrate extracts to <2mL on the N-Evap. Take extracts to a **2mL final volume** in Hexane.
12. Perform a **Florisil clean-up** on the 1mL of the extract.
13. Concentrate extracts to <1mL on the N-Evap. Take extracts to 1mL and place into a yellow vial.
14. Archive remaining 1mL of un-cleaned extract and record the location.

Additional Prep Information For Pest/PCB Water
EPA Method 3520

Service Request #_____ **Work Group #:**_____

Solvents/Reagents used:

DCM Lot #_____

Continuous Start (Time/Date/Initial):_____

Continuous Stop (Time/Date/Initial):_____

Cleanups:

Solvent Exchanged To Hexane (Date/Time/Initial) _____ **Hexane Lot #:**_____

S-Evap Therm ID:_____ **S-Evap (Date/Time/Initial):**_____
Temp as measured:_____**°C Correction factor:**_____ **°C Adjusted temp:**_____ **°C**

N-Evap Therm ID:_____ **N-Evap (Date/Time/Initial):**_____
Temp as measured:_____**°C Correction factor:**_____ **°C Adjusted temp:**_____ **°C**

Sulfuric Acid Clean-up (Date/Time/Initial):_____ **Acid Lot #:**_____

Clean-up #2:_____ ☐ **all samples** ☐ **some samples:**_____
Clean-up #3:_____ ☐ **all samples** ☐ **some samples:**_____

Pest Vial:_____ **Vial Storage:**_____
PCB Vial:_____ **Vial Storage:**_____

Archived Extract Storage:_____

Comments/Observations:_____

_____

Bench Sheet Review Check List
☐ Hold Times Met (if no, Reason: _____)
☐ Prep date, dept, method, product code correct in stealth
☐ Spike Information correct
☐ Weights/Volumes and units correct on raw and final bench sheets
☐ Sample IDs have been checked—Bottle numbers appended if required
☐ Names present for: Started by, Completed by, relinquished by, and witnessed by.
☐ Training has been circled
☐ Extract Storage recorded
☐ Additional Prep Sheet completely filled out (NA or line out Blanks)
☐ All clean-ups have been noted on additional prep sheet
☐ Signed service request with Form V, if applicable, has been attached

ALS Environmental

ORGANOCHLORINE PESTICIDES AND PCBs IN WATER

EPA Method 3520

1. Mark the meniscus on the side of the sample container for later determination of sample volume. Note any sediment layer in sample bottle. Use 1L of DI water for the method blank and laboratory control sample.

2. Add 500mL of DCM to the continuous extractor. Add several boiling chips to the flask.

3. Pour entire sample into continuous extractor.

4. Rinse the bottle with DCM and add to the extractor.

5. Add 100uL of 8081/8082 surrogate and 50ul of 8081 matrix spike to pesticide QC samples and 50uL of 8082-AR matrix spike to PCB QC samples.

6. Check pH of the sample with wide-range pH paper, and adjust to pH of 5-9 with 1:1 H_2SO_4, or 10 N NaOH.

7. Add sufficient DI water to the extractor to ensure proper operation, and extract for 18-24 hours.

8. Record start and stop times on the bench sheet.

9. Decant the extract into a K-D apparatus. If there is visible water in the flask, pour through a funnel with a small amount of muffled anhydrous sodium sulfate. Rinse flask three times with DCM adding to sample in KD.

10. Evaporate on the S-Evap, keeping temperature between 70-75°C. When extracts are at about 10mL, squirt about 5mL of hexane down top of Snyder column, keeping collector in the hot water of the bath. If the extract is allowed to cool, it will be necessary to add another boiling chip to the extract. Let the extracts solvent exchange on the S-Evap (at 70-75°C) into hexane for about 20 minutes or until solvent stops dripping, making sure they do not go dry.

11. Take to 2mL final volume in hexane and split if 8082-AR is required. Place exactly 1mL in a yellow vial for the Pesticides and mark the meniscus.

12. If PCB's are needed, do an acid cleanup on the remaining extract. After acid cleanup put the PCB extract in green vials and mark the meniscus.

Additional Prep Information Congener Water
EPA Method 3520

Service Request #_____ **Work Group #:**_____

Solvents/Reagents used:

DCM Lot #_____

Continuous Start (Time/Date/Initial):_____

Continuous Stop (Time/Date/Initial):_____

Cleanups:

Solvent Exchanged To Hexane (Date/Time/Initial)_____ **Hexane Lot #:**_____

S-Evap Thermometer ID:_____ **S-Evap (Date/Time/Initial):**_____
Temp as measured:_____**°C Correction factor:**_____**°C Adjusted temp:**_____**°C**

N-Evap Thermometer ID:_____ **N-Evap (Date/Time/Initial):**_____
Temp as measured:_____**°C Correction factor:**_____**°C Adjusted temp:**_____**°C**

Sulfuric Acid Clean-up (Date/Time/Initial):_____ **Acid Lot #:**_____

Clean-up #2:_____ ☐ **all samples** ☐ **some samples:**_____
Clean-up #3:_____ ☐ **all samples** ☐ **some samples:**_____

PCB Vial:_____ **Vial Storage:**_____

Archived Extract Storage:_____

Comments/Observations:_____

_____

Bench Sheet Review Check List
❑ Hold Times Met (if no, Reason: _____)
❑ Prep date, dept, method, product code correct in stealth
❑ Spike Information correct
❑ Weights/Volumes and units correct on raw and final bench sheets
❑ Sample IDs have been checked—Bottle numbers appended if required
❑ Names present for: Started by, Completed by, relinquished by, and witnessed by.
❑ Training has been circled
❑ Extract Storage recorded
❑ Additional Prep Sheet completely filled out (NA or line out Blanks)
❑ All clean-ups have been noted on additional prep sheet
❑ Signed service request with Form V, if applicable, has been attached

ALS Environmental

CONGENERS IN WATER

Methods EPA 3520

1. Mark the meniscus on the side of the sample container for later determination of sample volume. Note any sediment layer in sample bottle. Use 1L of DI water for the method blank and laboratory control sample.

2. Add 500mL of DCM to the continuous extractor. Add several boiling chips to the flask.

3. Pour entire sample into continuous extractor.

4. Rinse the bottle with DCM and add to the extractor.

5. Add congener surrogate to all samples and Congener matrix spike to QC samples.

6. Check pH of the sample with wide-range pH paper, and adjust to pH of 5-9 with 1:1 H_2SO_4, or 10 N NaOH.

7. Add sufficient DI water to the extractor to ensure proper operation, and extract for 18-24 hours.

8. Record start and stop times on the bench sheet.

9. Decant the extract into a K-D apparatus. If there is visible water in the flask, pour through a funnel with a small amount of muffled anhydrous sodium sulfate. Rinse flask three times with DCM adding to sample in KD.

10. Evaporate on the S-Evap, keeping temperature at 70-75°C. When extracts are at about 10mL, squirt about 5mL of hexane down top of Snyder column, keeping collector in the hot water of the bath. If the extract is allowed to cool, it will be necessary to add another boiling chip to the extract. Let the extracts solvent exchange on the S-Evap (at 70-75°C) into hexane for about 20 minutes or until solvent stops dripping, making sure they do not go dry.

11. Take to 1mL final volume in hexane and do an acid cleanup. After acid cleanup put exactly 500uL of Congener extract in green vials and mark the meniscus.

ALS Environmental

DOCUMENT TITLE:	***DETERMINATION OF METALS AND TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY (ICP-MS)***
REFERENCED METHOD:	***EPA 6020, 6020A***
SOP ID:	***MET-6020***
REV. NUMBER:	***15***
EFFECTIVE DATE:	***07/31/2013***

DETERMIMATION OF METALS AND TRACE ELEMENTS BY INDUCTIVELY COUOPLED PLASMA-MASS SPECTROMETRY (ICP-MS)

MET-6020

ALS-KELSO

SOP ID:	MET-6020	Rev. Number:	15	Effective Date:	07/31/2013

Approved By: _____ Date: 7/16/13
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Approved By: _____ Date: 7/16/13
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Issue Date:	_____	Doc Control ID#:	_____	Issued To:	_____

Standard Operating Procedure

For

DETERMIMATION OF METALS AND TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY (ICP-MS) METHOD 6020

1. SCOPE AND APPLICATION

1.1 This procedure is used to determine the concentrations of certain elements in water, soil, tissues, aqueous and non-aqueous wastes, and sediment samples using EPA Method 6020 or 6020A. Table 1 indicates analytes that are typically determined by this procedure and lists the standard Method Reporting Limits (MRLs) for each analyte in water and soil. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. Project-specific MRLs may apply, and if lower than standard MRLs, it is demonstrated through method detection limit determinations and analysis of MRL standards that the MRL is achievable. Method Detection Limits (MDLs) that have been achieved are listed in Table 1. These may change as new studies are performed.

1.2 The complexity of the technique generally requires outside study of appropriate literature as well as specialized training by a qualified spectroscopist. The scope of this document does not allow for the in-depth descriptions of the relevant spectroscopic principles required for gaining a complete level of competence in this scientific discipline.

2 SUMMARY OF METHOD

2.1 Prior to analysis, samples must be digested using appropriate sample preparation methods. The digestate is analyzed for the elements of interest using ICP-mass spectrometry (ICP-MS).

2.2 Methods 6020 and 6020A describe the multi-elemental determination of analytes by ICP-MS. The method measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

3 DEFINITIONS

3.1 **Batch** – A batch of samples is a group of environmental samples that are prepared and/or analyzed together as a unit with the same process and personnel using the same lot(s) of reagents. It is the basic unit for analytical quality control.

3.1.1 Preparation Batch – A preparation batch is composed of one to twenty field samples, all of the same matrix, and with a maximum time between the start of processing of the first and last samples in the batch to be 24 hours.

3.2 Analysis Batch – Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration (initial or continuing verification) followed by sample extracts interspersed with calibration standards (CCBs, CCVs, etc.) The sequence ends when the set of samples has been analyzed or when qualitative and/or quantitative QC criteria indicate an out-of-control situation.

3.3 **Sample**

3.3.1 Field Sample – An environmental sample collected and delivered to the laboratory for analysis; a.k.a., client's sample.

3.3.2 Laboratory Sample – A representative portion, aliquot, or subsample of a field sample upon which laboratory analyses are made and results generated.

3.4 **Quality System Matrix** – The *matrix* of an environmental sample is distinguished by its physical and/or chemical state and by the program for which the results are intended. The following sections describe the matrix distinctions. These matrices shall be used for purpose of batch and quality control requirements.

3.4.1 Aqueous – Any groundwater sample, surface water sample, effluent sample, and TCLP or other extract. Specifically excluded are samples of the drinking water matrix and the saline/estuarine water matrix.

3.4.2 Drinking water – Any aqueous sample that has been designated a potable or potential potable water source.

3.4.3 Saline/Estuarine water – Any aqueous sample from an ocean or estuary or other salt-water source.

3.4.4 Nonaqueous Liquid – Any organic liquid with <15% settleable solids.

3.4.5 Animal tissue – Any tissue sample of an animal, invertebrate, marine organism, or other origin; such as fish tissue/organs, shellfish, worms, or animal material.

3.4.6 Solids – Any solid sample such as soil, sediment, sludge, and other materials with >15% settleable solids.

3.4.7 Chemical waste – Any sample of a product or by-product of an industrial process that results in a matrix not described in one of the matrices in Sections 3.4.1 through 3.4.6. These can be such matrices as non-aqueous liquids, solvents, oil, etc.

3.4.8 Miscellaneous matrices – Samples of any composition not listed in 3.4.1 – 3.4.7. These can be such matrices as plant material, paper/paperboard, wood, auto fluff, mechanical parts, filters, wipes, etc. Such samples shall be batched/grouped according to their specific matrix.

3.5 Matrix Spike/Duplicate Matrix Spike (MS/DMS) Analysis – In the matrix spike analysis, predetermined quantities of target analytes are added to a sample matrix prior to sample preparation and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the method used for the analysis. Duplicate samples are spiked, and analyzed as a MS/DMS pair. Percent recoveries are calculated for each of the analytes detected. The relative percent difference (RPD) between the duplicate spikes (or samples) is calculated and used to assess analytical precision. The concentration of the spike should be at the mid point of the calibration range or at levels specified by a project analysis plan.

3.6 Laboratory Duplicates (DUP) – Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. The relative percent difference (RPD) between the sample and its duplicate is calculated and used to assess analytical precision.

3.7 Surrogate – Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to extraction and analysis. Percent recoveries are calculated for each surrogate.

3.8 Method Blank (MB) – The method blank is an artificial sample composed of analyte-free water or solid matrix and is designed to monitor the introduction of artifacts into the analytical process. The method blank is carried through the entire analytical procedure.

3.9 Laboratory Control Samples (LCS) – The LCS is an aliquot of analyte free water or analyte free solid to which known amounts target analytes are added. The LCS is prepared and analyzed in exactly the same manner as the samples. The percent recovery is compared to established limits and assists in determining whether the batch is in control.

3.10 Independent Verification Standard (ICV) – A pre-mixed, purchased, second-source standard analyzed after the calibration curve. This is used to verify the validity of the initial calibration standards

3.11 Continuing Calibration Verification Standard (CCV) – A mid-level standard analyzed at specified intervals. Used to verify that the initial calibration curve is still valid for quantitative purposes.

3.12 Duplicates and Duplicate Matrix Spikes are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. Depending on the method of analysis, either a duplicate analysis (and/or a matrix spiked sample) or a matrix spiked sample and duplicate matrix spiked sample (MS/DMS) are analyzed.

3.13 Standard Reference Material (SRM) – A material with specific certification criteria and is issued with a certificate or certificate of analysis that reports the results of its characterizations and provides information regarding the appropriate use(s) of the material.

An SRM is prepared and used for three main purposes: (1) to help develop accurate methods of analysis; (2) to calibrate measurement systems used to facilitate exchange of goods, institute quality control, determine performance characteristics, or measure a property at the state-of-the-art limit; and (3) to ensure the long-term adequacy and integrity of measurement quality assurance programs.

4 INTERFERENCES

4.1 Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z). A data system must be used to correct for these interferences. This involves determining the signal for another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal. Attention should be given to circumstances where very high ion currents at adjacent masses may contribute to ion signals at the mass of interest. Matrices exhibiting a significant problem of this type may require resolution improvement, matrix separation, or analysis using another isoptope.

4.2 Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences that could affect ICP-MS determinations have been identified in the literature. Refer to Method 6020/A for further discussion.

5 SAFETY

5.1 All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2 Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in MSDSs where available. Refer to the ALS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3 Hydrochloric and/or Nitric Acid are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

5.4 High Voltage – The RF generator supplies up to 2000 watts to maintain an ICP. The power is transferred through the load coil located in the torch box. Contact with the load coil while generator is in operation will likely result in death. When performing maintenance on the RF generator, appropriate grounding of all HV capacitors must be performed as per manufacturer.

5.5 UV Light – The plasma is an intense source of UV emission, and must not be viewed with the naked eye. Protective lenses are in place on the instrument. Glasses with special protective lenses are available when direct viewing of the plasma is necessary.

6 SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1 Aqueous samples are typically collected in plastic containers. Aqueous samples are preserved with nitric acid (pH<2), then refrigerated at 4 ± 2°C from receipt until digestion.

Soil or solid samples may be collected in plastic or glass jars. Non-aqueous samples are refrigerated at 4 ± 2°C from receipt until digestion.

6.2 Samples are prepared via procedures in SOPs MET-DIG, MET-3020A, or MET-3050 depending on matrix and project specifications.

6.3 Digestates are stored in the appropriate volumetric containers. Following analysis, digestates are stored until all results have been reviewed. Digestates are neutralized prior to disposal through the sewer system, 2 weeks after data is reviewed.

7 APPARATUS & EQUIPMENT

7.1 Instruments: Thermo Elemental ExCell (K-ICP-MS-02) Serial # EX191, Thermo Elemental X-Series (K-ICP-MS-03) Serial # X0193, and NexION 300D (K-ICP-MS-04).

7.2 Thermo Meinhard type (Part # 1201318)

7.3 Thermo Impact Bead Quartz Spray Chamber (Part # 3600170)

7.4 Thermo X7 Nickel Sample Cone (1.0 mm orifice) (Part # 3004661), or Xi sample cone (part # 3600812)

7.5 Thermo X7 Nickel Skimmer Cone (0.75 mm orifice) (Part # 3200860) or Xi skimmer cone(part # 3600811)

8 STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1 All standards are prepared from NIST traceable standards. The expiration dates are assigned according to the EPA method and the vendor's assigned expiration dates. For example, working ICS solutions are prepared weekly in accordance with Method 6020, Section 5.6.1.

8.1.1 1000 ppm Single Element Stock Standard Solutions: Each stock standard is store at room temperature on shelves located in room 113 of the metals lab. The manufacturer, lot number, and expiration date of each stock standard is recorded in a bound logbook also located in room 113. Additionally each stock standard is given a unique, identifying name.

8.1.2 Intermediate Standard Solutions: Intermediate mixed stock solutions are made from the individual stock standards described above. The individual component of each mixed solution is recorded in a bound logbook located in the ICP-MS laboratory and mixed solution is given a unique, identifying name. The expiration date for the intermediate standard is the earlier of any one of its stock components.

8.1.3 Calibration Standards: Calibration standards are made fresh daily from the intermediate standard solutions. Each individual intermediate standard used in the calibration standard is recorded in a bound logbook located in the ICP-MS laboratory, and the calibration standard solution is given a unique, identifying name. The calibration standards unique name is used on the raw data to link the data to the subsequent prepared standards and ultimately the original purchased stock standard.

8.2 Standards Preparation

8.2.1 Expiration of all standard solutions defaults to the earliest expiration date of an individual component unless otherwise specified.

8.2.2 Calibration Standards

The calibration standard is prepared from two intermediate stock solutions. These solutions are prepared in acid rinsed 1000 mL Class A volumetric flasks following the formulations laid out on the attached example standard sheet (see Attachments). The working calibration standard is made daily by aliquoting 2.5 mL of each of the intermediate solutions in to a 100 mL Class A volumetric flask and diluting to volume with 1% HNO3. This standard is also used as the Continuing Calibration Verification (CCV).

8.2.3 Initial Calibration Verification (ICV)

8.2.3.1 The ICV intermediate stock solution is prepared in an acid rinsed 100 mL Class A volumetric flask. The solution is prepared by adding 2.0 mL of Inorganic Ventures QCP-CICV-1, 1.0 mL each of QCP-CICV-2 and QCP-CICV-3, 0.5 mL of 1000 ppm Molybdenum stock solution, 0.5 mL of 1000 ppm Uranium stock solution, and 0.5mL of 1000ppm B, Bi, Sr, Ti solution and diluting to volume with 1% HNO3.

8.2.3.2 The working ICV solution is prepared by aliquoting 0.5 mL of the mixed ICV intermediate solution into an acid rinsed 100 mL Class A volumetric flask and diluting to volume with 1% HNO3.

NOTE: The ICV solution is not at the midpoint of the linear range which may be as high as 1000 µg/L for some elements. The ICV solution used is a premixed standard purchased from Inorganic Ventures and contains the elements of interest between 2.5 and 100 µg/L. This solution provides calibration confirmation at more representative levels, given that most ICP-MS analyses are quantifying analytes in the low-ppb to sub-ppb range.

8.2.4 Interference Check Solutions (ICSA and ICSAB)

8.2.4.1 The ICSA is prepared in an acid rinsed 50 mL Class B volumetric flask by aliquoting 1.0 mL of Elements ICSAm (CS-CAK02) solution and diluting to volume with 1% HNO3.

8.2.4.2 The ICSAB is prepared in an acid rinsed 50 mL Class B volumetric flask by aliquoting 1.0 mL of Elements ICSAm (CS-CAK02), 0.125 mL of Inorganic Ventures 6020ICS-9B, and 0.250 mL of 10 ppm Molybdenum solutions and diluting to volume with 1% HNO3.

8.2.5 Post-digestion spikes are performed by adding appropriate amounts of the calibration intermediate solutions to aliquots of the sample digestate. The volumes of each standard used vary based on the native concentrations found in the field samples. Refer to the post-digestion spike in Section 12 for details.

8.2.6 Refer to the appropriate digestion SOP for details of LCSW and matrix spike solution composition and preparation.

8.2.7 Tuning / Mass Calibration Solution

8.2.7.1 A 1ppm intermediate solution containing Be, Bi, Ce, Co, In, Li, Pb, Mg, and U is prepared by adding 1.0 mL of each from 1000 ppm stock standards to an acid rinsed 1000 mL volumetric flask and diluting to volume with 1% nitric acid. The expiration date for the intermediate solution is the earliest of any one of its stock components.

8.2.7.2 The working solution is prepared in three ways:

- For the ExCell (K-ICP-MS-02) a 1.0 ppb tune/mass calibration solution is prepared by adding 1.0 mL of intermediate solution to an acid rinsed 1000 mL volumetric flask and diluting to volume with 1% nitric acid.
- For the X-Series (K-ICP-MS-03) instrument a 5.0 ppb tune/mass calibration solution is prepared by adding 5.0 mL of intermediate solution to an acid rinsed 1000 mL volumetric flask and diluting to volume with 1% nitric acid.
- For the NexION (K-ICP-MS-04) instrument a 2.0 ppb tune/mass calibration solution is prepared by adding 2.0 mL of intermediate solution to an acid rinsed 1000 mL volumetric flask and diluting to volume with 1% nitric acid.
- The expiration date for this solution is taken from the intermediate stock above.

8.3 Internal Standards Stock Solution – Prepare a 10 µg/mL solution containing ^{71}Ga, ^{115}In, ^{6}Li, ^{175}Lu, ^{103}Rh ^{45}Sc, and ^{89}Y by adding 10.0 mL of each 1000 ppm single element stock solution to a acid rinsed 1000 mL volumetric flask and diluting to volume with 1% nitric. Use this solution for addition to blanks, calibration standards and samples at a ration of 0.5 mL of internal standard to 100 mL of solution, or dilute by an appropriate amount using 1% (v/v) nitric acid, if the internal standards are being added by peristaltic pump.

8.4 Additional Reagents

8.4.1 Reagent water, ASTM Type II

8.4.2 "OmniTrace Ultra" Concentrated Nitric Acid (EM Science # NX0408-2)

8.4.3 Argon (Airgas Industrial Grade – 99.999% pure, bulk delivered)

9 PREVENTIVE MAINTENANCE

9.1 All maintenance is documented in the instrument logbook. ALS/Kelso maintains a service contract with the instrument manufacturer that allows for an unlimited number of service calls and full reimbursement of all parts and labor.

9.2 Most routine maintenance and troubleshooting is performed by ALS staff. Preventive maintenance activities listed below should be performed when needed as determined by instrument performance (i.e. stability, sensitivity, etc.) or by visual inspection. Other

maintenance or repairs may, or may not require factory service, depending on the nature of the task.

- cone removal and cleaning
- removal and cleaning of ICP glassware and fittings
- checking and cleaning RF contact strips
- checking air filters and cleaning if necessary
- checking the oil mist filters and cleaning if necessary
- checking the rotary pump oil and adding or changing if necessary
- removal and cleaning of extraction lens
- removal and cleaning of ion lens stack
- replace the electron multiplier as necessary

10 RESPONSIBILITIES

10.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2 It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in the SOP for Documentation of Training, is also the responsibility of the department supervisor/manager.

11 PROCEDURE

11.1 Refer to method 6020 (or 6020A) and the instrument manuals for detailed instruction on implementation of the following daily procedures preceding an analytical run.

11.2 After the instrument has been placed in the "Operate" mode, begin completing the daily instrument log (see Attachments). Refer to the instrument manuals for the optimum settings for each instrument.

11.3 The following parameters are monitored to assure awareness of changes in the instrumentation that serve as signals that optimum performance is not being achieved, or as indicators of the physical condition of certain consumable components (i.e. EMT and cones).

11.3.1 Multiplier Voltages

11.3.2 Gas Flows - Coolant Ar

11.3.3 The nebulizer and auxiliary flows are adjusted later as part of the optimizing procedure.

11.4 Optimization

11.4.1 Gas Flows

11.4.1.1Allow a period of not less than 30 minutes for the instrument to warm up.

11.4.1.2 Aspirate a mixed tune solution into the plasma and monitor the instrument output signal of In at mass 115 on the ratemeter. Adjust the nebulizer and auxiliary flows to obtain maximum signal. Adjust the tension screw on the peristaltic pump to obtain minimum noise in the analytical signal. Record flow rates and note any large variances.

Note: Significant differences in flow rates will be observed for different torches and cones.

11.4.2 Tuning

11.4.2.1 Ion Lens Setting - While monitoring the output signal of a mixed tune solution at mass 115 on the ratemeter, adjust the ion lenses to obtain maximum sensitivity. Refer to the instrument manual for details on performing the adjustments.

11.4.2.2 Mass Calibration - Aspirate the tune / mass calibration solution described in section 8.2.7.2 and perform the mass calibration using the instrument's Mass Calibration program. (Refer to the instrument manual for details pertaining to the mass calibration procedure.) The acceptance criteria for the mass calibration is <0.1 amu from the true value. If the mass calibration fails criteria re-tune the instrument and perform the mass calibration procedure again.

11.4.2.3 Resolution Check - Using the spectra created during the mass calibration procedure; perform the resolution check to assure the resolution is less than 0.9 AMU at 10% peak height. If the resolution does not pass criteria adjust the instrument's resolution settings, run a new scan of the mass calibration solution and recheck.

11.4.2.4 Stability Check - Using the tune / mass calibration solution, perform a short-term stability check as per EPA Method 6020 or 6020A. The relative standard deviations of five scans for each element in the tune solution must be < 5%. If the test does not pass criteria determine the cause (i.e. dirty cones, improper tune, etc.) correct the problem and re-run the test.

11.5 Analytical Run

11.5.1 Calibrate the instrument using a calibration blank (Standard 0), composed of reagent water and 1% nitric acid, and the working calibration standard (8.2.2). The masses typically monitored and those used for quantification are listed in Table 1. These masses are set as defaults in the instrument's analytical procedures. To begin select the correct method. Nebulize Standard 0 (Blank) into the plasma. Allow 1-2 minutes for system to equilibrate prior to establishing baseline. Follow directions on computer screen to perform standardization. Nebulize the working calibration standard into the plasma. The operator must sign and date the first page of standardization.

11.5.2 After the first CCB and before the ICS standards a CRA (MRL / LLICV / LLCCV) standard is analyzed. Method 6020 requires the detection to be > the MDL but < 2x

the MRL. For 6020A, the criteria are 70-130% recovery. For DoD projects, the CRA criteria are 80-120%.

Note: For 6020A the LLCCV must also be analyzed at the end on the analytical run sequence.

11.5.3 Perform the analysis in the order listed below. A daily run log of all samples analyzed is maintained.

Initial Calibration Verification (ICV)
Continuing Calibration Verification (CCV)
Initial Calibration Blank (ICB)
Continuing Calibration Blank (CCB)
CRA (MRL / LLICV / LLCCV)
ICSA
ICSAB
Analyze 10 Samples
CCV
CCB
Analyze 10 Samples
CCV
CCB
Repeat sequence as required to complete analytical run, analyzing CCVs/CCBs every 10 analyses and at the end of the run.

12 QA/QC REQUIREMENTS

12.1 Initial Precision and Recovery Validation

The accuracy and precision of the procedure must be validated before analysis of samples begins, or whenever significant changes to the procedures have been made. To do this, four LCS aliquots are prepared and analyzed. The average percent recovery of for each analyte must be 85-115% (for water, and within the LCS limits for soils) and the RSD <20%.

12.2 Method Detection Limits

12.2.1 A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank matrices at a level near or below the MRL. Follow the procedures starting in Section 11 to analyze the samples. Refer to CE-QA011, *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification* details of performing the MDL study.

12.2.2 Calculate the average concentration found (x) and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. MDL's must be verified annually or whenever there is a significant change in the background or instrument response.

12.3 For method 6020A, an LLQC sample (a CRA that is carried through the digestion) must be analyzed to verify accuracy at the MRL. The recovery must be 70-130%.

12.4 Instrument Detection Limits (IDLs) and linear ranges studies are performed quarterly. These will be calculated and made available to the ICP-MS operator. Linear range studies determine the Linear Dynamic Range (LDR) of the each instrument by analysis of a high concentration standard with results with ± 10% of the expected value. For non-DoD projects samples may be quantified between the MRL and 90% of the LDR without flagging. The Linear Calibration Range (LCR) is established by the highest calibration standard.

- **Note:** IDLs must be < LOD for DOD projects. DoD project samples with concentrations above the calibration standard must be diluted to bring results within the quantitation range. The LOQ and cal standard establish the quantitation range. The lab may report a sample result above quantitation range if the lab runs and passes a CCV that is > sample result.

12.5 The Initial Calibration Verification (ICV) standard is analyzed immediately after calibration. The results of the ICV must agree within ±10% of the expected value. If the control limits are exceeded, the problem will be identified and the instrument recalibrated.

12.6 A Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB) are analyzed after calibration then every 10 samples thereafter with a final CCV/CCB closing the final samples of the analytical run.

12.6.1 The results of the CCV must agree within ±10% of the expected value.

12.6.2 The CCB measured values must be less than the MRL / LOQ for each element for standard applications. Other project-specific criteria may apply (for DoD QSM projects CCB can have no analytes > the LOD).

12.6.3 If the control limits are exceeded, the problem will be identified and corrective action taken. The instrument recalibrated. The previous 10 samples must be reanalyzed.

12.7 The ICSA and ICSAB solutions are analyzed after calibration and before any field samples. The solutions are then reanalyzed every 12 hours. Results of the ICSA are used by the analyst to identify the impact of potential interferences on the quality of the data. Based on these results appropriate action should be taken when interferences are suspected in an field sample including, but not limited to, selecting and alternative isotope for quantification, manual correction of the data, elevating the MRL, selection of an alternative method (e.g. optical ICP, GFAA) or flagging the result as estimated when no other action is possible. Results for the spiked analytes in the ICSAB solution must agree with ± 20% of the expected value.

INTERFERENCE CHECK SAMPLE COMPONENTS AND CONCENTRATIONS

	Solution A Concentrations (mg/L)	**Solution B** Concentrations (mg/L)
Al	20.0	20.0
Ca	60.0	60.0

Fe	50.0	50.0
Mg	20.0	20.0
Na	50.0	50.0
P	20.0	20.0
K	20.0	20.0
S	20.0	20.0
C	40.0	40.0
Cl	424	424
Mo	0.05	0.05
Ti	0.40	0.40
As	0.0	0.025
Cd	0.0	0.025
Cr	0.0	0.050
Co	0.0	0.050
Cu	0.0	0.050
Mn	0.0	0.050
Ni	0.0	0.050
Se	0.0	0.025
Ag	0.0	0.0125
V	0.0	0.050
Zn	0.0	0.025

NOTE: The concentration of interfering elements in the ICSA and ICSAB solutions are spiked at levels 5 times lower than recommended in Table 1 of Method 6020A. Running the full strength solutions as described in 6020A introduces too much material approximately 0.35 % dissolved solids into the ICP-MS system when trying to conduct low level analysis. Since the ICP-MS instrumentation is able to handle a maximum of 0.2% solids, the 6020A ICSA solution is higher in interfering components than any sample that would run through the instrument. However, the ICS solutions will be analyzed at levels that will provide approximately 0.1% dissolved solids.

12.8 Internal standards are used to correct for physical interferences. Masses used as internal standards include; ^{71}Ga, ^{115}In, ^{6}Li, ^{175}Lu, ^{103}Rh ^{45}Sc, and ^{89}Y. These internal standards are used in combination to cover the appropriate mass ranges. Internal standard correction is applied to the analytical isotopes via interpolation of the responses from nearest internal standard isotopes (Thermo instruments) or direct correlation of analyte to IS (NexION). This function is performed in real-time by the instruments operating system. Internal standards must be run within 50 AMU of the masses that are analyzed. Internal standard recoveries must fall between 30% and 125% when running method 6020, or 70% to 125% when running method 6020A Revision 1. If not, then the sample must be reanalyzed after a fivefold or greater dilution has been performed.

12.9 A method blank is digested and analyzed with every batch of 20 (or fewer) samples to demonstrate that there are no method interferences. If the method blank shows any hits above the MRL for standard applications, or >½ the MRL for DoD projects or > 1/10 the sample result, corrective action must be taken. The MB can only be rerun once. Corrective action includes recalculation, reanalysis, system cleaning, or re-extraction and reanalysis.

12.10 Laboratory Control Samples are analyzed at a frequency of 5% or one per batch, whichever is greater. See the Attachments for a listing of control limits. For method 6020A, the LCS recovery limits are 80-120%. If statistical in-house limits are used, they must fall within the 80-120% range. Project, QAPP, or client-specific control limits may supersede the limits listed, but laboratory limits should be consistent with specified limits in order to establish that the specified limits can be achieved. If the control limits are exceeded, the associated batch of samples will be re-digested and reanalyzed.

12.11 A digested duplicate and matrix spike are analyzed at a frequency of 5% or one per batch, whichever is greater. The matrix spike recovery and relative percent difference will be calculated while analysis is in progress. See the Attachments for a listing of control limits. Project, QAPP, or client-specific control limits may supersede the limits listed. If the control limits are exceeded, the samples will be re-digested and reanalyzed, unless matrix interference or sample non-homogeneity is established as cause. In these instances, the data and the report will be flagged accordingly.

12.12 A Matrix Spike sample is digested one per batch, or per 20 samples (i.e. 5%). Default spike concentrations are listed in the sample digestion SOPs. Spike concentrations may be adjusted to meet project requirements. The matrix spike recovery will be calculated while the job is in progress. Where specified by project requirements, a matrix spike duplicate may be required. Matrix spike recovery criteria are derived from lab data and are listed in Table 2. For method 6020A, the recovery limits are 75-125%. If statistical in-house limits are used, they must fall within the 75-125% range. In some cases, project-specific QC limits may be required. Unless specified otherwise, for DoD QSM projects the project LCS criteria will be used for evaluation of matrix spikes. If an analyte recovery is outside acceptance limits proceed with the additional quality control tests described in sections 12.13 and 12.14. Based on results of these tests, the physical nature of the sample (e.g. homogeneity), and any specific project requirements, a determination can then be made as to appropriate corrective action (e.g. re-digestion, reporting with a qualifier, alternative methodologies, etc.). If the analyte concentration is >4x the spike level the spike control limit is no longer applicable and no action is required. For specifics on the preparation and composition of matrix spike solutions refer to the appropriate digestion SOP.

Note: For DOD projects a MS/MSD is required with every extraction batch. The %RSD should be < 20%.

12.13 Post Digestion Spike Test: When analysis is conducted via 6020 a post digestion spike must be performed for each matrix and each batch of sample. The prepared sample or its dilution is spiked for each element of interest at a concentration sufficiently high to be observed. Typically 20 µL of 10,000 ppb intermediate stock is added to a 10 mL aliquot of sample. If analyte concentrations are elevated in the sample, spiking at a higher concentration may be required. The post spike should be recovered to within 75-125% of the known value or within the laboratory derived acceptance criteria. When analysis is conducted via 6020A, the post digestion spike test is performed whenever matrix spike or replicate criteria are exceeded. An analyte spike is added to a portion of a prepared sample, or its dilution, and should be recovered to within 80% to 120% of the known value. If this spike fails, then the dilution test (Sec. 12.14) should be run on this sample. If both the matrix spike and the post digestion spike fail, then matrix effects are confirmed.

12.14 Dilution Test: When analysis is conducted via 6020, a serial dilution test must be performed for each matrix and each batch of sample. For sample concentrations that are sufficiently

high (minimally, a factor of greater than 100 times the MDL), the analysis of a fivefold (1+4) dilution must agree within ± 10% of the original determination. When analysis is conducted via 6020A, the dilution test is performed whenever matrix spike or replicate criteria and post digestion spike criteria are exceeded. If the dilution test fails then a chemical or physical effect should be suspected. Corrective action can include additional dilution of the sample, the use of alternate methodologies, etc. or the data can be flagged and reported. The exact course of action will be dependent on the nature of the samples and project requirements and should be discussed with the project manager.

12.15 Instrument blanks should be evaluated for potential carryover and rinse times need to bring the analyte signal to within the CCB criteria discussed above in section 12.6.2. Results from instrument blanks run after standards or control samples should be used to establish levels at which carryover in samples may occur. Samples exhibiting similar effects of carryover should be reanalyzed.

12.16 Refer to the Quality Control section of EPA Methods 6020 and 6020A for additional information describing required QA/QC. Note that the nomenclature of certain QC samples in the method differs from that of the CLP SOW, but the function of those samples is equivalent in both cases.

13 DATA REDUCTION, REPORTING, AND REVIEW

13.1 Calculations

Calculate sample results using the data system printouts and digestion information. the digestion and dilution information is entered into the data system. The data system then uses the calculations below to generate a sample result.

Aqueous samples are reported in µg/L:

$$\mu g / L\ (Sample) = C^{*} \text{ x Digestion Dilution Factor x Post Digestion Dilution Factor}$$

C*= Concentration of analyte as measured at the instrument in ug/L (in digestate).

Solid samples are reported in mg/Kg:

$$mg/Kg\ (Sample) = C^{*} \text{ x Post Digestion Dilution Factor } x \frac{Digestion\ Vol.(ml)}{Sample\ wt.(g)} x \frac{1mg}{1000ug} x \frac{1L}{1000ml} x \frac{1000g}{1Kg}$$

C*= Concentration of analyte as measured at the instrument in ug/L (in digestate).

NOTE: If results are to be reported on a dry weight basis, determine the dry weight of a separate aliquot of the sample, using the SOP for Total Solids.

13.2 Common isobaric interferences are corrected using equations equivalent to those listed in EPA Methods 6020, 6020A, and 200.8. Monitoring of multiple isotopes for a single element provides a mechanism for identifying isobaric interferences. Refer to the Interferences section of EPA methods for additional descriptions of possible interferences and the mechanisms required for adequately compensating for their effects.

13.3 Data Review and Reporting

13.3.1 The ICP-MS operator reviews the MS data and signs and dates the Data Review Form. A qualified senior staff spectroscopist performs a secondary review of the data and the Data Review Form is signed and dated. The data is then delivered to the report generation area where it is filed in the service request file. Once all of the data for the service request is complete, a CAR is generated.

13.3.2 The data is saved on the local hard drive and is also copied to the appropriate directory on the network. The data directories are located at r:\icp\wip\data. The data is kept on the local directory for 1 month. The network files are periodically backed up on disc or network tape.

13.3.3 For "non-production" work (such as method development or research/development studies) the analyses are performed under the direction of a senior spectroscopist. All associated data is scrutinized by the senior spectroscopist. Original raw data and associated records are archived in the analytical project file.

13.3.4 The final review and approval of all data is performed by qualified spectroscopists.

14 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

14.1 Refer to the SOP for *Non Conformance and Corrective Action (CE-QA008)* for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2 Handling out-of-control or unacceptable data

14.2.1 On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, runlogs, for example.

14.2.2 Some examples when documentation of a nonconformity is required using a Nonconformity and Corrective Action Report (NCAR):

- Quality control results outside acceptance limits for accuracy and precision
- Method blanks or continuing calibration blanks (CCBs) with target analytes above acceptable levels
- Sample holding time missed due to laboratory error or operations
- Deviations from SOPs or project requirements
- Laboratory analysis errors impacting sample or QC results
- Miscellaneous laboratory errors (spilled sample, incorrect spiking, etc)
- Sample preservation or handling discrepancies due to laboratory or operations error

15 METHOD PERFORMANCE

15.1 This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional available method performance data.

The method detection limit (MDL), limit of detection (LOD) and limit of quantitation (LOQ) are established using procedures described in CE-QA011, *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification.* Method Reporting Limits are established for this method based on MDL studies and as specified in the ALS, Kelso Quality Assurance Manual.

16 TRAINING

16.1 A minimum of two senior level spectroscopists are to be maintained on staff at all times. Senior spectroscopists are defined as individuals with a minimum of ten years combined education and experience in, or related to atomic spectroscopy. Of those ten years, a minimum of two years of ICP-MS experience is required.

16.2 All technical staff is encouraged to attend one technical seminar per year. In addition to the technical seminars, senior spectroscopists are required to complete a one week training session offered by the instrument manufacturer.

16.3 Training outline

16.3.1 Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

16.3.2 The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

16.3.3 Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

16.4 Training and proficiency is documented in accordance with the SOP ADM-TRANDOC.

17 POLLUTION PREVENTION AND WASTE MANAGEMENT

17.1 It is the laboratory's practice to minimize the amount of solvents, acids, and reagents used to perform this method wherever feasibly possible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvents and/or reagents used in this method can be minimized when recycled or disposed of properly.

17.2 The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Environmental Health and Safety Manual.

18 METHOD MODIFICATIONS

18.1 There are no known modifications in this laboratory standard operating procedure from the reference method.

19 CHANGES SINCE THE LAST REVISION

19.1 Reformatted to ALS branding.
19.2 Replaced "CAS" references with "ALS".
19.3 Updated SOP references.
19.4 Sec. 1.1: Removed reference to annual studies, replaced with "new".
19.5 Sec. 7.1: Added NexION 300D.
19.6 Sec 8.2.7.1: Removed Ba and Tl from the intermediate solution; added Ce.
19.7 Sec 8.2.7.2: Added NexION working solution prep.
19.8 Sec. 8.3.2.1: Revised ICV int. stock sol. prep instructions.
19.9 Sec. 8.2.7.1/2: Updated solution prep instructions.
19.10 Sec. 12.8: Added description of NexION IS correction.
19.11 Sec. 18: New.

20 REFERENCES

20.1 USEPA, Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Update III Method 6020, Revision 0, September 1994.

20.2 USEPA, Test Methods for Evaluating Solid Waste, SW-846, Update IV, Method 6020A, Revision 1, February 2007.

20.3 VG and Thermo Elemental Instrument Manuals

TABLE 1
Method Reporting Limits and Method Detection Limits – Water Matrix

	Water (ug/L)			Water (ug/L)			Seawater (ug/L)		
	CLP Digestion			3020 Digestion			Reductive Precipitation		
Analyte	MRL (DoD)	**MRL**	**MDL**	MRL (DoD)	**MRL**	**MDL**	MRL (DoD)	**MRL**	**MDL**
Aluminum	2	**2**	**0.3**	2.4	**2**	**0.8**	–	**–**	**–**
Antimony	0.09	**0.05**	**0.03**	0.09	**0.05**	**0.03**	–	**–**	**–**
Arsenic	0.5	**0.5**	**0.08**	0.5	**0.5**	**0.07**	0.5	**0.5**	**0.02**
Barium	0.06	**0.05**	**0.02**	0.12	**0.05**	**0.04**			
Beryllium	0.02	**0.02**	**0.008**	0.02	**0.02**	**0.006**	0.02	**0.02**	**0.0007**
Bismuth	0.1	**0.1**	**0.02**	–	**–**	**–**	–	**–**	**–**
Boron	0.9	**0.5**	**0.3**	–	**–**	**–**	–	**–**	**–**
Cadmium	0.02	**0.02**	**0.008**	0.06	**0.02**	**0.02**	0.02	**0.02**	**0.006**
Chromium	0.2	**0.2**	**0.07**	0.2	**0.2**	**0.05**	0.2	**0.2**	**0.03**
Cobalt	0.02	**0.02**	**0.005**	0.02	**0.02**	**0.005**	0.02	**0.02**	**0.001**
Copper	0.1	**0.1**	**0.02**	0.21	**0.1**	**0.07**	0.1	**0.1**	**0.03**
Lead	0.03	**0.02**	**0.009**	0.06	**0.05**	**0.02**	0.02	**0.02**	**0.003**
Manganese	0.06	**0.05**	**0.02**	0.05	**0.05**	**0.01**	–	**–**	**–**
Molybdenum	0.09	**0.05**	**0.03**	0.09	**0.05**	**0.03**	–	**–**	**–**
Nickel	0.2	**0.2**	**0.07**	0.2	**0.2**	**0.05**	0.2	**0.2**	**0.03**
Selenium	1.2	**1**	**0.4**	1	**1**	**0.2**	–	**–**	**–**
Silver	0.03	**0.02**	**0.009**	0.03	**0.02**	**0.009**	0.02	**0.02**	**0.004**
Thallium	0.02	**0.02**	**0.003**	0.02	**0.02**	**0.004**	0.02	**0.02**	**0.0009**
Tin	0.1	**0.1**	**0.04**	–	**–**	**–**	–	**–**	**–**
Uranium	0.02	**0.02**	**0.005**	0.02	**0.02**	**0.004**	–	**–**	**–**
Vanadium	0.2	**0.2**	**0.08**	0.2	**0.2**	**0.05**	–	**–**	**–**
Zinc	0.5	**0.5**	**0.1**	0.6	**0.5**	**0.2**	0.5	**0.5**	**0.04**

TABLE 1 (continued)
Method Reporting Limits and Method Detection Limits – Solid Matrix

	Soil/Sediment (mg/kg)			Tissue (mg/kg, dry basis)		
	3050 Digestion			PSEP		
Analyte	MRL (DoD)	**MRL**	**MDL**	MRL (DoD)	**MRL**	**MDL**
Aluminum	2	**2**	**0.5**	2	**2**	**0.4**
Antimony	0.09	**0.05**	**0.03**	0.06	**0.05**	**0.02**
Arsenic	0.5	**0.5**	**0.1**	0.5	**0.5**	**0.05**
Barium	0.09	**0.05**	**0.03**	0.15	**0.05**	**0.05**
Beryllium	0.06	**0.02**	**0.02**	0.02	**0.02**	**0.005**
Bismuth	0.1	**0.1**	**0.02**	–	**–**	**–**
Cadmium	0.02	**0.02**	**0.008**	0.03	**0.02**	**0.01**
Chromium	0.2	**0.2**	**0.04**	–	**–**	**–**
Cobalt	0.02	**0.02**	**0.003**	0.02	**0.02**	**0.006**
Copper	0.3	**0.1**	**0.1**	0.1	**0.1**	**0.03**
Lead	0.06	**0.05**	**0.02**	0.02	**0.02**	**0.006**
Manganese	0.12	**0.05**	**0.04**	0.06	**0.05**	**0.02**
Molybdenum	0.15	**0.05**	**0.05**	0.06	**0.05**	**0.02**
Nickel	0.2	**0.2**	**0.05**	0.2	**0.2**	**0.03**
Selenium	2	**1**	**0.4**	–	**–**	**–**
Silver	0.06	**0.02**	**0.02**	0.02	**0.02**	**0.006**
Thallium	0.02	**0.02**	**0.003**	0.02	**0.02**	**0.005**
Tin	0.2	**0.1**	**0.06**	–	**–**	**–**
Uranium	0.02	**0.02**	**0.004**	0.02	**0.02**	**0.007**
Vanadium	0.2	**0.2**	**0.04**	0.2	**0.2**	**0.04**
Zinc	0.6	**0.5**	**0.2**	1.2	**0.5**	**0.4**

ATTACHMENTS

List of Target Element Masses

Example Standard Sheet

QC Acceptance Criteria

Analyte	ISOTOPES ANALYZED	ISOTOPE REPORTED
Aluminum	27	27
Antimony	121,123	123
Arsenic	75	75
Barium	135,137,138	137
Beryllium	9	9
Cadmium	111,112,114	111
Chromium	52,53	52
Cobalt	59	59
Copper	63,65	65
Lead	206,207,208	208
Manganese	55	55
Molybdenum	95,97,98	98
Nickel	60,61,62	60
Selenium	77,78,82	82
Silver	107,109	107
Thallium	203,205	205
Uranium	238	238
Vanadium	51	51
Zinc	66,67,68	66

SOLUTION: ICP-MS, 200.8 INTERMEDIATE STOCK
MATRIX: 5% HNO3
VOLUME: 1000 ml.

	ELEMENT			ALIQUOT OF 1000 ppm Std./1000ml		CONCENTRATION (µg/L)
	HNO3			50.0 ml.		5%
	Al			1.0 ml.		1000
	Sb			1.0 ml.		1000
	As			1.0 ml.		1000
	Ba			1.0 ml.		1000
	Be			1.0 ml.		1000
	Cd			1.0 ml.		1000
	Cr			1.0 ml.		1000
	Co			1.0 ml.		1000
	Cu			1.0 ml.		1000
	Pb			1.0 ml.		1000
	Mn			1.0 ml.		1000
	Mo			1.0 ml.		1000
	Ni			1.0 ml.		1000
	Se			1.0 ml.		1000
	Tl			1.0 ml.		1000
	V			1.0 ml.		1000
	U			1.0 ml.		1000
	Zn			1.0 ml.		1000

SOLUTION: ICP-MS, 200.8 SILVER INTERMEDIATE STOCK
MATRIX: 5% HNO3
VOLUME: 1000 ml.

	ELEMENT			ALIQUOT OF 1000 ppm Std./1000ml	CHECK OFF	CONCENTRATION (µg/L)
	HNO3			50.0		5%
	Ag			1.0		1000

SOLUTION: ICP-MS 25ppb Calibration Standard and CCV
MATRIX: 1% HNO3
VOLUME: 100 ml.

SOURCE	ALIQUOT PER 100 ml.		CONCENTRATION (µg/L)
HNO3 (Ultrex)	1.0		1%
INTERMEDIATE STOCK	2.5		25.0
SILVER INTERMEDIATE STOCK	2.5		25.0

SOLUTION: ICP-MS, 200.8 INTERMEDIATE STOCK
MATRIX: 5% HNO3
VOLUME: 1000 ml.

ELEMENT			ALIQUOT OF 1000 ppm Std./1000ml		CONCENTRATION (µg/L)
HNO3			50.0 ml.		5%
Al			1.0 ml.		1000
Sb			1.0 ml.		1000
As			1.0 ml.		1000
Ba			1.0 ml.		1000
Be			1.0 ml.		1000
Cd			1.0 ml.		1000
Cr			1.0 ml.		1000
Co			1.0 ml.		1000
Cu			1.0 ml.		1000
Pb			1.0 ml.		1000
Mn			1.0 ml.		1000
Mo			1.0 ml.		1000
Ni			1.0 ml.		1000
Se			1.0 ml.		1000
Tl			1.0 ml.		1000
V			1.0 ml.		1000
U			1.0 ml.		1000
Zn			1.0 ml.		1000

SOLUTION: ICP-MS, 200.8 SILVER INTERMEDIATE STOCK
MATRIX: 5% HNO3
VOLUME: 1000 ml.

ELEMENT			ALIQUOT OF 1000 ppm Std./1000ml	CHECK OFF	CONCENTRATION (µg/L)
HNO3			50.0		5%
Ag			1.0		1000

SOLUTION: ICP-MS 25ppb Calibration Standard and CCV
MATRIX: 1% HNO3
VOLUME: 100 ml.

SOURCE	ALIQUOT PER 100 ml.		CONCENTRATION (µg/L)
HNO3 (Ultrex)	1.0		1%
INTERMEDIATE STOCK	2.5		25.0
SILVER INTERMEDIATE STOCK	2.5		25.0

SOLUTION: ICP-MS, 200.8 INTERMEDIATE STOCK
MATRIX: 5% HNO3
VOLUME: 1000 ml.

ELEMENT			ALIQUOT OF 1000 ppm Std./1000ml		CONCENTRATION (µg/L)
HNO3			50.0 ml.		5%
Al			1.0 ml.		1000
Sb			1.0 ml.		1000
As			1.0 ml.		1000
Ba			1.0 ml.		1000
Be			1.0 ml.		1000
Cd			1.0 ml.		1000
Cr			1.0 ml.		1000
Co			1.0 ml.		1000
Cu			1.0 ml.		1000
Pb			1.0 ml.		1000
Mn			1.0 ml.		1000
Mo			1.0 ml.		1000
Ni			1.0 ml.		1000
Se			1.0 ml.		1000
Tl			1.0 ml.		1000
V			1.0 ml.		1000
U			1.0 ml.		1000
Zn			1.0 ml.		1000

SOLUTION: ICP-MS, 200.8 SILVER INTERMEDIATE STOCK
MATRIX: 5% HNO3
VOLUME: 1000 ml.

ELEMENT			ALIQUOT OF 1000 ppm Std./1000ml	CHECK OFF	CONCENTRATION (µg/L)
HNO3			50.0		5%
Ag			1.0		1000

SOLUTION: ICP-MS 25ppb Calibration Standard and CCV
MATRIX: 1% HNO3
VOLUME: 100 ml.

SOURCE	ALIQUOT PER 100 ml.		CONCENTRATION (µg/L)
HNO3 (Ultrex)	1.0		1%
INTERMEDIATE STOCK	2.5		25.0
SILVER INTERMEDIATE STOCK	2.5		25.0

SOLUTION: ICP-MS, 200.8 INTERMEDIATE STOCK
MATRIX: 5% HNO3
VOLUME: 1000 ml.

ELEMENT			ALIQUOT OF 1000 ppm Std./1000ml		CONCENTRATION (µg/L)
HNO3			50.0 ml.		5%
Al			1.0 ml.		1000
Sb			1.0 ml.		1000
As			1.0 ml.		1000
Ba			1.0 ml.		1000
Be			1.0 ml.		1000
Cd			1.0 ml.		1000
Cr			1.0 ml.		1000
Co			1.0 ml.		1000
Cu			1.0 ml.		1000
Pb			1.0 ml.		1000
Mn			1.0 ml.		1000
Mo			1.0 ml.		1000
Ni			1.0 ml.		1000
Se			1.0 ml.		1000
Tl			1.0 ml.		1000
V			1.0 ml.		1000
U			1.0 ml.		1000
Zn			1.0 ml.		1000

SOLUTION: ICP-MS, 200.8 SILVER INTERMEDIATE STOCK
MATRIX: 5% HNO3
VOLUME: 1000 ml.

ELEMENT			ALIQUOT OF 1000 ppm Std./1000ml	CHECK OFF	CONCENTRATION (µg/L)
HNO3			50.0		5%
Ag			1.0		1000

SOLUTION: ICP-MS 25ppb Calibration Standard and CCV
MATRIX: 1% HNO3
VOLUME: 100 ml.

SOURCE	ALIQUOT PER 100 ml.		CONCENTRATION (µg/L)
HNO3 (Ultrex)	1.0		1%
INTERMEDIATE STOCK	2.5		25.0
SILVER INTERMEDIATE STOCK	2.5		25.0

METALS ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**LCS Accuracy (% Rec.)**	**Matrix Spike (% Rec.)**	**Precision (RPD)**
6020	3050B	Soil	Aluminum	41-158	75-125*	20
6020	3050B	Soil	Antimony	50-150	10-103	20
6020	3050B	Soil	Arsenic	78-122	57-133	20
6020	3050B	Soil	Barium	81-119	54-173	20
6020	3050B	Soil	Beryllium	83-117	64-133	20
6020	3050B	Soil	Boron	67-133	75-125*	20
6020	3050B	Soil	Cadmium	81-119	68-137	20
6020	3050B	Soil	Chromium	80-119	34-175	20
6020	3050B	Soil	Cobalt	82-118	74-118	20
6020	3050B	Soil	Copper	83-116	22-181	20
6020	3050B	Soil	Lead	79-121	27-178	20
6020	3050B	Soil	Manganese	81-119	75-125*	20
6020	3050B	Soil	Molybdenum	75-125	53-143	20
6020	3050B	Soil	Nickel	81-118	59-132	20
6020	3050B	Soil	Selenium	80-120	65-125	20
6020	3050B	Soil	Silver	66-134	62-131	20
6020	3050B	Soil	Thallium	79-120	70-128	20
6020	3050B	Soil	Uranium	80-120*	75-125*	20
6020	3050B	Soil	Vanadium	79-121	59-142	20
6020	3050B	Soil	Zinc	73-121	37-162	20
6020	CLP/3020A	Water	Aluminum	85-120	56-143	20
6020	CLP/3020A	Water	Antimony	91-112	66-133	20
6020	CLP/3020A	Water	Arsenic	89-112	72-129	20
6020	CLP/3020A	Water	Barium	92-111	86-117	20
6020	CLP/3020A	Water	Beryllium	81-122	73-125	20
6020	CLP/3020A	Water	Cadmium	92-111	87-113	20
6020	CLP/3020A	Water	Chromium	88-113	60-136	20
6020	CLP/3020A	Water	Cobalt	87-114	84-115	20
6020	CLP/3020A	Water	Copper	89-113	62-130	20
6020	CLP/3020A	Water	Lead	90-112	76-117	20
6020	CLP/3020A	Water	Manganese	89-115	25-180	20
6020	CLP/3020A	Water	Molybdenum	66-135	67-138	20
6020	CLP/3020A	Water	Nickel	89-113	78-117	20
6020	CLP/3020A	Water	Selenium	87-115	47-150	20
6020	CLP/3020A	Water	Silver	64-134	55-136	20
6020	CLP/3020A	Water	Thallium	78-123	75-121	20
6020	CLP/3020A	Water	Vanadium	87-113	82-119	20
6020	CLP/3020A	Water	Zinc	86-119	65-126	20

STANDARD OPERATING PROCEDURE

DOCUMENT TITLE:	***MERCURY IN LIQUID WASTE***
REFERENCED METHOD:	***EPA 7470A***
SOP ID:	***MET-7470A***
REV. NUMBER:	***15***
EFFECTIVE DATE:	***1/31/2013***

MERCURY IN LIQUID WASTE METHOD 7470A

MET-7470A

ALS-KELSO

SOP ID:	MET-7470A	Rev. Number:	15	Effective Date:	1/31/2013

Approved By: _____ Date: 12/31/12
Department Supervisor - Jeff Coronado

Approved By: _____ Date: 12/31/12
QA Manager - Suzanne LeMay

Approved By: _____ Date: 12/31/12
Laboratory Director - Jeff Grindstaff

Issue Date:	_____	Doc Control ID#:	_____	Issued To:	_____

Standard Operating Procedure

for

MERCURY IN LIQUID WASTE – 7470A

1. SCOPE AND APPLICATION

1.1. This procedure is used to determine the concentrations of Mercury in aqueous samples, including mobility-procedure extractions, aqueous wastes, and ground water, using EPA Method 7470A. Method 7470A is a cold-vapor atomic absorption procedure.

1.2. The Method Reporting Limit (MRL) is 0.2ug/L. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. The reported MRL may be adjusted if required for specific project requirements; however, the capability of achieving other reported MRLs must be demonstrated. The current MDL for 7470A is 0.02µg/L. The MDL may change slightly as annual studies are performed

2. METHOD SUMMARY

1.3. A representative aliquot of sample is prepared as described in this procedure. The mercury is reduced to its elemental state and aerated from solution and measured with an atomic absorption spectrometer. The mercury vapor passes through a cell positioned in the light path of the AA where absorbance is measured as a function of mercury concentration.

3. DEFINITIONS

3.1. **Batch** - A batch of samples is a group of environmental samples that are prepared and/or analyzed together as a unit with the same process and personnel using the same lot(s) of reagents. It is the basic unit for analytical quality control.

3.1.1. Preparation Batch - A preparation batch is composed of one to twenty field samples, all of the same matrix, and with a maximum time between the start of processing of the first and last samples in the batch to be 24 hours.

3.1.2. Analysis Batch - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration (initial or continuing verification) followed by sample extracts interspersed with calibration standards (CCBs, CCVs, etc.) The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria indicate an out-of-control situation.

3.2. **Sample**

3.2.1. Field Sample - An environmental sample collected and delivered to the laboratory for analysis; a.k.a., client's sample.

3.2.2. Laboratory Sample - A representative portion, aliquot, or subsample of a field sample upon which laboratory analyses are made and results generated.

3.3. **Quality System Matrix** - The *matrix* of an environmental sample is distinguished by its physical and/or chemical state and by the program for which the results are intended. The following sections describe the matrix distinctions. These matrices shall be used for purpose of batch and quality control requirements.

3.3.1. Aqueous - Any groundwater sample, surface water sample, effluent sample, and TCLP or other extract. Specifically excluded are samples of the drinking water matrix and the saline/estuarine water matrix.

3.3.2. Drinking water - Any aqueous sample that has been designated a potable or potential potable water source.

3.3.3. Saline/Estuarine water - Any aqueous sample from an ocean or estuary or other salt-water source.

3.3.4. Non-aqueous Liquid - Any organic liquid with <15% settleable solids.

3.3.5. Animal tissue - Any tissue sample of an animal, invertebrate, marine organism, or other origin; such as fish tissue/organs, shellfish, worms, or animal material.

3.3.6. Solids - Any solid sample such as soil, sediment, sludge, and other materials with >15% settleable solids.

3.3.7. Chemical waste - Any sample of a product or by-product of an industrial process that results in a matrix not described in one of the matrices in Sections 3.3.1 through 3.3.6. These can be such matrices as non-aqueous liquids, solvents, oil, etc.

3.3.8. Miscellaneous matrices – Samples of any composition not listed in 3.3.1 – 3.3.7. These can be such matrices as plant material, paper/paperboard, wood, auto fluff, mechanical parts, filters, wipes, etc. Such samples shall be batched/grouped according to their specific matrix.

3.4. Matrix Spike/Duplicate Matrix Spike (MS/DMS) Analysis - In the matrix spike analysis, predetermined quantities of target analytes are added to a sample matrix prior to sample preparation and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the method used for the analysis. Duplicate samples are spiked, and analyzed as a MS/DMS pair. Percent recoveries are calculated for each of the analytes detected. The relative percent difference (RPD) between the duplicate spikes (or samples) is calculated and used to assess analytical precision. The concentration of the spike should be at the mid point of the calibration range or at levels specified by a project analysis plan.

3.5. Laboratory Duplicates (DUP) – Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. The relative percent difference (RPD) between the sample and its duplicate is calculated and used to assess analytical precision.

3.6. Surrogate - Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples and

spiked samples prior to extraction and analysis. Percent recoveries are calculated for each surrogate.

3.7. Method Blank (MB) - The method blank is an artificial sample composed of analyte-free water or solid matrix and is designed to monitor the introduction of artifacts into the analytical process. The method blank is carried through the entire analytical procedure.

3.8. Laboratory Control Samples (LCS) – The LCS is an aliquot of analyte free water or analyte free solid to which known amounts target analytes are added. The LCS is prepared and analyzed in exactly the same manner as the samples. The percent recovery is compared to established limits and assists in determining whether the batch is in control.

3.9. Independent Verification Standard (ICV) - A mid-level standard injected into the instrument after the calibration curve and prepared from a different source than the initial calibration standards. This is used to verify the validity of the initial calibration standards

3.10. Continuing Calibration Verification Standard (CCV) - A mid-level standard analyzed at specified intervals. Used to verify that the initial calibration curve is still valid for quantitative purposes.

3.11. Instrument Blank (CCB) - The instrument blank (also called continuing calibration blank) is a volume of clean solvent analyzed on each column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrumental analysis itself, particularly with regard to the carry-over of analytes from standards or highly contaminated samples into subsequent sample analyses.

3.12. Duplicates and Duplicate Matrix Spikes are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. Depending on the method of analysis, either a duplicate analysis (and/or a matrix spiked sample) or a matrix spiked sample and duplicate matrix spiked sample (MS/DMS) are analyzed.

3.13. Standard Reference Material (SRM) – A material with specific certification criteria and is issued with a certificate or certificate of analysis that reports the results of its characterizations and provides information regarding the appropriate use(s) of the material. An SRM is prepared and used for three main purposes: (1) to help develop accurate methods of analysis; (2) to calibrate measurement systems used to facilitate exchange of goods, institute quality control, determine performance characteristics, or measure a property at the state-of-the-art limit; and (3) to ensure the long-term adequacy and integrity of measurement quality assurance programs.

4. INTERFERENCES

4.1. Potassium permanganate is added to eliminate possible interference from sulfide. Samples high in chlorides require additional permanganate because, during the oxidation step, chlorides are converted to free chlorine, which absorbs radiation at 253 nm.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in MSDSs where available. Refer to the ALS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. Hydrochloric and/or Nitric Acid are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

6. SAMPLE PRESERVATION AND STORAGE

6.1. Aqueous samples are preserved with nitric acid (pH<2), then refrigerated at 4 ± 2ºC from receipt until analysis.

6.2. The maximum holding for mercury in aqueous samples is 28 days.

7. APPARATUS AND EQUIPMENT

7.1. CETAC M-6000A Mercury Analyzer. (See Attachments for instrument parameters).

7.2. Evergreen polypropylene tubes with disposable caps, 50 mL

7.3. Modified block digesters, 50 and 100 ml.

7.4. Pipettors, Eppendorf and Finnpipette fixed and adjustable volume

7.5. Polypropylene graduated cylinders, 25 mL

7.6. 125 mL CENTRIFUGE TUBE

8. STANDARDS AND REAGENTS

8.1. Mercury stock solution (1,000mg/L). Commercially prepared certified solution stored at room temperature. The expiration date determined by manufacturer.

8.2. Mercury intermediate stock solution (10mg/L). Prepared from the stock solution listed above. Store at room temperature and assign a one month expiration date.

8.3. Mercury working standard (100µg/L). Prepared from the intermediate stock solution listed above. Store at room temperature and prepare a new standard daily.

8.4. See section 11.2.2 for details on preparation of calibration and ICV standards. See section 12 for QC sample preparation.

8.5. Reagent water - ASTM Type II water (laboratory deionized water).

8.6. Concentrated nitric and sulfuric acids. Purity of acids must be established by the laboratory as being high enough to eliminate the introduction of contamination above the Method Reporting Limit.

8.7. Potassium permanganate solution, 5% w/v. To prepare, add 50g of solid reagent to 1000mL of D.I. water and place on magnetic stir plate for a approximately 30 minutes until dissolved.

8.8. Potassium persulfate solution, 5% w/v. To prepare, add 50g of solid reagent to 1000mL of D.I. water and warm in water bath for approximately 10 minutes. Place the warmed solution on a magnetic stir plate for approximately 10 minutes until dissolved.

8.9. Sodium chloride/hydroxylamine hydrochloride solution, 12% w/v each. To prepare, add 120g sodium chloride and 120g of hydroxylamine hydrochloride to 1000mL of D.I. water and place on magnetic stir plate for a approximately 15 minutes until dissolved.

8.10. Stannous chloride, 10% w/v in HCl (7% v/v). To prepare, add 100g stannous chloride crystals and 70mL of concentrated hydrochloric acid in 1000mL of D.I. water. Seal lid on mixing bottle and shake until the stannous chloride is dissolved.

9. PREVENTIVE MAINTENANCE

9.1. ALS staff performs all routine maintenance and troubleshooting. Preventative maintenance activities listed below should be performed when needed as determined by instrument performance (i.e. stability, sensitivity, etc.) or by visual inspection. Repairs of an extraordinary nature may or may not require factory service, depending on the nature of the task. All maintenance activities are recorded in a maintenance logbook kept for each instrument.

9.2. Keep the instrument free of dust, deposits, and chemical spills.

9.3. Replace the peristaltic and autosampler rinse tubing.

9.4. Remove and clean the Gas-Liquid Separator.

9.5. Remove, dismantle, and clean the optical cells (sample cell and reference cell) including the sapphire windows.

9.6. Replace the Hg lamp bulb when the lamp current reaches 13 mA.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in 7470A, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Sample Preparation

11.1.1. Shake the sample and measure 20 mL into a 50 mL centrifuge tube. Add 1 mL of H2SO4 and 0.5 mL of concentrated HNO3, mixing after each addition. Add 3 mL of potassium permanganate solution to each tube and shake. If the purple color does not persist for 15 minutes add additional potassium permanganate until it does so. Any additional potassium permanganate solution must also be added to the blanks and standards in equal proportion. Note: Spiking solution is added prior to acidification.

11.1.2. Add 1.6 ml of potassium persulfate to each bottle and heat for 2 hr in a modified block maintained at 95ºC. The temperature of each block is monitored with a calibrated thermometer. Cool and add 1.2 ml of sodium chloride-hydroxylamine hydrochloride solution.

11.1.3. The samples are now ready to be analyzed. The analyzer does the final step of adding the stannous chloride solution automatically.

11.2. Calibration

11.2.1. To prepare calibration standards a 10 ppm intermediate stock solution is first prepared by aliquoting 1.0 mL of commercially prepared 1000 ppm stock standard into an acid rinsed 100 mL Class A volumetric flask and diluting to volume with 1% HNO3 (This solution must be prepared monthly). Next, a 100 ppb working solution is prepared by aliquoting 1.0 mL of the 10 ppm intermediate stock solution into an acid rinsed 100 mL Class A volumetric flask and diluting to volume with 1% HNO3. This solution must be prepared daily.

Note: All standard aliquots are measured using calibrated fixed or adjustable volume autopipettors or calibrated disposable 2.5 or 5 mL pipettes.

11.2.2. Transfer 0, 0.1, 0.25, 0.5, 2.5, and 5.0 ml aliquots of the working solution to a series of labeled 125 mL centrifuge tubes. Add the appropriate amount of reagent water to bring each bottle to a final volume of 50 ml. The final concentrations of the prepared standards are 0, 0.2, 0.5, 1.0, 5.0, 10.0 ppb. Standards may be prepared at 100 mL final volume rather than 50 mL volume to ensure sufficient quantity is available for recalibrations as well as aliquoting for CCV, CCB and CRA samples in the analytical run. The proportions of reagents and their sources are identical to those of the samples.

11.2.3. The Initial Calibration Verification (ICV) is prepared by first making a 1000 ppb intermediate solution. 0.10 mL of commercially prepared 1000 ppm stock standard, from a different manufacturer and lot than the calibration standard, is aliquoted into an acid rinsed 100 mL Class A volumetric flask and diluting to volume with 1% HNO3. This solution must be prepared monthly. Prepare the ICV standard by aliquoting 0.25 mL to a labeled 125 mL centrifuge tube. Add 49.75 mL of reagent water to bring the final volume to 50 mL.

11.2.4. Mix thoroughly and add 2.5 ml of concentrated H_2SO_4 and 1.25 ml of concentrated HNO_3 to each bottle. Add 7.5 ml of potassium permanganate to each bottle and let stand for 15 minutes. Add 4 ml of potassium persulfate to each bottle and heat in the modified block for 2 hours at 95ºC. Cool and add 3 ml of sodium chloride-

hydroxylamine hydrochloride solution.

11.2.5. CETAC Calibration and Sample Analysis

11.2.5.1. Turn on the CETAC instrument, including the Hg lamp, and autosampler. After this is done turn open the operating software (Mercury Analyzer 1.5.1.1).

11.2.5.2. The rinse station for the autosampler turns on automatically, but the peristaltic pump must be started manually. Make sure all sample uptake and drain tubes are placed correctly on the pump and are secured with the appropriate tension. Place the reagent uptake tube in the stannous chloride and start the pump.

11.2.5.3. From the software's main screen select the "Worksheet" button and then the "Template" button. Select the "Kelso Mercury Program".

Note: The CETAC software will not allow "0" to be entered as a true value for a "Standard". The software template includes the "0" standard as the "calibration blank".

11.2.5.4. Go to the "Labels" tab and enter the QC and field samples to be analyzed in the appropriate order.

11.2.5.5. Transfer the solutions to be analyzed to labeled 12mL polyethylene test tubes and place them in the appropriate spaces on the autosampler trays.

11.2.5.6. Transfer the calibration blank and standards (0.2, 0.5, 1.0, 5.0, and 10 ppb) from their Centrifuge tubes to the standard tubes located behind the autosampler trays. The calibration blank is placed in the left most tube and the other standards are placed in ascending order to the right.

11.2.5.7. Return to the software and go to the "Analysis" tab. At this point the analysis is ready to begin. Click on the start button. In the dialog box that appears be sure the following are checked:

- Calibrate before first sample.
- New output file before first sample.
- Zero before first sample.

Click start and the analysis will begin.

11.2.6. After the calibration standards have run the software will use linear regression to create a calibration curve based on the concentration and measured absorbance of each standard. The form of regression line is y = mx + b. If the correlation coefficient of the curve is greater than 0.995 the analysis will continue, if not the analysis will be terminated and corrective action will be needed by the analyst.

11.3. As the analysis sequence proceeds, next analyze the following QC standards.

- ICV (5.0 ppb standard prepared from a second source)
- ICB
- CCV (5.0 ppb calibration standard)
- CCB
- CRA (0.2 ppb calibration standard)

11.4. If either the ICV or CCV are different from their true values by more than 10% the software will terminate the analysis. If either the ICB or CCB is greater than the MRL of 0.2 µg/L the software will terminate the analysis. No control limit is applied to the CRA except under specific project requirements.

Note: For projects falling under **DoD** QSM requirements, the QSM criteria for CCV standards is ±20% and for ICB and CCB standards no analytes detected > LOD. (The ICV limit is as listed above.)

11.5. Sample Analysis

11.5.1. The samples are analyzed with the CETAC analyzer in the same manner as the calibration standards. The analyzer does the step of adding the stannous chloride solution automatically. Check the baseline between samples to verify that the spectrometer reading has stabilized at the normal baseline level.

11.5.2. The analytical sequence should be set up to include all samples, QC samples, blanks, and calibration verification standards at necessary intervals. Refer to the SOP for Sample Batches.

11.5.3. Sample digestion batches are analyzed with a set of CCV and CCB standards which are run at the beginning and end of the analytical run and at a minimum every 10 samples during the run. The same criteria listed above are applied to the CCVs and CCBs. If outside these limits the analysis is terminated.

12. QUALITY CONTROL

12.1. Method Detection Limits

12.1.1. A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank replicates with a MDL spiking solution near the MRL and analyze. Refer to the ALS *SOP for The Determination of Method Detection Limits and Limits of Detection* (ADM-MDL).

12.1.2. Calculate the average concentration found (x) in the sample concentration, and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. The MDL study should be done annually. The MDL study and MDL verification check should be analyzed annually or whenever there are major changes in the instrument or procedure is implemented.

12.2. Ongoing QC Samples required are described in the ALS-Kelso Quality Assurance Manual, in the *SOP for Sample Batches* (ADM-BATCH). For this analysis, these include:

12.2.1. Prepare one method blank (MB) per digestion batch, or per 20 samples, or per EPA SDG group, whichever is more frequent. Use D.I. water and follow the digestion procedures. The Method Blank should be < MRL. Re-digest the associated samples if sample levels are <20X MB level.

12.2.2. **DoD** QSM Method Blank Requirements– The Method Blank will be considered contaminated if:

12.2.2.1. Prepare one duplicate and matrix spike sample per each digestion batch, or per The concentration of any target analyte in the blank exceeds ½ the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).

12.2.2.2. The concentration of any common laboratory contaminant in the blank exceeds the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).

12.2.2.3. The blank result otherwise affects the samples results as per the test method requirements or the project-specific objectives.

12.2.3. Prepare one duplicate and matrix spike sample per each digestion batch, or per twenty samples, or per EPA SDG group, whichever is more frequent. At times, specific samples will be assigned as duplicates or spikes depending on client requirements. The matrix spike is prepared by aliquoting 0.2 mL of the 100 ppb working standard (section 11.2.1) to the 20 mL sample designated as the matrix spike, resulting in a spike concentration of 1ppb.

Note: Duplicate samples are routinely analyzed, however all **DoD** projects require a MSD with every preparation batch. The MSD sample is prepare as described above.

The RPD criterion for duplicates is 20% RPD. If not, flag the data or redigest samples. Apply Matrix spike recovery criterion listed in the DQO Table, unless project-specific limits are required. For **DoD** QSM work, MS recoveries are assessed using the QSM LCS control limits. If the MS (and/or MSD where applicable) recovery is outside acceptance limits proceed with the additional interference tests described in section 12.2.4. Based on results of these tests, the physical nature of the sample (e.g. homogeneity), and any specific project requirements, a determination can then be made as to appropriate corrective action (e.g. re-digestion, reporting with a qualifier, alternative methodologies, etc.). If the analyte concentration is >4x the spike level the spike control limit is no longer applicable and no action is required.

12.2.4. Prepare one Laboratory Control Sample (LCS) per digestion batch, or per 20 samples. The LCSW is prepared by aliquoting 0.1 mL of the 1000 ppb ICV intermediate solution (section 11.2.2) to 20 mL of reagent water, resulting in a concentration of 5ppb, and processing as per the procedure.

Apply LCS recovery criteria listed in the DQO Table, unless project-specific limits are required. If the LCS fails the acceptance criteria, re-digest the batch of samples. An LCS recovery criterion for **DoD** QSM projects is 80-120%.

12.2.4.1.Calculate the LCS recovery as follows:

$$\%R = X/TV \times 100$$

Where X = Concentration of the analyte recovered
TV = True value of amount spiked

12.2.4 Interference Tests: Prepare one post spike for every batch of samples and if samples are sufficiently high (10x the MRL/LOQ) a serial dilution. The serial dilution must agree within 10% of the original sample result. Post spike recovery acceptance limits for method 7470A are 85-115% under SW846 Update 3, and 80-120% for project falling under SW846 Update 4. When both the post spike and dilution tests fail all of the samples in the associated preparation batch must be quantified via Method of Standard Additions (MSA).

13. CALCULATIONS, DATA REDUCTION, AND REPORTING

13.1. Solution concentrations are calculated by the Mercury Analyzer software based on the linear regression calibration curve created when the calibration standards are analyzed. The absorbance measured for each sample is applied to the linear regression curve and the final solution concentration is determined and displayed as the instrument result.

13.2. Calculate sample results using the data system printouts and digestion information. The digestion and dilution information is entered into the data system. The data system then uses the calculations below to generate a sample result.

Aqueous samples are reported in µg/L:

$$\mu g / L\ (Sample) = C^{*} \times Digestion\ Dilution\ Factor \times Post\ Digestion\ Dilution\ Factor$$

C*= Concentration of analyte as measured at the instrument in mg/L (in digestate).

13.3. A daily run log of all samples analyzed is maintained. All CLP data should be printed and stored after operator has checked for evenness of burns. A copy of this document will go with each package of Tier III or higher data run that day.

13.4. It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified in section 12.

13.5. Record all sample volumes and dilutions on an A.A. benchsheet (see Attachments).

13.6. Record all concentrations determined at the instrument and calculate the final results in µg/L. Record the final results on the A.A. benchsheet.

13.7. The data packet for the sequence is submitted for review by supervisor or designee. The results are transferred to the appropriate report form located in the ALS network directory R:\ICP\WIP. Once the results are transferred, the report is reviewed.

13.8. Refer to the *SOP for Laboratory Data Review Process* (ADM-DREV) for general instructions for data review.

14. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

14.1. Refer to the SOP for *Corrective Action (CE-QA008)* for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2. Handling out-of-control or unacceptable data

14.2.1. On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, runlogs, for example.

14.2.2. Some examples when documentation of a nonconformity is required using a Nonconformity and Corrective Action Report (NCAR):

- Quality control results outside acceptance limits for accuracy and precision
- Method blanks or continuing calibration blanks (CCBs) with target analytes above acceptable levels
- Sample holding time missed due to laboratory error or operations
- Deviations from SOPs or project requirements
- Laboratory analysis errors impacting sample or QC results
- Miscellaneous laboratory errors (spilled sample, incorrect spiking, etc)
- Sample preservation or handling discrepancies due to laboratory or operations error

15. METHOD PERFORMANCE

15.1. This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional available method performance data.

15.2. The method detection limit (MDL), limit of detection (LOD), and limit of quantitation (LOQ) are established using the procedure described in the SOP for *The Determination of Method Detection Limits and Limits of Detection* (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the ALS Quality Assurance Manual.

16. POLLUTION PREVENTION AND WASTE MANAGEMENT

16.1. It is the laboratory's practice to minimize the amount of solvents, acids, and reagents used to perform this method wherever feasibly possible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvents and/or reagents used in this method can be minimized when recycled or disposed of properly.

16.2. The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Environmental Health and Safety Manual.

16.3. This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 5-9 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be

documented on the treatment by generator record. See the ALS EH&S Manual for details.

17. TRAINING OUTLINE

17.1. Review literature (see References section). Review the SOP. Also review safety procedures. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

17.2. The next training step is to assist in the procedure under the guidance of an experienced analyst for a period of 1-2 months. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

17.3. Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

17.4. Training is documented following the *ALS Kelso Training Procedure* (ADM-TRAIN) SOP for documentation of training.

17.5. **NOTE:** When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

18. METHOD MODIFICATIONS

18.1. There are no known modifications in this laboratory standard operating procedure from the reference method.

19. CHANGES SINCE THE LAST REVISION

19.1. Updated the Definitions section.
19.2. Reformatted the SOP to ALS stlye.
19.3. Updated references to ALS corporate SOPs.
19.4. Added Instrument parameter and benchsheet attachments
19.5. Added Table 1.

20. REFERENCES

20.1. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*. EPA SW-846, 3rd Edition, Final Update II, Method 7470A, September 1994.
20.2. *DoD Quality Systems Manual for Environmental Laboratories* Version 4.1 4/22/2009

ATTACHMENT 1
Instrument Parameters

Analysis Parameters

Instrument M-6100 Mercury Analyzer

Conditions

Gas flow (mL/min)	Sample Uptake (s)	Rinse (s)	Read delay (s)	Replicates (#)	Replicate time (s)	Pump speed (%)	Wavelength (nm)
40	30.00	60.00	50.00	4	2.00		253.65

Instrumental Zero

Zero before first sample: No

Zero periodically: Yes
Before each calibration.

Baseline Correction

#1 Start time (s)	#1 End time (s)	#2 Start time (s)	#2 End time (s)
5.00	10.00		

Standby Mode

Enabled: Yes

Standby Options: gas off, lamp off

Autodilution

Enabled: No

Condition:

Tube # range:

If no autodilution tubes remaining

Calibration

Settings

Algorithm	Through blank	Weighted fit	Cal. Type	Racalibration rate	Reslope rate	Reslope standard
Linear	Yes	No	Normal	0	0	N/A

Limits

Calibration slope		Reslope		Coeff. of
Lower (%)	Upper (%)	Lower (%)	Upper (%)	Determination
75	125	75	125	0.99500

Error action: Stop analysis

QC

GLP Override: Yes

QC Tests

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ATTACHMENT 1
Benchsheet

COLUMBIA ANALYTICAL SERVICES, INC.
ANALYTICAL WORKSHEET

PRINTOUT WITH:_____

Method: (Circle One) 7470A 7471B 245.1	**Service Request # :**
Analysis For: Hg	

DATA

Pos.	SAMPLE NUMBER	Initial Sample (g) or (mL)	Initial Dilution (mL)	Dilution Factor	Measured (µg/L)	Sample Actual (mg/kg)	Sample Actual (µg/L)
1	Cal. Blk.	0.00	50	~	0.00		0.00
2	Std 0.2	*0.1	50	~	0.20		0.20
3	Std 0.5	*0.25	50	~	0.50		0.50
4	Std 1.0	*0.5	50	~	1.00		1.00
5	Std 5.0	*2.5	50	~	5.00		5.00
6	Std 10.0	*5.0	50	~	10.00		10.00
7		~	~	~			
8		~	~	~			
9		~	~	~			
10		~	~	~			
11		~	~	~			
12				~			
13				~			
14				~			
15				~			
16				~			
17				~			
18				~			
19				~			
20				~			
21				~			
22		~	~	~			
23		~	~	~			
24				~			
25				~			

Comments: Reporting Levels: **Cal. Inter. Std* (100ppb)**_____
Soil/Tissue Spike Level: **2nd Source Inter Std** (1ppm)**_____
Post Spike Level: 1.0 ppb

Method	Spike Level	MRL	LCS Limit	MS Limit	RPD
7470A Water	1.0 µg/L	0.2 µg/L	83-117%	80-120%	20%
245.1 Water	1.0 µg/L	0.2 µg/L	85-115%	70-130%	20%
7470A TCLP	5.0 µg/L	1.0 µg/L	85-115%	75-125%	20%
7471A Soil LCSS	3.73 mg/kg	0.02 mg/kg	72-128%	80-120%	30%
7471A Tissue Tort	0.27 mg/kg	0.02 mg/kg	63-130%	80-120%	30%

Analyst:	Date:	Page Number: 1

[Controlled - HgWaterRunForm 7-11-11] HG1.XLS

TABLE 1

Summary of Corrective Actions

Method Reference	Control	Specification and Frequency	Acceptance Criteria	Corrective Action
EPA 7470A	ICAL	Prior to sample analysis	R2 ≥ 0.995	Correct problem then repeat ICAL
EPA 7470A	ICV	After ICAL	±10%	Correct problem and verify second source standard; rerun second source verification. If fails, correct problem and repeat initial calibration.
EPA 7470A	CCV	Prior to sample analysis	±10%	Correct problem then repeat CCV or repeat ICAL
EPA 7470A	Method Blank	Include with each analysis batch (up to 20 samples)	<MRL	If target exceeds MRL, reanalyze to determine if instrument was cause. If still noncompliant then: Re-extract or reanalyze samples containing contaminate, unless samples contain > 20x amount in blank.
EPA 7470A	Laboratory Control Sample	Include with each analysis batch (up to 20 samples)	See DQO Table	If exceeds limits, re-extract and re-analyze
EPA 7470A	Matrix Spike	Include with each analysis batch (up to 20 samples)	See DQO Table	Evaluate data to determine if the there is a matrix effect or analytical error
EPA 7470A	Sample Duplicates	Include with each analysis batch (up to 20 samples)	RPD ≤ 20	Re-homogenize and re-analyze if result is > 5 X the MRL

ALS Environmental

DOCUMENT TITLE:	***METALS DIGESTION OF AQUEOUS SAMPLES***
REFERENCED METHOD:	
SOP ID:	***MET-DIG***
REV. NUMBER:	***14***
EFFECTIVE DATE:	***07/31/2013***

METALS DIGESTION OF AQUEOUS SAMPLES

MET-DIG

ALS-KELSO

SOP ID:	MET-DIG	Rev. Number:	14	Effective Date:	07/31/2013

Approved By: _____ Date: 7/8/13
Department Supervisor - Jeff Coronado

Approved By: _____ Date: 7/8/13
QA Manager - Suzanne LeMay

Approved By: _____ Date: 7/9/13
Laboratory Director - Jeff Grindstaff

Issue Date:	_____	Doc Control ID#:	_____	Issued To:	_____

Standard Operating Procedure

for

METALS DIGESTION OF AQUEOUS SAMPLES

1. SCOPE AND APPLICATION

1.1 This procedure is used to prepare aqueous samples for determination of metals using ICP, ICP/MS, and GFAA methodologies. This procedure is the ALS default water digestion procedure and is based on methods described in EPA CLP ILM04.0, EPA 200.8, EPA 200.7 and EPA 200.9.

1.2 This procedure is applicable to all "CLP" scope of elements plus boron and molybdenum.

1.3 This procedure is used for the determination of dissolved as well as total recoverable metals.

1.4 This procedure is applicable to drinking water and non-potable water sample matrices.

2 METHOD SUMMARY

2.1 A representative aliquot of aqueous sample is digested in nitric, hydrochloric acid, hydrogen peroxide or a mix of acids/reagents. After cooling, the sample is made up to volume prior to analysis.

2.2 This digestion procedure is carried out in a Class-10,000 clean room and "Clean" procedures are utilized throughout.

3 DEFINITIONS

3.1 Batch - A batch of samples is a group of environmental samples that are prepared and/or analyzed together as a unit with the same process and personnel using the same lot(s) of reagents. It is the basic unit for analytical quality control.

3.2 Preparation Batch - A preparation batch is composed of one to twenty field samples, all of the same matrix, meeting the criteria in Section 3.3 and with a maximum time between the start of processing of the first and last samples in the batch to be 24 hours.

3.3 Sample

3.3.1 Field Sample - An environmental sample collected and delivered to the laboratory for analysis; a.k.a., client's sample.

3.3.2 Laboratory Sample - A representative portion, aliquot, or subsample of a field sample upon which laboratory analyses are made and results generated.

3.4 Quality System Matrix - The matrix of an environmental sample is distinguished by its physical and/or chemical state and by the program for which the results are intended. The

following sections describe the matrix distinctions. These matrices shall be used for purpose of batch and quality control requirements.

3.4.1 Aqueous - Any groundwater sample, surface water sample, effluent sample, and TCLP or other extract. Specifically excluded are samples of the drinking water matrix and the saline/estuarine water matrix.

3.4.2 Drinking water - Any aqueous sample that has been designated a potable or potential potable water source.

3.4.3 Saline/Estuarine water - Any aqueous sample from an ocean or estuary or other salt-water source.

3.5 Method Blank (MB) - The method blank is an artificial sample composed of analyte-free water or solid matrix and is designed to monitor the introduction of artifacts into the analytical process. The method blank is carried through the entire analytical procedure.

3.6 Laboratory Control Samples (LCS) – The LCS is an aliquot of analyte free water or analyte free solid to which known amounts target analytes are added. The LCS is prepared and analyzed in exactly the same manner as the samples. The percent recovery is compared to established limits and assists in determining whether the batch is in control.

3.7 Matrix Spike/Duplicate Matrix Spike (MS/DMS) Analysis - In the matrix spike analysis, predetermined quantities of target analytes are added to a sample matrix prior to sample preparation and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the method used for the analysis. Samples are split into duplicates, spiked, and analyzed as a MS/DMS pair. Percent recoveries are calculated for each of the analytes detected. The relative percent difference (RPD) between the duplicate spikes (or samples) is calculated and used to assess analytical precision. The concentration of the spike should be at 3- 5 times the method reporting limit or at levels specified by a project analysis plan.

3.8 Laboratory Duplicates (DUP) – Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. The relative percent difference (RPD) between the sample and its duplicate is calculated and used to assess analytical precision.

4 INTERFERENCES

Refer to the determinative method for a discussion of interferences.

5 SAFETY

5.1 All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2 Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in MSDSs where available. Refer to the ALS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3 Hydrochloric and/or Nitric Acid are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

5.4 Hydrogen peroxide is an irritant of the eyes, mucous membranes, and skin. Inhalation of high concentrations of the vapor or mist may cause extreme irritation of the nose and throat. Lab coat, gloves and safety eyewear must be worn while working with this reagent.

6 SAMPLE PRESERVATION AND STORAGE

6.1 Aqueous samples are preserved with nitric acid (pH<2), then refrigerated at 4± 2°C from receipt until analysis. If properly acid preserved, the sample can be held up to 6 months before analysis.

6.2 Samples acidified at the laboratory must be held for 24 hours, then the pH verified as < 2 prior to digestion.

6.3 Metals holding time is six months from sample collection until analysis.

7 APPARATUS AND EQUIPMENT

7.1 Class 10,000 clean room equipped with Class 100 High Efficiency Particulate Air (HEPA) filter equipped laminar flow hoods.

7.2 Hot Blocks

7.3 Powder free PVC gloves

7.4 100ml Plastic beaker cups

7.5 Plastic Watch glasses, ribbed (large enough to cover plastic beaker cups.

7.6 Evergreen disposable tubes and caps, 50 mL. Check tubes for accuracy on a per batch basis by filling a tube to the 50 mL mark and measuring the water's mass. The measured mass must be accurate to ± 3%, if not obtain a new lot of tubes and retest. Refer to the SOP for *Checking Volumetric Labware (ADM-VOLWARE)*, for detailed instructions on performing the accuracy test.

8 STANDARDS AND REAGENTS

8.1 Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination. The preparation for all laboratory prepared reagents and solutions must be documented in a laboratory logbook. Refer to the SOP *Reagent/Standards Login and Tracking (ADM-RTL)* for the complete procedure and documentation requirements.

8.1.1 Reagent water: ASTM Type I water (resistivity ≥18 MΩ-cm, conductivity ≤0.056 uS/cm).

8.1.2 Concentrated Nitric Acid: J.T. Baker "Instra-analyzed", Trace Metals Grade - should be demonstrated to be free of impurities

8.1.3 Ultrex concentrated nitric acid - should be demonstrated to be free of impurities

8.1.4 Ultrex concentrated hydrochloric acid - should be demonstrated to be free of impurities

8.1.5 Hydrogen peroxide (30%) - H_2O_2. should be demonstrated to be free of impurities.

8.2 Standards

8.2.1 Stock standard solutions may be purchased from a number of vendors. All reference standards, where possible, must be traceable to SI units or NIST certified reference materials. The vendor-assigned expiration date is used.

8.2.2 Metals spiking solutions: 14 Solutions are needed to prepare the matrix spiking standards: SS1, SS2, SS3, SS4, SS5, GLFLCSW, QCP-CICV-1, QCP-CICV-2, QCP-CICV-3, U- 10ppm and Mo - 10ppm.

8.2.2.1 QCP-CICV-1, QCP-CICV-2, and QCP-CICV-3 are purchased as prepared solutions.

8.2.3 Follow the formulations laid out on the "Metals Spike Form" and "ICPMS LCSW and Spiking Solutions Form" (see Table A). These solutions are prepared in acid rinsed Class A volumetric flasks using purchased custom mixed standards or 1000 ppm single analyte standards. Aliquots are made using acid rinsed Class A volumetric pipettes of the appropriate size.

8.2.3.1 SS1 (Al, Ag, Ba, Be, Cd, Co, Cr, Cu, Fe, Pb, Mn, Ni, Sb, V, and Zn): Fill a 1000 mL volumetric flask approximately half full with reagent water, add 50 mL of nitric acid and mix. Next add 100 mL of the custom mixed standard (CAS-CAL-14) purchased from "Inorganic Ventures". In addition add 50 mL of 1000 ppm Antimony. Dilute to volume with reagent water, mix thoroughly and transfer to a 1000 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

8.2.3.2 SS3 (As, Se, and Tl): Fill a 500 mL volumetric flask approximately half full with reagent water, add 25 mL of nitric acid and mix. Next add 50 mL each of 1000 ppm Arsenic, Selenium, and Thallium. Dilute to volume with reagent water, mix thoroughly and transfer to a 500 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

8.2.3.3 SS4 (B, Mo): Fill a 500 mL volumetric flask approximately half full with reagent water, add 25 mL of nitric acid and mix. Next add 50 mL each of 1000 ppm Boron and Molybdenum. Dilute to volume with reagent water, mix thoroughly and transfer to a 500 mL Teflon bottle for storage. The solution's expiration date is determined by the earliest expiration date of any single component in the solution.

8.2.3.4 SS5 (K, Na, Mg, Ca): Fill a 1000 mL volumetric flask approximately half full with reagent water, add 10 mL of nitric acid and mix. Next add 100 mL of the custom mixed standard (CAS-CAL-14) purchased from "Inorganic Ventures". In addition add 20 mL of 10000 ppm Phosphorus, Sodium, Magnesium, Calcium. Dilute to volume with reagent water, mix thoroughly and transfer to a 1000 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

8.2.3.5 SS2 (As, Cd, Pb, Se, Tl and Cu): Fill a 500 mL volumetric flask approximately half full with reagent water, add 25 mL of nitric acid and mix. Next add 2.0 mL each of 1000 ppm Arsenic, Cadmium, Lead, Selenium, Thallium and Copper. Dilute to volume with reagent water, mix thoroughly and transfer to a 500 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

8.2.3.6 k-met 1/100 QCP-CICV-1: Fill a 500 mL volumetric flask approximately half full with reagent water. Next add 5.0 mL of QCP-CICV-1 and 5 mL of Ultrex nitric acid. Dilute with reagent water mix thoroughly and transfer to a 500 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

8.2.3.7 k-met 1/100 QCP-CICV-2: Fill a 500 mL volumetric flask approximately half full with reagent water. Next add 2.5 mL of 1000 ppm Sb and 5 mL of Ultrex nitric acid. Dilute with reagent water mix thoroughly and transfer to a 500 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

8.2.3.8 k-met 1/100 QCP-CICV-3: Fill a 500 mL volumetric flask approximately half full with reagent water. Next add 5.0 mL of QCP-CICV-3 and 5 mL of Ultrex nitric acid. Dilute with reagent water mix thoroughly and transfer to a 500 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

8.2.3.9 K-met U-10ppm: Fill a 500 mL volumetric flask approximately half full with reagent water, add 5.0 mL of nitric acid and mix. Next add 5.0 mL of 1000ppm Uranium dilute with reagent water mix thoroughly and transfer to a 500 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

8.2.3.10 k-met Mo-10ppm: Fill a 500 mL volumetric flask approximately half full with reagent water, add 5.0 mL of nitric acid and mix. Next add 5.0 mL of 1000ppm Molybdenum dilute with reagent water mix thoroughly and transfer to a 500 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

9 PREVENTIVE MAINTENANCE

9.1 Facility and Equipment Preparation

9.1.1 Laboratory equipment (i.e. centrifuge tubes, filtration apparatus, etc.) which comes in contact with the sample or digestate during the analysis must be thoroughly pre-cleaned with 1:4 HCl, and rinsed with DI water. All laboratory equipment used for trace metals analysis shall be stored in the clean room, and shall not be used for any other purpose.

9.1.2 All clean room work areas, including bench tops and laminar flow hoods, should be frequently washed and wiped dry with lint free; class-100 wipes to remove contamination.

9.2 Hot Block temperatures are monitored on sample batch basis.

10 RESPONSIBILITIES

10.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2 It is the responsibility of the department supervisor/manager to document analyst training.

11 PROCEDURE

11.1 Sample Digestion (go to section 11.2 if sample originate from Arizona)

11.1.1 Set up the sample batch in LIMs and print the *Preparation Information Sheet*. All digestion and sample information on this benchsheet.

11.1.2 Centrifuge tubes used for ICP-OES or ICP-MS analysis require pre-cleaning. This is done by adding 5-10 mL of 25% HCl to the centrifuge tube, shaking to completely 'wash' the centrifuge tube and its cap. Dispose of the rinse acid into an acid waste container. Rinse the centrifuge tube and cap 3 times with DI water, then air dry.

11.1.3 Shake the sample and measure a 50ml aliquot into a 50ml centrifuge tube. Use DI water for the MB and LCS.

11.1.4 Add the appropriate spiking solutions directly into the designated MS and LCS samples prior to addition of reagents. The amount and mix of spiking solutions are determined during the initial batch set up in LIMs. Typically this is 0.1 ml - 0.5 mL appropriate spiking solution. Fill out a spiking data sheet and keep it with the digestion data sheets.

11.1.4.1 Pipette tips used for ICP-OES or ICP-MS analysis require pre-cleaning. This is done by rinsing 3 times with Ultrex HNO_3 and then 3 times with DI water. Dispose of the rinse into an acid waste container.

11.1.5 The amount of type of reagents added for digestion will vary depending upon the method used for the final determination. The follow table list the amount and type of reagent to add:

Method	HNO_3	Ultrex HNO_3	HCL	Ultrex HCL	30% H_2O_2
200.7 & 6010	0.5 mL	-	2.5 mL		-
200.8	-	0.5 mL	-	-	-
6020	-	0.5 mL	-	-	-
200.9	0.5 mL	-	-	-	2.0 mL
7010	0.5 mL	-	-	-	2.0 mL

11.1.5.1 If the sample is being prepared for analysis by ICP, add 0.5ml of concentrated HNO_3, and 2.5 mL of concentrated HCL. Cap centrifuge tube with open reflux caps.

11.1.5.2 If the sample is being prepared for analysis by Graphite Furnace, add 0.5 mL of HNO_3 and 2.0 ml of 30% H_2O_2. Cap centrifuge tube with open reflux caps.

11.1.5.3 If sample is being prepared for ICP-MS the 50 mL aliquot into an acid rinsed centrifuge tube (sec 11.1.2) and add 0.5mL of *Ultrex* concentrated nitric acid. Cap centrifuge tube with centrifuge tube caps.

11.1.6 Place the centrifuge tube containing the sample in the "Block Digester", located in the Clean Room laminar flow hood, and heat at 95˚C for two hours, or until the volume has been reduced to between 15 and 25 mL.

11.1.7 Allow the sample to cool and dilute to the 50 mL mark with ASTM Type I - DI water.

11.1.8 Insoluble material is allowed to settle overnight, or the digestate may be centrifuged.

11.1.9 The digestates are ready for analysis

11.2 Procedure for Arizona Samples

Note: For Arizona samples and DW samples with turbidity > 1 NTU, digestion by the EPA Method 200.8 procedure is required.

11.2.1 For the determination of total recoverable analytes in aqueous samples, transfer a 100 mL (±1 mL) aliquot from a well mixed, acid preserved sample to an acid/DI rinsed 150 mL borosilicate beaker.

11.2.2 Add 1 mL concentrated nitric acid and 0.5 mL concentrated hydrochloric acid to the tube containing the measured volume of sample. Place the tube on a 95˚C Hot block digester, located in a clean room laminar flow hood, for solution evaporation. The tube should be covered with an acid/DI rinsed disposable watch glass to prevent sample contamination from the fume hood environment.

11.2.3 Reduce the volume of the sample aliquot to about 20 mL by gentle heating. DO NOT BOIL. This step takes about two hours for a 100 mL aliquot with the rate of evaporation rapidly increasing as the sample volume approaches 20 mL.

11.2.4 Cover the lip of the tube with a plastic watch glass to reduce additional evaporation and gently reflux the sample for 30 minutes. (Slight boiling may occur, but vigorous boiling must be avoided to prevent loss of the HCl-H2O azeotrope.)

11.2.5 Allow the tube to cool then dilute with reagent water to a final volume of 50 mL. Seal the tube with an acid/DI rinsed cap and mix.

11.2.6 Allow any undissolved material to settle overnight, or centrifuge a portion of the prepared sample until clear. (If after centrifuging or standing overnight the sample contains suspended solids that would clog the nebulizer, a portion of the sample may be filtered for their removal prior to analysis. However, care should be exercised to avoid potential contamination from filtration.)

11.2.7 Prior to analysis, adjust the chloride concentration by diluting the sample 2.0 fold. (If the dissolved solids in this solution are >0.2%, additional dilution may be required to prevent clogging of the extraction and/or skimmer cones.) The sample is now ready for analysis. Because the effects of various matrices on the stability of diluted samples cannot be characterized, all analyses should be performed as soon as possible after the completed preparation.

12 QA/QC REQUIREMENTS

12.1 Initial Precision and Recovery Validation

12.1.1 The accuracy and precision of the procedure must be validated before analyses of samples begin, or whenever significant changes to the procedures have been made. To do this, four water samples are spiked with the LCS spike solution, then prepared and analyzed.

12.2 Ongoing QC Samples required are described in the ALS-Kelso Quality Assurance Manual and in the SOP for Sample Batches. Additional QC Samples may be required in project specific quality assurance plans (QAPP). General QA requirements for DoD QSM are defined in the laboratory SOP, Department *of Defense Projects – Laboratory Practices and Project Management (ADM-DOD).* General QC Samples are:

12.2.1 Method Blank

12.2.1.1 A method blank is extracted and analyzed with every batch of 20 (or fewer) samples to demonstrate that there are no method interferences. If the method blank shows any hits above the reporting limit, corrective action must be taken. Corrective action includes recalculation, reanalysis, system cleaning, or re-extraction and reanalysis. For some project specific needs, exceptions may be noted and method blank results above the MRL may be reported for common lab contaminants.

12.2.2 Lab Control Sample (LCS)

12.2.2.1 Digest one laboratory control sample with each sample batch. Use the appropriate dilution of Inorganic Ventures ICV solutions for the liquid laboratory control sample (LCSW.)

12.2.3 Matrix Spike and Sample Duplicates

12.2.3.1 Digest one duplicate and one spiked sample with each sample matrix. For 6010, 6020, and 7010, prepare one duplicate and spiked sample on a five percent frequency per twenty samples. For 200.7, 200.8 and 200.9 prepare a duplicate and spiked sample on a ten percent frequency per twenty samples, or per SDG group, whichever is more frequent. Specific samples may be assigned as duplicates or spikes depending on client requirements.

13 REPORTING

13.1 Digestion data sheets including sample weights and volumes used are completed and a batch lot number is assigned and attached to the data sheet. The Manufacturer's lot numbers for the reagents used are added to the digestion data sheet as well as the spiking solutions and amount.

14 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

14.1. Refer to the SOP for *Non Conformance and Corrective Action (CE-QA008)* for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2. Handling out-of-control or unacceptable data

14.2.1. On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, runlogs, for example.

14.2.2. Some examples when documentation of a nonconformity is required using a Nonconformity and Corrective Action Report (NCAR):

- Quality control results outside acceptance limits for accuracy and precision
- Method blanks or continuing calibration blanks (CCBs) with target analytes above acceptable levels
- Sample holding time missed due to laboratory error or operations
- Deviations from SOPs or project requirements
- Laboratory analysis errors impacting sample or QC results
- Miscellaneous laboratory errors (spilled sample, incorrect spiking, etc)
- Sample preservation or handling discrepancies due to laboratory or operations error

15. POLLUTION PREVENTION AND WASTE MANAGEMENT

15.1. It is the laboratory's practice to minimize the amount of solvents, acids, and reagents used to perform this method wherever feasibly possible. Standards are prepared in volumes

consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvents and/or reagents used in this method can be minimized when recycled or disposed of properly.

15.2. The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Environmental Health and Safety Manual.

15.3. This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 2.5-12 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the ALS EH&S Manual for details.

16. METHOD PERFORMANCE

16.1. Available method performance data is given in the reference method. In addition, this procedure was validated through single laboratory studies of accuracy and precision as in the determinative procedure. The method detection limit(s) and method reporting limit(s) are established for the determinative procedure. See CE-QA011, *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation.*

17. TRAINING

17.1. Training outline

17.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

17.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

17.1.3. Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

17.2. Training is documented following the SOP for Documentation of Training.

17.2.1. When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

18. METHOD MODIFICATIONS

There are no known modifications in this laboratory standard operating procedure from the reference method.

19. REFERENCES

19.1. EPA Contract Laboratory Program, ILM04.0

19.2. DETERMINATION OF TRACE ELEMENTS IN WATERS AND WASTES BY INDUCTIVELY COUPLED PLASMA - MASS SPECTROMETRY, Method 200.8, Revision 5.4, 1994

19.3. DETERMINATION OF TRACE ELEMENTS BY STABILIZED TEMPERATURE GRAPHITE FURNACE ATOMIC ABSORPTION, Method 200.9, Rev 2.2, 1994

19.4. DETERMINATION OF METALS AND TRACE ELEMENTS IN WATER AND WASTES BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY, Method 200.7, Rev 4.4, 1994

20. CHANGES SINCE THE LAST REVISION

20.1. Reformatted SOP to ALS branding.
20.2. Replaced “CAS” references with “ALS”.
20.3. Updated SOP references.
20.4. Sec. 11.2: Added DW, turb. >1 NTU.
20.5. Sec. 11.2.1: Changed digestion tube to beaker.
20.6. Sec. 11.2.2: Revised acid volumes and added requirement for concentrated acid.
20.7. Sec. 11.2.7: Revised sample dilution.
20.8. Added changes per Procedural Change form dated 8/1/13.

Table A

METALS SPIKING SOLUTIONS CONCENTRATIONS FORM

Solution Name	Element	mLs of 1000ppm Solution	Final Volume	Solution Conc. mg/L	Concentration in the digest mg/L
SS1	HNO3	50.0	1000ml	-	
	Al	100*	1000ml	200	2
	Ag	100*	1000ml	5	0.05
	Ba	100*	1000ml	200	2
	Be	100*	1000ml	5	0.05
	Cd	100*	1000ml	5	0.05
	Co	100*	1000ml	50	0.5
	Cr	100*	1000ml	20	0.2
	Cu	100*	1000ml	25	0.25
	Fe	100*	1000ml	100	1
	Pb	100*	1000ml	50	0.5
	Mn	100*	1000ml	50	0.5
	Ni	100*	1000ml	50	0.5
	Sb	50	1000ml	50	0.5
	V	100*	1000ml	50	0.5
	Zn	100*	1000ml	50	0.5
SS2	HNO3	25.0	500ml	-	
GFAA SPIKE	As	2.0	500ml	4	0.04
	Cd	2.0	500ml	4	0.04
	Pb	2.0	500ml	4	0.04
	Se	2.0	500ml	4	0.04
	Tl	2.0	500ml	4	0.04
	Cu	2.0	500ml	4	0.04
SS3	HNO3	25.0	500ml	-	
	As	50.0	500ml	100	1
	Se	50.0	500ml	100	1
	Tl	50.0	500ml	100	1
SS4	HNO3	25	500ml	-	
	B	50	500ml	100	1
	Mo	50	500ml	100	1
SS5	HNO3	10.0	200ml	-	
	K**	20	200ml	1000	10
	Na**	20	200ml	1000	10
	Mg**	20	200ml	1000	10
	Ca**	20	200ml	1000	10

Table A –cont.

METALS SPIKING SOLUTIONS CONCENTRATIONS FORM

Solution Name	Element	mLs of 1000ppm Solution	Final Volume	Solution Conc. mg/L	Concentration in the digest mg/L
GFAA ONLY	As, Pb, Se, Tl	5.0	1000ml	2.5	0.025
	Cd	-	-	1.25	0.0125
	Cu	2.5	1000ml	2.5	0.025
QCP-CICV-1 ICP ONLY	Ca, Mg, Na, K	no dilution	-	2500	12.5
	Al, Ba	no dilution	-	1000	5
	Fe	no dilution	-	500	2.5
	Co, Mn, Ni, V, Zn	no dilution	-	250	1.25
	Cu, Ag	no dilution	-	125	0.625
	Cr	no dilution	-	100	0.5
	Be	no dilution	-	25	0.125
QCP-CICV-2 ICP ONLY	Sb	no dilution	-	500	2.5
QCP-CICV-3 ICP ONLY	As, Pb, Se, Tl	no dilution	-	500	2.5
	Cd	no dilution	-	250	1.25

* Denotes volume of mixed stock standard.
** Denotes 10,000 ppm individual stock standards.

Table A –cont.
ICPMS LCSW AND SPKING SOLUTIONS

5.00mL to 500mL Dilution of Inorganics Ventures QCP-CICV-1

k-met 1/100 QCP-CCV-1

Analyte	Concentration in solution (ppb)	Concentration in digest (ppb)
Al	10000	100
Ba	10000	100
Co	2500	25
Mn	2500	25
Ni	2500	25
V	2500	25
Zn	2500	25
Cu	1250	12.5
Ag	1250	12.5
Cr	1000	10
Be	250	2.5

5.00mL to 500mL Dilution of Inorganics Ventures QCP-CICV-2

k-met 1/100 QCP-CICV-2

Analyte	Concentration in solution (ppb)	Concentration in digest (ppb)
Sb	5000	50

5.00mL to 500mL Dilution of Inorganics Ventures QCP-CICV-3

k-met 1/100 QCP-CICV-3

Analyte	Concentration in solution (ppb)	Concentration in digest (ppb)
As	5000	50
Pb	5000	50
Se	5000	50
Tl	5000	50
Cd	2500	25

1.00mL to 100mL Dilution of Inorganic Ventures 1,000 ppm Mo

k-met Mo 10ppm

Analyte	Concentration in solution (ppb)	Concentration in digest (ppb)
Mo	10000	20

5.00mL to 500mL Dilution of High Purity 1,000 ppm U

k-met U 10ppm

Analyte	Concentration in solution (ppb)	Concentration in digest (ppb)
U	10000	20

Table B: Metals Water Digestion Spiking Solution & Acid Quantities

Method	200.7 & 6010	200.7LL & 6010LL	200.8 & 6020	200.8LL	200.9	7010
Solution						
K-MET SS1	0.5 mL	0.5 mL				
K-MET SS2					0.5 mL	0.5 mL
K-MET SS3	0.5 mL	0.5 mL				
K-MET SS4	0.5 mL	0.5 mL				
K-MET SS5	0.5 mL	0.5 mL				
K-MET GFLCSW					0.5 mL	0.5 mL
K-MET QCP-CICV-1	0.25 mL	0.25 mL				
K-MET QCP-CICV-2	0.25 mL	0.25 mL				
K-MET QCP-CICV-3	0.25 mL	0.25 mL				
k-met 1/100 QCP-CICV-1			0.5 mL	0.5 mL		
k-met 1/100 QCP-CICV-2			0.5 mL	0.5 mL		
k-met 1/100 QCP-CICV-3			0.5 mL	0.5 mL		
k-met Mo 10ppm			0.1 mL	0.1 mL		
k-met U 10 ppm			0.1 mL	0.1 mL		
k-met Alt. 200.8 Spiking Solution			0.1 mL	0.1 mL		
Acid						
HNO3	0.5 mL	0.5 mL			0.5 mL	0.5 mL
HCL	2.5 mL	2.5 mL				
H2O2 (30%)					2 mL	2 mL
HNO3 ULTREX			0.5 mL	0.5 mL		

5% QC for 6010, 6010LL, 6020, 7010.
10% QC for 200.7, 200.7LL, 200.8, 200.8LL, 200.9.
Matrix spike quantities that have a reduced final volume of 25 mL require half the normal spiking amount.

STANDARD OPERATING PROCEDURE

For

GASOLINE RANGE ORGANICS BY GAS CHROMATOGRAPHY

SOP No.: PET-GRO

Revision: 9

November 22, 2010

Approved by: _____ Supervisor — 11/2/10 Date

_____ QA Manager — 11/2/10 Date

_____ Laboratory Manager — 11/2/10 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL
NUMBER: _____
Initials: _____ Date: _____

Standard Operating Procedure
for
GASOLINE RANGE ORGANICS BY GAS CHROMATOGRAPHY
SOP NO.: PET-GRO
Revision 9
Date: November 22, 2010

Procedure Change Form

SOP Section Number	Date Procedure Change Implemented	Description of Procedure Change	Supervisor Approval and Notification of Other Analysts
2.2	12/2/10	No Low-level direct purge mentioned anywhere else Take out ↑ (Minor change)	KA 8/11-11

1. SCOPE AND APPLICATION

1.1. This Standard Operating Procedure (SOP) describes the method used for analysis of purgeable petroleum hydrocarbons by methods EPA 8015C, AK101, NWTPH-Gx and CA-LUFT. This procedure describes both the extraction and chromatographic procedures used to determine the target analytes listed in Table 1.

1.2. This procedure is used to determine the analytes of interest in water, soil, sediment, and oil matrices. The procedure may be applied to other miscellaneous sample matrices providing that the analyst demonstrates the ability of the procedure to give data of acceptable quality in that matrix. The Method Reporting Limits (MRLs) for target analytes in water and soil are presented in Table 2. Method Detection Limits (MDLs) which have been achieved are also given, and are subject to change as studies are updated.

1.3. This procedure is primarily used to report Gasoline Range Organics (GRO) quantified using a gasoline standard. It may be used for purgeable fuel identification. This procedure is not capable of distinguishing mixtures of hydrocarbon products and is not recommended for use on samples containing complex mixtures of volatile fuels or solvents.

2. METHOD SUMMARY

2.1. Samples are prepared and analyzed for volatile organic contaminants using gas chromatography (GC) and quantified using a flame ionization detector (FID).

2.2. Soil samples are typically analyzed by performing a methanol extraction, followed by dilution into reagent water and GC analysis. A low level direct-purge option is also given. Water samples are analyzed by direct purge (unheated) with helium. Target analytes are adsorbed and concentrated on a carbon based trap. Upon completion of the purge step, the trap is heated and target analytes are desorbed into the GC system. Analyte separation is accomplished using a capillary chromatographic column and quantitation is made by comparison of target analyte response to the response of calibration standards over a known concentration range.

2.3. The procedure is applicable for the analysis of target analytes, and meets method criteria for the methods listed in Table 1.

3. DEFINITIONS

3.1. Analytical Batch: A group of field samples, QC samples, standards and blanks analyzed together under a common and unique identification number. Analytical batches must contain blanks, QC samples, and calibration standards interspersed at the required frequency. Analytical batch identification numbers are assigned to each batch.

3.2. Analytical Sequence: A batch of samples analyzed in sequential order. A sequence starts with an initial calibration or continuing calibration verification (CCV) standard, followed by a system blank, followed by ten samples, followed by a continuing calibration verification standard. The closing continuing calibration verification standard for one group of samples may serve as the opening continuing calibration verification standard for the next group of samples. This pattern may be repeated until the sequence is complete, or until a continuing calibration verification standard fails acceptance criteria. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria indicate an out-of-control situation.

3.3. Calibration: procedure used to quantify analyte amount in the sample.

3.3.1. Continuing Calibration Blank (CCB): system blank analyzed at a routine frequency throughout the sequence to establish that the instrument is free of target analyte contamination.

3.3.2. Continuing Calibration Verification (CCV): standard analyzed at a routine frequency throughout the sequence to establish the instrument is meeting calibration criteria.

3.3.3. Independent Calibration Verification (ICV): standard analyzed from a source that is different than that used to calibrate the instrument. The ICV is a check on the accuracy of the calibration standards.

3.4. Extraction Batch: A group of field samples and QA/QC samples prepared together under a common and unique identification number. Extraction batch identification numbers are limited to 10 characters. Extraction batches are limited in size to 20 field samples per set of QA/QC samples.

3.5. Hydrocarbon Marker Mix: A solution of individual marker compounds whose retention times are used in defining the window of integration for range compounds.

3.6. Method Detection Limit (MDL): A statistically derived value representing the lowest level of target analyte that may be measured by the instrument with 99% confidence that the value is greater than zero.

3.7. Method Reporting Limit (MRL): The minimum amount of a target analyte that can be measured and reported quantitatively (supported by a low calibration point). The MRL is equivalent to Practical Quantitation Level (PQL) and Estimated Quantitation Level (EQL).

3.8. QA/QC Samples: Samples added to a sample preparation batch, or an analytical batch to provide quality assurance checks on the analysis.

3.8.1. Duplicate Sample (DUP): A laboratory duplicate. The duplicate sample is a separate sample aliquot that is processed in an identical manner as the sample proper.

3.8.2. Laboratory Control Sample (LCS): A laboratory blank that has been fortified with target analyte.

3.8.3. Laboratory Control Sample/Duplicate Laboratory Control Sample (LCS/DLCS): Laboratory control samples performed in duplicate to evaluate precision within the batch. If insufficient sample amount is provided for DUP or DMS analysis, a DLCS is required.

3.8.4. Method Blank (MB): A laboratory blank that is used to check that reagents are free of target analytes. Method Blanks for aqueous samples are prepared using laboratory reagent (deionized) water. Method Blanks for solid samples are prepared using sand, baked overnight at 400^{o}C. Method Blanks for soluble waste samples are prepared using solvent (methanol).

3.8.5. Matrix Spike Sample (MS): A duplicate sample aliquot that has been fortified with target analyte.

3.8.6. Matrix Spike Sample/Duplicate Matrix Spike Sample (MS/DMS): Matrix spike samples prepared in duplicate.

3.9. Retention Time Window: The chromatographic time period established within which a target analyte is qualitatively determined to be present in the sample or sample extract.

3.10. Hydrocarbon Range: A retention time window that encompasses an extended time period.

3.10.1. Hydrocarbon Range Start Time (Inclusive): absolute retention time of the appropriate marker compound minus 0.1 minutes.

3.10.2. Hydrocarbon Range Start Time (Exclusive): absolute retention time of the appropriate marker compound plus 0.1 minutes.

3.10.3. Hydrocarbon Range End Time (Inclusive): absolute retention time of the appropriate marker compound plus 0.1 minutes.

3.10.4. Hydrocarbon Range End Time (Exclusive): absolute retention time of the appropriate marker compound minus 0.1 minutes.

3.11. Surrogate: A non-target analyte that is added to all samples and QA/QC samples that is chemically similar to the compounds of interest. The surrogate is used to evaluate the effectiveness of the analysis.

3.12. Target Analyte: A compound, or substance of interest for which the method is capable of measuring.

4. INTERFERENCES

4.1. Samples can become contaminated by diffusion of volatile organics during shipment and storage. If a trip blank is received with the samples, it can serve as check for such contamination.

4.2. Contamination by carryover can occur when high level samples immediately proceed samples containing significantly lower levels of contamination. This can be minimized by running system blanks after samples that are suspected to contain high levels of target analyte. For purposes of evaluating sequencing criteria during an analytical batch, these system blanks are not defined as samples and therefore are not counted in the ten sample groupings.

4.3. High levels of heavier petroleum products such as diesel fuel will cause a positive bias for Gasoline Range Organics when integrated using the techniques described in this procedure. If the response in the Gasoline range is obviously not due to gasoline hydrocarbons, the data may be flagged to indicate that the fingerprint did not match typical gasoline.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. The use of pressurized gases is required for this procedure. Care should be taken when moving cylinders. All gas cylinders must be secured to a wall or an immovable counter with a chain or a cylinder clamp at all times. Sources of flammable gases (e.g., pressurized hydrogen) should be clearly labeled.

5.4. The proper use of syringes and glass pipettes should be part of employee training. Care should be taken to avoid personal injury as a result of improper handling techniques.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Aqueous Samples should be collected (received) in 40 mL VOA vials with zero headspace. Samples should be preserved to pH <2 with hydrochloric acid. Ideally, three VOA vials will be received for each sample. Samples will be refrigerated to 4 ± 2°C upon sample login. To monitor for possible contamination, a trip blank prepared from organic-free reagent water should accompany the sample containers throughout sampling, shipping, and storage. For laboratory handling and analysis, trip blanks are treated the same as field samples. Trip blanks and field blanks are mandatory for CA-LUFT analyses.

6.2. Solid samples are collected in a vial containing methanol and cooled to 4 ± 2°C and extracted as described in the SOP VOC-5035. Samples shipped in glass jars with Teflon lined septum and packed tightly to minimize interstitial space and reduce headspace will be extracted by method 5030A.

6.3. Samples will be refrigerated to 4 ± 2°C upon sample login (CA-LUFT requires soil samples to be frozen until analysis). Where field-preservation with methanol is used, a trip blank containing methanol preservative should accompany the sample containers throughout sampling, shipping, and storage. For laboratory handling and analysis, trip blanks are treated the same as field samples.

6.4. Methanol preserved soil and sediments (e.g., soils collected for Alaska DEC Method AK 101) should be received in a pre-weighed 4-ounce jar with a Teflon lined septum fused to the lid. The samples should be submerged in a pre-measured volume of methanol containing a known concentration of surrogate compound. Samples will be refrigerated to 4 ± 2°C upon sample login.

6.5. Waste samples such as oils can be collected (received) in a variety of sample containers. Typically, glass bottles with Teflon lined septum are preferred. Headspace should be kept to a minimum.

6.6. Analytical Holding Times: See Table 7 for guidelines on method specific maximum recommended holding times.

NOTE: For AK101, samples received unpreserved are not to be used for compliance purposes.

7. APPARATUS AND EQUIPMENT

7.1. Balances

7.1.1. Analytical Balance

7.1.2. Top Loading Balance

7.2. Centrifuge

7.3. Gas Chromatography System

7.3.1. Gas Chromatograph (GC): Analytical system equipped with gas supplies, column inlet, programmable oven, heated detectors (PID/FID in series), and a data system for determining peak areas. Hewlett-Packard 5890 Series II, Varian 3300, or equivalent.

7.3.2. Chromatographic Columns

7.3.2.1.DB-624: 30 m x 0.53 mm ID, 3 μm film thickness; or equivalent.

7.3.2.2.JW-VRX: 30 m x 0.45 mm ID, 2.55 μm film thickness; or equivalent

7.3.2.3.RTX-VGC: 30 m x 0.45 mm ID, 2.55 μm film thickness; or equivalent.

7.3.3. Autosampler Device: Dynatech PTA 30 and Varian Archon systems meet the minimum requirements for automatic sampling from 40 mL VOA vials.

7.3.4. Purge and Trap Device: OI 4460A, or equivalent, which meets the minimum requirements for GC purge and trap analysis.

7.3.5. Trap: Supelco J trap (part number 2-1145), or equivalent trap that meets the minimum requirements for the procedure.

7.3.6. Compressed Gases or Gas Generators: Hydrogen (fuel gas), Air (pneumatic drive and fuel gas), Helium (purge gas and column flow).

7.4. Glassware and Syringes

7.4.1. Disposable Pipettes: Pasteur and serological (1mL, 2 mL, 5 mL).

7.4.2. Glass Beads: approximately 5 mm in diameter.

7.4.3. Graduated Cylinders with Ground Glass Stoppers: 50 mL and 100 mL.

7.4.4. Microreaction Vials with Pushpin Syringe Port.

7.4.5. Microsyringes: various sizes (10 μl, 25 μl, 100 μl, 250 μl, 500 μl, 1000 μl).

7.4.6. VOA Vials: 20 mL

7.4.7. VOA vials (40 mL) with Teflon Lined Septum

7.4.8. Volumetric Flasks: various sizes (5 mL, 10 mL, 25 mL, 50 mL, 100 mL).

7.5. pH Paper: wide range for measuring aqueous sample pH after analysis.

7.6. Stainless Steel Scoopula

7.7. Ultrasonic Bath

7.8. Vortex

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1. Reagents

8.1.1. Methanol: purge and trap grade

8.1.2. Reagent Water: Laboratory deionized water, produced by passing water through a series of deionizers followed by activated carbon filters in series. The water must be shown to be free of volatile contaminants of interest.

8.2. Standards: Unless otherwise noted, standards used in this procedure are prepared in methanol. Standards should be labeled as follows: logbook ID number, compounds or compound mix, concentration, solvent, date prepared, expiration date, analyst's initials.

8.2.1. Stock Standards: purchased as solutions from a reliable source or prepared in high concentration from neat material. A stock standard is defined as any standard that requires additional dilution prior to being used in the analysis. To prepare stock standards from neat material, accurately weigh the neat material using a four place analytical balance into a tared volumetric flask. Dilute to volume using methanol. Stopper and invert several times to mix. Transfer the solution to an appropriate storage vial (e.g., a microreaction vial), label and refrigerate. When preparing stock standard mixes from neat material, it is recommended that each component of the mix be prepared individually at high concentration then combined as a complete mix at a secondary dilution. Stock standards used for quantitative analysis are given an expiration date equal to the manufacturer's recommendation (purchased, sealed solutions). The expiration date on unopened ampules is the manufacturer's recommendation. The expiration period for opened ampules is 3 months. The following stock standards are used in this procedure.

8.2.1.1. Internal Standard Solution: Purchased solution containing α,α,α - Trifluorotoluene at 25000 ppm in methanol. Required for 8021B analysis.

8.2.1.2. Purge Surrogate Solution: Purchased solution containing 1,4-Difluorobenzene at 25000 ppm in methanol.

8.2.1.3. Soil Extraction Surrogate: Purchased solution containing 4-Bromofluorobenzene at 25000 ppm in methanol.

8.2.1.4. Hydrocarbon Marker Mix: Purchased solution containing Hexane, 2-Methylpentane, Benzene, Toluene, Decane, 1,2,4-Trimethylbenzene, Dodecane, and Naphthalene at 1000 ppm in methanol.

8.2.1.5. Gasoline: Prepared or purchased solution containing gasoline at 5000 ppm in methanol.

8.2.2. Working Standards:

8.2.2.1. Working standards are purchased as solutions from a reliable source or prepared from stock standards. A working standard is defined as any standard that is used directly in sample analysis. Working standards are

prepared from stock solutions by accurately transferring a volume of stock standard into a volumetric flask or graduated cylinder partially filled with the dilution solvent (methanol or reagent water). The volume is further adjusted using the dilution solvent to a known level. Working standards not used immediately are transferred to an appropriate storage vial, stored under minimal headspace, labeled and refrigerated.

8.2.2.2. Working standards used for quantitative analysis are given an expiration date equal to the manufacture's recommendation (purchased, sealed solutions), 7 days for purchased solutions that have been opened and for laboratory prepared solutions in methanol, and 24 hours for laboratory prepared solutions in reagent water. The expiration date for surrogate working standards is one month.

8.2.2.3. The concentration and spike amounts for working standards used for QC analyses are listed in Table 5.

Note: When stock standards are used as working standards (e.g., used in spiking QA/QC samples, the addition of internal standard or surrogates directly to samples, etc.) they will be considered stock standards for purposes of assigning expiration dates.

8.2.3. Calibration Standards: A series of standards prepared from a common stock ranging in concentration from a value representing the method reporting limit (or lower) to a value near the high end of the linear calibration range of the detector. Calibration standards (and continuing calibration standards) are prepared in reagent water. Calibrations will contain a minimum of five concentration levels. Recommended calibration ranges are listed in Table 3.

8.2.4. Independent Calibration Verification Standards (ICV): A second source independent of that used in preparing the calibration curve should be prepared as stock standards and subsequently used to create working standards. Concentrations are listed in Table 3.

9. PREVENTIVE MAINTENANCE

9.1. A maintenance log will be kept documenting maintenance performed on each analytical system. Log entries will include the date maintenance was performed, symptoms of the problem, serial numbers of major equipment upgrades or replacements (defined as any non-consumable part essential to operation), phone logs from technical support contacts, a description of the maintenance performed, a description of the check performed to assure the system has returned to acceptable levels of operation, and the analyst's initials.

9.2. Autosampler: Routine maintenance is generally limited to changing the Teflon lined O-ring seal on the syringe plunger, cleaning the rods on the Archon with methanol, and replacing the waste valve when leaks develop.

9.3. Purge and Trap: Routine maintenance is generally limited to changing the trap, cleaning or replacing the sparge vessel, and adjusting the purge flow. Trap breakdown is generally indicated by poor recovery of all target analytes. If purge flow is too fast, lighter end compounds will exhibit a reduced recovery. If purge flow is too slow, heavier end compounds will exhibit a reduced recovery.

9.4. GC System: Maintenance of the GC system is generally limited to trimming or changing the column and cleaning the FID. Poor chromatography and reduced resolution of closely eluting peaks is an indication of column breakdown. The FID will rarely require maintenance for this analysis.

9.5. Cleaning of Glassware: Glassware used in sample analysis is washed and baked for several hours at 70-100^{o}C prior to use.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in the SOP for Documentation of Training, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Sample Preparation: An extraction batch is defined as a group of field samples and QA/QC samples processed together with the same reagents. Extraction batches are limited to 20 field samples.

Each sample preparation batch for aqueous and solid matrices will contain a maximum of 20 field samples, one MB, one LCS, and a precision component (DUP, DMS, or DLCS). Add additional QC samples such as one DLCS, a MS, and either a DMS or sample DUP, depending on method requirements. Additional QC analyses are performed at a rate of one set per 20 field samples. Batches may not extend over more than a one-day period. The analyst should refer to the CAS *SOP for Sample Batches.*

Note: NWTPH-Gx analyses are limited to 10 field samples per MB and DUP.

11.1.1. Aqueous Samples: Direct Purge (unheated)

11.1.1.1. Aqueous samples may be aliquotted from a VOA vial by the autosampler and directly purged into the gas chromatograph. Program the autosampler to aliquot a 10 mL sample volume into the purge and trap sparge vessel.

If dilutions are required, prepare an adequate volume to fill a 40 mL VOA with no headspace.

11.1.1.2. Surrogate spike is added by the autosampler prior to purging. Add the appropriate matrix spike solution to LCS, DLCS, MS, and DMS samples. See Table 5 for appropriate spike amounts. Matrix spiked samples are prepared by adding a single glass bead to each aliquot to be spiked. The appropriate spike is added via a syringe. Invert the sample to mix (the glass bead helps to homogenize the spike).

11.1.1.3. Prepare a Method Blank using reagent water.

11.1.1.4. Following analysis, check and record sample pH using wide range pH paper. Samples should be preserved to <2. NWTPH-Gx and CA-LUFT allow unpreserved samples to be analyzed within 7 days of collection.

11.1.2. Solid Samples: The method-prescribed sample preparation is used unless otherwise specified for the project.

11.1.2.1. Methanol Extraction of bulk soil samples

11.1.2.1.1. A portion of sample is removed from the sample container by coring down into the center of the sample and obtaining a representative cross section of the sample.

11.1.2.1.2. Weigh approximately 5g (10g for AK101) of sample (dry wt.) into a 20 mL VOA vial. Record weight to the nearest 0.01 gram. Prepare a Method Blank and LCS/DLCS using sand. Quickly add 10 mL methanol and the surrogate spike. Spike all field and QC samples with soil surrogate.

11.1.2.1.3. Add the appropriate matrix spike solutions to LCS, DLCS, MS, and DMS samples. See Table 5 for appropriate spike amounts. Recap vials and vortex. Allow samples to equilibrate before diluting. Extracts require refrigeration during storage. It is useful to mark the meniscus on the vial to serve as an indication if solvent evaporation has occurred over time.

11.1.2.1.4. Using reagent water, dilute 1mL of the methanol extract to 50mL in a stoppered graduated cylinder. Invert the cylinder several times to mix. Fill a clean VOA vial with the diluted extract so that no headspace is present. Additional dilutions may be prepared by using smaller volumes of methanol extract diluted in the same manner as described above.

Note: AK101 samples that are not field-preserved may only be reported if they are not for compliance purposes. The results for unpreserved samples should be flagged as not to be used for compliance purposes.

11.1.2.2.Solid Samples: Methanol Extraction, Field Preserved

Note: EPA method 5035 recommends field preservation of solid samples for GRO analysis. Alaska DEC requires field preservation for AK101 compliance samples. For 8015 and CA-LUFT, Encore samples are preserved in methanol, or field-preserved (methanol) samples are processed as per the 5035 procedure.

11.1.2.2.1.Weigh the sample container and contents. Record the weight to the nearest 0.1 gram. Subtract the weight of the sample container + MeOH (available from field notes or laboratory records). Calculate the weight of sample and record to the nearest 0.1 gram. Record the extraction solvent volume (available from field notes).

NOTE: The methanol used for extraction contains the proper concentration of extraction surrogate. For 8015 and Alaska Method AK101, soil surrogate should be included in the field preservative and therefore is already in the sample upon receipt from the field. Do not add more surrogate to these samples.

11.1.2.2.2.Matrix spikes are not performed on methanol preserved samples. Duplicate laboratory control samples should be prepared using approximately 10g sand extracted with 10 mL methanol. Spike laboratory control samples with surrogate and matrix spike prior to diluting the methanol into water. See Table 5 for appropriate spike amounts.

11.1.2.2.3.Prepare a Method Blank using 10g sand extracted with 10 mL methanol. Spike the method blank with surrogate prior to diluting the methanol into water. See Table 5 for appropriate spike amounts.

11.1.2.2.4.Using reagent water, dilute 1mL of the methanol extract to 50mL in a stoppered graduated cylinder. Invert the cylinder several times to mix. Fill a clean VOA vial with the diluted extract so that no headspace is present. Additional dilutions may be prepared by using smaller volumes of methanol extract diluted in the same manner as described above.

11.1.3. Methanol Soluble Wastes

11.1.3.1.Weigh 1gram sample to a 20 mL scintillation vial. Record weight to the nearest 0.1 gram. Quickly add 10 mL methanol. Cap vial and vortex.

11.1.3.2. Spike all samples and QC samples with soil surrogate. Matrix spikes are not performed on methanol soluble waste samples. Add the appropriate matrix spike solutions to LCS and DLCS samples. See Table 5 for appropriate spike amounts. Recap vials and vortex. Extracts require refrigeration during storage. It is useful to mark the meniscus on the vial to serve as an indication if solvent evaporation has occurred over time.

11.1.3.3. Prepare a Method Blank using 10 mL methanol.

11.1.3.4. Using reagent water, dilute 1mL of the methanol extract to 50mL in a stoppered graduated cylinder. Invert the cylinder several times to mix. Fill a clean VOA vial with the diluted extract so that no headspace is present. Additional dilutions may be prepared by using smaller volumes of methanol extract diluted in the same manner as described above.

11.2. Gas Chromatography

11.2.1. Program the autosampler. Refer to operator's manual for specifics on programming the various modes on the device. For sampling of aqueous samples and dilutions, program the device to sample a fixed volume appropriate for the analysis. Fill the rinse reservoir with reagent water. Empty the waste reservoir. Check that the system is pressurized before initiating the analysis.

11.2.2. See Table 4 for purge and trap operating conditions and suggested GC operating conditions.

11.2.3. Establish retention time windows for surrogates and hydrocarbon ranges.

11.2.3.1. For hydrocarbon range analytes, start and end times are established by analyzing a marker solution containing individual compounds that define the quantitation range. See Table 6 for guidance on establishing windows for the various method specified ranges. The hydrocarbon marker solution should be analyzed at least once within each analytical sequence containing samples being analyzed for hydrocarbon range analytes to verify no shift has occurred with respect to start and end times for the quantitation ranges.

11.2.3.2. Update daily retention times based on the analysis of an opening CCV and the hydrocarbon marker mix. For qualitative determination of a target analyte to be accepted, each set of ten samples in the analytical sequence should be bracketed by a CCV for which the absolute retention times have not shifted by more than one half the retention time delta (indicated by an "f" flag on the quantitation report). If this criteria is not met, the analyst must take measures to determine the reason for the retention time shifts. The affected samples (i.e., samples with tentative identification of target analyte) should be reanalyzed after the problem has been fixed.

11.3. Calibration

The calibration procedure(s) and options chosen must follow the CAS protocols. Any exceptions to the calibration procedures detailed in the CAS SOP for *Calibration of Instruments for Organics Chromatographic Analyses* are described as follows:

11.3.1. Analyze a minimum of 5 calibration standards as described in section 8.2.3. SOP (see Table 3). Refer to the *SOP for Calibration of Instruments for Organic Chromatographic Analyses* (SOC-CAL) for guidelines on instrument calibration procedures. However, method-specific calibration procedures must be followed.

11.3.1.1. Average of Response Factor (external standard calibration): Calibration factors are calculated as the total area of the peak or integration range divided by the amount injected. For external calibration, the calibration factor is calculated as:

$$Cf = \frac{Rx}{Ax}$$

Where: Cf = Calibration factor
Rx = Response of the analyte (area)*
Ax = Concentration of analyte purged (ug/L).

*Note: For fuel analytes the integrated area for the surrogate compound (and I.S. for 8021) is subtracted from the total area for the fuel pattern. This is done to eliminate the contribution of the surrogate or I.S. area within the fuel range. The Enviroquant software automatically performs this operation.

If the percent relative standard deviation (%RSD) of all points is <20% (25% for AK 101), linearity through the origin may be assumed and the average calibration factor may be used in quantifying sample data. When average of response factor is used to quantitate sample results, the calibration factor (*Cf*) is calculated as the average response factor for the curve (sum of all calibration factors divided by the number of points in the curve).

11.3.1.2. Linear Regression Calibration Curve: To use this option the correlation coefficient must be ≥ 0.995 ($r \geq 0.995$, $r^2 \geq 0.990$). This is the required calibration criteria for NWTPH-Gx. For NWTPH-Gx, each point must be within ± 15% of the true value.

This method of quantitation uses the equation of a line ($y=mx+b$). When a linear calibration curve is used to quantitate sample results, the line is plotted using response versus amount. The cumulative calibration factor (*Cf*) is equal to the linear term (i.e., slope of the line: *m*).

11.3.2. Verify the calibration by analyzing an ICV. The measured concentration must be ±20% of the true value (25% for AK101).

11.3.3. Analyze a system blank to establish that the instrument is free of target analyte contamination. Unless stated in project specific data quality objectives, the system is considered free of target analyte contamination if the level detected is less than half the method reporting limit.

11.3.4. Continuing Calibration Verification

11.3.4.1. The working calibration curve or calibration factor must be verified on each analytical sequence by the analysis of one or more mid-range calibration standards (CCV). A mid-level standard (CCV) must be injected at the start of each sequence and after each set of sample extracts (every 10 samples or every 12 hours, whichever is first) in the analysis sequence.

11.3.4.2. The acceptance criteria for all analytes in the CCV, as compared to the initial calibration, are listed below:

AK101	± 25% D *
NWTPH-Gx	± 20% D
8015	± 20% D
CA-LUFT	± 10% D

Note: For AK101, the surrogate recovery in the CCV must be 60-120% (see Table 9).

11.3.4.3. Use the mid-level standards interspersed throughout the analysis sequence to evaluate the qualitative performance of the GC system.

11.4. Sample Analysis

11.4.1. Analyze the samples and QC samples using the chromatographic conditions used for calibration acquisition method. Bracket samples with the appropriate calibration standards as described above.

11.4.2. Process the data using the software functions for identification, integration, and quantification. Evaluate the analytical sequence as follows:

11.4.2.1. Check retention times in the hydrocarbon marker mix and in the continuing calibration verification standards. Update retention times as needed. Section 11.2 discusses guidelines for evaluating the significance of a retention time shift during the analytical sequence.

11.4.2.2. Check that CCVs and CCBs were interspersed throughout the sequence at the proper frequency and that acceptance criteria are met. The CCV acceptance criteria are listed in 11.3.4. The CCBs should be free of all target analyte contamination at half of the MRL.

11.4.2.3. Evaluate QC sample results as described in section 12.2.

11.4.3. Evaluation of Sample Analysis

11.4.3.1. Evaluate the surrogate recovery. If the recovery is outside acceptance criteria, reanalysis may be necessary (see 12.2.3.1).

11.4.3.2. Examine solution concentrations of target analytes in the samples. If the concentration is greater than the high calibration standard, reanalyze the sample at a dilution. Dilutions should be performed to keep target analytes in the top half of the calibration range.

11.4.3.3. Check for possible carryover. Pay particular attention to situations where samples containing low levels of target analyte were analyzed one or two runs after samples containing levels of target analyte near the high end of the calibration range. Reanalyze as needed if carryover is suspected.

11.4.3.4. Check peak integrations. All integrations should be performed consistent with integration of the corresponding calibration standards and documented according to the CAS *SOP for Manual Integration of Chromatographic Peaks*. The following guidelines are provided:

- Hydrocarbon ranges are integrated baseline to baseline, dropped to the lowest point in the chromatographic window.

- Once the chromatogram is integrated for the fuel pattern specified, the integrated area for the surrogate and I.S. is subtracted from the total area for the fuel pattern.

In all manual integration situations the analyst must use professional judgment in deciding if the peak exhibits sufficient resolution from interferences to be reported quantitatively or if the MRL should be elevated above the level indicated by the integration. Obtaining a second opinion from an experienced analyst or the supervisor is recommended.

11.4.3.5. This method is primarily used to report Gasoline Range Organics (GRO). It may also be used as a fingerprinting procedure for purgeable hydrocarbon products. Interpretation of GRO fingerprints for EPA Method 8015C, NWTPH-Gx, and AK101 are provided below.

11.4.3.5.1. Gasoline: Hydrocarbon response is characterized as gasoline when the fingerprint matches the fuel pattern exhibited by a gasoline standard. The reported values are flagged with an "F'" qualifier. Specifically, the pattern should show significant amounts of BTEX, trimethylbenzenes, and a distinctive pattern of alkane and aromatic response in the C9 - C12 range. As Gasoline weathers, benzene and toluene are often missing from this fingerprint. However, the distinctive ethylbenzene, m,p-xylene, and o-xylene pattern should be present in addition to the characteristic relationship of peaks eluting in the C9 - C12 range. Once identified, the integration of the chromatogram is based on the method-specified ranges (see Table 6).

11.4.3.5.2. If the chromatographic response cannot be matched to the fingerprint described above, yet shows distinct characteristics of a petroleum product and elutes in the same carbon range as gasoline, report the result as Gasoline and flag the data with a "Y" qualifer. Examples include a highly weathered gasoline fingerprint that can no longer be recognized, or mixtures of hydrocarbon products.

11.4.3.5.3. If the chromatographic response does not resemble a petroleum product, report the result as Gasoline and flag the data with a "Z" qualifier. For volatile range hydrocarbons the best example of this involves descrete peaks (e.g., solvents) with no discernable alkane hydrocarbon fingerprint.

11.4.3.5.4. If the chromatographic response cannot be matched the fingerprint described above, yet shows distinct characteristics of a petroleum product and elutes in the back half of the chromatogram, report the result as Gasoline, flag the data with a "H" qualifier. Examples include mineral spirits, kerosene, and diesel.

11.4.4. Secondary Confirmation of Individual Analytes - Confirmation of hydrocarbon range analytes is not required.

12. QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

Note: The analyst should refer to the CAS *SOP for Sample Batches (ADM-Batch)* and the CAS *SOP Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification (ADM-MDL)*

12.1. Method Performance

12.1.1. Initial Precision and Recovery Validation

12.1.1.1. The accuracy and precision of the procedure must be validated before analysis of samples begins, for each matrix, and whenever significant changes to the procedures have been made. IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability. Initial Precision and Recovery studies should be performed as part of analyst training. Copies of the studies should be maintained in the lab and in the analyst's training file.

12.1.1.2. Perform IPR studies by preparing and analyzing four replicate laboratory control samples spiked at a concentration within the working calibration range. Calculate average percent recovery and relative standard deviation for the four replicate analyses and evaluate as described in EPA SW-846 or by method-specific criteria. Unless specified by the method, acceptance criteria are 70-130% for average percent recovery and 30% RSD.

12.1.2. A method detection limit (MDL) study must be undertaken before analysis of samples can begin or when there is a significant change to the extraction or analytical system. The studies should be performed using preparation procedures appropriate to commonly requested analyses. Copies of MDL studies should be forwarded to the QA Department, and maintained in the lab. Refer to the CAS *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification (ADM-MDL).* The MDL study must be verified annually for each matrix.

12.1.3. Retention time window determinations should be performed with each new column installed. Section 11.2 provides guidelines for establishing retention time windows.

12.2. Ongoing QC Analyses: See Table 8 for minimum method-required QC samples. Project-specified QC requirements may also be given, and take precedence over standard QC.

12.2.1. Refer to Table 9 for the acceptance criteria for QC analyses. These criteria are established based on method requirements or from statistical calculations using

historical results. Unless criteria are required by the method or stated in project-specific data quality objectives, CAS statistical acceptance criteria are used in evaluating QC data. Statistical acceptance criteria are subject to change as periodic updates are made. Refer to the SOP for Control Limits (ADM-CTRL_LIM) for determination of statistical control limits. Based on various method-specifications (AK101, NWTPH-GX, etc.) statistical control limits for LCS recoveries should not exceed the 50-150% recovery range.

12.2.2. Corrective Action: Guidelines for appropriate corrective action when nonconformance or an out of control analysis occurs are provided below and throughout this SOP. A nonconformance is defined as a procedure that is performed contradictory to standard operating procedure (unless stated in project specific data quality objectives). Nonconformances must be documented on a Nonconformance and Corrective Action Report (NCAR).

12.2.3. Evaluate QC sample results as follows:

12.2.3.1. Calculate surrogate recovery as the amount detected in the sample (solution concentration) divided by the true value (solution concentration). If the recovery is outside acceptance criteria, examine the chromatogram for obvious matrix interferences such as high levels of target analyte or coeluting peaks. If the problem is the result of obvious matrix interference, flag the recovery value as outside acceptance criteria due to matrix interference. If no obvious interference is observed, reanalyze (or reextract and reanalyze if deemed more appropriate) the sample. If the recovery is again outside criteria, report the original value and flag the recovery as being outside of acceptance criteria due to matrix interference. Otherwise, report the reanalysis.

12.2.3.2. Check Method Blanks for laboratory contamination. Method Blanks should be free of all target analyte contamination at a level less than half the MRL. In the event that a Method Blank contains target analyte greater than or equal to half the MRL, samples testing positive for that compound at levels less than 20 times the level detected in the Method Blank should be reanalyzed or the data must be flagged to indicate potential laboratory contribution.

12.2.3.3. Check batch QC samples against acceptance criteria. Acceptance criteria are listed in Table 9.

12.2.3.3.1. If matrix spike recoveries are outside acceptance criteria, examine corresponding sample results for potential matrix interferences such as high levels of target analyte. If no obvious matrix interferences are observed, evaluate the recovery of the laboratory control sample (LCS). Reanalyze if deemed appropriate. Do not reanalyze the matrix spikes unless the problem can be traced to analyst error or system failure.

12.2.3.3.2. If LCS recoveries are outside acceptance criteria evaluate the chromatogram for potential purge or injection problems. Reanalyze if appropriate. If reanalysis does not correct the problem, reextract and reanalyze the associated samples. **NOTE: Alaska Method AK101 requires acceptable recovery for the LCS/DLCS pair for associated sample data to be acceptable.**

12.2.3.3.3. Check RPD values on duplicate sample analyses (sample/duplicate or MS/DMS). If values exceed 30% for aqueous samples or 40% for solid and miscible waste samples, examine the chromatograms and bench sheets for potential matrix interferences. Examples include product layers on aqueous samples that may result in non-homogenous subsampling, non-homogenous solid samples, chromatographic interferences resulting in poor peak resolution and inconsistent integrations, inefficient extraction or sample purge in one sample compared to another as indicated by significantly different surrogate recoveries and internal standard response. Reanalyze (or reextract and reanalyze if deemed more appropriate) only those samples in the batch that do not meet surrogate recovery acceptance criteria. Flag RPD values that are outside of acceptance criteria and explain the anomalies in the form of a case narrative.

12.2.3.3.4. Check RPD values on duplicate LCS/DLCS analyses (when applicable). If values exceed 20%, examine the data for obvious problems. Reanalyze if deemed appropriate. If reanalysis does not correct the problem, reextract and reanalyze the associated samples.

13. DATA REDUCTION AND REPORTING

13.1. The concentration of each compound in the sample is determined by calculating the amount of analyte purged using the calibration curve or the calibration factor. Calculations for determining specific analyte concentrations in the sample are described below.

13.1.1. The solution is calculated as:

$$Cx = \frac{Ax}{Cf}$$

Where: C*x* = Solution concentration of the compound (ug/L)
Ax = Response (area) for the compound (or fuel pattern)
Cf = Calibration factor from the curve

13.1.2. To calculate sample concentration of the analyte detected, use one of the calculations below depending on matrix.

Aqueous Samples: $\mathrm{Result}(ug/L) = Cx \times Df$

Where: *Cx*= Solution concentration of the compound (ug/L)
Df = Dilution factor (if no dilution was performed *Df* = 1)

Solid samples (MeOH Extract):

$$\mathrm{Result}(ug/Kg) = Cx \times \frac{(V_{ext} \times V_{Dil} \times Df)}{(ASWt)(Aliquot)(Solids)}$$

Where: *Rsult* = Sample result (ppb)
Cx= Solution concentration of the compound (ug/L)
ASWt = Actual Sample Weight in grams
V_{ext} = Extract Volume (MeOH) in mL*
V_{Dil} = Initial Dilution Volume (DI H2O) in mL
Aliquot = The amount of MeOH extract used in mL
Df = Dilution factor (if no dilution was performed *Df* = 1)
Solids = Percent Solids/100

* The water contained in the native sample is accounted for when determining the final extract volume. The final volume of the methanol extract is the total volume of the methanol/water mixture. Calculate the final volume as follows:

$$Final Volume\ Methanol/Water = mL\ of\ solvent + \left(\frac{\%Moisture\ x\ Sample\ Wt.(g)}{100} \right)$$

13.1.3. Results of extracted solid and waste samples are usually reported in mg/Kg (ppm). The conversion is made by dividing the calculated result (ug/Kg) by 1000.

13.1.4. Apply any necessary flags to the data. AK101 samples that are not field-preserved may only be reported if they are not for compliance purposes. The results for unpreserved AK101 samples should be flagged as not to be used for compliance purposes.

13.2. Data Review

Following primary data interpretation and calculations, all data is peer reviewed by a second analyst. Following generation of the report, the report is reviewed by the department supervisor or a designated analyst trained in performing final review of data packages. Refer to the *SOP for Laboratory Data Review Process* for details.

14. CORRECTIVE ACTION

14.1. Refer to the *SOP for Corrective Action* for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2. Handling out-of-control or unacceptable data

14.2.1. On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, runlogs, for example.

14.2.2. Documentation of a nonconformity must be done using a Nonconformity and Corrective Action Report (NCAR) when: a) corrective action is not taken or not possible b) corrective action fails to correct an out-of-control problem on a laboratory QC or calibration analysis c) reanalysis corrects the nonconformity but is not a procedurally compliant analysis.

15. METHOD PERFORMANCE

15.1. This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available. In addition, this procedure was validated through single laboratory studies of accuracy and precision as specified in Section 12.1.

15.2. The method detection limit (MDL) is established using the procedure described in the SOP for *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification* (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

16. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents, acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvent and reagents used in this method can be minimized when recycled or disposed of properly.

17. WASTE MANAGEMENT

17.1. The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

17.2. This method uses non-halogenated solvents and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and disposed of in accordance with Federal and State regulations.

18. TRAINING

18.1. Training Outline

18.1.1. Review literature by reading references. Review state specific methods for Gasoline Range Organics and Total Petroleum Hydrocarbons as Gasoline. Review the SOP. Also review MSDS for methanol.

18.1.2. Observe the procedure as performed by an experienced analyst at least three times.

18.1.3. Assist in the procedure under the guidance of an experienced analyst. During this training process, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

18.1.4. Following the training period the analyst is expected to complete an initial precision and recovery (IPR) study (see section 12) for both solid and water samples. Summaries of the IPR are reviewed and signed by the supervisor and forwarded to the employee's training file.

18.2. Training is documented following the *SOP for Documentation of Training*.

NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

19. REFERENCES

19.1. EPA Method 5030B, SW-846 Update III. "Purge and Trap", USEPA, December, 1996.

19.2. EPA Method 5035, SW-846 Update III. "Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples", USEPA, December, 1996.

19.3. EPA Method 8015C, Rev 3, SW-846 Update IV. "Nonhalogenated Volatile Organics by Gas Chromatography", USEPA, February 2007.

19.4. Alaska DEC Method AK 101, Contaminated Sites Program, "Method AK101 for the Determination of Gasoline Range Organics", April, 2002.

19.5. California DHS Method TPH-G, Leaking Underground Fuel Tank Field Manual, Appendix C, "Total Petroleum Hydrocarbons as Gasoline", May 1988.

19.6. Analytical Methods for Petroleum Hydrocarbons, "Northwest TPH-Gx", Washington State Department of Ecology, Toxics Cleanup Program and The Ecology Environmental Laboratory, Publication No. ECY 97-602, June 1997.

20. CHANGES SINCE THE LAST REVISION

20.1. Updated EPA reference throughout sop for 8015B to 8015C.
20.2. Sec 1.1 added applicable methods
20.3. Updated Tables 3, 4, 5 and 7
20.4. Sec 6.2 re-written to comply with method 5035 and WA-DOE Memo #5
20.5. The previous section 6.3 is removed
20.6. Sec 11.1 Removed AFCEE batch definition
20.7. Sec 11.3.1.2 removed the step to recalculate curve since included in Cal reports.
20.8. Sec 11.1.1.4 changed narrow range pH paper to wide range.
20.9. Sec 11.1.2.1.2 added LCS/DLCS
20.10. Old sec 11.1.3 removed – direct purge no longer performed
20.11. Sec 11.3 Update reference to SOC-CAL
20.12. Sec 11.3.2 Updated ICV criteria from 15% to 20%.
20.13. Sec 11.3.4.2 Updated CCV criteria for method 8015C.
20.14. Sec 12.1.2 updated to reflect lab standards.
20.15. Sec 12.2.3.3 removed old USACE note. All DoD covered by DoD QSM
20.16. Sec 15.2 updated sop reference

Table 1

Applicable Method References, Target Analytes, Detector of Quanititation, and Calibration Technique for GRO Analyses

Method	Target Analytes	Detector	Calibration
EPA Methods 5030B (or 5035)/8015B	Gasoline Range Organics (GRO)	FID	External
Alaska DEC Method AK 101	Gasoline Range Organics (GRO)	FID	External
NWTPH-Gx	Gasoline Range Organics (GRO)	FID	External
California DHS LUFT Method TPH-G	Gasoline Range Organics (GRO)	FID	External

Table 2

Method Reporting Limits and Method Detection Limits for GRO Analyses

Compound	Aqueous Sample		Solid/Waste Sample	
	MRL (ug/L)	MDL (ug/L)	MRL (mg/Kg)	MDL (mg/Kg)
GRO - 8015B	50	13	5	1.3
GRO - AK101	100	13	20	1.5
GRO - NWTPH-Gx	250	13	5	1.5

Table 3

Suggested Concentration of Calibration Standards (ug/L)*

Compound	Type	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	ICV
4-Bromofluorobenzene	Surrogate	25	30	40	50	100	150	-	NA
1,4-Difluorobenzene	Surrogate	25	30	40	50	100	150	-	NA
Gasoline	Target	50	100	200	500	1000	5000	10000	500

***** Levels 1-5 required for NWTPH-Gx

Table 4

Suggested Purge/Trap and GC Operating Conditions

Event	GC06	GC07
Purge and Trap		
Purge Gas	Helium	Helium
Purge Temperature (°C)	Room Temp.	Room Temp.
Purge Time (min.)	11	11
Dry Purge Time (min.)	1	1
Desorb Temperature (°C)	220	220
Desorb Time (min.)	1.5	1.5
Bake Temperature (°C)	250	250
Bake Time	10	10
GC System		
Carrier Gas	Helium	Helium
Injection Mode	NA	NA
Split Flow	NA	NA
Septum Purge On Time (min.)	NA	NA
Initial Temperature (°C)	40	40
Initial Hold Time (min.)	2	2
Ramp Rate 1 (°C/min.):Temp Break (°C)	12:NA	12:NA
Ramp Rate 2 (°C/min.):Temp Break (°C)	NA	NA
Ramp Rate 3 (°C/min.)	NA	NA
Final Temperature (°C)	165	165
Final Hold Time (min.)	4	4
Inlet Temperature (°C)	250	250
Detector Temperature (°C) (FID/PID)	250/250	250/250
Pressure Program		
Constant Flow (✓)	NA	NA
Constant Pressure (✓)	NA	NA
Initial Pressure (psi)	NA	NA
Pulse Rate (psi/min):Pulse Max (psi)	NA	NA
Pulse Max Hold Time (min.)	NA	NA
Pulse Return Rate (psi/min):Pulse Min (psi)	NA	NA
Pressure Rate (psi/min.)	NA	NA
Pressure Max (psi):Hold Time (min.)	NA	NA
Approximate Cycle Time (min.)	33	31

Table 5

Recommended Spike Amounts

Analyte	Description	Spike Concentration (ug/mL)	Spike Amount (ul)	Notes
Surrogates				
1,4-Difluorobenzene	10 mL dir. purge, dual column (GC06, GC07)	1000	1	Added by the autosampler (nominal amounts)
4-Bromofluorobenzene	MeOH extracts	250	100	Added to MeOH Extract
Aqueous Spikes				
Gasoline	Matrix Spike	1000	21.5	Spiked into a 43 mL VOA
	LCS	1000	25	Spiked into a 50 mL grad cyl
Mid Level Soil Matrix Spikes				
Gasoline	Matrix Spike	5000	100	Added to the MeOH Extract
	LCS	5000	100	Added to the MeOH Extract

Table 6

Method-Specific Hydrocarbon Ranges for GRO Analyses

Method	Starting Marker	Closing Marker
GRO – 8015C	2-Methylpentane (inclusive)	1,2,4-Trimethylbenzene (inclusive)
GRO - AK101	n-Hexane (inclusive)	n-Decane (exclusive)
GRO - NWTPH-Gx	Toluene (inclusive)	Naphthalene (inclusive)
GRO - CA LUFT	2-Methylpentane (inclusive)	1,2,4-Trimethylbenzene (inclusive)

Table 7

Maximum Recommended Analytical Holding Times and Preservatives

Method	Matrix	Preservative	Extraction Holding Time	Analysis Holding Time
EPA 5030B/8015C	Aqueous	pH < 2 with HCL; 4°C	NA	14 Days (collection)
	Solid	4°C	14 Days (collection)	14 Days (collection)
	Waste	None	14 Days (collection)	14 Days (collection)
EPA 5035 (Encore)/8015C	Solid	4°C	Transfer to preservative within 48 hours[a]	14 Days (collection)
EPA 5035 (field preserved)/8015C	Solid[b]	4°C	14 Days (collection)	14 Days (collection)
Alaska DEC Method AK 101	Aqueous	pH < 2 with HCL; 4°C	NA	14 Days (collection)
	Solid (preserved)	Methanol	NA	28 Days (collection)
	Waste	None	14 Days (collection)	14 Days (collection)
NWTPH-Gx	Aqueous	pH < 2 with HCL; 4°C	NA	14 Days (collection)
	Aqueous (unpreserved)	4°C	NA	7 Days (collection)
	Solid[b]	4°C	14 Days (collection)	14 Days (collection)
California DHS LUFT	Aqueous (preserved)	pH < 2 with HCL; 4°C	NA	14 Days (collection)
	Aqueous (unpreserved)	4°C	NA	7 Days (collection)
	Solid	Frozen	14 Days (collection)	14 Days (collection)

a If samples are frozen the holding time is 7 days from collection to analysis.
b Field preserved with Methanol

Table 8

QA/QC Samples by Method

Method	Matrix	Level*	MB	DUP	MS/DMS	LCS	DLCS
EPA 5030B/8015C	Aqueous	Low	X		X	X	
	Solid	Med	X		X	X	
	Waste	Med	X			X	X
EPA 5035 (Encore or Bisulfate)/8015C	Solid	Med	X		X	X	
EPA 5035 (MeOH Extracted)/8015C	Solid	Med	X		X	X	
EPA 5035 (MeOH Preserved)/8015C	Solid	Med	X			X	X
Alaska DEC Method AK 101	Aqueous	Low	X			X	X
	Solid	Med	X			X	X
	Waste	Med	X			X	X
NWTPH-Gx	Aqueous	Low	X	X		X	
	Solid	Med	X	X		X	
	Waste	Med	X			X	X
California DHS LUFT	Aqueous	Low	X		X	X	
	Solid	Med	X		X	X	
	Waste	Med	X			X	X

- Low = Direct Purge; Med = MeOH Extract

Table 9
Data Quality Objectives

QC Parameter	Analytical Method	Aqueous Sample	Solid/Waste Sample
Accuracy		**Recovery**	
Gasoline – Laboratory Control Sample	8015	76-120	63-130
Gasoline – Laboratory Control Sample	NWTPH-Gx	77-122	81-111
Gasoline – Laboratory Control Sample	AK101	60-120[m]	60-120[m]
Gasoline – Matrix Spike	8015	52-139	54-127
Gasoline – Matrix Spike	NWTPH-Gx	71-128	32-154
Gasoline – Matrix Spike	AK101	73-127	64-138
1-4-Difluorobenzene - Surrogate	8015	82-123	NA
	NWTPH-Gx	50-150[m]	NA
	AK101 - Samples	50-150[m]	NA
	AK101 – LCS, CCV	60-120[m]	NA
4-Bromofluorobenzene - Surrogate	8015	NA	65-132
	NWTPH-Gx	NA	50-150[m]
	AK101 - Samples	NA	50-150[m]
	AK101 – LCS, CCV	NA	60-120[m]
Precision		**Relative Percent Difference**	
Gasoline	Matrix Spike Duplicates	30%	40%
Gasoline	AK101 – LCS Duplicates	20%	20%

M – Method required criteria
MD – Method default, pending acquisition of sufficient data points to establish statistical limits.

STANDARD OPERATING PROCEDURE

for

ANALYSIS OF WATER, SOLIDS AND SOLUBLE WASTE SAMPLES FOR SEMI-VOLATILE FUEL HYDROCARBONS

SOP No.: PET-SVF
Revision: 12

Effective Date: June 13, 2011

Approved by: Tom E Portwood — Supervisor — 5/13/11 — Date

Julie Fish — QA Manager — 5/13/11 — Date

Laboratory Director — 5/13/11 — Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
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DOCUMENT CONTROL	
NUMBER:	
Initials:	Date:

ANALYSIS OF WATER, SOLIDS AND SOLUBLE WASTE SAMPLES FOR SEMI-VOLATILE FUEL HYDROCARBONS

1. SCOPE AND APPLICATION

1.1. This Standard Operating Procedure (SOP) describes the method used for analysis of semi-volatile petroleum hydrocarbons using EPA Method 8015C and state-specific methods, as listed in Table 1.

1.2. This SOP describes both the sample preparation and chromatographic procedures used to determine the target analytes in water, soil, sediment and unknown waste (product) matrices. The SOP may be applied to other miscellaneous sample matrices providing that the analyst demonstrates the ability of the SOP to give data of acceptable quality in that matrix. The Method Reporting Limits (MRLs) for target analytes in water and soil are presented in Table 2. Method Detection Limits which have been achieved are also given.

2. METHOD SUMMARY

2.1. The analysis is performed in a way to allow for detection and quantification of semi-volatile fuel hydrocarbons ranging from gasoline through fuel oils and lubricating oils. The SOP is applicable for quantitative analysis of Total Petroleum Hydrocarbons as Diesel, Diesel Range Organics, Motor Oil and Residual Range Organics. The SOP is applicable for semi-quantitative analysis of more volatile petroleum products ranging chiefly from n-C8 through n-C11 (e.g., Gasoline). The SOP is also applicable for qualitative identification of petroleum products ranging from n-C8 through n-C40. Normally only the following analytes are reported under this SOP: Diesel Range Organics (various ranges), Residual Range Organics (various ranges) and C8-C12 Gasoline Range Organics. Other petroleum products are reported (quantitatively, semi-quantitatively or qualitatively) when and as requested by the client.

2.2. Samples are solvent extracted using methylene chloride; the extracts are injected into the GC system using a micro-syringe. Target analytes are separated using a chromatographic column and quantitation is made by comparison of target analyte response to the response of calibration standards over a known concentration range. Integrations are performed over an extended hydrocarbon range. Chromatographic fingerprints are compared to known fuel and other hydrocarbon and petroleum product fingerprints for qualitative identification. Table 3 provides a list of fingerprints maintained on file in the lab. More fingerprints are generated as necessary, from purchased materials and from suspected contaminants often supplied by clients.

2.3. The SOP is applicable to the analysis of target analytes and meets method criteria for the methods listed in Table 1. The SOP may be applied to qualitative analysis and identification of a wide variety of hydrocarbon products.

2.4. The requested target analyte list is analyzed meeting established method criteria for the compounds of interest as set forth in sections 11 and 12 of this SOP. Calibration standards used to quantitate the various target analytes are listed in Table 1.

3. DEFINITIONS

3.1. Analytical Batch: A group of field samples, QC samples, standards and blanks analyzed together under a common and unique identification number. Analytical batches are limited to one continuous analytical sequence.

3.2. Analytical Sequence: A batch of samples analyzed in sequential order. A sequence starts with an initial calibration or a continuing calibration verification standard (CCV) and a system blank, followed by ten samples (inclusive of QC samples), followed by a CCV. The closing CCV for one group of samples may serve as the opening CCV for the next group of samples. This pattern may be repeated until the sequence is complete, or until a continuing CCV fails acceptance criteria. No more than twelve hours are to elapse between successive passing CCVs. Subsequent sample analyses must be initiated with a new calibration or a passing CCV.

3.3. Calibration: Procedure used to quantify analyte amount in the sample.

3.3.1. Continuing Calibration Blank (CCB): System blank analyzed at a routine frequency throughout the sequence to establish that the instrument is free of target analyte contamination. Unless stated in project specific data quality objectives, the system is considered free of target analyte contamination if the level detected is less than the method reporting limit.

3.3.2. Continuing Calibration Verification (CCV): Standard analyzed at a routine frequency throughout the sequence to establish the instrument is meeting calibration criteria.

3.3.3. Independent Calibration Verification (ICV) or Second Source Verification (SSV): Standard analyzed from a source that is different than that used to calibrate the instrument. The ICV is a check on the purity and accuracy of the calibration standards and can be used in preparing matrix spike solutions for spiking of QC samples.

3.4. Extraction Batch: A group of field samples and QC samples prepared together under a common and unique identification number limited to 10 characters. Extraction batches are limited in size to 20 field samples per set of QC samples.

3.5. Hydrocarbon Marker Mix: A solution of individual marker compounds whose retention times are used in defining the window of integration for hydrocarbon ranges, or the retention time of discrete compounds of interest (e.g., pyrene).

3.6. Method Detection Limit (MDL): A statistically derived value representing the lowest level of target analyte that may be measured by the instrument with 99% confidence that the value is greater than zero.

3.7. Method Reporting Limit (MRL): The minimum amount of a target analyte that can be measured and reported quantitatively. The MRL is equivalent to Estimated Quantitation Level (EQL).

3.8. Quality Control (QC) Samples: Samples added to a sample preparation batch, or an analytical batch to provide quality control checks on the analysis. For purposes of evaluating sequencing criteria during an analytical batch, QC samples are defined as samples.

3.8.1. Duplicate Sample (DUP): A laboratory duplicate. The duplicate sample is a separate client sample aliquot that is processed in an identical manner as the client sample proper.

3.8.2. Laboratory Control Sample (LCS): A laboratory blank that has been fortified with target analyte(s).

3.8.3. Laboratory Control Sample/Duplicate Laboratory Control Sample (LCS/DLCS): Laboratory control samples performed in duplicate to evaluate reproducibility within the batch. This QC sample set is a requirement for Alaska Methods AK 102 and AK 103.

3.8.4. Method Blank (MB): A laboratory blank that is used to check that reagents and labware are free of target analytes, and in order to demonstrate that the sample preparation procedure itself does not result in the introduction of analytes or unacceptable interferences. Method Blanks for aqueous samples are prepared using laboratory reagent water. Method Blanks for solid samples are prepared using clean sodium sulfate (baked overnight at 400^{o}C). Method Blanks for soluble waste samples are prepared using solvent (methylene chloride).

3.8.5. Matrix Spike Sample (MS): A duplicate sample aliquot that has been fortified with target analyte(s).

3.8.6. Matrix Spike Sample/Duplicate Matrix Spike Sample (MS/DMS): Matrix spike samples prepared in duplicate.

3.9. Retention Time Window: The time period established within which a target analyte is qualitatively determined to be present in the sample or sample extract.

3.9.1. Hydrocarbon Range: A retention time window that encompasses an extended time period.

3.9.1.1. Hydrocarbon Range Start Time (Inclusive): Absolute retention time of the appropriate marker compound minus 0.1 minute.

3.9.1.2. Hydrocarbon Range Start Time (Exclusive): Absolute retention time of the appropriate marker compound plus 0.1 minute.

3.9.1.3. Hydrocarbon Range End Time (Inclusive): Absolute retention time of the appropriate marker compound plus 0.1 minute.

3.9.1.4. Hydrocarbon Range End Time (Exclusive): Absolute retention time of the appropriate marker compound minus 0.1 minute.

3.10. Surrogate: A non-target compound that is added to all samples and QC samples that is chemically and physically similar to the compounds of interest. The surrogate is used to evaluate the effectiveness of the analysis.

3.11. Target Analyte: A range of compounds of interest which the method is capable of measuring.

3.12. n-Alkane: A straight chain saturated hydrocarbon. The length of the chain is designated with a "C" followed by the number of carbons in the molecule (e.g., C12; the 'n-' is implied in this SOP). Chain length is approximately indicative of molecular weight, boiling point range, and elution order of the analyte. For the purposes of this SOP, all hydrocarbon ranges are based on normal alkanes.

4. INTERFERENCES

4.1. Solvents, reagents and labware may yield artifacts that will cause responses in various hydrocarbon ranges integrated in the SOP. These materials should be checked and cleaned to prevent positive biases to the data. For interpretive analyses, obvious artifacts resulting from contaminated solvents, reagents, and glassware will not be reported as petroleum products.

4.2. Direct exposure of samples and sample extracts to unregulated labware (such as polymeric tubing attached to a laboratory reagent water spigot), personal protective equipment (such as surfactant-coated nitrile gloves) and human contact (such as skin oils and personal hygiene products) generally yields artifacts that will cause responses in various hydrocarbon ranges integrated in the SOP. Such exposures are to be avoided.

4.3. Contamination by carryover can occur when high level samples immediately proceed samples containing significantly lower levels of contamination. This can be minimized by running system blanks after samples that are suspected to contain high levels of target analyte. For purposes of evaluating sequencing criteria during an analytical batch, these system blanks are not defined as samples and therefore are not counted in the ten sample groupings.

4.4. An optional sulfuric acid/silica gel cleanup procedure is provided for removal of chiefly biogenic polar interferences. A state specific (AK 102 and AK 103 tests only), silica gel only cleanup procedure is also provided for, usually to be performed only upon client request.

4.5. This SOP provides for the qualitative identification and quantitative or semi-quantitative measurement of various petroleum products in samples. In practice, a great variety of different organic compounds, many of which are not part of petroleum products, are analyzed. In all cases, all chromatographic responses (exclusive of those due to surrogate compounds) that occur within a hydrocarbon range are reported; a qualitative evaluation of the result is then made and an appropriate Petroleum Hydrocarbon Specific Data Qualifier is added to the reported result.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this SOP. This includes the use of personal protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. This method uses Methylene Chloride, a suspected human carcinogen. Viton gloves should be used while rinsing, pouring or transferring the solvent; if solvent is spilled on the glove surface, remove the glove immediately. Nitrile gloves are more practical for sample preparation but provide only momentary protection; if solvent (especially methylene chloride) is spilled on the glove surface, remove the glove immediately. Latex and vinyl gloves do not provide adequate protection from methylene chloride and should not be worn during procedures involving large quantities of solvent. All procedures involving the transfer of organic solvents should be performed in a fume hood.

In the case of a major solvent or reagent spill (greater than 1 L), evacuate the lab and contact the lab safety officer. Minor spills (less than 1 L) may be absorbed using a spill pillow if the solvent is in an easily accessible area. Do not attempt to clean spills that have infiltrated behind counters and equipment. This should be performed by a trained spill response expert. Contact the safety officer. Document spills in excess of 500 ml in the form of an incident/accident report.

5.4. The use of pressurized gases is required for this SOP. Care should be taken when moving gas cylinders. All cylinders must be secured to a wall or an immovable counter with a chain or a cylinder clamp at all times. Sources of flammable gases (e.g., pressurized hydrogen) should be clearly labeled.

5.5. The proper use of syringes and glass pipettes should be part of employee training. Care should be taken to avoid personal injury as a result of improper handling techniques.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Aqueous Samples should be collected (received) in 500mL glass amber bottles. Samples should be preserved to pH <2 with hydrochloric acid. Samples will be refrigerated to 4 ±2 C upon sample login.

6.2. Solid samples should be collected (received) in glass jars with PTFE-lined septa. Alternatively, soil samples may be received in brass sleeves prepared in the field. Samples will be refrigerated to 4 ± 2°C upon sample login.

6.3. Waste samples such as products can be collected (received) in a variety of sample containers. Typically, glass bottles with PTFE-lined septa are preferred.

6.4. Analytical and Sample Preparation Holding Times: See Table 4 for guidelines on method specific maximum recommended holding times.

7. APPARATUS AND EQUIPMENT

7.1. Balance

7.1.1. Analytical Balance

7.1.2. Top Loading Balance

7.2. Centrifuge

7.3. Gas Chromatography System

7.3.1. Gas Chromatograph (GC): analytical system equipped with gas supplies, column inlet, programmable oven, heated detectors (FID), and a data system for determining peak areas. Hewlett-Packard 5890, Agilent 6890, or equivalent.

7.3.2. Chromatographic Column: Phenomenex ZB-1, 15 m x 0.25 mm ID, 1.0 µm film thickness, or equivalent column of comparable chromatographic performance.

7.3.3. Autosampler Device: Designed for operation in conjunction with the gas chromatographic systems used in this SOP, Hewlett-Packard 7673 or Agilent 7683.

7.3.4. Compressed Gases or Gas Generators: Hydrogen (column flow and flame gas), Air (flame gas), and Nitrogen or Helium (make-up gas).

7.4. Labware and Syringes

7.4.1. Beakers: various sizes (125 ml, 250 ml, 400 ml, 600 ml)

7.4.2. Disposable Test Tubes: 15 ml (100 mm x 10 mm), 20 ml (150 mm x 10 mm)

7.4.3. Disposable Test Tubes Caps: Polypropylene, 10 mm

7.4.4. Disposable Pipettes: Pasteur and serological (1ml, 2 ml, 5 ml, 10 ml)

7.4.5. Erlenmeyer Flasks: 500 ml

7.4.6. Brandtech Pipetters (or equivalent): 50µl, 100µl, 500 µl, and 1000 µl

7.4.7. Fleakers: 1000 ml and 500 ml

7.4.8. Graduated cylinders, 1 liter and 50 mL, Class A, TC

7.4.9. PTFE stoppers: of appropriate size for 50 ml graduated cylinder

7.4.10. Inject Vials: 1.8 ml glass autoinject vials with aluminum crimp tops with PTFE-lined natural rubber and silicone septa

7.4.11. Kuderna Danish (KD) Concentration Units:

7.4.11.1. Flask: 500 ml

7.4.11.2. Keck Clip (blue)

7.4.11.3. Receiving Tube (collector): 15ml

7.4.11.4. Snyder Column: 3 or 2 Ball

7.4.12. Microsyringes: various sizes (10 µl, 25 µl, 100 µl, 250 µl, 500 µl, 1000 µl)

7.4.13. Separatory Funnels: 1 liter, 2 liter

7.4.14. VOA vials (40 ml): capped, with PTFE-Lined Septa

7.4.15. Volumetric Flasks: various sizes (1 ml, 2 ml, 5 ml, 10 ml, 25 ml, 50 ml, 100 ml)

7.4.16. Large stainless steel tablespoon

7.5. N-EVAP Nitrogen Evaporation Unit

7.6. pH Paper: narrow range (0-4 pH units) for measuring aqueous sample pH

7.7. PTFE Filters: leur-locking, 0.45 micron

7.8. S-EVAP Concentration Unit

7.9. Sonication Device

7.9.1. Lab Jack

7.9.2. Sonic Controller

7.9.3. Sonic Horn (4 x 1/4" microtip couple or ¾" tip)

7.9.4. Sonibox

7.10. Stainless Steel Scoopulas

7.11. Ultrasonic Bath

7.12. For AK 102 and AK 103 cleanup: Pre-packed Restek silica gel column: 0.5 g silica gel in a 3 mL total volume, PP barrel with PE frits.

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1. Reagents

Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination. The preparation for all laboratory prepared reagents and solutions must be documented in a laboratory logbook. Refer to the SOP *Reagent/Standards Login and Tracking (ADM-RTL)* for the complete procedure and documentation requirements.

8.1.1. Acids and Bases

8.1.1.1. Hydrochloric Acid, concentrated

8.1.1.2. Sulfuric Acid, concentrated

8.1.2. Glass wool: baked overnight at 280^{o}C.

8.1.3. Reagent Water: Laboratory reagent water that is free of contaminants of interest. The laboratory reagent water is produced by passing water through a series of deionizers followed by activated carbon filters in series.

8.1.4. Sodium Sulfate (Na_2SO_4): Powdered and granular, baked overnight at 400°C.

8.1.5. Solvents: Reagent Grade acetone, hexane and methylene chloride (DCM). Histological grade acetone (for rinsing prior to DCM rinsing only).

8.1.6. Silica Gel: 100-200 Mesh, Baked for 4 hours at 130°C before use.

8.2. Standards: Standards should be labeled as follows: logbook ID number, compounds or compound mix, concentration, solvent, date prepared, expiration date, analyst's initials.

8.2.1. Neat Standard Material: non-fuel standards are purchased from a reliable source (e.g., Chem Service). Neat petroleum product is only routinely used to calibrate for Residual Range Organics (30 weight motor oil, 30/40 weight motor oil mix), but can be used when requested to calibrate for a suspected petroleum product contaminant (e.g., mineral spirits). Neat material used for quantitative analysis is given an expiration date equal to the manufacturer's recommendation, or if no expiration date is provided, the expiration date shall be five years.

8.2.2. Stock Standards: Purchased as solutions from a reliable source (e.g., Supelco) or prepared in high concentration from neat material. Stock standards are generally prepared in methylene chloride. A stock standard is defined as any standard that requires additional dilution prior to being used in the analysis. To prepare stock standards from neat material, accurately weigh the neat material using a four-place analytical balance into a tared volumetric flask. Dilute to volume using methylene chloride. Stopper and invert several times to mix. Transfer the solution to an appropriate storage vial, label and refrigerate. When preparing stock standard mixes from neat material, it is recommended that each component of the mix be prepared individually at high concentration and then combined as a complete mix at a secondary dilution. Stock standards used for quantitative analysis are given an expiration date equal to the manufacturer's recommendation (purchased, sealed solutions) or one year for laboratory prepared stocks and purchased solutions that have been opened for use. The following stock standards are used in this SOP:

8.2.2.1. Hydrocarbon Marker Mix #1: Multi-State hydrocarbon window defining standard from AccuStandard (or equivalent vendor) containing n-Alkanes from C8-C40.

8.2.2.2. Hydrocarbon Marker Mix #2: Purchased or prepared solution containing discrete aromatic and non-aromatic hydrocarbons at a minimum of 10 ppm in methylene chloride. The solution must include Ethylbenzene, *o*-Xylene, *m*-Xylene, *p*-Xylene, 1,2,3-Trimethylbenzene, Fluoranthene, Pyrene, α-Pinene, β-Pinene. It is useful, but not necessary, to include other compounds for characterization of products.

8.2.2.3. Gasoline: Purchased solution containing a three-source composite of gasoline at 50000 ppm (total) in methanol. Alternately, may be purchased at 5000 ppm (total).

8.2.2.4. Kerosene: Purchased solution containing kerosene at 50000 ppm in methylene chloride. Alternately, may be purchased at 20000 ppm.

8.2.2.5. Diesel #2: Purchased solution containing a three-source composite of diesel #2 at 50000 ppm (total) in methylene chloride. Alternately, may be purchased at 20000 ppm (total).

8.2.2.6. SAE 30 Motor Oil: Prepared solution containing 30-weight motor oil at 10000 ppm in methylene chloride.

8.2.2.7. SAE 30/SAE 40 Motor Oil Mixture: Prepared solution containing 30-weight motor oil and 40-weight motor oil at 5000 ppm (each) in methylene chloride.

8.2.2.8. Other petroleum products (e.g., Mineral Spirits, Bunker C) may be purchased at a high concentration from a reliable vendor (usually in methylene chloride), or may be prepared from neat material.

8.2.2.9. Fuel Fingerprints: A wide variety of petroleum and other hydrocarbon products are characterizable by this SOP. Table 3 lists fuel fingerprints currently on file or available for qualitative analysis.

8.2.3. Working Standards: Purchased as solutions from a reliable source (e.g., Supelco) or prepared from stock standards. A working standard is defined as any standard that is used directly in sample analysis. Working standards are prepared from stock solutions by accurately transferring a volume of stock standard into a volumetric flask or graduated cylinder partially filled with the dilution solvent (usually methylene chloride). The volume is further adjusted using the dilution solvent to a known level. Working standards used for quantitative analysis are given an expiration date equal to the manufacturer's recommendation (purchased, sealed solutions), or 6 months for purchased solutions that have been opened and for laboratory prepared solutions.

8.2.3.1. Calibration Standards: A series of standards prepared from a common stock ranging in concentration from a value representing the method reporting limit (or lower) to a value near the high end of the linear calibration range. Calibration standards (and continuing calibration standards) are prepared in methylene chloride. Calibrations will contain a minimum of five concentration levels. Recommended calibration standards are listed in Table 5.

8.2.3.2. Surrogate Solution: Purchased solution (custom from Restek) containing 500 ppm each of *o*-Terphenyl, 4-Bromofluorobenzene, and n-Triacontane in methylene chloride. Since the solution is in a relatively volatile solvent, it is suggested that the working solution be stored in a bottle with a neck that is as narrow as practical, in order to minimize concentration. The solution may also be prepared from neat material.

8.2.3.3. Working Analyte Spike Solution: Using neat products, prepare a solution in methylene chloride containing Diesel #2 at a concentration of 16,000 ppm and 30-weight Motor Oil at a concentration of 8,000 ppm. Since the solution is in a relatively volatile solvent, it is suggested that the working solution be stored in bottles with necks that are as narrow as practical, in order to minimize concentration.

8.2.3.4. Working AK Analyte Spike Solution: Using neat products, prepare a solution in methylene chloride containing Diesel #2 at 16,000 ppm, 30-weight motor oil at 4,000 ppm, and 40-weight motor oil at 4,000 ppm.

8.2.4. Independent Calibration Verification Standard (ICV), also known as Second Source Verification Standard (SSV): A stock standard should be prepared for each analyte from a second source independent of that used in preparing the calibration curve; this is subsequently used to create a working standard to verify the curve accuracy. Recommended ICVs for the various calibration standards used in this SOP are listed in Table 1.

9. PREVENTIVE MAINTENANCE

9.1. A maintenance log will be kept documenting maintenance performed on each analytical system. Log entries will include the date maintenance was performed, symptoms of the problem, serial numbers of major equipment upgrades or replacements (defined as any non-consumable part essential to operation), phone logs from technical support contacts, a description of the maintenance performed, a description of the check performed to assure the system has returned to acceptable levels of operation, and the analyst's initials.

9.2. Autosampler: Maintenance is generally limited to cleaning or replacing dirty, clogged or otherwise malfunctioning syringes, and cleaning other moving components with methanol when necessary.

9.3. GC System: Maintenance of the GC system is generally limited to, in declining order of frequency:

9.3.1. Replacement or cleaning of the injection port liner, or (more frequently) replacement of the glass wool plug therein. The need for this is usually indicated by a significant drop in heavier range product (Motor Oil) and surrogate

(Triacontane) responses without a coincident drop in lighter range product and surrogate responses.

9.3.2. Replacement of the injection port septum. Leaks may occur here.

9.3.3. Replacement or cleaning of the injection port liner (usually gold) seal. The need for this can be indicated by a change in surrogate responses without a coincident or proportionate change in hydrocarbon product responses. Note that the seal usually functions satisfactorily even when obviously dirty in appearance because petroleum products are generally composed exclusively of 'non-polar' components and are thus not significantly affected by active sites; two notable exceptions are brake fluids, which are composed exclusively of glycol ethers, and Fuel Oil #6 (Bunker C), which contains significant quantities of sulfur-containing compounds.

9.3.4. Column trimming. The need for this can be indicated by surrogate peak broadening. Removal of less than two inches of the column at a time is usually sufficient.

9.3.5. Detector cleaning – scrubbing of the detector jet, collector and (less importantly) chimney. The need for this can be indicated by a steady increase or decrease (depending on the nature of the acquisition method) in all responses over time with no other discernable cause. Its need can also be indicated by the appearance of random spikes in chromatograms.

9.3.6. Replacement of ferrules or o-rings (or tightening associated connections). Leaks may occur at these points.

9.3.7. Column replacement. The need for this is usually indicated by significant broadening and diminution of responses of early-eluting peaks (and responses of products), especially at lower concentrations (resulting in quadratic curves). Ready disintegration of the column upon handling is another indication.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in the CAS *SOP for Documentation of Training (ADM-TRANDOC)*, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Cleaning of Labware: Non-disposable labware (except for syringes) used in sample analyses is washed with hot soapy water, rinsed three times with hot tap water, rinsed three times with reagent water, and either allowed to air dry or rinsed with histological grade acetone. Immediately prior to sample preparation, labware must be rinsed with methylene chloride. Syringes are rinsed with the appropriate solvent(s).

11.2. Sample Preparation: A preparation (or extraction) batch is defined as a group of field samples and QC samples processed together with the same reagents. Each preparation batch will consist of no more than 20 field samples. Every preparation batch will include one laboratory method blank (MB) and one laboratory control sample (LCS) at a frequency of not less than one each per 20 client samples. Additional QC sample requirements depend on the methodology (or matrix) employed:

- EPA 8015C: matrix spike (MS) and duplicate matrix spike (DMS) samples are required at a frequency of not fewer than one each per 20 client samples. If insufficient sample is available for a DMS or both MS and DMS, a duplicate laboratory control sample (DLCS) must be prepared at the same frequency.

- AK102/AK103: MS/DMS and DLCS are all required at a frequency of not fewer than one each per 20 client samples.

- NWTPH-DX, including NWTPH-HCID: a duplicate client sample (DUP) is required at a frequency of not fewer than one per 10 client samples (note higher frequency requirement). If insufficient sample is available for a DUP a DLCS must be prepared at a frequency of not less than one per 20 client samples.

- WASTE or PRODUCT dilutions, all methods: a duplicate client sample (DUP) should be prepared with each batch. It is not necessary to prepare a MS or MS/DMS set (but do prepare a DLCS for EPA 8015C and AK102/AK103).

11.2.1. Aqueous Samples: EPA 3510 Liquid-Liquid Extraction

11.2.1.1. Prepare KD extract collection apparatuses: For each sample, tightly fit a 500 mL KD flask with a 15 mL collector, and secure these with a blue Keck clip. Place this assembly in a secure rack. Drop a clean PTFE boiling chip into the collector. Prepare a modified funnel thus: place a small glass wool plug at the bottom of the funnel (rinse the assembly 3 times at this point with methylene chloride, as the glass wool can be a significant source of contamination), and add about 3 or 4 heaping tablespoons of muffled, powdered Na_2SO_4 to the funnel. Place the funnel in the top of the KD flask. Arrange the KD apparatuses in a fume hood.

11.2.1.2.Allow the sample to warm to room temperature and shake to mix. (Note that mixing of the sample may in some cases not be desirable. If a significant portion of the sample volume is solid material, consult with the supervisor or project manager for guidance). Mark the meniscus on the side of the bottle and pour the entire contents of the bottle into a 1L separatory funnel (2L funnel if more than approximately 600 mL of sample was received in one bottle). Save the bottle for solvent rinsing and sample volume determination (11.2.1.5). Laboratory QC (MB, LCS and DLCS) are each prepared from 500 mL (or 1L, if any client samples are greater than approximately 600 mL in volume) laboratory reagent water.

11.2.1.3.Check pH of sample. If sample pH is not < 2, acidify to pH < 2 with concentrated HCl.

11.2.1.4.Add 60 mL of methylene chloride (DCM) to the empty sample bottle, swirl (in order to extract organics adhering to the bottle), and then pour the mixture into the funnel containing the sample; for laboratory QC, add 60 mL directly to the funnel.

11.2.1.5.Determine the sample volume by filling the sample bottle with water to the marked meniscus. Transfer the contents of the bottle to a graduated cylinder to measure the volume of the sample prepared. Record the volume prepared to at least 2 significant figures.

11.2.1.6.Add the working surrogate spike to all client and QC samples. Add the working analyte (matrix) spike to all MS, DMS, LCS and DLCS samples. Use the working AK analyte (matrix) spiking solution for the AK103 method.

11.2.1.7.Stopper and shake the funnel for 1-2 minutes with periodic venting. Allow the solvent layers to separate.

11.2.1.8.Filter the extract into a KD apparatus then rinse the top of the Na_2SO_4 bed with approximately 20 mL of DCM.

11.2.1.9.If an unresolvable emulsion is encountered, first drain the emulsion into another vessel for mechanical resolution, preliminary drying employing granular Na_2SO_4, or centrifugation, followed by pipetting off of aqueous layer; then collect the DCM layer as in 11.2.1.7. It is sometimes helpful to place 2-4 heaping tablespoons of muffled, granular Na_2SO_4 on top of the powdered Na_2SO_4 bed to help resolve some less severe emulsions.

11.2.1.10.Repeat the extraction (steps 11.2.1.7 through 11.2.1.9) twice more in sequence, starting each with a fresh 60 mL portion of DCM added directly to the sample in the separatory funnel. Combine the 3 extracts in the same

KD apparatus for concentration. A vacuum pump may be employed after the third extraction to maximize extract collection in the KD apparatus.

11.2.1.11. Remove the modified funnel from the KD and fit the KD with a Snyder column. Concentrate the extract by means of a hot water bath (S-Evap) at 70°C-75°C. Remove the KD apparatus from the bath when the apparent solvent level in the collector is approximately 5 mL. Do not allow the extract in the collector to evaporate to dryness.

11.2.1.12. Carefully remove the collector from the KD and further concentrate the extract, using a gentle N_2 stream and < 40°C water bath (N-Evap), to 1 mL in DCM (if the sample volume was approximately 500 mL; 2 mL if sample volume was approximately 1L). Again, do not allow the extract to evaporate to dryness. If the extract is particularly dark, viscous and/or obnoxious smelling, it may be advisable to take the extract to a higher final volume. A graduated, disposable pipet or syringe may be used to measure the final volume.

11.2.1.13. Place 1 mL of extract in a 1.8 mL autosampler vial and cap tightly with a PTFE-lined, silicone septum (natural rubber septa introduce irremovable, MDL/MRL level artifacts into the extract upon injection, and should therefore be avoided for extracts not known to have relatively high concentrations of extractable organics). Archive any remaining extract in a capped, disposable test tube.

11.2.1.14. Mark the extract meniscuses if necessary. Complete paperwork, taking care to note all problems, difficulties or deviations from the standard operating procedure.

11.2.2. Solid Samples: Sonication

11.2.2.1. Prepare and arrange KD extract collection apparatuses as in 11.2.1.1.

11.2.2.2. Thoroughly mix the sample. Discard large rocks, sticks, leaves and other organic matter unless they are the primary make-up of the sample.

11.2.2.3. Weigh 30 g of soil into an appropriately sized beaker: a 250 mL beaker is usually large enough for a sample consisting of at least 80% total solids; for wetter samples, or for samples with much organic matter, a 400 mL or 600 mL beaker is usually necessary. Less sample may be prepared if the sample obviously contains high levels of petroleum product, and a homogeneous mixture can be attained. Muffled laboratory matrix sand is employed as the LCS/DLCS matrix; no matrix is employed for the MB (except a typically employed amount of muffled Na_2SO_4, which is added to all samples). Record weight prepared to at least 3 significant figures.

11.2.2.4.Add approximately 30 g muffled, powdered Na_2SO_4 to the sample in the beaker, stir the mixture thoroughly with a scoopula until the mixture is dry and free flowing, and no material adheres to the beaker or scoopula. More Na_2SO_4 may be added as necessary. Some muffled, granular Na_2SO_4 may be employed in order to facilitate mixing. After initial mixing, it will be necessary to allow the mixture to sit at room temperature for at least 15 minutes (often longer), followed by further mixing, in order to ensure that the Na_2SO_4 has been allowed to dry the sample as effectively as possible.

11.2.2.5.Tune the sonic horn controller(s) in accordance with the manufacturer's instructions and record the action on the benchsheet.

11.2.2.6.Arrange the sample mixtures in a fume hood and keep them in the hood during the following steps (11.2.2.7 through 11.2.2.14) in order to minimize personal solvent exposure. Add working surrogate spike to all client and QC samples. Add working analyte (matrix) spike to all MS, DMS, LCS and DLCS samples.

Note: The AK103 methods require the use of the working analyte matrix spiking solution described in sec 8.2.3.4.

11.2.2.7.As soon as practicable, add approximately 100 mL of DCM to the mixture. Add more or less solvent as necessary, such that the solid mixture is immersed under at least 2 cm of solvent. Since DCM is quite volatile, it will be necessary to cover those sample mixtures (with Al foil or watch glasses, e.g.) which are not immediately taken to the next step in order to minimize evaporation and consequent exposure.

11.2.2.8.Place the sonic horn tip in the solvent layer above the top of the solid mixture and sonicate for 3 minutes at a 2 second pulse interval.

11.2.2.9.Decant as much of the extract as possible off the solid mixture into a KD apparatus.

11.2.2.10.Rinse the top of the Na_2SO_4 bed in the funnel (and whatever solid mixture was transferred with the extract) with approximately 20 mL of DCM.

11.2.2.11.Repeat the extraction (steps 11.2.2.8 through 11.2.2.10) twice more in sequence, starting each with a fresh portion of DCM sufficient to cover the solid mixture under at least 2 cm of solvent. Combine the 3 extracts in the same KD apparatus for concentration.

11.2.2.12. After the third decantation, and before rinsing the Na_2SO_4 bed, transfer the entire solid matrix (or as much as will fit) to the modified funnel (a stream of DCM squirted into the beaker as it is held over the funnel is usually sufficient to accomplish the transfer); then thoroughly rinse the mixture in the funnel with approximately 30 mL of DCM. A vacuum pump may be employed to maximize extract collection in the KD apparatus.

11.2.2.13. Remove the modified funnel from the KD and fit the KD with a Snyder column. Concentrate the extract by means of a hot water bath (S-Evap) at 70°C-75°C. Remove the KD apparatus from the bath when the apparent solvent level in the collector is approximately 5 mL. Do not allow the extract in the collector to evaporate to dryness.

11.2.2.14. Carefully remove the collector from the KD and take the extract to 10 mL in DCM in a disposable test tube. If the extract is particularly dark, viscous and/or obnoxious smelling, it may be advisable to take the extract to a higher final volume. If further concentration of the extract is necessary, use a gentle N_2 stream and < 40°C water bath (N-Evap). Again, and more importantly at this point, do not allow the extract to evaporate to dryness. A graduated, disposable pipet is used to measure the final volume.

11.2.2.15. Place 1 mL of extract in a 1.8 mL autosampler vial and cap tightly with a PTFE-lined, silicone septum (see note about natural rubber septa in 11.2.1.13). Cap and archive the test tube containing the balance of the extract.

11.2.2.16. Mark the extract meniscuses if necessary. Complete paperwork, taking care to note all problems, difficulties or deviations from the standard operating procedure.

11.2.3. Extract Cleanups (for removal of mostly biological interferences)

Note: If a sample cleanup is performed, a portion of each of all associated laboratory QC and common client QC must also undergo the same cleanup.

11.2.3.1. Sulfuric Acid/Silica Gel (for EPA 8015C and NWTPH methods):

Note: if an auto-pipettor or other dispenser is used to dispense H_2SO_4, it will be necessary to draw off and discard the first 2-3 mL of the acid before adding any to extracts. Failure to do so will result in poor results for the first extract cleaned up.

11.2.3.2. Measure a volume (usually 1 or 2 mL) of the extract at final volume into a disposable test tube. Note: if the extract is not in DCM, it will be necessary

to exchange the portion to be cleaned up by this method into DCM before proceeding further.

11.2.3.3. Add 2-3 mL of concentrated H_2SO_4. Tightly cap the test tube and vigorously but carefully vortex the mixture for approximately 15 seconds.

11.2.3.4. Centrifuge the mixture (at least 200 G for 5 minutes is recommended) in order to maximize separation of the layers; the extract will be the top layer.

11.2.3.5. Add 1-2 g activated silica gel to a separate test tube (cooling to room temperature, if directly from oven). Carefully (avoiding the H_2SO_4) draw off as much of the extract layer as practicable from the acid layer in the H_2SO_4-cleanup tube and transfer it to the silica gel-cleanup tube.

11.2.3.6. Tightly cap the test tube and gently swirl the contents. Allow the mixture to sit for at least 30 minutes at room temperature.

11.2.3.7. Carefully (avoiding the silica gel) transfer an aliquot of the extract to a 1.8 mL autosampler vial. Cap the vial tightly with a PTFE-lined, silicone septum (see note about latex septa in 11.2.1.13).

11.2.3.8. Mark the extract meniscus if necessary. Complete paperwork, taking care to note all problems, difficulties or deviations from standard operation procedure.

11.2.4. Silica Gel (ADEC Technical Memorandum 06-001 for AK102/AK103):

11.2.4.1. **Note**: Alaska DEC does not permit an acid clean-up step.

11.2.4.2. **Note**: Alaska DEC requires both pre- and post-silica gel cleanup results be provided. The extract(s) must be analyzed per the standard AK102/AK103 methods, silica gel cleanup performed, re-analyzed and both results reported. Therefore, LIMS test codes AK_DRO_SGT or AK_RRO_SGT mean that both the non-SGT and SGT treated analyses will be reported.

11.2.4.3. **Note**: DO NOT HURRY.

In general, **NO VACUUM OR PRESSURE** is required to elute solvent or sample extracts through the pre-packed columns employed in this method; gravity is sufficient (fast enough).

NO VACUUM OR PRESSURE should be used unless the column is clearly blocked by sample extract matrix (the eluent drip rate is less than 5 drops/minute): if means other than gravity are used in order to effect elution, **THE RATE OF DRIPPING MUST NEVER EXCEED 10 DROPS/MINUTE.**

FINALLY: if the extract is not in DCM, it will be necessary to exchange the portion to be cleaned up by this method into DCM before proceeding further.

11.2.4.4. Place a pre-packed Restek silica gel column (0.5 g silica gel in a 3 mL total volume PP barrel with PE frits) in a disposable, 150 mm x 10 mm (length x diameter) test tube (the waste collection vessel). The lip at the top of the barrel will allow it to sit at the top of the test tube without falling in.

11.2.4.5. Precondition the column with at least 12 mL DCM, draining eluent to waste: **DO NOT FORCE THE SOLVENT THROUGH THE COLUMN;** allow the solvent to drip under the force of gravity **ONLY**. After the last of the DCM has eluted (the PE frit atop silica gel can go dry; it takes about a minute for the silica gel itself to start drying), remove the column from the waste collection vessel and place it in an extract collection vessel (another 150 mm x 10 mm test tube).

Note: The entire silica gel bed at this point should have a translucent appearance, and it should also appear darker than when new.

11.2.4.6. Introduce exactly 0.50 mL extract to the column, collecting the eluent. Allow the PE frit to just go dry.

11.2.4.7. Add approximately 1.5 mL DCM to column, collecting the eluent. Allow the PE frit to just go dry.

11.2.4.8. Repeat step 11.2.4.4. Stop collecting eluent once it stops dripping from column: **UNDER NO CIRCUMSTANCES SHOULD THE COLUMN BE FORCED TO DRYNESS** by vacuum or pressure. Remove the column from the extract collection vessel: there will still be some solvent adhering to column exit. This is normal, and its presence does not imply the loss of any desired compounds of interest.

11.2.4.9. Gently concentrate the now cleaned-up (approximately 3.5 mL) extract, using a gentle N_2 stream and < 40°C water bath (N-Evap), to 0.50 mL in DCM, and transfer to a 1.8 mL autosampler vial. Do not allow the extract to evaporate to dryness. Cap the vial tightly with a teflon-lined, silicone septum (see note about natural rubber septa in 11.2.1.13).

11.2.4.10. Mark the extract meniscus if necessary. Complete paperwork, taking care to note all problems, difficulties or deviations from standard operation procedure.

11.2.5. TCLP leachates are prepared as aqueous samples. Prepare a leachate blank in addition to the laboratory blank.

11.3. Gas Chromatography

11.3.1. See Table 6 for suggested GC operating conditions. Samples are analyzed as part of an analytical sequence. Sequencing guidelines are described in section 3.2. Sequences are programmed in the GC operating software and run on user command. Prior to initiating an analytical sequence, check that gas supplies and autosampler rinse cup reservoirs are adequate to last the entire sequence.

11.3.2. Establish retention time windows for hydrocarbon ranges. For hydrocarbon range analytes, start and end times are established by analyzing a marker solution containing individual compounds that define the quantitation range. See Table 7 for guidance on establishing windows for the various method specified ranges. The hydrocarbon marker mix #1 (alkane mix) should be analyzed at least once every 24 hours to verify no significant retention time shift has occurred with respect to start and end times for the quantitation ranges. The hydrocarbon marker mix #2 (aromatic mix) should be analyzed at least once every 24 hours.

11.3.3. Method 8015C also describes background subtraction that should be performed, as follows: "Because the chromatographic conditions employed for DRO analysis can result in significant column bleed and a resulting rise in the baseline, it is appropriate to perform a subtraction of the column bleed from the area of the DRO chromatogram. In order to accomplish this subtraction, analyze a methylene chlorine blank during each 12-hour analytical shift which samples are analyzed for DRO. Measure the area of this chromatogram in the same fashion as is used for samples by projecting a horizontal baseline across the retention time range for DRO. Then subtract this area from the area measured for the sample and use the difference in areas to calculate the DRO concentration". Automated software functions (e.g. the instrument's column compensation feature) may be used to perform this column bleed subtraction from a blank run.

11.3.4. Calibration

NOTE: The calibration procedure(s) and options chosen must follow the CAS protocols. Any exceptions to the calibration procedures detailed in the CAS SOP for *Calibration of Instruments for Organics Chromatographic Analyses* are described as follows:

11.3.4.1. Average of Response Factor: Calibration factors are calculated as the total area of the peak or integration range divided by the amount injected. If the percent relative standard deviation (%RSD) of all points is less than 20% (25% for Alaska Methods AK 102 and AK 103), linearity through the origin may be assumed and the average calibration factor may be used in quantifying sample data.

***Note:** For fuel analytes the integrated area for the surrogate compound is subtracted from the total area for the fuel pattern. This is done to eliminate the contribution of the surrogate area within the fuel range. The Enviroquant software (in use as of this writing) automatically performs this operation.

11.3.4.2. Calibration Curve, Linear Regression: This option may be used if the correlation coefficient is greater than or equal to 0.995 ($r \geq 0.995$, $r^2 \geq 0.990$). This method of quantitation uses the equation of a line ($y=mx+b$). It will generally provide an acceptable fit when %RSD is < 30% and is therefore a useful option in evaluating the acceptability of the calibration curve for generating quantitative values.

11.3.5. Evaluation of calibration curves for use in quantitative analysis should be performed in the following manner.

11.3.5.1. If %RSD for the compound is less than 20% (25% for Alaska Methods AK 102 and AK 103) use average of response factor from the calibration. If %RSD exceeds this value but is less than 30%, plot a linear regression.

11.3.5.2. For the NWTPH method: the measured result of every calibration standard employed in the initial calibration must be within 15% of its true value. For the 8015C method, the measured result of the lowest concentration calibration standards used must be within 30% of its true value; other calibration standards must be within 20% of the true value.

11.3.5.3. If these guidelines are exceeded, obtain a secondary opinion from a senior analyst or supervisor. The calibration may need to be reanalyzed.

11.3.6. Verify the calibration by analyzing an independent calibration verification standard. Acceptance criteria for the ICV are ±20% (25% for Alaska Methods AK 102 and AK 103; 15 % for NWTPH).

11.3.6.1. Analyze a CCB to establish that the instrument is free of target analyte contamination.

11.3.6.2. The first set of 10 samples in an analytical sequence may be analyzed immediately following an acceptable calibration and analysis of a CCB. This set of samples is bracketed with continuing calibration verification (CCV) standard. This pattern is continued until the sequence is complete, or a CCV fails to meet acceptance criteria described below.

11.3.6.3. Verify the calibration each working day by the analysis of a CCV (mid-level concentration of the calibration curve). A mid-level standard (CCV)

must be injected at the start of each sequence and after each set of sample extracts (every 10 samples or every 12 hours, whichever is first) in the analysis sequence. Table 8 lists method specific criteria for evaluating the acceptability of a CCV. See the SOP for *Calibration of Instruments for Organic Chromatographic Analyses* (SOC-CAL) for allowable exceptions.

11.3.7. Evaluation of the Analytical Sequence

11.3.7.1. Check that CCVs and CCBs were interspersed throughout the sequence at the proper frequency. Nonconformances must be documented and obtain supervisory approval before data is accepted.

11.3.7.2. Check retention times in the hydrocarbon marker mix #1 (alkane mix). Update retention times as needed

11.3.7.3. Check that CCVs and CCBs are acceptable. CCV acceptance criteria are described in Table 8. CCBs should be free of all target analyte contamination at a level less than the MRL.

11.3.7.4. Check Method Blanks for potential extraction batch contamination. Extraction Method Blanks should be free of all target analyte contamination at a level less than the MRL. In the event that a Method Blank contains target analyte greater than or equal to the MRL, samples testing positive for that compound at levels less than 20 times the level detected in the Method Blank should be reanalyzed or the associated data must be flagged to indicate potential laboratory contribution.

Note For DOD Projects: If the Method Blank contains target analyte(s) greater than or equal to ½ the MRL, the associated samples, where the analyte(s) are detected at a concentration greater than or equal to the LOD and is less than 10 times the amount detected in the Method Blank, require re-analysis or the data must be flagged with appropriate qualifiers.

11.3.7.5. Check Method Blank and LCS surrogate recoveries. Surrogate recoveries for MBs and LCSs are used to make inferences about associated environmental samples. Low recoveries in the MB and/or LCS constitute an out-of-control event for the entire batch of samples and all samples in the batch testing positive for any target analyte should be re-analyzed or the data must be flagged to indicate potential laboratory contribution. High recoveries in the MB and/or LCS show a potential high bias. However, if all target analyte concentrations (in associated samples) are below the MRL, no corrective action is required other than flagging the high surrogate recoveries in the analytical report.

11.3.7.6.Check extraction batch QC samples against acceptance criteria. Acceptance criteria are listed in Table 9.

11.3.7.6.1.In order to report the associated sample results, all LCS recoveries must be within acceptance criteria. If the LCS recovery is outside the criteria due to chromatographic problems (injection problem, etc.) take the necessary corrective action and reanalyze. If reanalysis does not correct the problem or if the LCS recovery is outside the criteria due to extraction/preparation failure, re-extract and reanalyze the associated samples.

11.3.7.6.2.If matrix spike recoveries are outside acceptance criteria, examine corresponding sample results for potential matrix interferences such as high levels of target analyte (spike levels should be a minimum of 4 times the background concentration). If no obvious matrix interferences are observed, evaluate the recovery of the laboratory control sample (LCS). Reanalyze if deemed appropriate. Do not re-extract the matrix spike unless required by the project.

11.3.7.6.3.Check RPD values on duplicate sample analyses (sample/duplicate or MS/DMS). If values exceed the criteria, examine the chromatograms, bench sheet comments and the actual sample for potential matrix interferences. Examples include product layers on aqueous samples that may result in non-homogenous sub-sampling, severe emulsions, non-homogenous solid samples, inefficient extraction in one sample compared to another as indicated by significantly different surrogate recoveries. Flag RPD values that are outside of acceptance criteria and explain the anomalies in the form of a case narrative. Reanalysis and/or re-extraction and reanalysis may be required, see supervisor.

11.3.7.6.4.Check RPD values on duplicate LCS/DLCS analyses (when applicable). If values exceed criteria, examine the data for obvious problems. Reanalyze one or the other LCS if deemed appropriate. If reanalysis does not correct the problem, re-extract and reanalyze the associated samples.

11.3.8. Evaluation of Sample Analysis

CAS acceptance criteria are established based on either the method criteria or on control charting of analytical results. Unless specific criteria are required by the method, or stated in project specific data quality objectives, CAS-generated criteria will be used in evaluating acceptability of the analysis. The acceptance criteria for

surrogate recovery, matrix spike recovery, and relative percent difference on duplicate analyses are listed in Table 9.

11.3.8.1. Calculate surrogate recovery as the amount detected in the sample (solution concentration) divided by the true value (solution concentration). If surrogate recovery is outside acceptance criteria examine the chromatogram for obvious matrix interferences such as high levels of target analyte or co-eluting peaks. If the problem is the result of obvious matrix interference, flag the recovery value as outside acceptance criteria due to matrix interference. If no obvious interference is observed, reanalyze (or re-extract and reanalyze if deemed more appropriate) the sample. If surrogate recovery is again outside criteria (or acceptable but within 5% of the primary analysis), report the original value and flag the recovery as being outside of acceptance criteria due to matrix interference. Otherwise, report the reanalysis.

11.3.8.2. Examine solution concentrations of target analytes in the samples. If the concentration is greater than the high calibration standard, reanalyze the sample at a dilution. Preferably, dilutions should be performed to keep target analytes in the top half of the calibration range.

11.3.8.3. Check for possible carryover. Pay particular attention to situations where samples containing low levels of target analyte were analyzed one or two runs after samples containing levels of target analyte near or above the high end of the calibration range. Reanalyze as needed.

11.3.8.4. Check integrations. Where possible, all integrations should be performed consistent with integration of the corresponding calibration standards. Hydrocarbon ranges are integrated baseline to baseline, dropped to the lowest point in the chromatographic window. The integration software currently in use is usually capable of performing the integrations (and subtracting any responses contributed by added surrogates) satisfactorily.

11.3.8.5. Perform qualitative evaluation of the chromatographic fingerprint. This is done by comparing the fingerprint of the sample to the two marker mixes and the fingerprint library. The marker mixes are used to assess qualitatively the presence of common fuel components. The qualitative evaluation is used to report specific hydrocarbon products in the interpretive analyses (EPA Method 8015C) and to qualitatively flag the results for hydrocarbon range analyses. The following guidelines are provided:

- Gasoline is present if ethylbenzene, xylenes, and 1,2,3-trimethylbenzene, and a small hump of unresolved responses eluting about and after 1,2,3-trimethylbenzene are observed. Ethylbenzene and

the xylenes should have the same pattern as the calibration standard (i.e. relative peak height to each other). Weathered gasoline is indicated by relative ethylbenzene and xylene responses that are different than in the calibration standard.

- Diesel is present if the fingerprint shows a recognizable n-alkane pattern that includes the isoprenes pristane and phytane, and a large hump of unresolved responses that elute from C10-C23 with its apex at about C15. 1,2,3-trimethylbenzene is usually distinguishable. Weathered diesel is indicated by (1) pristane/C17 and phytane/C18 response ratios greater than those in the calibration standard, (2) the later shifting of the unresolved response hump relative to that in the standard (yet pristane and phytane are observed) or (3) the presence of pristane and phytane with the lack of any recognizable n-alkanes. In the latter case, the unresolved hump is typically later eluting than in the standard, but should still be primarily within the diesel window; if most of the hump is outside the diesel window, the responses may be due to another fuel oil.

- Bunker C is present if the fingerprint shows a recognizable n-alkane pattern that includes pristane, phytane and contains fluoranthene and pyrene in approximately the correct ratio for Bunker C. Note that the aromatic compounds are more susceptible to weathering than the isoprenes, yet less susceptible to weathering than the n-alkanes.

- Lubricating range oil is present if the responses are chiefly a large hump of unresolved responses that resembles that of the 30-wt. Motor Oil calibration standard and usually elutes in approximately the same or heavier carbon range as that of the hump in the standard. The humps of lubricating oils of higher viscosity than the motor oil calibration standard elute later than that of the standard while those of lighter viscosity elute earlier, but still mostly within the motor oil range.

- Hydraulic fluids fingerprints resemble that of motor oil, and can indeed resemble 30-wt. motor oil in appearance and retention times, but often elute earlier, mostly in the latter half of the diesel range, and sometimes possess more than one, yet unresolved, hump (a large hump with a large shoulder).

- Transformer oil fingerprints also resemble that of motor oil, and are typically one simple hump, but elute earlier than hydraulic fluids, entirely within the early to middle regions of the diesel range.

- Turpentine is present if alpha pinene and beta pinene are observed in the chromatogram, in high concentrations (especially alpha pinene) relative to other extracted organics in the approximate elution range.

- Creosote is present if the chromatographic responses are due chiefly to PAHs, with many resolvable yet unidentifiable individual responses from C12-C36.

- Mineral spirits elute in a very short window approximately between C8-C14 with, in a relatively unweathered sample, the top of the hump centered between C10-C11, and the individual n-alkanes between C9-C12 especially prominent.

- Kerosene (and Jet A fuel; the two are chromatographically indistinguishable) is present if the fingerprint shows a recognizable n-alkane pattern that includes the isoprenes pristane and phytane (though all components eluting this late are at very low relative concentration and thus may not be observed if the concentration is relatively low), and a large hump of unresolved responses that elute from C8-C17 with its apex at about C12. 1,2,3-trimethylbenzene is more prominent than in diesel.

- Military jet fuels: Fingerprints can vary from site to site (and from supplier to supplier), and can have additives not present in kerosene, specifically ethylbenzene and xylenes. JP-5 tends to resemble kerosene, eluting at a slightly (extent can vary) earlier retention time; pristane/C17 and phytane/C18 are just distinguishable if the concentration is high enough. JP-4 resembles kerosene much less, and elutes at a much earlier retention time; pristane/C17 and phytane/C18 are not usually observed. JP-8 tends to resemble kerosene, eluting within the same window but with a flatter appearing hump; pristane/C17 can be observed but phytane/C18 usually cannot be observed.

- Weathered products: All fuels generally demonstrate weathering characteristics similar to those enumerated above for diesel. As a fuel is weathered, (1) its hump tends to shift later in the chromatogram, due to evaporation, (2) individual, semi-resolved, peaks in the hump become less resolved (the hump looks smoother) due to microbial weathering, (3) n-alkanes become much less prominent or disappear, also due to microbial action, and (4) individual marker compounds become more prominent relative to the total chromatographic responses due to their greater resistance to microbial weathering. For the purposes of this SOP (that is, among those marker compounds identifiable in this SOP), survivability of compounds increase in the following order:

1. n-alkanes < aromatics < branched alkanes

2. branched alkanes include the isoprenes pristane and phytane.

- Client samples often contain mixtures of many different petroleum products. Interpretation of petroleum product mixtures includes a significant amount of analytical judgment and should be confirmed by an analyst experienced with interpretation or the supervisor.

Note: Alaska DEC requires both pre- and post-silica gel cleanup results be provided. The extract(s) must be analyzed per the standard AK102/AK103 methods, silica gel cleanup performed, re-analyzed and both results reported. Therefore, LIMS test codes AK_DRO_SGT or AK_RRO_SGT mean that both the non-SGT and SGT treated analyses will be reported.

12. QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

12.1. Initial Precision and Recovery Validation

12.1.1. The accuracy and precision of the procedure must be validated before analysis of the samples begins, or whenever significant changes to the procedures have been made. IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

12.1.2. Perform IPR studies by preparing and analyzing four replicate laboratory control samples spiked at a level of 1 to 4 times the MRL. Calculate average percent recovery and relative standard deviation for the four replicate analyses. Unless specified by the method, acceptance criteria are the control limits used for LCS percent recovery and % RSD.

12.1.3. Calculate the average concentration found (x) in µg/mL, and the standard deviation of the concentrations (s) in µg/mL for each analyte. Calculate the MDL for each analyte. Refer to the CAS *SOP Performing Method Detection Limit Studies and Establishing Limits of detection and Quantification (ADM-MDL).* The MDL study must be verified annually.

12.2. Limits of Quantification (LOQ)

12.2.1. The laboratory establishes a LOQ for each analyte as the lowest reliable laboratory reporting concentration or in most cases the lowest point in the calibration curve which is less than or equal to the desired regulatory action levels, based on the stated project requirements. Analysis of a standard or extract prepared at the lowest point calibration standard provides confirmation of the established sensitivity of the method. The LOQ recoveries must be within laboratory established acceptance range to verify the data reporting limit. Refer to the CAS SOP *Performing Method*

Detection Limit Studies and Establishing Limits of detection and Quantification (ADM-MDL)

12.3. The Method Reporting Limits (MRLs) used at CAS are the routinely reported lower limits of quantitation which take into account day-to-day fluctuations in instrument sensitivity as well as other factors. These MRLs are the levels to which CAS routinely reports results in order to minimize false positive or false negative results. The MRL is normally two to ten times the method detection limit.

12.4. Ongoing QC Samples required are described in the CAS-Kelso Quality Assurance Manual and in the SOP for Sample Batches. Additional QC Samples may be required in project specific quality assurance plans (QAPP). For example projects managed under the DoD ELAP must follow requirements defined in the DoD *Quality Systems Manual for Environmental Laboratories.* General QA requirements for DoD QSM are defined in the laboratory SOP, Department *of Defense Projects – Laboratory Practices and Project Management (ADM-DOD).* General QC Samples are:

12.4.1. QA/QC Sample Analysis: Each sample preparation batch will contain a maximum of 20 field samples, one Method Blank, and one Laboratory Control Sample. Additional QC analyses are performed at a rate of one set per 20 field samples. Unless stated in project specific data quality objectives, the standard QA/QC sample set for aqueous and solid matrices is Matrix Spike/Duplicate Matrix Spike. A Duplicate client sample is required for the NWTPH method at a rate of no less than one per 10 field samples. A Duplicate Laboratory Control Sample is required for Alaska Methods AK 102 and AK 103, and when the appropriate Matrix Spike/Duplicate Matrix Spike or Duplicate client sample requirements cannot be met (due to limited sample volume). The standard QC sample set for miscible waste samples is a Duplicate client sample.

12.4.2. QC Analyses: The acceptance criteria for QC analyses are listed in Table 9. The acceptance criteria are established based on method-required criteria or from control charting of analytical results. Surrogate recoveries for new compounds or analyses not charted are set at 70-130%. Unless criteria are required by the method or stated in project specific data quality objectives, CAS criteria determined from historical results will be used in evaluating acceptability of the analysis.

12.4.3. Section 11 provides guidelines for corrective action when QC sample recoveries are outside acceptance criteria.

13. DATA REDUCTION AND REPORTING

13.1. External Standard Calibration: The concentration of each compound in the sample is determined by calculating the amount of analyte injected using the calibration curve or the

calibration factor. Calculations for determining specific analyte concentrations in the sample are described below.

Calibration Factors

For external calibration, the calibration factor is calculated as:

$$Cf = \frac{Rx}{Ax}$$

Where: *Cf* = Calibration factor
Rx = Response of the analyte (area)
Ax = Concentration of analyte injected (ug/mL).

Solution Concentration of Analyte Detected

Results based on average of response factor or a linear regression is calculated as:

$$Csol = \frac{Rcmpd}{Cf}$$

Where: *Csol* = Solution concentration of the compound (mg/L)
Rcmpd = Response of the compound (area)
Cf = Calibration factor

Sample Concentration of Analyte Detected

Aqueous Samples: $$Rsult(ug/L) = Csol \times \frac{(Fvol \times Df)}{Vsmp}$$

Solid and Waste Sample: $$Rsult(mg/Kg) = Csol \times \frac{(Fvol \times Df)}{Wsmp}$$

Where: *Rsult* = Sample result (ppm)
Csol = Solution concentration of the compound (mg/L)
Fvol = Final volume of the extraction (ml)
Df = Dilution factor (if no dilution was performed *Df* = 1)
Cf = Calibration factor
Vsmp = Volume of sample extracted (L)
Wsmp = Weight of sample extracted (g), in Dry Weight or Wet Weight, depending on the reporting basis of the data

Results of extracted solid and waste samples are reported in mg/Kg (ppm). Solid sample results are usually reported on a dry-weight basis.

13.2. Data Review

Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the *SOP for Laboratory Data Review Process* for details.

13.3. Reporting

13.3.1. Reports are generated in the CAS LIMS or Stealth reporting software by compiling the SMO login, sample prep database, instrument date, and client-specified report requirements (when specified). This compilation is then transferred to a file which Excel© uses to generate a report. The forms generated may be CAS standard reports, DOD, or client-specific reports. The compiled data from LIMS are also used to create EDDs.

13.3.2. As an alternative, reports are generated using Excel© templates located in R:\PHC\forms. The analyst should choose the appropriate form and QC pages to correspond to required tier level and deliverables requirements. The detected analytes, surrogate and matrix spikes are then transferred, by hand or electronically, to the templates.

13.3.3. Sample concentrations are reported when all QC criteria for the analysis have been met or the results are qualified with an appropriate footnote. Refer to the *SOP for Data Reporting and Report Generation* for specific procedures and guidelines used for reporting analytical results and generating analytical reports.

13.3.4. Data Qualifiers

13.3.4.1. Data should be qualified when significant information regarding the analysis needs to be communicated to assist the user in determining the usability of the results. Acceptable qualifiers should comply with standard CAS (Kelso) data qualifiers.

13.3.4.2. When it is requested that results for Gasoline or lighter analytes be reported, these results are to be reported as estimated values and appropriately narrated.

13.3.4.3. Always use data qualifiers to describe hydrocarbon products. Refer to the *SOP for Data Reporting and Report Generation,* Appendix B for the specific hydrocarbon qualifiers:

- F-The chromatographic fingerprint of the range in question approximately matches that of the calibration standard used to quantify the result.

- Y-The chromatographic fingerprint of the range in resembles a petroleum product that elutes approximately within the range of integration of the calibration standard used to quantify the result but does not match the chromatographic fingerprint of the standard. A sufficiently weathered product that would otherwise match the appropriate calibration standard also falls into this category.

- L,H-The chromatographic fingerprint of the range in question mostly matches that of a petroleum product, but is actually a partial contribution of a product which elutes mostly in a lighter or heavier range (e.g., motor oil partially eluting in the diesel range), respectively.

- O-The chromatographic fingerprint in a range later than the Diesel range resembles that of oil, but does not match that of the calibration standard used to quantify the result.

- Z-The hydrocarbon responses in the range in question are primarily not due to response from a petroleum product.

14. CORRECTIVE ACTION

14.1. Refer to the *SOP for Corrective Action* for corrective action procedures. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2. Handling out-of-control or unacceptable data

14.2.1. On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks and runlogs, for example.

14.2.2. Documentation of a nonconformity must be done using a Nonconformity and Corrective Action Report (NCAR) when:

- Corrective action is not taken or not possible
- Corrective action fails to correct an out-of-control problem on a laboratory QC or calibration analysis.
- Reanalysis corrects the nonconformity but is not a procedurally compliant analysis.

15. METHOD PERFORMANCE

Available method performance data is given in the reference method. In addition, this procedure was validated through single laboratory studies of accuracy and precision as specified in Section 12. The method detection limit(s) and method reporting limit(s) were established for this method as specified in Section 12.

16. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents, acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvent and reagents used in this method can be minimized when recycled or disposed of properly.

17. WASTE MANAGEMENT

17.1. The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

17.2. This method uses Methylene Chloride. Any waste generated from this solvent must be placed in the dedicated collection cans in the lab. Solvent accumulations will be regularly transferred to the hazardous waste storage area and ultimately disposed of in accordance with Federal and State regulations.

17.3. This method uses non-halogenated solvents. Any waste generated from these solvents must be placed in the dedicated collection cans in the lab. Solvent accumulations will be regularly transferred to the hazardous waste storage area and ultimately disposed of in accordance with Federal and State regulations.

17.4. This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 5-9 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the CAS EH&S Manual for details.

18. TRAINING

18.1. Training outline

18.1.1. Review literature (see references section). Review the SOP. Also review all appropriate MSDSs, particularly that for methylene chloride.

18.1.2. Observe the procedure as performed by an experienced analyst at least three times.

18.1.3. Assist in the procedure under the guidance of an experienced analyst. During this training process, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

18.1.4. Following the training period the analyst is expected to complete an initial precision and recovery (IPR) study as described in section 12 for solid samples by sonication (diesel, motor oil), and water samples by liquid-liquid extraction (diesel, motor oil). Summaries of the IPR are reviewed and signed by the supervisor and forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

18.2. Training is documented following the *SOP for Documentation of Training*. When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor acknowledges that the analyst has read and understands this SOP and that

adequate training has been given to the analyst to competently perform the analysis independently.

19. REFERENCES

19.1. EPA Method 3500C, SW-846 Update IV. *Organic Extraction and Sample Preparation*, USEPA, 02/2007.
19.2. EPA Method 3510C, SW-846 Update III. *Separatory Funnel Liquid-Liquid Extraction*, USEPA, December, 1996.
19.3. EPA Method 3540C, SW-846 Update III. *Soxhlet Extraction*, USEPA, December, 1996.
19.4. EPA Method 3550C, SW-846 Update IV. *Ultrasonic Extraction*, USEPA, December, 02/2007.
19.5. EPA Method 3580A, SW-846 Update I. *Waste Dilution*, USEPA, July 1992.
19.6. EPA Method 3600C, SW-846 Update III. *Cleanup*, USEPA, December, 1996.
19.7. EPA Method 3611B, SW-846 Update III. *Alumina Column Cleanup and Separation of Petroleum Wastes*, USEPA, December, 1996.
19.8. EPA Method 3630C, SW-846 Update III. *Silica Gel Cleanup*, USEPA, December, 1996.
19.9. EPA Method 3650B, SW-846 Update III. *Acid-Base Partition Cleanup*, USEPA, December, 1996.
19.10. EPA Method 8000C, SW-846 On-Line. *Gas Chromatography*, USEPA, 03/2003
19.11. EPA Method 8015C, SW-846 Update IV *Nonhalogenated Organics by Gas Chromatography*, USEPA, February, 2007.
19.12. Alaska DEC Method AK 102, Contaminated Sites Program, *Method AK102 for the Determination of Diesel Range Organics*, April 2002.
19.13. Alaska DEC Method AK 103, Underground Storage Tank/LUST Program, *Method for the Determination of Residual Range Organics*, April 2002.
19.14. Alaska DEC Technical Memorandum 06-001, Alaska Department of Environmental Conservation, Division of Spill Prevention and Response, Contaminated Sites Remediation Program, *Biogenic Interference and Silica Gel Cleanup,* May 2006.
19.15. NWTPH-Dx, Semi-Volatile Petroleum Products Method for Soil and Water, Analytical Methods for Petroleum Hydrocarbons, Publication No. ECY 97-602, June 1997.

20. CHANGES SINCE LAST REVISION

20.1. Update correct Titles for Corporate SOPs throughout the document.
20.2. Sec 3.1 removed last sentence
20.3. Sec 3.7 remove PQL, no longer defined as equal to MRL
20.4. Sec 6.1 added temp range
20.5. EPA Method changes from 8015B to 8015C throughout the document.
20.6. Sec 8.1 paragraph under title is new
20.7. Section 8.2: Updates made to Standards expiration dates; updated standards list and working analyte spike solutions(s).
20.8. 11.2: Updated reference to the working spike solution for AK103.
20.9. 11.2: Added the comment that Alaska DEC does not permit a sulfuric acid clean-up.
20.10. Sec 11.2.2.6 Note with this section is new
20.11. 11.3:Re-worded section on background subtraction for DRO anlaysis.
20.12. Corrected numbering for section 11.
20.13. Section 11: Added 8015C instruction for the lowest calibration standard – 30% True value.
20.14. Section 11: Re-worded section for DoD requirements. Removed USACE HTRW project instructions.
20.15. Sec 11.3.4.1 from previous sop removed – redundant in SOC-CAL
20.16. Sec 11.3.4.3.1.2 from previous sop removed – redundant in SOC-CAL
20.17. Sec 11.3.6.1 & 11.3.6.2 replaced the word *system blank* with CCB
20.18. Sec 11.3.7.4 broke out DoD program specific requirements into a Note under this section
20.19. Sec 12 completely re-organized to fit CAS format
20.20. Sec 12.3 from previous sop deleted – not applicable to this section
20.21. 13.3.1: Added Stealth reporting software to the reporting section.
20.22. 13.3: Corrected the order of Data Qualifiers in the sentence.
20.23. Sec 19 updated references
20.24. Updates made to Tables – 1; 2,4,5, 7,8, and 9 to reflect current practices.

Table 1 - Applicable Method References, Target Analytes, Standard of Quantitation, Suggested Independent Calibration Verification Standards, and Calibration Technique.

Method	Target Analytes	Standard of Quantitation	Suggested ICV	Calibration
EPA Method 8015C	C10-C28 Diesel Range Organics	Diesel #2 composite	Diesel #2	External
EPA Method 8015C	Diesel Range Organics (various ranges) Residual Range Organics (various ranges)	Diesel #2 composite 30 wt. Motor Oil	Diesel #2 30 wt. Motor Oil	External
Alaska DEC Method AK 102	C10-C25 DRO (Diesel Range Organics)	Diesel #2 composite	Diesel #2	External
Alaska DEC Method AK 103	C25-C36 RRO (Residual Range Organics)	30/40wt. Motor Oil Mix	30/40 wt. Motor Oil Mix	External
Northwest TPH-Dx	C12-C25 Diesel Range Organics C25-C36 Residual Range Organics	Diesel #2 composite 30wt. Motor Oil Mix	Diesel #2 30 wt. Motor Oil	External
Northwest TPH-HCID	C12-C25 Diesel Range Organics C25-C36 Residual Range Organics C8-C12 Gasoline Range Organics	Diesel #2 composite 30 wt. Motor Oil Unleaded Gasoline Composite	Diesel #2 30 wt. Motor Oil Unleaded Gasoline Composite	External

Table 2 - Method Reporting Limits and Method Detection Limits

Compound	Aqueous Sample	Aqueous Sample	Solid/Waste Sample	Solid/Waste Sample
	MRL (ug/L)	MDL (ug/L)	MRL(mg/kg)	MDL (mg/kg)
8015C TPH-Diesel Range Hydrocarbons	50	15	25	1.6
NW TPH-Diesel Range Hydrocarbons	250	11	25	1.2
NW TPH-Residual Range Hydrocarbons	500	19	100	2.9
AK102 Diesel Range Hydrocarbons	800*	11	20*	1.3
AK103 Residual Range Hydrocarbons	500*	19	100*	2.9

*Required MRLs for Alaska fuels methods.

Table 3 - Library of Fuel Fingerprints

Class	Product	Class	Product
Fuels and Oils	Unleaded Gasoline	**Fuels and Oils**	Hydraulic Oil (Standard Fluid)
	Jet A		Hydraulic Oil (Tractor Fluid)
	JP 4		Hydraulic Oil Composite
	JP 5		90 wt Gear Oil
	JP 8		Crude Oil
	White Gas	**Petroleum Solvents**	Petroleum Naphtha
	Kerosene		Mineral Spirits
	Diesel #1		Stoddard Solvent
	Diesel #2		410 Thinner
	#4 Fuel Oil		Commercial Paint Thinner
	#5 Fuel Oil		Turpentine
	#6 Fuel Oil (Bunker C)	**Waxes, Greases, Residuals**	Paraffin Wax
	Stove Oil		Vacuum Grease
	Transformer Oil		Petroleum Jelly
	Mineral Oil (Light Grade)		Bearing Grease
	Mineral Oil (Heavy Grade)		Pine Tar
	Machine Oil		Asphalt
	Cutting Oil		Creosote
	Chain Bar Oil		
	30 wt Motor Oil		
	Multi-viscosity Motor Oils		

Table 4 - Maximum Recommended Analytical Holding Time and Preservatives

Method	Matrix	Preservative	Extraction Holding Time	Analysis Holding Time
EPA Method 8015C	Aqueous	pH < 2 with HCL; 4°C	7 Days (collection)	40 Days (extraction)
	Solid	4°C	14 Days (collection)	40 Days (extraction)
	Waste	None	28 Days (collection)	40 Days (extraction)
Alaska DEC Method AK 102	Aqueous	pH < 2 with HCL; 4°C	14 Days (collection)	40 Days (extraction)
	Solid	4°C	14 Days (collection)	40 Days (extraction)
Alaska DEC Method AK 103	Solid	4°C	14 Days (collection)	40 Days (extraction)
Alaska DEC Method AK 103	Aqueous	pH < 2 with HCL, 4°C	14 Days (collection)	40 Days (extraction)
NWTPH-Dx	Aqueous	pH < 2 with HCL; 4°C	7 Days (collection) 14 Days (collection) if preserved	40 Days (extraction)
	Solid	4°C	14 Days (collection)	40 Days (extraction)

Table 5. Suggested Concentrations of Calibration Standards. Concentrations are in ug/ml (ppm)

Compound	Type	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	ICV
4-Bromofluorobenzene	Surrogate	1	2.5	10	25	100	250	NA	NA	NA
o-Terphenyl	Surrogate	1	2.5	10	25	100	250	NA	NA	NA
Triacontane	Surrogate	1	2.5	10	25	100	250	NA	NA	NA
Gasoline	Target Analyte	20	50	200	500	2000	5000	NA	NA	1000
Kerosene	Target Analyte	20	50	200	500	2000	5000	NA	NA	1000
Diesel #2	Target Analyte	20	50	200	500	2000	5000	20000	50000	1000
30 wt. Motor Oil	Target Analyte	NA	50	200	500	2000	5000	NA	NA	1000
30wt./40wt. Motor Oil Mixture	Target Analyte	NA	50	200	500	2000	5000	NA	NA	1000

Table 6. Suggested GC Operating Conditions

Event	GC21 (Front Injector)	GC21 (Back Injector)
GC System		
Carrier Gas	Hydrogen	Hydrogen
Injection Mode	Splitless	Splitless
Septum Purge On Time (min)	0.10	0.10
Septum Purge Flow (ml/min)	50	50
Initial Temperature (°C)	60	60
Initial Hold Time (min)	1.5	1.5
Ramp Rate 1 (°C/min)	45	45
Temp Break 1 (°C)	280	280
Ramp Rate 2 (°C/min)	25	25
Temp Break 2 (°C)	315	315
Ramp Rate 3 (°C/min)	10	10
Final Temperature (°C)	330	330
Final Hold Time (min)	7.00	7.00
Inlet Temperature (°C)	280	280
Detector Temperature (°C)	340	340
Pressure Program		
Constant Flow (✓)	NA	NA
Constant Pressure (✓)	NA	NA
Initial Pressure (psi)	10	10
Pulse Rate (psi/min):	150	150
Pulse Maximum (psi)	30	30
Pulse Maximum Hold Time (min)	0	0
Pulse Return Rate (psi/min)	150	150
Pulse Minimum (psi)	3.0	3.0
Pressure Hold Time (min)	0.5	0.5
Pressure Rate (psi/min)	4.7	4.7
Pressure Maximum (psi)	75	75
Pressure Hold Time (min)	0	0
Approximate Cycle Time (min)	22	22

Table 7. Nominal Hydrocarbon Ranges for Petroleum Products

Method	Target Analyte	Starting Marker	Closing Marker
Nominal EPA Method 8015C	Diesel	Decane (inclusive)	Octacosane (inclusive)
8015C	Gasoline	Octane (inclusive)	Dodecane (exclusive)
8015C	Kerosene	Nonane (inclusive)	Octadecane (inclusive)
8015C when reported with separate heavy range	Diesel*	Dodecane (inclusive)	Pentacosane (exclusive)
8015C	Residual (or Heavy) Range Organics*	Pentacosane (inclusive)	Hexatriacontane (inclusive)
8015C	Motor Oil	Pentacosane (inclusive)	Hexatriacontane (inclusive)
8015C	#6 Fuel Oil (Bunker C)	Dodecane (inclusive)	Hexatriacontane (inclusive)
Alaska Method AK 102	Diesel Range Organics	Decane (inclusive)	Pentacosane (exclusive)
Alaska Method AK 103	Residual Range Organics	Pentacosane (inclusive)	Hexatriacontane (inclusive)
Washington NWTPH-Dx	Diesel Range Organics	Dodecane (inclusive)	Pentacosane (exclusive)
Washington NWTPH-Dx	Residual Range Organics	Pentacosane (inclusive)	Hexatriacontane (inclusive)

* These ranges may be defined slightly differently based on client requests/requirements

Table 8. Initial and Continuing Calibration Acceptance Criteria

Method	Initial Calibration Criteria	CCV Criteria		Notes
EPA Method 8015C	< 20 % RSD (or plot)	± 20 %		None
Alaska DEC Method AK 102	< 25 % RSD (or plot)	± 25 %		Surrogate in CCV 60-120%
Alaska DEC Method AK 103	< 25 % RSD (or plot)	± 25 %		Surrogate in CCV 60-120%
NWTPH-Dx	<20% RSD (or plot: r≥0.990)	± 15 %		Method sets criteria on linear regression. All points must be within 15% of true value.

Table 9. Data Quality Objectives: Surrogate Recoveries, Matrix Spike Recoveries, and Relative Percent Difference for Duplicate Analyses.

Compound	Type	Aqueous Sample	Solid/Waste Sample
Spike Recovery	**Accuracy**		
Diesel #2 - Method 8015C	Lab Control Sample	58-127	63-121
Diesel #2 – Method AK102	Lab Control Sample	75-125[m,c]	75-125[m,c]
Diesel #2 - Method 8015C	Matrix Spike	44-133	43-146
Diesel #2 – Method AK102	Matrix Spike	75-125	60-140
Motor Oil – Method 8015C	Lab Control Sample	60-134	57-136
Motor Oil – Method AK103	Lab Control Sample	60-120[m]	60-120[m]
4-Bromofluorobenzene – Method 8015C	Surrogate	35-145	10-154
o-Terphenyl – Method 8015C	Surrogate	50-136	51-126
o-Terphenyl – Method NWTPH-Dx	Surrogate	50-150[m]	50-150[m]
o-Terphenyl – Method AK102	Surrogate (MB,LCS,CCV)	60-120[m,c]	60-120[m,c]
o-Terphenyl – Method AK102	Surrogate (Samples)	50-150[m]	50-150[m]
Triacontane – Method AK103	Surrogate (MB,LCS,CCV)	60-120[m]	60-120[m]
Triacontane – Method AK103	Surrogate (Samples)	50-150[m]	50-150[m]
Relative Percent Difference	**Precision**		
Diesel #2 - Method 8015C, NWTPH-Dx	Matrix Spike/Sample Duplicates	30%	40%
Diesel #2 – Method AK102	Lab Control Sample Dup.	20%[m]	20%[m]
Motor Oil – Method AK103	Lab Control Sample Dup.	20%[m]	20%[m]

M – Method required criteria
C – For AK102 with Silica Gel cleanup, 70-125% LCS recovery, 70-125% recovery for surrogate in MB/LCS/CCV.

ALS Environmental

DOCUMENT TITLE:	*PCBS AS AROCLORS*
REFERENCED METHOD:	*METHOD 8082A*
SOP ID:	*SOC-8082AR*
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PCBS AS AROCLORS

METHOD 8082A

ALS-KELSO

SOPID:	SOC-8082Ar	Rev. Number:	16	Effective Date:	06/30/2013

Approved By: Loren E Portwood Date: 6/26/13
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Approved By: Suzanne LeMay Date: 6/26/13
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STANDARD OPERATING PROCEDURE

for

PCBS AS AROCLORS

1. SCOPE AND APPLICATION

This procedure is used to determine the concentrations of PCBs as Aroclors using EPA Method 8082A. This procedure is typically applied to water, sediment, and soil matrices but may also be applicable to tissue or various miscellaneous waste samples. Table 1 lists the analytes that are determined by this procedure and lists the method reporting limits (MRLs) for each compound in water and soil. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL). Therefore, MRL=EQL. The reported MRL may be adjusted if required for specific project requirements; however, the capability of achieving other reported MRLs must be demonstrated. Method Detection Limits that have been achieved are given in Table 1. MDLs may change as repeat studies are conducted.

2. METHOD SUMMARY

2.1. This procedure provides gas chromatographic conditions for the detection of parts-per-billion (ppb) levels of PCBs. The target PCBs are extracted from samples using the appropriate procedure for the sample matrix (see applicable SOP), analyzed, and reported as Aroclors. Liquid samples are extracted using solid phase extraction (Method 3535, EXT-3535). Liquid samples containing solid material may be extracted by continuous liquid-liquid extraction (Method 3520, EXT-3520). Soil/sediment samples are extracted using Soxhlet (Method 3540, EXT-3540), automated Soxhlet extraction (Method 3541, EXT-3541) or by Ultrasonic extraction (Method 3550, EXT-3550). An aliquot of the extract is injected into the gas chromatograph (GC). The compounds are separated on a fused silica capillary column. Compounds of interest are detected by an electron capture detector. Identification of the analytes of interest is performed by comparing the retention times of the analytes with the respective retention times of an authentic standard and by comparison of elution patterns to those of Aroclor standards. Quantitative analysis is performed by using the authentic standard to produce a calibration factor or calibration curve, and using the calibration data to determine the concentration of an analyte in the extract. The concentration in the sample is calculated using the sample weight or volume and the extract volume.

2.2. The sensitivity of this method usually depends on the level of interferences rather than on instrument limitations. If interferences prevent detection of the analytes, GPC, florisil column cleanup, sulfur cleanup, or concentrated sulfuric acid cleanups are used to eliminate interferences in the analysis. Refer to section 4.2 for cleanup procedure references.

2.3. In cases where there is a project-specific quality assurance plan (QAPP), the project manager identifies and communicates the QAPP-specific requirements to the laboratory. In general, project specific QAPP's supersede method specified requirements. An example of this are projects falling under DoD ELAP. QC requirements defined in the SOP *Department of Defense*

Projects – Laboratory Practices and Project Management (ADM-DOD) may supersede the requirements defined in this SOP.

3. DEFINITIONS

3.1. **Analysis Sequence** - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample extracts interspersed with calibration standards. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

3.2. **Batch** - A batch of samples is a group of environmental samples that are prepared and/or analyzed together as a unit with the same process and personnel using the same lot(s) of reagents. It is the basic unit for analytical quality control.

3.2.1. Preparation Batch - A preparation batch is composed of one to twenty field samples, all of the same matrix, and with a maximum time between the start of processing of the first and last samples in the batch to be 24 hours.

3.3. Analysis Batch - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration (initial or continuing verification) followed by sample extracts interspersed with calibration standards (CCBs, CCVs, etc.) The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria indicate an out-of-control situation.

3.4. **Sample**

3.4.1. Field Sample - An environmental sample collected and delivered to the laboratory for analysis; a.k.a., client's sample.

3.4.2. Laboratory Sample - A representative portion, aliquot, or subsample of a field sample upon which laboratory analyses are made and results generated.

3.5. **Quality System Matrix** - The *matrix* of an environmental sample is distinguished by its physical and/or chemical state and by the program for which the results are intended. The following sections describe the matrix distinctions. These matrices shall be used for purpose of batch and quality control requirements.

3.5.1. Aqueous - Any groundwater sample, surface water sample, effluent sample, and TCLP or other extract. Specifically excluded are samples of the drinking water matrix and the saline/estuarine water matrix.

3.5.2. Drinking water - Any aqueous sample that has been designated a potable or potential potable water source.

3.5.3. Saline/Estuarine water - Any aqueous sample from an ocean or estuary or other salt-water source.

3.5.4. Nonaqueous Liquid - Any organic liquid with <15% settleable solids.

3.5.5. Animal tissue - Any tissue sample of an animal, invertebrate, marine organism, or other origin; such as fish tissue/organs, shellfish, worms, or animal material.

3.5.6. Solids - Any solid sample such as soil, sediment, sludge, and other materials with >15% settleable solids.

3.5.7. Chemical waste - Any sample of a product or by-product of an industrial process that results in a matrix not described in one of the matrices in Sections 3.3.1 through 3.3.6. These can be such matrices as non-aqueous liquids, solvents, oil, etc.

3.5.8. Miscellaneous matrices – Samples of any composition not listed in 3.3.1 – 3.3.7. These can be such matrices as plant material, paper/paperboard, wood, auto fluff, mechanical parts, filters, wipes, etc. Such samples shall be batched/grouped according to their specific matrix.

3.6. Matrix Spike/Duplicate Matrix Spike (MS/DMS) Analysis - In the matrix spike analysis, predetermined quantities of target analytes are added to a sample matrix prior to sample preparation and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the method used for the analysis. Duplicate samples are spiked, and analyzed as a MS/DMS pair. Percent recoveries are calculated for each of the analytes detected. The relative percent difference (RPD) between the duplicate spikes (or samples) is calculated and used to assess analytical precision. The concentration of the spike should be at the mid point of the calibration range or at levels specified by a project analysis plan.

3.7. Laboratory Duplicates (DUP) – Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. The relative percent difference (RPD) between the sample and its duplicate is calculated and used to assess analytical precision.

3.8. Surrogate - Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to extraction and analysis. Percent recoveries are calculated for each surrogate.

3.9. Method Blank (MB) - The method blank is an artificial sample composed of analyte-free water or solid matrix and is designed to monitor the introduction of artifacts into the analytical process. The method blank is carried through the entire analytical procedure.

3.10. Laboratory Control Samples (LCS) – The LCS is an aliquot of analyte free water or analyte free solid to which known amounts target analytes are added. The LCS is prepared and analyzed in exactly the same manner as the samples. The percent recovery is compared to established limits and assists in determining whether the batch is in control.

3.11. Independent Verification Standard (ICV) - A mid-level standard injected into the instrument after the calibration curve and prepared from a different source than the initial calibration standards. This is used to verify the validity of the initial calibration standards

3.12. Continuing Calibration Verification Standard (CCV) - A mid-level standard analyzed at specified intervals. Used to verify that the initial calibration curve is still valid for quantitative purposes.

3.13. Instrument Blank (CCB) - The instrument blank (also called continuing calibration blank) is a volume of clean solvent analyzed on each column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrumental analysis itself, particularly with regard to the carry-over of analytes from standards or highly contaminated samples into subsequent sample analyses.

3.14. Duplicates and Duplicate Matrix Spikes are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. Depending on the method of analysis, either a duplicate analysis (and/or a matrix spiked sample) or a matrix spiked sample and duplicate matrix spiked sample (MS/DMS) are analyzed.

3.15. Standard Reference Material (SRM) – A material with specific certification criteria and is issued with a certificate or certificate of analysis that reports the results of its characterizations and provides information regarding the appropriate use(s) of the material. An SRM is prepared and used for three main purposes: (1) to help develop accurate methods of analysis; (2) to calibrate measurement systems used to facilitate exchange of goods, institute quality control, determine performance characteristics, or measure a property at the state-of-the-art limit; and (3) to ensure the long-term adequacy and integrity of measurement quality assurance programs.

4. INTERFERENCES

4.1. Interferences by phthalate esters can pose a major problem in PCB determinations when using the electron capture detector. These compounds generally appear in the chromatogram as large, late-eluting peaks, especially in the 15% and 50% fractions from the florisil cleanup. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Phthalate contamination is not usually a problem in our laboratory operation.

4.2. Co-extractables such as lipids, waxes, etc., can be removed via GPC cleanup (SOC-3640A). Certain fractionization cleanups can be used to selectively remove organochlorine pesticides, aiding in Aroclor determination (SOC-3665). The presence of elemental sulfur will result in interferences for most Aroclors. If GPC cleanup is insufficient, cleanup via Method 3660 (SOC-3660) may be used for the removal of sulfur.

4.3. A standard of the DDT analogs should be injected with each initial calibration to determine which of the PCB or Aroclor peaks may be subject to interferences on the analytical columns used. There may be substantial DDT interference with the last major Aroclor 1254 peak in some soil and sediment samples.

5. SAFETY

5.1. The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level.

5.2. Follow all applicable safety procedures as described in the ALS Safety Manual. A reference file of material safety data sheets is available to all personnel involved in these analyses. ALS also maintains a file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Containers used to collect samples should be purchased pre-cleaned containers. Alternatively, containers may be soap and water washed followed by methanol (or isopropanol) rinsing. The sample containers should be of glass or teflon and have screw-top covers with teflon liners. In situations where teflon is not available, solvent-rinsed aluminum foil may be used as a liner. Highly acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may not be used for

the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic.

6.2. Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples should not be collected or stored in the presence of exhaust fumes. If the sample comes in contact with the sampler (e.g., if an automatic sampler is used), run reagent water through the sampler and use the rinseate as a field blank.

6.3. Samples should be tested for residual chlorine at the time of sampling. For aqueous samples with residual chlorine present, add 3-mL 10% sodium thiosulfate solution per gallon (0.008%).

6.4. Water and soil samples must be iced or refrigerated at 4 ± 2ºC from time of collection until extraction. Tissue samples should be stored in accordance with project requirements, typically refrigerated or frozen.

6.5. There are no holding time requirements for this method.

7. APPARATUS AND EQUIPMENT

7.1. Gas Chromatograph (GC)

7.1.1. Analytical system complete with gas chromatograph suitable for splitless or on-column automated injection into a wide bore capillary column with an electron capture detector (ECD). Use of Large Volume Injection (LVI) is optional. Helium is used as the carrier gas; argon/methane mixture is used for the detector makeup gas (auxiliary gas). Current instrumental systems are identified as follows:

Instrument I.D.	Analytical System	Routine Matrix
GC22	Agilent 6890	Water-LL/Tissue/Soil
GC32	Agilent 6890	Water/Soil/Tissue

7.1.2. GC Autosampler: The GC system should be configured with a compatible autosampler for automated injection of standards, samples, and QC samples.

7.1.3. GC Columns - fused silica capillary columns
Column 1: DB-35MS, 30-m x 0.53mm, 1.0um film thickness, or equivalent.
Column 2: DB-XLB, 30-m x 0.53mm, 1.5um film thickness, or equivalent.

Note: Column diameter and film thickness varies depending on the column. Refer to the instrument maintenance logbook for the column used for a specific instrument configuration.

7.1.4. Data System - A computer data system must be interfaced to the GC/ECD. The system allows the continuous acquisition and storage on machine-readable media of chromatographic data obtained throughout the duration of the analysis program. The computer must have software that includes automated calibration, identification, and quantitation routines. The software must also be capable of integrating the chromatographic peaks abundances. The current version of the manufacturer's software is preferred (Target or HP Chemstation/Enviroquant).

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1. Solvents: Hexane, acetone, methylene chloride, isooctane, and methanol. Pesticide grade or equivalent.

8.2. Standards

8.2.1. Stock standard solutions may be purchased from a number of vendors. All reference standards, where possible, must be traceable to SI units or NIST certified reference materials.

8.2.2. Aroclor stock standard solutions are purchased from AccuStandard at 1000 ug/mL. Other vendors may be used providing they meet the requirements in sec 8.2.1. Transfer stock standard solutions into Teflon-sealed screw-cap bottles. Stock standard solutions are stored at -10ºC, or at ambient temperature as recommended by the vendor, and protected from light. The expiration date for unopened ampules is the manufacturer's assigned expiration date. If the manufacturer does not assign a date, an expiration date of 1 year from receipt is assigned. Check stock standards frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

8.2.2.1. Intermediate Aroclor calibration standard solutions are made by diluting 1000 ug/mL stock standards 1:20 in hexane. An intermediate surrogate standard is prepared at 5ug/mL by diluting the stock 1:40 in hexane.

8.2.2.2. Prepare calibration standards at a minimum of five concentration levels containing equal concentrations of both Aroclors 1016 and 1260 by dilution of the intermediate standards with hexane. One of the concentration levels should be at or below a concentration representing the method reporting limit (MRL). The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should define the working range of the GC. See Table 4 for preparation and concentrations, including standards designated as CCVs. A calibration standard of mid-range concentration is used for the CCV.

8.2.2.3. Calibration standard solutions are stored at 4 ± 2ºC and must be replaced after six months, or sooner, if comparison with check standards indicate a problem.

8.2.3. The independent calibration verification (ICV) standards are prepared purchased from Ultra Scientific at 100 ug/mL. Other vendors may be used providing they meet the requirements in sec 8.2.1. ICV solutions are stored at -10ºC, or at ambient temperature as recommended by the vendor, and protected from light. The expiration date for unopened ampules is the manufacturer's assigned expiration date. If the manufacturer does not assign a date, an expiration date of 1 year from receipt is assigned.

8.2.3.1. Working ICV standards at 1000 ug/mL are prepared as described in Table 4.

8.2.4. Surrogate solutions are prepared from stock solutions purchased from Ultra Scientific at 200 ugmL. Other vendors may be used providing they meet the requirements in sec 8.2.1.

8.2.4.1. The procedure for adding the surrogate solution to the calibration standards is outlined in Table 4.

8.2.4.2. A surrogate spiking solution is prepared at 2 ug/mL by making a 1:100 dilution of the surrogate stock standard in acetone. The surrogate solution is stored in the refrigerator for up to six months.

8.2.5. Standards of the other Aroclors are prepared for use as retention time/pattern standards and to establish calibration factors for these Aroclors (see Table 4).

8.2.6. Matrix spike solution: Prepare a spiking solution at 40 ug/mL containing both Aroclor 1016 and 1260 by diluting the 1000 ug/mL stock standards 1:25 with acetone.

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument. Pertinent information (serial numbers, instrument I.D., etc.) must be in the logbook. This includes the routine maintenance described in section 9. The entry in the log must include: date of event, the initials of who performed the work, and a reference to analytical control.

9.2. Carrier gas - Inline purifiers or scubbers should be in place for all sources of carrier gas. These are selected to remove water, oxygen, and hydrocarbons. Purifiers should be changed as recommended by the supplier.

9.3. Gas Chromatograph

9.3.1. Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column. Injection port maintenance includes changing the injection port liner, seal, washer, o-ring, septum, column ferrule, and autosampler syringe as needed. Liners and seals should be changed when recent sample analyses predict a problem with chomatographic performance. In some cases liners and seals may be cleaned and re-used.

9.3.2. Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and “clean” (uniform, without fragmentation) by using the proper column cutting tool.

9.3.3. Over time, the column will exhibit poorer overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced. This is especially true when evident in conjunction with calibration difficulties.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in 8082A, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Sample Preparation

11.1.1. Water samples (1L) are extracted at a pH of 5-9 with methylene chloride, using Method 3520 (EXT-3520) or using solid phase extraction (EXT-3535). Refer to the applicable extraction SOP. For extraction by 3535, acidification of the sample prior to extraction may be allowable if project objectives and performance requirements of methods 3535 and 8081 are met. An ultra low-level water option may be used, where a 1L sample amount and a final extract volume of 2mL is used. Large Volume Injectors are typically used with this option.

Note: Project-specific or regulation-specific extraction methods may apply. For projects originating from South Carolina and under the SC DHEC lab certification, use the 3520 extraction method only.

11.1.2. Soil/sediment samples are extracted using either EPA Method 3540 (EXT-3540), EPA Method 3541 (EXT-3541) or EPA Method 3550 (EXT-3550). Refer to the applicable extraction SOP. A low-level sediment option may be used where the sample weight of 40g (20g dry weight) and a final extract volume of 4mL are used.

11.1.3. Additional sample cleanup procedures may be employed as appropriate for the samples. Refer to the section on interferences and the appropriate ALS SOP.

11.2. Calibration

Note: The calibration procedure(s) and options chosen must follow the ALS protocols. Any exceptions to the calibration procedures detailed in SOC-CAL, *Calibration of Instruments for Organics Chromatographic Analyses* are described as follows:

Note: Certain state or program protocols have specific procedures for calibration. The analyst must ensure that the correct procedures are used. Known exceptions are as follows:

- The use of quadratic regression calibration is not allowed for projects (samples) originating from South Carolina and under the SC DHEC lab certification.

11.2.1. Prepare a minimum of 5 calibration standards containing equal concentrations of both Aroclor 1016 and 1260 by dilution of the stock standard(s) with isooctane or hexane. Single standards of each of the other target Aroclors are required to aid the analyst in pattern recognition. Once the linearity of the detector has been demonstrated using Aroclor 1016/1260 standards, the single standards of the remaining target Aroclors are also used to determine the calibration factor for each Aroclor. Prepare a standard for each of the other Aroclors. The concentrations should correspond to the mid-point of the linear range of the detector.

Note: DoD QSM requires the quantitation for Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point.

A minimum of 3 peaks must be chosen for each Aroclor, and preferably 6 peaks. The peaks must be characteristic of the Aroclor in question. Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak and does not coelute with any of the DDT analogs. For each Aroclor, the set of 3 to 6 peaks should include at least one quantitation peak that is unique to that Aroclor. Use at least five peaks for the Aroclor 1016/1260 mixture, none of which should be found in both of these Aroclors. Establish the retention time window position using the mid point of the ICAL range before processing the calibration curve.

11.2.2. Calibrate the system immediately prior to conducting any analyses. Refer to Table 3 for instrument conditions. Starting with the standard of lowest concentration, analyze each 1016/1260 calibration standard and tabulate response (peak area) versus the concentration in the standard. Calculate the ratio of the response to the amount injected the (calibration factor) for each analyte at each standard concentration. For 1016/1260 and DCB, the Relative Standard Deviation (RSD) must be less than 20% when average response factor is used.

11.2.3. Analyze each of the single-point calibration standards of the other target Aroclors. Calculate the calibration factor (CF) for each analyte at each standard concentration.

11.2.4. Each calibration of each Aroclor is verified by an independent source. Prepare an independent calibration verification standard (ICV) by dilution of a stock solution purchased from a different vendor and analyze immediately after each initial calibration. Calculate the concentration using the typical procedure used for quantitation. Calculate the percent difference (%D) from the ICV true value. Evaluate the ICV as described in SOC-CAL, *Calibration of Instruments for Organics Chromatographic Analyses.*

11.3. Calibration Verification

11.3.1. The working calibration curve or calibration factor must be verified on each analytical sequence by the analysis of one or more mid-range calibration standards (CCV). A standard (CCV) must be injected at the start of each sequence and after each set of sample extracts (every 10 samples or every 12 hours, whichever is first) in the analysis sequence.

Note: DoD projects require a CCV analysis every 10 field samples.

11.4. Retention Time Windows

11.4.1. Pattern recognition/matching and retention times are used for the identification of PCBs as Aroclors.

11.4.2. Establish retention time windows for the peaks used for quantitation with the GC system in acceptable operating condition. Make three injections of all analytes throughout the course of a 72-hour period. Serial injections over less than a 72-hour period may result in retention time windows that are too tight. Using retention times from these analyses, calculate retention time windows. Refer to EPA Method 8000C for detailed instructions.

11.4.3. Plus or minus three times the standard deviation of the absolute retention times for each standard will be used to define the retention time window; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms. In those cases where the standard deviation for a particular standard is zero, the laboratory may use a default window of $\pm$ 0.03 minutes. If the peak width is $>$ 0.06 minutes, use a default window of 0.1 minutes.

11.4.4. Calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. Retain this data in the method file.

11.5. Gas Chromatography

11.5.1. Set up an analytical sequence for the standards and samples to be analyzed. Calibrate the system as described in Section 11.2. Refer to Table 3 for typical instrument operating conditions. The same conditions must be used for samples as for calibration and QC analyses. Ensure that the instrument configuration is correct and that any necessary maintenance has been performed. Figure 1 shows a typical analysis sequence.

Note: For DoD projects, the CCB must be analyzed following the CCV. Instrument blanks/CCBs may not be analyzed prior to QC samples or standards.

11.5.2. Evaluate the CCVs as indicated in Section 11.3. Use the standards interspersed throughout the sample analysis sequence to evaluate the qualitative performance of the GC system including positioning of the retention time window. If any retention time shift which would impede analyte identification is evident (as shown by Aroclor pattern irregularities or the surrogate falling outside of the retention time window), evaluate the chromatogram for possible causes such as carryover from a highly contaminated sample. If a problem related to GC system has been determined to be the cause of retention time shift, perform whatever maintenance is necessary before reanalyzing the CCV or recalibrating and proceeding with sample analysis. All samples that were injected after the sample exceeded the criteria must be reinjected if initial analysis indicated the presence of any analytes of interest.

FIGURE 1

Analysis Sequence

Initial Calibration Blank	ICB
Standard 1	1221 midpoint
Standard 2	1232 midpoint
Standard 3	1242 midpoint
Standard 4	1248 midpoint
Standard 5	1254 midpoint
Standard 6	1262 midpoint
Standard 7	1268 midpoint
Standards 8-12	1016/1260 ICAL standards
Standards 13-20	ICVs for 1016, 1221, 1232, 1242, 1254, 1260, 1262, 1268
Continuing Calibration Verification	CCV
Method Blank	
Laboratory Control Sample	LCS
Sample 1 - 8	
Matrix Spike	
Duplicate Matrix Spike	
Continuing Calibration Verification	CCV
Continuing Calibration Blank	CCB
Sample 9 - 18	
Continuing Calibration Verification	CCV
Continuing Calibration Blank	CCB

Note: For DoD projects, the CCB must be analyzed following the CCV. Instrument blanks and CCBs may not be analyzed prior to QC samples or standards.

11.6. Troubleshooting

11.6.1. Initial calibration – If the initial calibration fails to meet the criteria, or the ICV fails (indicating a calibration problem), the following steps may be taken, depending on nature of the problem.

- Recheck the information entered into the software used for calibration and quantitation. Verify the standard values are correct and datafiles are correct. If incorrect, repeat the calibration with the correct information.
- Recheck standards preparation to ensure that standards are correct. Re-prepare and reanalyze if needed.
- Ensure that proper preventive maintenance was performed. Repeat the preventive maintenance if necessary and reanalyze the calibration.
- If calibration problems persist or more substantial calibration problems exist, corrective maintenance or repair may be needed. This includes such measures as column changes, detector maintenance, or GC repair. This will depend on the nature of the problem. Following any such maintenance, repeat the calibration.

11.6.2. Continuing calibration – If the CCV analysis fails to meet the criteria, the following steps may be taken, depending on nature of the problem.

- Recheck the information entered into the software used for calibration and quantitation. Verify the standard values are correct and datafiles are correct. If incorrect, repeat the calibration with the correct information.
- Recheck standards preparation to ensure that standards are correct and that the correct standard is used as the CCV. Re-prepare and reanalyze if needed. Note that NELAC and DoD requirements apply when multiple CCVs are analyzed.
- Ensure that proper preventive maintenance was performed. Repeat the preventive maintenance if necessary and reanalyze the CCV.
- If calibration problems persist or more substantial calibration problems exist, corrective maintenance or repair may be needed. This includes such measures as column changes, detector maintenance, or GC repair. This will depend on the nature of the problem. Following any such maintenance, repeat analysis of the CCV and necessary samples. Major maintenance will require recalibration. Note that some samples may quickly deteriorate the system to the point that closing CCVs will not pass. This should be verified through a second run of the samples and documented.

12. QA/QC REQUIREMENTS

12.1. Initial Precision and Recovery Validation

12.1.1. The accuracy and precision of the procedure must be validated before analyses of samples begin, or whenever significant changes to the procedures have been made. To do this, four water samples are spiked with the LCS spike solution, then prepared and analyzed.

12.2. Method Detection Limits and Method Reporting Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike seven blank matrix (water or soil) samples with MDL spiking solution at a level below the MRL. Follow the analysis procedures in Section 11 to analyze the samples.

12.2.2. Calculate the average concentration found (x) in µg/mL, and the standard deviation of the concentrations (s) in µg/mL for each analyte. Calculate the MDL for each analyte. Refer to CE-QA011, *Performing Method Detection Limit Studies and Establishing Limits of detection and Quantification.* The MDL study must be verified annually.

12.2.3. Limits of Quantification (LOQ)

12.2.3.1. The laboratory establishes a LOQ for each analyte as the lowest reliable laboratory reporting concentration or in most cases the lowest point in the calibration curve which is less than or equal to the desired regulatory action levels, based on the stated project requirements. Analysis at the lowest point calibration level provides confirmation of the established sensitivity of the method. The LOQ recoveries should be within 50% of the true values to verify the data reporting limit. Refer to CE-QA011, *Performing Method Detection Limit Studies and Establishing Limits of detection and Quantification.*

12.2.4. The Method Reporting Limits (MRLs) used at ALS are the routinely reported lower limits of quantitation which take into account day-to-day fluctuations in instrument sensitivity as well as other factors. These MRLs are the levels to which ALS routinely reports results in order to minimize false positive or false negative results. The MRL is normally two to ten times the method detection limit.

12.3. Ongoing QC Samples required are described in the ALS-Kelso Quality Assurance Manual and in the SOP for Sample Batches. Additional QC Samples may be required in project specific quality assurance plans (QAPP). For example projects managed under the DoD ELAP must follow requirements defined in the DoD *Quality Systems Manual for Environmental Laboratories.* General QA requirements for DoD QSM are defined in the laboratory SOP, Department *of Defense Projects – Laboratory Practices and Project Management (ADM-DOD).* General QC Samples are:

12.3.1. Method blank - A method blank is extracted and analyzed with every batch of 20 or fewer samples to demonstrate that there are no method interferences. The method blank must demonstrate that interferences from the analytical and preparation steps minimized. No target analytes should be detected above the MRL in the method blank.

12.3.1.1. If the method blank fails to meet the criteria, the sample data in the associated batch should be examined. If all samples and QC have hits for the analyte, samples and QC should be re-extracted and reanalyzed as necessary (samples with higher level hits may not need reanalysis). It should be verified through the analysis of instrument blanks that the problem is isolated to either the GC of the sample preparation. If the problem is isolated to the MB, the data may be flagged and narrated. Also refer to the QA Manual for additional corrective action.

12.3.1.2. The source of MB contamination should be isolated and corrected as soon as possible to prevent further failures.

Note: DoD projects require no analytes detected > ½ the RL or > 1/10 the regulatory limit.

12.3.2. A lab control sample (LCS) must be extracted and analyzed with every batch of 20 samples. The water LCS is prepared by adding 50 µL of the matrix spike solution to 1L of reagent water, resulting in a concentration of 2.0 ug/L. The soil LCS is

prepared by adding 100 µL of spike solution to 20g of sand, resulting in a concentration of 200 ug/kg. For project-specific low-level extractions, spiking amounts can be adjusted accordingly. Calculate percent recovery (%R) as follows:

%R = X/TV x 100

Where X = Concentration of the analyte recovered
TV = True value of amount spiked

Acceptance criteria for lab control samples are listed in Table 1.

12.3.3. Project-specific or program-specific acceptance criteria may supersede ALS criteria. For example, for samples requiring South Carolina DHEC certification the acceptance criteria are 70-130 % recovery. If the lab control sample (LCS) fails acceptance limits for any of the compounds, the analyst must evaluate the system and calibration. If no problems are found, corrective action must be taken. The acceptance criteria listed are current criteria, but are subject to change as control limits are updated.

12.3.4. A matrix spike/duplicate matrix spike (MS/DMS) must be extracted and analyzed with every batch of 20 or fewer samples. The MS/DMS is prepared by adding the same volume of the matrix spike solution to the sample as listed for the LCS, then proceeding with the entire extraction and analysis. Calculate percent recovery (%R) as follows:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

Calculate Relative Percent Difference (RPD) as:

$$RPD = \frac{|R1 - R2|}{(R1 + R2)/2} \times 100$$

Where R1 = % recovery of the MS
R2 = % recovery of the DMS

Acceptance criteria for matrix spikes are listed in Table 1. If the MS/DMS recovery is out of acceptance limits for reasons other than matrix effects, corrective action must be taken. The acceptance criteria listed are current criteria, but are subject to change as control limits are updated.

12.3.5. Surrogate spike is added to every sample, blank and spike prior to extraction. Two surrogate standards (tetrachloro-m-xylene and decachlorobiphenyl) are added to each sample. For water, 100µL of the surrogate spike is added to 1L, resulting in 0.2 ug/L. For soil, 200µL of the surrogate spike is added to 20g, resulting in 20 ug/kg. Calculate surrogate percent recovery (%R) as:

%R = S/V x 100
Where S = The amount of surrogate recovered

V = The amount spiked/final volume

The acceptance limits for the surrogates are given in Table 1. Both surrogate recoveries must be within the acceptance limits. If either (or both) surrogate is outside of acceptance limits for reasons other than matrix interferences, corrective action must be taken. Corrective actions include recalculation, reanalysis, or reextraction and reanalysis. The acceptance criteria listed are current criteria, but are subject to change as control limits are updated.

12.3.6. Control charts should be maintained for QC results. The charts should be reviewed periodically for trends in results. Control limits for QC analyses may be determined using the control charts or similar mechanism on an annual basis.

13. DATA REDUCTION, REVIEW, AND REPORTING

13.1. Identification of PCBs as Aroclors

13.1.1. To identify Aroclors, compare the chromatographic pattern of the sample to known Aroclor standards. Tentative identification of PCBs as Aroclors is made when the pattern of peaks in the sample chromatogram matches the pattern of peaks in the Aroclor standard itself. There also needs to be agreement between the retention times and response ratios of the 3-6 selected quantitation peaks in the sample chromatogram and the Aroclor standard.

13.1.2. Tentative identification of analytes must be confirmed using a second GC column of dissimilar phase. Identify the Aroclor by comparing the chromatographic pattern of the sample to known Aroclor standards analyzed on the same column. Confirmation of the Aroclor is made when the sample chromatogram matches the pattern of peaks in the Aroclor being confirmed. Quantitations for the 2 columns must agree ($\leq$ 40%RPD) to confirm the identification. If interferences or other sample anomalies make the RPD value >40% but the analyst makes a positive identification, the basis of the identification must be documented and the data user notified of the discrepancy (see section 13.2).

13.2. Sample matrix difficulties

13.2.1. Weathering of PCBs in the environment and changes resulting from waste treatment processes may alter the pattern of a specific Aroclor so it does not closely match an Aroclor standard. The earlier eluting peaks will often diminish in comparison to the later eluting peaks. If this is observed, alternate peaks may be selected to aid identification to reduce quantitation bias.

13.2.2. Metabolism by organisms may also alter the pattern since individual PCB congeners are metabolized at different rates. When working with tissue samples, the 40% RPD criteria for confirmation may not be met.

13.2.3. Samples may also include mixtures of two or more Aroclors. To the extent possible, identify and quantify each Aroclor.

13.2.4. High amounts of organochlorine pesticides in the sample may interfere with identification. If this is observed, alternate peaks may be selected to aid identification to reduce quantitation bias. Certain fractionization cleanups can be used to selectively remove organochlorine pesticides, aiding in Aroclor determination (Acid cleanup, SOC-3665).

13.2.5. For all of these reasons a high level of analyst expertise is required to interpret complex chromatograms.

13.3. Quantitation of PCBs as Aroclors:

13.3.1. The quantitation of PCBs as Aroclors is accomplished by comparison of the sample chromatogram to that of the most similar Aroclor standard or standards. All calibration acceptance criteria as described in section 11 must be met before reporting any results. Sample results should then be reported according to the organics confirmation SOP (SOC-CONF). Results may be reported from either column if all calibration acceptance criteria as described in section 11are met.

13.3.2. Once the Aroclor pattern has been identified, compare the responses of 3 to 6 major peaks in the calibration standard of 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 6 peaks and the calibration model selected in section 11. The concentration is determined using the 3 to 6 characteristic peaks and then the concentrations are averaged to determine the concentration of the Aroclor. If there are interfering peaks with the 3 to 6 quantitation peaks that cause Aroclor average to be falsely overstated, then that interference peak is Q-deleted using the data system and the average is recalculated so that the average more truly represents the concentration in the sample. This often occurs when there are more than one Aroclor in a sample extract or if pesticides are present. Quantitation of mixed Aroclors will require the selection of peaks that are not shared in common by both Aroclors.

13.3.3. For samples with severe matrix interferences, the quantitation may be performed by measuring the total area of the PCB pattern and quantifying on the basis of the Aroclor standard that is most similar to the sample. Any peaks that are not identifiable as PCBs should be subtracted from the total area. When the quantitation option is used, the sample problems should be described for the data user and quantification procedure documented.

13.3.4. Using the data system, calculate the concentration in the extract using the calibration model chosen for calibration (see SOC-CAL).

13.3.5. Using the data system, calculate the concentration of each analyte in the sample extract (Cex) µg/ml units using the calibration factor or calibration curve (Section 11). The sample concentration computed using the following equations:

Aqueous Samples:

$$Concentration\ (\mu g / L) = \frac{(Cex)\ (Vf)\ (D)}{(Vs)}$$

Where Cex = Concentration in extract in µg/ml
Vf = Final volume of extract in ml
D = Dilution factor
Vs = Volume of sample extracted, liters

Nonaqueous Samples:

$$Concentration\ (mg / Kg) = \frac{(Cex)\ (Vf)\ (D)\ x\ 1{,}000}{(W)\ x\ 1{,}000}$$

Where Cex = Concentration in extract in µg/ml
Vf = Final volume of extract in ml
D = Dilution factor
W = Weight of sample extracted. The wet or dry weight may be used, depending upon the specific client requirements.

13.4. Sample concentrations are reported when all QC criteria for the analysis have been met or the results are qualified with a footnote.

13.5. Data Review

13.5.1. Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer ADM-DREV, *Laboratory Data Review Process* for details. The person responsible for final review of the data report and/or data package should assess the overall validity and quality of the results and provide any appropriate comments and information to the Project Chemist to inclusion in the report narrative.

13.6. Reporting

13.6.1. Reports are generated using the STEALTH Data Reporting System which compiles the SMO login information. This compilation is then transferred to a file, which STEALTH uses to generate a report. The forms generated may be ALS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13.6.2. As an alternative, reports are generated using Excel© templates located in R:\SVG\forms. The analyst should choose the appropriate form and QC pages to correspond to required tier level and deliverables requirements. The detected analytes, surrogate and matrix spikes are then transferred, by hand or electronically, to the templates.

13.6.3. Sample concentrations are reported when all QC criteria for the analysis have been met or the results are qualified with an appropriate footnote. For Arizona projects the appropriate Arizona qualifier must be used.

14. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

14.1. Refer to the SOP for *Non Conformance and Corrective Action (CE-QA008)* for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2. Handling out-of-control or unacceptable data

14.2.1. On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, runlogs, for example.

14.2.2. Some examples when documentation of a nonconformity is required using a Nonconformity and Corrective Action Report (NCAR):

- Quality control results outside acceptance limits for accuracy and precision
- Method blanks or continuing calibration blanks (CCBs) with target analytes above acceptable levels
- Sample holding time missed due to laboratory error or operations
- Deviations from SOPs or project requirements
- Laboratory analysis errors impacting sample or QC results
- Miscellaneous laboratory errors (spilled sample, incorrect spiking, etc)
- Sample preservation or handling discrepancies due to laboratory or operations error

15. METHOD PERFORMANCE

15.1. This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

15.2. The method detection limit (MDL) is established using the procedure described in CE-QA011, *Performing Method Detection Limit Studies and Establishing Limits of detection and Quantification*. Method Reporting Limits are established for this method based on MDL studies and as specified in the ALS Quality Assurance Manual.

16. TRAINING

16.1. Training outline

16.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

16.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst for a period of 3 months. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

16.1.3. Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

16.2. Training is documented following the SOP for Documentation of Technical Personnel Training.

16.2.1. NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

17. POLLUTION PREVENTION AND WASTE MANAGEMENT

17.1. It is the laboratory's practice to minimize the amount of solvents, acids, and reagents used to perform this method wherever feasibly possible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept

on site. The threat to the environment from solvents and/or reagents used in this method can be minimized when recycled or disposed of properly.

17.2. The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Environmental Health and Safety Manual.

18. METHOD MODIFICATIONS

18.1. There are no known modifications in this laboratory standard operating procedure from the reference method.

19. REFERENCES

18.1. Polychlorinated Biphenyls (PCBs) as Aroclors, Method 8082A, Revision 1, February 2007, EPA Test Methods for Evaluating Solid Waste, SW-846, Update IV

18.2. Determinative Chromatographic Separations, EPA SW846, Test Methods For Evaluating Solid Waste, On-Line, Method 8000C, Revision 3, March 2003.

18.3. 8000C Method criteria, Arizona DHS, 2/13/2007. Available online at http://www.azdhs.gov/lab/license/tech/8000cmethod.pdf

18.4. *DoD Quality Systems Manual for Environmental Laboratories* Version 4.2 10/25/10.

20. CHANGES SINCE THE LAST REVISION

20.1. Reformatted to ALS branding.
20.2. Replaced “CAS” references with “ALS”.
20.3. Updated SOP references.
20.4. Sec. 7.1: removed reference to GC09.
20.5. Sec. 11.2.1: Added clarification for choosing peaks.
20.6. Updated limits in Table 1.
20.7. Added Table 2.
20.8. Made extensive updates to Table 3.

TABLE 1

TARGET COMPOUNDS, MRLs, and MDLs

Analyte	Method Detection Limit		Method Reporting Limit	
	Water ug/L	Soil mg/kg	Water ug/L	Soil mg/kg
Aroclor 1016	0.0021	0.0085	0.005	0.1
Aroclor 1221	0.0021	0.0085	0.01	0.2
Aroclor 1232	0.0021	0.0085	0.005	0.1
Aroclor 1242	0.0021	0.0085	0.005	0.1
Aroclor 1248	0.0021	0.0085	0.005	0.1
Aroclor 1254	0.0021	0.0085	0.005	0.1
Aroclor 1260	0.0021	0.0085	0.005	0.1
Aroclor 1262	0.0021	0.0085	0.005	0.1
Aroclor 1268	0.0021	0.0085	0.005	0.1

TABLE 2

Summary of Corrective Actions

Method Reference	Control	Specification and Frequency	Acceptance Criteria	Corrective Action
EPA 8082A	ICAL	Prior to sample analysis	% RSD ≤ 20 R2 ≥ 0.995 COD ≥ 0.990	Correct problem then repeat ICAL
EPA 8082A	ICV	After ICAL	± 20% Diff	Correct problem and verify second source standard; rerun second source verification. If fails, correct problem and repeat initial calibration.
EPA 8082A	CCV	Prior to sample analysis, every 10 samplesor 12 hours	± 20% Diff	Correct problem then repeat CCV or repeat ICAL
EPA 8082A	Method Blank	Include with each analysis batch (up to 20 samples)	<MRL	If target exceeds MRL, reanalyze to determine if instrument was cause. If still noncompliant then: Re-extract or reanalyze samples containing contaminate, unless samples contain > 20x amount in blank.
EPA 8082A	Laboratory Control Sample	Include with each analysis batch (up to 20 samples)	Refer to DQO Tables	If exceeds limits, re-extract and re-analyze
EPA 8082A	Matrix Spike	Include with each analysis batch (up to 20 samples)	Refer to DQO Tables	Evaluate data to determine if the there is a matrix effect or analytical error
EPA 8082A	Sample Duplicates	Include with each analysis batch (up to 20 samples)	W, RPD ≤ 30 S, RPD ≤ 40	Re-homogenize and re-analyze if result is > 5 X the MRL

TABLE 3

Gas Chromatograph Operating Conditions*

Gas Chromatograph:	Hewlett-Packard Model 6890 or equivalent w/ECD
Injection Port Temperature:	Initial Temp 90˚C for 0.5 min., 250˚C/min ramp to 325˚C for 5.0 min., 20˚C/min ramp to 250˚C for 5.0 min.
Oven Temperature Program:	90˚C for 0.5 min., 5ºC/min ramp to 230˚C for 0.5 min., then 7ºC/min. to 315˚C, hold 0.06 min.
Detector Temperature:	325˚C
Injection Volume:	1 µL
Column:	30 m, DB-35MS and 30 m DB-XLB*
Carrier Gas:	Hydrogen
Auxillary Gas:	Nitrogen
Data System:	HP Chemstation (acquisition) and Target (data)

* The instrument temperatures may be modified depending on the instrument used. Also, the GC column diameter and film thickness depend on the instrument used. All conditions must be the same for initial calibration, continuing calibration, sample, and QC analyses.

TABLE 4

CALIBRATION STANDARD PREPARATION

1016/1260 Initial Calibration Standards (prepared in hexane)

Aroclor 1016 50 ug/mL Intermediate	Aroclor 1260 50 ug/mL Intermediate	Surrogate 5 ug/mL Intermediate	Final Volume	Final Concentration Aroclors	Final Concentration Surrogates
12.5 uL	12.5 uL	12.5 uL	25 mL	25 ug/L	2.5 ug/L
25 uL	25 uL	25 uL	25 mL	50 ug/L	5.0 ug/L
250 uL	250 uL	250 uL	25 mL	500 ug/L	50 ug/L
500 uL	500 uL	500 uL	25 mL	1000 ug/L*	100 ug/L*
1000 uL	1000 uL	1000 uL	25 mL	2000 ug/L	200 ug/L
2500 uL	2500 uL	2500 uL	25 mL	5000 ug/L	500 ug/L

* CCV Standard

Single-Point Calibration Standards

Intermediate Standard (1000 ug/mL)	Aliquot	Final Volume	Solvent	Final Concentration
Aroclor 1221	25 uL	25 mL	Hexane	1000 ug/L
Aroclor 1232	25 uL	25 mL		1000 ug/L
Aroclor 1242	25 uL	25 mL		1000 ug/L
Aroclor 1248	25 uL	25 mL		1000 ug/L
Aroclor 1254	25 uL	25 mL		1000 ug/L
Aroclor 1262	25 uL	25 mL		1000 ug/L
Aroclor 1268	25 uL	25 mL		1000 ug/L

ICV Standards

ICV Stock Standard (100 ug/mL)	Aliquot	Final Volume	Solvent	Final Concentration
Aroclor 1016	250 uL	25 mL	Hexane	1000 ug/L
Aroclor 1221	250 uL	25 mL		1000 ug/L
Aroclor 1232	250 uL	25 mL		1000 ug/L
Aroclor 1242	250 uL	25 mL		1000 ug/L
Aroclor 1248	250 uL	25 mL		1000 ug/L
Aroclor 1254	250 uL	25 mL		1000 ug/L
Aroclor 1260	250 uL	25 mL		1000 ug/L
Aroclor 1262	250 uL	25 mL		1000 ug/L
Aroclor 1268	250 uL	25 mL		1000 ug/L

STANDARD OPERATING PROCEDURE

SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS SELECTIVE ION MONITORING

EPA Method 8270D SIM

SVM-8270S
Revision 6

Effective Date: October 31, 2011

Approved By:

Supervisor	Date: 10/6/11
QA Manager	Date: 10/6/11
Laboratory Manager	Date: 10/6/11

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DOCUMENT CONTROL	
NUMBER:	
Initials:	Date:

SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS SELECTIVE ION MONITORING
Method 8270D SIM

1. SCOPE AND APPLICATION

1.1. This procedure is used to determine the concentrations of Semi-Volatile Organic Compounds in water, soil, and tissue matrices using EPA Method 8270D and Selected Ion Monitoring (SIM). This procedure may also be applicable to various miscellaneous waste samples. Table 4 lists compounds commonly determined by this method and lists nominal method reporting limits (MRLs) in water, soil, and tissue. Other compounds may be analyzed to meet project requirements. Due to the nature of this analysis and its applications, MRLs may vary due to project specifications. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL). Therefore, MRL=EQL. Table 4 lists Method Detection Limits (MDLs) that have been achieved, however, MDLs may change as MDL studies are performed, and may vary depending on the instrument used and preparation method.

1.2. The procedure is intended for samples containing trace-level amounts of target compounds. Samples containing high concentrations of target analyte will not be analyzed undiluted. Extracts may be screened using GC/FID to estimate the hydrocarbon content and concentrations of individual polynuclear aromatic hydrocarbons (PAHs). Samples containing PAHs are diluted prior to analysis. All MRLs will be adjusted in accordance with this dilution. Therefore, samples containing high levels of PAHs will not be analyzed to achieve the optimum MRLs for the analysis.

1.3. This procedure can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone phase. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons, phthalate esters, nitrosamines, haloethers, ketones, anilines, aromatic nitro compounds, and phenols, including nitrophenols. Most acidic compounds are deriviatized in tissue samples prior to analysis to aid sensitivity and chromatographic resolution.

1.4. Other compounds than those listed in Table 4 may be analyzed. However, analytes not summarized in Table 4 may not been validated with a method detection limit study. Therefore, the lab will not use this procedure to analyze for non-routine analytes unless a similar analyte has been validated with a MDL study. As a general rule, the MRL for these compounds will equal the MRL of a similar compound in the routine analyte list. Results will not be reported below this estimated MRL.

2. METHOD SUMMARY

2.1. This method provides Gas Chromatography/Mass Spectrometry (GC/MS) conditions for the detection of Semi-volatile Organic Compounds. Prior to the use of this method, an appropriate sample preparation method must be used to recover the analytes of interest. Soil, tissue, and colored water extracts may be cleaned using EPA Method 3640 (gel permeation chromatography) prior to analysis.

2.2. A 0.5-20 µL aliquot of the extract is injected into the gas chromatograph (GC). The compounds are separated on a fused silica capillary column. Compounds of interest are detected by a mass selective detector in the selective ion mode. Identification of the analytes of interest is performed by comparing the retention times of the analytes with the respective retention times of an authentic standard, and by comparing mass spectra of analytes with mass spectra of reference materials. Quantitative analysis is performed by using the authentic standard to produce a response factor and calibration curve, and using the calibration data to determine the concentration of an analyte in the extract. The concentration in the sample is calculated using the sample weight or volume and the extract volume. To achieve MRLs listed in Table 4 the initial sample amount, final extract volume, and injection aliquot are specified for each product.

2.3. The following compounds may require special treatment when being determined by this method. Benzidine, 3, 3'-Dichlorobenzidine, nitrophenols, Hexachlorocyclopentadiene, Pentachlorophenol, Benzoic Acid and Benzyl Alcohol are subject to poor extraction efficiency or erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

3. DEFINITIONS

3.1. **Analysis Sequence** - Samples are analyzed in a set referred to as an analysis sequence The sequence begins with injection of Decafluorotriphenylphosphine (DFTPP) acquired in full scan mode followed by initial calibration standard(s) acquired in SIM mode. Once calibrated, a CCV is evaluated and extracts can be run. The sequence ends after 12 hours based on the DFTPP acquisition time.

3.2. **Laboratory Control Sample (LCS)** - In the LCS analysis, predetermined quantities of standard solutions of all analytes are added to a blank matrix prior to sample extraction and analysis. The purpose of the LCS is to monitor analytical control for the sample batch. Percent recoveries are calculated for each of the analytes.

3.3. **Matrix Spike/Duplicate Matrix Spike Analysis** - In the matrix spike analysis, predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Samples are split into duplicates, then spiked and analyzed. Percent recoveries are calculated for

each of the controlled analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

3.4. **Standard Curve** - A standard curve is a plot of concentrations of a known analyte standard versus the instrument response to the analyte.

3.5. **Surrogate** - Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate. A subset of those surrogates listed in the procedure may be reported, depending on target analytes.

3.6. **Method Blank** - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

3.7. **Continuing Calibration Verification Standard (CCV)** - A mid-level standard injected into the instrument at specified intervals and is used to verify the validity of the initial calibration.

3.8. **Second Source Verification Standard or Independent Verification Standard (SSV or ICV)** - A mid-level standard injected into the instrument after the calibration curve and prepared from a different source than the initial calibration standards. This is used to verify the validity of the initial calibration standards.

3.9. **Selective Ion Monitoring (SIM)** – Mass spectrometry technique where ions resulting from fragmentation are selectively monitrored, therefore excluding other ions. The technique enhances sensitivity as compared to full scan analysis. Because the analysis results in significantly less mass spectral information, this gain in sensitivity is made at the expense of analyte selectivity. Therefore, the use of SIM results in significantly lower instrument detection limits, but increases the uncertainty associated with the analysis.

4. INTERFERENCES

4.1. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation of the samples. Corrective action should be taken to eliminate the interferences.

4.2. Accurate determination of phthalate esters can pose difficulties when using this methodology. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Cross contamination of clean glassware may occur when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from

phthalates can best be minimized by avoiding contact with any plastic materials. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

4.3. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of an instrument blank to check for cross contamination.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personal protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. This method uses Methylene Chloride, a known human carcinogen. Viton brand gloves should be used while rinsing, pouring or transferring the solvent

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Certified clean containers should be purchased for sample collection. Alternatively, containers used to collect samples for the determination of semivolatile organic compounds should be soap and water washed followed by methanol (or isopropanol) rinsing. The sample containers should be of glass or teflon and have screw-top covers with teflon liners. In situations where teflon is not available, solvent-rinsed aluminum foil may be used as a liner. Highly acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may not be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic.

6.2. Water and soil samples should be iced or refrigerated at 4 ± 2°C from time of collection until extraction. Tissue samples are stored frozen until extraction.

6.3. Water samples must be extracted within 7 days. Soil samples must be extracted within 14 days. Holding times for tissues are typically defined by project specifications, otherwise tissue samples may be held frozen up to one year before extraction. All extracts must be analyzed within 40 days following extraction. All extracts are stored at < -10^{o}C.

7. APPARATUS AND MATERIALS

7.1. Gas Chromatograph/Mass Spectrometer System

7.1.1. Gas Chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless, split, or large-volume injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

7.1.2. Column: 5% Dipenyl, 95% Dimethyl Polysiloxane - 30 m x 0.25 mm ID x 0.25 µm film thickness silicone-coated fused-silica capillary column or equivalent. Some projects may require a similar column, but with a different phase, to achieve improved separation.

7.1.3. Mass Spectrometer - Capable of scanning from 35 to 500 amu every 1 second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode, and capable of operating in the SIM mode.

7.1.4. GC/MS Interface - Any GC-to-MS interface that gives acceptable calibration points for each compound of interest and achieves acceptable tuning performance criteria may be used.

7.1.5. Data System - A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits.

7.2. Appropriate analytical balance (0.0001 g), volumetric flasks, syringes, vials, and bottles for standards preparation.

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1. Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination. The preparation for all laboratory prepared reagents and solutions must be documented in a laboratory logbook. Refer to the SOP *Reagent/Standards Login and Tracking (ADM-RTL)* for the complete procedure and documentation requirements.

8.2. BSTFA + TMCS 99:1, Supelco 33154-U: This is used to derivatize the phenols in extracts.

8.3. Stock Standard Solutions

8.3.1. Stock standard solutions may be purchased from a number of vendors. All reference standards, where possible, must be traceable to SI units or NIST certified reference materials. Commercially prepared stock standards are typically used when available at a concentration of 100 ug/ml or more. Standard concentrations can be verified by comparison versus an independently prepared standard. Alternatively, prepare stock standard solutions at a concentration of 1000 µg/ml by dissolving 0.0100 g of reference material in methylene chloride or other suitable solvent and diluting to volume in a 10mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard.

8.3.2. Transfer the stock standard solutions into amber Teflon-sealed crimp top autosampler vials at -10°C and protect from light, or store as recommended by the manufacturer. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. The expiration date is 1 year.

8.3.3. Unopened stock standards and neat materials have an expiration date equal to the manufacturer's recommendation. Neat material that does not have a manufacturer's recommended expiration date should be replaced after five years. Stock standard solutions received in sealed ampules with manufacture expiration dates in excess of 1 year have an expiration date of 1 year from the date of opening the sealed ampule.

8.4. Internal Standard Solutions - The internal standards are 1,4-dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12} (See Table 2 for corresponding compounds). The nominal concentration of the standard is 100ppb. For ultra low-level analyses, use 10ppb. Each 100 uL of sample extract undergoing analysis should be spiked with 1 µL of the internal standard solution. Store at -10°C or less when not being used. When using premixed certified solutions, store according to the manufacturer's recommendations. The expiration date is 1 year.

8.5. GC/MS Tuning Standard - A methylene chloride solution containing decafluorotriphenylphosphine (DFTPP) and pentachlorophenol is prepared. The concentration used (ng injected) will vary depending on the instrument model and/or sensitivity, but generally is 10-50ng. Store at -10°C or less when not being used, or store according to the manufacturer's recommendations. The expiration date is 1 year.

8.6. Calibration Standards

8.6.1. A minimum of five initial calibration standards must be prepared from stock solutions (note that a seven point calibration is recommended). One of the

calibration standards must be at a concentration at or below the method reporting limit. The others should correspond to the range of concentrations found in samples, but should not exceed the working range of the GC/MS system. Each 1 ml aliquot of calibration standards should be spiked with 10 µL of the internal standard solution prior to analysis. All calibration standards should be stored at -10°C or less and should be freshly prepared from stocks every 365 days, or sooner if check standards indicate a problem.

8.6.2. A calibration range of 10-5000 ng/ml is recommended. Higher concentrations may be prepared for poorer performing analytes. Calibration levels and injection amount may be adjusted to meet project-specific requirements.

8.6.3. The daily calibration standard (CCV) is prepared at a mid-level concentration from stock solutions. The CCV is prepared weekly and can be stored at 4 ± 2°C, or as recommended by the manufacturer.

8.7. QC Standards

8.7.1. Surrogates: Prepare a working solution in methanol containing 2-fluorophenol, phenol-d6, and 2,4,6-tribromophenol at 150 ng/µL and nitrobenzene-d5, 2-fluorobiphenyl, and terphenyl-d14 at 100 ng/µL. This solution may be combined with the surrogate solution used to monitor analyses for PAHs only (Biphenyl-d8, Fluorene-d10, and Fluoranthene-d10). Custom project-specific surrogate solutions may also be used. Aliquots of the solution are spiked into all extracted samples, blanks, and QC samples according to the extraction SOP used.

8.7.2. Matrix Spike Standards: Prepare a working solution in methanol containing all analytes of interest ("full list spike"). Base-Neutral analytes and acid analytes are prepared at a concentration of 100 ng/ul. Aliquots of the solution are spiked into the selected QC aliquots according to the extraction SOP used.

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument. Pertinent information (serial numbers, instrument I.D., etc.) must be in the logbook. This includes the routine maintenance described in section 9. The entry in the log must include: date of event, the initials of who performed the work, and a reference to analytical control.

9.2. Carrier gas - Inline purifiers or scubbers should be in place for all sources of carrier gas. These are selected to remove water, oxygen, and hydrocarbons. Purifiers should be changed as recommended by the supplier.

9.3. Gas Chromatograph

9.3.1. Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column. Injection port maintenance may include swabbing out the port, changing the injection port liner, seal, washer, o-ring, septum, column ferrule, and autosampler syringe as needed. Liners and seals should be changed when recent sample analyses predict a problem with chomatographic performance. In some cases liners and seals may be cleaned and re-used.

9.3.2. Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and "clean" (uniform, without fragmentation) by using the proper column cutting tool.

9.3.3. Over time, the column will exhibit poorer overall performance, as indicated by poor peak shape and reduced responses. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in performance is evident, more thorough maintenance is necessary. Some steps are to solvent rinse the split vent and septum lines with a mix of 20% methanol in DCM. When these and other maintenance options do not result in improvement, the column should be replaced. This is especially true when evident in conjunction with calibration difficulties.

9.4. Mass Spectrometer

9.4.1. For units under service contract, certain maintenance is performed by instrument service staff, including pump oil changed, vacuuming boards, etc., as recommended by the manufacturer.

9.4.2. MS source cleaning should be performed as needed, depending on the performance of the unit. This may be done by the analyst or by instrument service staff.

9.4.3. Tune the MS as needed to result in consistent and acceptable performance while meeting the required ion abundance criteria given in section 11.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in 8270D, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Sample Preparation

11.1.1. Water, soil, tissue and waste samples are prepared using the appropriate extraction and cleanup methods (refer to SOPs) and may be screened by GC/FID (see screening SOP). Soil, tissue, waste, and colored water extracts may be cleaned using EPA Method 3640 (GPC).

11.1.2. Tissue extracts and standards that are to be analyzed for phenols are derivatized by the following procedure: 100 ul of BSTFA+TMCS is added to 1.0 ml of the extract or standard in 2 ml autosampler vial. The vial is then sealed with a cap and heated to 60 C for 1.0 hour.

11.1.3. Following sample preparation, sample extracts are then transferred to the extract cold storage unit. Extracts must be analyzed within 40 days of extraction.

11.2. Typical GC/MS operating conditions are listed below, but should be optimized for specific instruments and projects to achieve optimal performance for the application.

Ion dwell time:	10 – 50 msec per ion
Initial temperature:	40°C, hold for 1 minutes
Temperature program:	40-150°C at 10°C/min hold for 2 minutes
Temperature program:	150-270°C at 15°C/min hold for 2 minutes
Final temperature:	270-300°C at 30°/min, hold for 4.33 minutes
Injector temperature:	275°C
Detector interface temp:	300°C
Injector:	Atas Optic, Agilent PTV or equivalent (parameters are method-specific)
Sample volume:	0.5-20 µL
Carrier gas:	helium at 1.3ml/min (constant flow)

11.3. Selected Ion Acquisition

11.3.1. Determine the ions to be monitored for the compounds of interest. Refer to Table 1 for characteristic ions. At a minimum, 2 ions (preferably 3 or more) are monitored for each compound, and a minimum of 3 monitored for halogenated compounds and compounds with more complex fragmentation patterns. Set the SIM windows in order to monitor the correct ions at the correct time, based on chromatographic elution of the compounds. This can be setup by analyzing a standard using a full scan analysis and using the GC conditions of the SIM analysis. This analysis will

give retention time and spectral information for determining the location of start times for the SIM groups or windows. This is often referred to as a "locator" analysis.

11.3.2. Select the dwell times to be used for each group of ions to be monitored. Dwell times should be selected in order to provide a sufficient number of measurements across the chromatographic peak to accurately define the peak shape. Too few measurements across the peak will result in poor definition of the peak and subsequently result in poor accuracy and precision of results. Too many measurements across the peak may result in inconsistent detector behavior over the calibration range. Significant differences in dwell times may also affect sensitivity.

11.4. Initial Calibration

NOTE: The calibration procedure(s) and options chosen must follow the CAS protocols. Any exceptions to the calibration procedures detailed in the CAS SOP for *Calibration of Instruments for Organics Chromatographic Analyses* (SOC-CAL) are described as follows:

11.4.1. Prior to calibration, analyze the GC/MS tuning standard using instrument conditions used for calibration. The DFTPP solution is analyzed to verify GC/MS tuning, injection port inertness, and GC column performance. This injection is acquired in full scan mode and evaluated in accordance with the method specified criteria.

11.4.2. Evaluate the spectrum obtained for DFTPP against the tuning criteria in Table 3. The GC/MS must meet the DFTPP ion abundance criteria prior to further analyses. Once the instrument is tuned, all subsequent analyses of standards, samples, and QA/QC samples must be analyzed using the identical mass spectrometer operating conditions. The following tuning options can be used.

- Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tune peak or part of any other peak eluting close to the tune peak.
- Use one scan at the apex of the peak. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tune peak or part of any other peak eluting close to the tune peak.

- Use one scan either directly preceding or following the apex of the peak. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tune peak or part of any other peak eluting close to the tune peak.
- Use the average across the entire peak up to a total of 5 scans. Peak integration must be consistent with standard operating procedure. If the peak is wider than 5 scans, the tune will consist of the peak apex scan and the two scans immediately preceding and following the apex. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tune peak or part of any other peak eluting close to the tune peak.

11.4.3. To assess column performance and injection port inertness, pentachlorophenol should be present at an acceptable level and peak tailing should not be excessive. Tailing is evaluated using a tailing factor. The tailing factor is calculated by the processing software as follows:

Tailing Factor = *PeakBack/PeakFront*

Where:

PeakBack is the time from Peak Apex to Peak End measured at 10% of the peak height
PeakFront is the time from Peak Start to Peak APEX measured at 10% of the peak height.

11.4.4. If excessive tailing or poor chromatography is noted, the injection port may require cleaning. It may also be necessary to remove the first 15-30 cm of the GC column. If hardware tuning criteria can not be met, the source may need cleaning, filaments replaced or other maintenance.

11.4.5. The internal standards should permit most of the components of interest in the chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Refer to Table 2 for internal standards and corresponding analytes assigned for quantitation. Use the base peak ion from the specific internal standard as the primary ion for quantitation (See Table 1). If interferences are noted, use the next most intense ion as the quantitaion ion (i.e. for 1,4-dichlorobenzene-d_4, use 152 m/z for quantitation).

11.4.6. Analyze each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each compound (as

indicated in Table 2). Calculate response factors (RFs) for each compound relative to one of the internal standards as follows:

$$RF = (A_x C_{is})/(A_{is} C_x)$$

where:

A_x = Area of the characteristic ion for compound being measured.
A_{is} = Area of the characteristic ion for specific internal standard.
C_{is} = Concentration of the specific internal standard (ng/µL).
C_x = Concentration of the compound being measured (ng/µL).

11.4.7. The recommended minimum acceptable average RF for target analytes is given in Table 1. If the RF does not meet these recommendations the GC system may require maintenance (see section 9) and recalibration.

11.4.8. The percent relative standard deviation (%RSD) should be less than 20% for each compound. The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time units.

$$\%RSD = \frac{SD}{\overline{RF}} \times 100$$

where:

RSD = relative standard deviation.

$\overline{RF}$ = mean of 5 initial RFs for a compound.
SD = standard deviation of average RFs for a compound.

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

where:

RF_i = RF for each of the 5 calibration levels
N = Number of RF values (i.e., 5)

11.4.9. Linearity - If the % RSD of any compound is 20% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

11.4.10. If the %RSD for a compound is >20%, then alternative calibration models should be used. See the SOP (SOC-CAL) *Calibration of Instruments for Organics Chromatographic Analysis* for further guidance.

11.5. Review of calibration curve

11.5.1. The calibration curve must be reviewed to ensure it represents the calibration data. This is done by re-fitting each calibration level against the true concentration of each calibration standard. See the SOP (SOC-CAL) *Calibration of Instruments for Organics Chromatographic Analysis* for further guidance.

11.6. Independent Calibration Verification

11.6.1. Following initial calibration, analyze an ICV standard. The ICV solution must contain all analytes in the calibration standards. Calculate the concentration using the typical procedure used for quantitation. Calculate the percent difference (%D) from the ICV true value. For each compound of interest, the calculated value must be ± 30% of the true value for the initial calibration to be valid.

11.7. Continuing Calibration

11.7.1. Following an acceptable tune, a calibration standard, or standards, at mid-concentration containing all semivolatile analytes, and all required surrogates, must be analyzed every 12 hours during analysis.

11.7.2. If the percent drift for each compound is less than or equal to 20%, the initial calibration is assumed to be valid.

11.7.3. If the criterion is not met (> 20% drift) for any one compound, corrective action must be taken. Problems similar to those listed. If no source of the problem can be determined after corrective action has been taken, a new initial calibration must be generated. This criterion must be met before sample analysis begins.

Calculate the percent drift using:

$$\%\ Drift = \frac{C_1 - C_c}{C_1} \times 100$$

where:

C_1 = Compound standard concentration.
C_c = Measured concentration using selected quantitation method.

11.7.4. Non-detected analytes can be reported from analyses when a CCV that does not meet the criteria. However, the CCV must exhibit a positive bias (i.e., outside the upper

control limit), no further documentation is required. This is considered a temporary measure and corrective action must be taken in order that subsequent CCVs may pass standard criteria.

11.7.5. The internal standard responses and retention times in the calibration check standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the midpoint standard of the most recent initial calibration sequence, the chromatographic system must be inspected for malfunctions and corrective action identified, as required. If the EICP area for any of the internal standards changes by a factor of two (50% to 200%) from that in the midpoint standard of the most recent initial calibration sequence, the chromatographic system must be inspected for malfunctions and corrective action identified, as appropriate. When corrective action is taken, reanalysis of samples analyzed while the system was malfunctioning is required. Update the reference spectra and retention times in the quanitiation database for the instrument method or ID file. The initial calibration average RF or calibration curve is then used in the quanitiation of subsequent analyses.

11.7.6. A blank (method blank, GPC blank, or solvent blank) should be analyzed after the CCV, or at any other time during the analytical shift, to prove the system is free of contaminants. If contaminants are found in a method blank or GPC blank, then a solvent blank should be analyzed to help isolate the source of contamination.

11.8. GC/MS Analysis

11.8.1. Evaluate FID screen and make proper dilution (See FID screening SOP).

11.8.2. Spike the 1 ml extract obtained from sample preparation with 10 µL of the internal standard solution just prior to analysis. Use the same operating conditions as were used for initial calibration.

11.8.3. If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, extract dilution must take place. Additional internal standard must be added to the diluted extract to maintain the required concentration of each internal standard in the extracted volume. The diluted extract must be reanalyzed.

11.8.4. Store the extracts at -10°C or less, protected from light in vials equipped with unpierced Teflon lined septa. Archive extract in freezer for 3 months after analysis in the instrument/date specific storage boxes.

12. QA/QC REQUIREMENTS

12.1. In addition to instrument criteria for calibration, the ability of each analyst/instrument to generate acceptable accuracy and precision must be documented prior to sample analysis (IPR study). This must be validated before analysis of samples begins, or whenever significant changes to the procedures have been made. To do this, four deionized water samples are spiked with each target analyte, extracted, and analyzed. Refer to Method 8270D Section 8.3 for this requirement and acceptance criteria.

12.2. Method Detection Limits

12.2.1. For projects that require reporting to the method detection limit (MDL), a method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank replicates with a MDL spiking solution (at a level below the MRL) for each target analyte, extract, and analyze. The MDL studies should be done for each matrix, prep method, and instrument. Refer to the CAS SOP *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification.*

12.2.2. Calculate the average concentration found (x) in the *sample concentration*, and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. The MDL study must be verified annually.

12.3. Ongoing QC Samples required are described in the CAS-Kelso Quality Assurance Manual and in the SOP for *Sample Batches*. In general, these include:

12.3.1. Method blank - A method blank is extracted and analyzed with every batch of 20 or fewer samples to demonstrate that there are no method interferences. The method blank must demonstrate that interferences from the analytical and preparation steps minimized. No target analytes should be detected above the MRL in the method blank. For some project specific needs, exceptions may be noted and method blank results above the MRL may be reported for common lab contaminants (phthalate esters, etc.).

12.3.2. A lab control sample (LCS) must be extracted and analyzed with every batch of 20 or fewer samples. The LCS is prepared by spiking a blank with the matrix spike solution, and going through the entire extraction and analysis. Calculate percent recovery (%R) as follows:

$$\%R = X/TV \times 100$$

Where X = Concentration of the analyte recovered
TV = True value of amount spiked

Acceptance criteria for lab control samples are listed in Table 4. If the LCS recovery for any control analyte fails acceptance limits, corrective action is required. If any other analyte fails the advisory acceptance limits, the analyst must evaluate the impact on data quality and take any necessary corrective action, which may include re-extraction of the associated samples. Project-specific requirements may require the use of project acceptance criteria.

12.3.3. A matrix spike/duplicate matrix spike (MS/DMS) must be extracted and analyzed with every batch of 20 or fewer samples. The MS is prepared by spiking a sample aliquot with the matrix spike solution, and going through the entire extraction and analysis. Calculate percent recovery (%R) as follows:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

Calculate Relative Percent Difference (RPD) as:

$$\%RPD = \frac{R1 - R2}{(R1 + R2)/2} \times 100$$

Where R1 = recovered amount in the higher result
R2 = recovered amount in the lower result

The acceptance limits for the MS/DMS recovery are given in Table 4. If the MS/DMS recovery is out of acceptance limits for reasons other than matrix effects, corrective action must be taken. The RPD acceptance limits are 30% for water and 40% for soils, sediments, and solids. Project-specific requirements may dictate the use of project acceptance criteria.

12.3.4. The acceptance limits for the surrogates are given in Table 4. If any surrogate recovery is outside acceptance criteria, the sample data must be closely evaluated for possible matrix interferences. If none are present, then corrective action must be taken. The sample should be re-analyzed if instrument factors (calibration, injection port) are suspected. If not, re-extraction and re-analysis is required,

except in cases of high recovery and no positive hits in the sample for the analyte class represented by the particular surrogate.

12.3.5. Additional QA/QC measures include control charting of QC sample results.

12.4. Corrective action – When a data quality objective is not met, the initial corrective action will include a review of the raw data for potential calculation and/or integration errors. If this review does not correct the problem, the following corrective actions will be performed.

12.4.1. Method Blank – No target analyte should be detected in a method blank at or above the method reporting limit (1/2 the MRL for DoD projects). If target analytes are detected in the method blank, the sample data must be reviewed for possible laboratory contribution. Detections of target analytes greater than the MRL require a Nonconformity and Corrective Action Report (NCAR). A decision to reextract the associated samples will depend on the level of the contamination, data quality implications, and the intended use of the data. This is especially true for positive detections of common laboratory contaminants (e.g., phthalate esters). At a minimum, all positive detections in the associated samples that are not more than 20X the concentration in the blank will be qualified with a "B". Also, as part of the corrective action, the problem will be discussed with the appropriate sample prep personnel in an effort to identify the contamination source.

12.4.2. Laboratory Control Sample – The analysis should include a full list LCS spike. All target analytes will be evaluated. The following cases require corrective action:

- If any control analytes do not meet acceptance criteria, the analytical batch should be considered out of control for that analyte. Corrective action may include reinjection to verify the result. If the result is confirmed, a NCAR will be filed. A decision to re-extract the associated samples will depend on the data quality implications and the intended use of the data. If re-extraction is not feasible, all reported results for that analyte will be qualified and the data quality implications will be discussed in the case narrative.
- In cases where a result is outside the upper control criterion, corrective action is only required if that analyte was also detected in field samples. The associated samples with positive results should be re-extracted. In cases where a result is outside the lower control criterion, the associated samples should be re-extracted. If re-extraction is not feasible, all reported results for that analyte will be qualified and the data quality implications will be discussed in the case narrative.

12.4.3. Matrix Spike and Duplicate Matrix Spike Samples – A subset of analytes, as specified in Method 8270D, are monitored in the matrix spike and duplicate matrix spike analyses. If a recovery is outside of control criteria, review the consistency between the two analyses. If the result is supported between the two analyses, the

outlier can be attributed to matrix interference. Do not reanalyze the extract. If the results do not support each other, reanalyze the extracts to verify the results. If the results confirm, review the LCS recovery and take corrective action accordingly. If the LCS recovery is acceptable, flag the matrix spike data and discuss potential data quality implications in the case narrative.

12.4.4. Relative Percent Difference – For MS/DMS or LCS/DLCS, no corrective action is required based on RPD data alone. However, the data should be reviewed for information that will help determine if the RPD problem is the result of a sample specific issue (e.g., the DMS was concentrated to dryness), or if the problem is representative of the entire analytical batch. When the problem is apparently universal to the batch, a NCAR will be filed and the batch will be re-extracted. If results of the re-extraction confirm the original analyses of the field samples, the original data is reported and the RPD problems are discussed in the case narrative. If results of the re-extraction confirm a problem in the original data, only the re-extracted data is reported.

12.4.5. Surrogates – Up to six surrogates are used to monitor the analysis (three base-neutral compounds and three acid compounds). If any surrogate is recovered less than 10%, the analysis should be considered out of control for that sample. Corrective action includes reinjection to verify the result. If the result is confirmed, a NCAR will be filed and the sample will be re-extracted. If the re-extraction confirms the original results are biased due to matrix interferences, report the original data. If re-extraction is not feasible, the surrogate will be qualified and the data quality implications will be discussed in the case narrative.

Note: If all target analytes are "acid" analytes, then only the acid surrogates are needed. If all target analytes are base/neutral analytes, then only the base/neutral surrogates are needed

13. DATA REDUCTION, REVIEW, AND REPORTING

13.1. Qualitative Analysis - The qualitative identification of compounds determined by this procedure is based on retention time, and comparison of the sample mass spectrum with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the instrument and conditions used for the sample analysis. The characteristic ions from the reference mass spectrum are defined to be the ions monitored in the SIM mode and typically are the two or three ions of greatest relative intensity. Compounds are identified as present when the criteria below are met.

13.2. Qualitative Analysis - The qualitative identification of compounds determined by this procedure is based on retention time, and comparison of the sample mass spectrum with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the instrument and conditions used for the

sample analysis. The characteristic ions from the reference mass spectrum are defined to be the ions monitored in the SIM mode and typically are the two or three ions of greatest relative intensity. Compounds are identified as present when the criteria below are met.

13.2.1. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

13.2.2. The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

13.2.3. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

13.2.4. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is <25% of the sum of the 2 peak heights. Otherwise, structural isomers are identified as isomeric pairs.

13.2.5. Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When the ion relative intensities do not agree within 30% due to these types of matrix interferences, the analyst uses professional judgement in determining whether the analyte is or is not present. The analyst will take into account other knowledge of the sample, presence of other compounds, and other presumptive evidence available for review. Difficulties will be noted in the case narrative.

13.2.6. Evaluate internal standard areas in each sample. If the area in the sample is less than 50% or greater than 200% the area of in the CCV, corrective action must be taken. Depending on the analysis, this corrective action may include reinection or dilution of the extract followed by reinjection.

13.3. Tentatively identified compound (TIC) cannot be reported using this method.

13.4. Quantitation and Calculations

13.4.1. The GC/MS data stations, in current use, all use the H-P RTE Integrator to generate the raw data used to calculate the standards $\overline{RF_x}$ values, the sample amounts, and the spike values. The software does three passes through each data file. The first two identify and integrate each internal standard and surrogate. The third pass uses the

time-drift information from the first two passes to search for all method analytes in the proper retention times and with the proper characteristic quantitation ions. When $\overline{RF_x}$ is used, calculate the extract concentration as follows:

$$C_{ex} = \frac{(Resp_x)(Amt_{ISTD})}{(Resp_{ISTD})(\overline{RF_x})}$$

Where: C_{ex} = the concentration in the sample extract (ppm);
$Resp_x$ = the peak area of the analytes of interest;
$Resp_{ISTD}$ = the peak area of the associated internal standard;
Amt_{ISTD} = the amount, in ppm, of internal standard added
$\overline{RF_x}$ = the average response from the initial calibration.

13.4.2. The concentration of analytes in the original sample is computed using the following equations:

Aqueous Samples: $Concentration\ (\mu g / L) = \frac{(Cex)\ (Vf)\ (D)}{(Vs)}$

Where	Cex	=	Concentration in extract in µg/mL
	Vf	=	Final volume of extract in mL
	D	=	Dilution factor
	Vs	=	Volume of sample extracted, liters

Nonaqueous Samples: $Concentration\ (mg / Kg) = \frac{(Cex)\ (Vf)\ (D)}{(W)}$

Where	Cex	=	Concentration in extract in µg/mL
	Vf	=	Final volume of extract in mL
	D	=	Dilution factor
	W	=	Weight of sample extracted in grams.

13.5. Data Review

Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the SOP for *Laboratory Data Review Process* for details.

13.6. Reporting

Reports are generated in the CAS LIMS by compiling the SMO login, sample prep database, instrument, date, and client-specified report requirements (when specified). This compilation is then transferred to a file which the Stealth reporting system uses to generate a report. The forms generated may be CAS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

14. CORRECTIVE ACTION

14.1. Refer to the SOP for *Corrective Action (ADM-CA)* for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2. Handling out-of-control or unacceptable data

14.2.1. On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, runlogs, for example.

14.2.2. Documentation of a nonconformity must be done using a Nonconformity and Corrective Action Report (NCAR) when:

- Corrective action is not taken or not possible
- Corrective action fails to correct an out-of-control problem on a laboratory QC or calibration analysis.
- Reanalysis corrects the nonconformity but is not a procedurally compliant analysis.

15. METHOD PERFORMANCE

15.1. This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

15.2. The method detection limit (MDL) is established using the procedure described in the SOP for *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification* (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

16. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible, and within method requirements. Standards are prepared in volumes consistent with laboratory use in order to minimize the volume of expired standards to be disposed of. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

17. WASTE MANAGEMENT

17.1. The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

17.2. This method uses methylene chloride and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and recycled off site.

18. TRAINING OUTLINE

18.1. The following items provide guidelines for training analysts.

18.1.1. Review applicable literature (method references, etc.) and this SOP. Review the MSDS for all chemicals used in the analysis.

18.1.2. Observe the procedure as performed by an experienced analyst at least three times.

18.1.3. Assist in the procedure under the guidance of an experienced analyst for at least one month, preferably three months. During this training period, the analyst is expected to progress from a role of assisting to a role of performing the procedure with minimal oversight.

18.2. Following this training period, the analyst is expected to complete an Initial Precision and Recovery (IPR) study as described in Section 12. Documentation of the IPR study should be forwarded to the analyst's training file. Refer to the SOP ADM-TRANDOC *Documentation of Training.*

19. METHOD MODIFICATIONS

19.1. There are no know modifications from the reference method

19. REFERENCES

Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Method 8270D, Revision 4, EPA Test Methods for Evaluating Solid Waste, SW-846, Final Update IV, February 2007.

20. CHANGES SINCE THE LAST REVISION

20.1. Updated to comply with EPA 8270D and references
20.2. Sec 1.1 removed PQL as equivalent to MRL
20.3. Sec 8.1 updated to comply with QA systems requirements
20.4. Sec 8.2 is new
20.5. Sec 9.1 updated to comply with QA systems requirements
20.6. Sec 11.1.2 is new
20.7. Sec 11 – updated % RSD from 15% to 20% and added referencing SOC-CAL for alternative curve fit. Removed the use of 'averaging' of RSD for evaluating curves
20.8. Sec 11.4.7 added reference to recommended RF
20.9. Sec 11.6.1 changed ICV acceptance from 20% to 30%
20.10. Sec 11.7.4 is new
20.11. Sec 14 updated
20.12. Updated Tables

TABLE 1
TARGET COMPOUNDS AND CORRESPONDING PRIMARY AND SECONDARY QUANTITATION IONS and RECOMMENDED MINIMUM RF

Compound	Min RF	Primary Ion	Secondary Ion
Phenol	0.800	94	66
1,3-Dichlorobenzene	0.050	146	148,111
1,4-Dichlorobenzene-d 4 (I.S.)	-	152	150
1,4-Dichlorobenzene	0.050	146	148,111
1,2-Dichlorobenzene	0.050	146	79148,111
Benzyl alcohol	0.050	79	108,77
2-Methylphenol	0.700	108	77
Hexachloroethane	0.050	117	119,121
3-and 4-Methylphenol (coelute)	0.600	108	77
2,4-Dimethylphenol	0.200	122	107
1,2,4-Trichlorobenzene	0.050	180	182,145
Naphthalene-d 8 (I.S.)	-	136	68
Naphthalene	0.700	128	127
Benzoic acid	0.050	122	105
Hexachlorobutadiene	0.010	225	227,223
2-Methylnaphthalene	0.400	141	142
Acenaphthylene	0.900	152	153
Dimethyl phthalate	0.01	163	164,194
Acenaphthlene-d 10 (I.S.)	-	164	162
Acenaphthlene	0.900	154	153
Dibenzofuran	0.800	168	139
Fluorene	0.900	166	165
Diethyl phthalate	0.010	149	177
N-Nitrosodiphenylamine	0.010	169	168,167
Hexachlorobenzene	0.100	284	249,142
Pentachlorophenol	0.050	266	268
Phenanthrene-d 10 (I.S.)	-	188	94
Phenanthrene	0.700	178	179
Anthracene	0.700	178	176
Di-n-butyl phthalate	0.010	149	150
Fluoranthene	0.600	202	203
Pyrene	0.600	202	203
Butyl Benzyl Phthalate	0.010	149	91,206
Benz (a) anthracene	0.800	228	226
Chrysene-d 12 (I.S.)	-	240	236
Chrysene	0.700	228	226

TABLE 1
TARGET COMPOUNDS AND CORRESPONDING PRIMARY AND SECONDARY QUANTITATION IONS and RECOMMENDED MINIMUM RF

(continued)

Bis (2-ethylhexyl) phthalate	0.010	149	167,279
Di-n-octyl phthalate	0.010	149	150,279
Benzo(b)fluoranthene	0.700	252	126
Benzo(k)fluoranthene	0.700	252	126
Benzo(a)pyrene	0.700	252	126
Perylene-d 12 (I.S.)	-	264	260
Indeno (1,2,3-cd) pyrene	0.500	276	277
Dibenz (a, h) anthracene	0.400	278	279
Benzo(g,h,i)perylene	0.500	276	277
2-Fluorophenol (surr.)	-	112	64
Phenol-d 6 (surr.)	-	99	71
Nitrobenzene-d 5 (surr.)	-	82	128
2-Fluorobiphenyl (surr.)	-	172	171
2,4,6-Tribromophenol (surr.)	-	330	332
Terphenyl-d 14 (surr.)	-	244	122
1,4-Dioxane	0.010	88	58,43
1,4-Dioxane-d8	0.010	96	64,48
Derivatized Phenols			
2-Methylphenol	0.05	151	166,152
4-Methylphenol	0.05	165	180,90
2-Chlorophenol	0.05	185	200,93
2,4-Dimethylphenol	0.05	194	179,105
Benzoic Acid	0.05	179	105,135,194
4-Chloro-3-methylphenol	0.05	199	214,201
2,4-Dichlorophenol	0.05	219	234,183
2-Nitrophenol	0.05	196	151,197
2,4,6-Trichlorophenol	0.05	253	255,270
2,4,5-Trichlorophenol	0.05	253	255,270
4-Nitrophenol	0.05	196	211,150
2,3,4,6-Tetrachlorophenol	0.05	289	287,304
Pentachlorophenol	0.05	323	325,338
2-Fluorophenol	0.05	91	184
Phenol-d6	0.05	156	171
2,4,6-Tribromophenol	0.05	389	404,227

TABLE 2

SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANITATION*

1,4-Dichlorobenzene-d4	**Naphthalene-d8**	**Acenaphthene-d10**
Phenol	Benzoic Acid	Acenaphthene
Benzyl Alcohol	2,4-Dimethylphenol	Acenaphthylene
1,3-Dichlorobenzene	Hexachlorobutadiene	Dibenzofuran
1,4-Dichlorobenzene	Naphthalene	Diethyl phthalate
1,2-Dichlorobenzene	1,2,4-Trichlorobenzene	Dimethyl phthalate
2-Fluorophenol (surr.)	2-Methylnaphthalene	Fluorene
Hexachloroethane		2-Fluorobiphenyl (surr.)
2-Methylphenol		Hexachlorocyclopentadiene
4-Methylphenol		N-Nitrosodiphenylamine
Phenol-d (6) (surr.)		
Nitrobenzene-d (5) (surr.)		

Phenanthrene-d10	**Chrysene-d12**	**Perylene-d12**
Anthracene	Benzo(a) anthracene	Benzo(b)fluoranthene
Di-n-butyl phthalate	Bis (2-ethylhexyl)phthalate	Benzo(k)fluoranthene
Hexachlorobenzene	Butyl benzyl phthalate	Benzo(g,h,i)perylene
Pentachlorophenol	Chrysene	Dibenz(a,h)anthracene
Phenanthrene	Pyrene	Benzo(a)pyrene
2,4,6-Tribromophenol (surr.)	Terphenyl-d (14) (surr.)	Indeno (1,2,3-ccd)pyrene
Fluoranthene		Di-n-octyl-phthalate

* Example list only

TABLE 3
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

TABLE 3A
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA
FOR **5973** GC/MS SYSTEMS

Mass	Ion Abundance Criteria
51	10-80% of mass 198
68	0-2% of mass 69
70	0-2% of mass 69
127	10-80% of 198
197	0-2% of 198
198	30-100% of 442 (alternate base)
199	5-9% of 198
275	10-60% of 198
365	1-50% of 442
441	0.01-100% of 443
442	30-100% of 198 (alternate base)
443	15-24% of 442

Alternate tuning criteria (from Method 525.2 or CLP OLM04.2) may be used provided that method performance is not adversely affected and that method performance criteria is met. The criteria used must be the same for **all** ion abundance criteria checks associated with a given analysis. For example, initial calibration, continuing calibration(s), QC, and sample analyses for a given sample must all use the same criteria.

TABLE 4

CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	LODb	LOQc	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)
8270 SIM	Pentachlorophenol	87-86-5	Soil	6	200	30	200	ug/kg	10-117	10-117	40
8270 SIM	1,4-Dioxane	123-91-1	Soil	0.48	5	1	5	ug/Kg	44-125	39-150	40
8270 SIM	1,4-Dioxane-d8 (Surr.)	17647-74-4	Soil	NA	NA	NA	NA	%	35-151	NA	NA
8270 SIM	PBDE 100	189084-64-8	Soil	0.014	0.1	0.03	0.1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 128	CASID30336	Soil	0.013	0.1	0.03	0.1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 138	182677-30-1	Soil	0.0059	0.1	0.01	0.1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 153	68631-49-2	Soil	0.011	0.1	0.03	0.1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 154	207122-15-4	Soil	0.007	0.1	0.02	0.1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 17	147217-75-2	Soil	0.011	0.1	0.03	0.1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 183	207122-16-5	Soil	0.03	0.1	0.03	0.1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 190	CASID30338	Soil	0.014	0.1	0.03	0.1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 203	CASID30339	Soil	0.016	0.1	0.03	0.1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 206	63936-56-1	Soil	0.018	1.0	0.03	1.0	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 209	1163-19-5	Soil	0.029	1.0	0.03	1.0	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 28	41318-75-6	Soil	0.015	0.1	0.03	0.1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 47	5436-43-1	Soil	0.12	0.1	0.03	0.1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 66	189084-61-5	Soil	0.022	0.1	0.03	0.1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 71	189084-62-6	Soil	0.0087	0.1	0.02	0.1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 85	182346-21-0	Soil	0.0081	0.1	0.02	0.1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 99	60348-60-9	Soil	0.019	0.1	0.03	0.1	ug/Kg	70-130	70-130	40
8270 SIM	Tetrabromobisphenol A	79-94-7	Soil	1.1	4			ug/Kg	70-130	70-130	40
8270 SIM	PBDE 47C13 (Surr.)	CASID30341	Soil	NA	NA	NA	NA	%	70-130	70-130	NA
8270 SIM	PBDE 99C13 (Surr.)	CASID30342	Soil	NA	NA	NA	NA	%	70-130	70-130	NA
8270 SIM	Tetrabromobisphenol A C13 (Surr.)	CASID30439	Soil	NA	NA	NA	NA	%	70-130	70-130	NA
8270 SIM	N-Nitrosodimethylamine-d6 (Surr.)	17829-05-9	Soil	NA	NA	NA	NA	%	23-156	NA	NA

TABLE 4 cont.

CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	LODb	LOQc	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)
8270 SIM	Pentachlorophenol	87-86-5	Water	0.017	1	0.2	1	ug/L	NA	NA	30
8270 SIM	1,4-Dioxane	123-91-1	Water	0.16	1	0.4	1	ug/L	52-105	40-114	30
8270 SIM	1,4-Dioxane-d8 (Surr.)	17647-74-4	Water	NA	NA	NA	NA	%	42-112	NA	NA
8270 SIM	N-Nitrosodimethylamine	62-75-9	Water	0.2	2	0.4	2	ng/L	70-130	70-130	30
8270 SIM	Di(proplyene glycol) butyl ether	29911-28-2	Water	0.06	0.25	0.1	0.25	ug/L	70-130	70-130	30
8270 SIM	PBDE 100	189084-64-8	Water	0.064	1.0	0.15	1.0	ng/L	61-103	61-103	30
8270 SIM	PBDE 128	CASID30336	Water	0.11	1.0	0.3	1.0	ng/L	50-111	50-111	30
8270 SIM	PBDE 138	182677-30-1	Water	0.1	1.0	0.3	1.0	ng/L	57-106	57-106	30
8270 SIM	PBDE 153	68631-49-2	Water	0.11	1.0	0.3	1.0	ng/L	60-109	60-109	30
8270 SIM	PBDE 154	207122-15-4	Water	0.075	1.0	0.15	1.0	ng/L	62-103	62-103	30
8270 SIM	PBDE 17	147217-75-2	Water	0.12	1.0	0.3	1.0	ng/L	64-103	64-103	30
8270 SIM	PBDE 183	207122-16-5	Water	0.1	1.0	0.3	1.0	ng/L	55-183	55-183	30
8270 SIM	PBDE 190	CASID30338	Water	0.12	1.0	0.3	1.0	ng/L	44-117	44-117	30
8270 SIM	PBDE 203	CASID30339	Water	0.095	1.0	0.15	1.0	ng/L	44-115	44-115	30
8270 SIM	PBDE 206	63936-56-1	Water	0.09	10	0.15	10	ng/L	31-141	31-141	30
8270 SIM	PBDE 209	1163-19-5	Water	0.39	10	0.5	10	ng/L	27-143	27-143	30
8270 SIM	PBDE 28	41318-75-6	Water	0.21	1.0	0.5	1.0	ng/L	62-104	62-104	30
8270 SIM	PBDE 47	5436-43-1	Water	0.11	1.0	0.3	1.0	ng/L	63-100	63-100	30
8270 SIM	PBDE 66	189084-61-5	Water	0.34	1.0	0.5	1.0	ng/L	60-105	60-105	30
8270 SIM	PBDE 71	189084-62-6	Water	0.1	1.0	0.3	1.0	ng/L	61-104	61-104	30
8270 SIM	PBDE 85	182346-21-0	Water	0.059	1.0	0.15	1.0	ng/L	58-106	58-106	30
8270 SIM	PBDE 99	60348-60-9	Water	0.12	1.0	0.3	1.0	ng/L	61-103	61-103	30
8270 SIM	PBDE 99C13 (Surr.)	CASID30342	Water	NA	NA	NA	NA	%	43-121		NA
8270 SIM	PBDE 47C13 (Surr.)	CASID30341	Water	NA	NA	NA	NA	%	41-121		NA

TABLE 4 cont.

CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)
8270 SIM	1,2,4,5-Tetrachlorobenzene	95-94-3	Tissue	6.0	40	ug/Kg	70-130	70-130	40
8270 SIM	1,2,4-Trichlorobenzene	120-82-1	Tissue	4.2	40	ug/Kg	1/10-8/11 Updated	36-119	40
8270 SIM	1,2-Dichlorobenzene	95-50-1	Tissue	6.5	40	ug/Kg	60-110	60-110	40
8270 SIM	1,2-Diphenylhydrazine	122-66-7	Tissue	2.5	40	ug/Kg	70-130	70-130	40
8270 SIM	1,3-Dichlorobenzene	541-73-1	Tissue	8.6	40	ug/Kg	59-109	59-109	40
8270 SIM	1,4-Dichlorobenzene	106-46-7	Tissue	7.6	40	ug/Kg	60-108	60-108	40
8270 SIM	2,3,4,5-Tetrachlorophenol	4901-51-3	Tissue	40	40	ug/Kg	70-130	70-130	40
8270 SIM	2,3,4,6-Tetrachlorophenol	58-90-2	Tissue	40	40	ug/Kg	48-136	48-136	40
8270 SIM	2,3,4-Trichlorophenol	15950-66-0	Tissue	40	40	ug/Kg	70-130	70-130	40
8270 SIM	2,3,5,6-Tetrachlorophenol	935-95-5	Tissue	40	40	ug/Kg	70-130	70-130	40
8270 SIM	2,3,5-Trichlorophenol	933-78-8	Tissue	40	40	ug/Kg	70-130	70-130	40
8270 SIM	2,3,6-Trichlorophenol	933-75-5	Tissue	40	40	ug/Kg	70-130	70-130	40
8270 SIM	2,4,5-Trichlorophenol	95-95-4	Tissue	3.1	40	ug/Kg	49-121	49-121	40
8270 SIM	2,4,6-Trichlorophenol	88-06-2	Tissue	3.1	40	ug/Kg	47-120	47-120	40
8270 SIM	2,4-Dichlorophenol	120-83-2	Tissue	5.8	40	ug/Kg	47-117	47-117	40
8270 SIM	2,4-Dimethylphenol	105-67-9	Tissue	17	40	ug/Kg	10-101	10-101	40
8270 SIM	2,4-Dinitrophenol	51-28-5	Tissue	180	500	ug/Kg	40-172	40-172	40
8270 SIM	2,4-Dinitrotoluene	121-14-2	Tissue	5.9	80	ug/Kg	54-109	54-109	40
8270 SIM	2,6-Dinitrotoluene	606-20-2	Tissue	5.8	40	ug/Kg	60-117	60-117	40
8270 SIM	2-Chloronaphthalene	91-58-7	Tissue	2.6	40	ug/Kg	70-130	70-130	40
8270 SIM	2-Chlorophenol	95-57-8	Tissue	2.8	40	ug/Kg	63-106	63-106	40
8270 SIM	2-Methyl-4,6-dinitrophenol	534-52-1	Tissue	260	500	ug/Kg	46-119	46-119	40
8270 SIM	2-Methylnaphthalene	91-57-6	Tissue	4.1	40	ug/Kg	76-159	76-159	40
8270 SIM	2-Methylphenol	95-48-7	Tissue	3.2	40	ug/Kg	20-119	20-119	40
8270 SIM	2-Nitroaniline	88-74-4	Tissue	7.6	200	ug/Kg	59-116	59-116	40
8270 SIM	2-Nitrophenol	88-75-5	Tissue	3.8	40	ug/Kg	37-135	37-135	40
8270 SIM	3,4,5-Trichlorophenol	609-19-8	Tissue	40	40	ug/Kg	70-130	70-130	40
8270 SIM	3-Nitroaniline	99-09-2	Tissue	56	400	ug/Kg	57-124	57-124	40
8270 SIM	4-Bromophenyl Phenyl Ether	101-55-3	Tissue	4.1	40	ug/Kg	42-118	42-118	40

8270 SIM	4-Chloro-3-methylphenol	59-50-7	Tissue	3.0	40	ug/Kg	44-123	44-123	40
8270 SIM	4-Chlorophenyl Phenyl Ether	7005-72-3	Tissue	3.0	40	ug/Kg	42-114	42-114	40
8270 SIM	4-Methylphenol	106-44-5	Tissue	11	40	ug/Kg	22-125	22-125	40
8270 SIM	4-Nitroaniline	100-01-6	Tissue	88	400	ug/Kg	50-135	50-135	40
8270 SIM	4-Nitrophenol	100-02-7	Tissue	6.3	40	ug/Kg	51-154	51-154	40
8270 SIM	Acenaphthene	83-32-9	Tissue	1.9	40	ug/Kg	46-122	54-104	40
8270 SIM	Acenaphthylene	208-96-8	Tissue	3.5	40	ug/Kg	52-124	52-110	40
8270 SIM	Acetophenone	98-86-2	Tissue	14	80	ug/Kg	70-130	70-130	40
8270 SIM	Anthracene	120-12-7	Tissue	3.5	40	ug/Kg	70-130	70-130	40
8270 SIM	Azobenzene	103-33-3	Tissue	2.5	40	ug/Kg	49-118	49-118	40
8270 SIM	Benz(a)anthracene	56-55-3	Tissue	6.6	40	ug/Kg	50-119	63-102	40
8270 SIM	Benzo(a)pyrene	50-32-8	Tissue	4.4	40	ug/Kg	56-127	52-112	40
8270 SIM	Benzo(b)fluoranthene	205-99-2	Tissue	3.0	40	ug/Kg	50-119	53-108	40
8270 SIM	Benzo(g,h,i)perylene	191-24-2	Tissue	2.7	40	ug/Kg	52-118	57-106	40
8270 SIM	Benzo(k)fluoranthene	207-08-9	Tissue	2.3	40	ug/Kg	51-119	55-106	40
8270 SIM	Benzyl Alcohol	100-51-6	Tissue	16	40	ug/Kg	37-138	37-138	40
8270 SIM	Biphenyl	92-52-4	Tissue	5.1	40	ug/Kg	55-115	49-117	40
8270 SIM	Bis(2-chloroethoxy)methane	111-91-1	Tissue	2.4	40	ug/Kg	63-113	63-113	40
8270 SIM	Bis(2-chloroethyl) Ether	111-44-4	Tissue	3.0	40	ug/Kg	59-112	59-112	40
8270 SIM	Bis(2-chloroisopropyl) Ether	39638-32-9	Tissue	15	40	ug/Kg	59-117	59-117	40
8270 SIM	Bis(2-ethylhexyl) Phthalate	117-81-7	Tissue	66	200	ug/Kg	56-136	56-136	40
8270 SIM	Butyl Benzyl Phthalate	85-68-7	Tissue	7.3	40	ug/Kg	44-141	44-141	40
8270 SIM	Caprolactam	105-60-2	Tissue	63	200	ug/Kg	70-130	70-130	40
8270 SIM	Carbazole	86-74-8	Tissue	7.7	40	ug/Kg	66-123	66-123	40
8270 SIM	Chrysene	218-01-9	Tissue	6.6	40	ug/Kg	55-117	62-100	40

TABLE 4 cont.

8270 SIM	Dibenz(a,h)anthracene	53-70-3	Tissue	3.5	40	ug/Kg	52-125	40-134	40
8270 SIM	Dibenzofuran	132-64-9	Tissue	2.6	40	ug/Kg	38-107	38-107	40
8270 SIM	Diethyl Phthalate	84-66-2	Tissue	9.0	40	ug/Kg	63-124	63-124	40
8270 SIM	Dimethyl Phthalate	131-11-3	Tissue	3.6	40	ug/Kg	62-119	62-119	40
8270 SIM	Di-n-butyl Phthalate	84-74-2	Tissue	100	100	ug/Kg	45-145	45-145	40
8270 SIM	Di-n-octyl Phthalate	117-84-0	Tissue	11	40	ug/Kg	51-145	51-145	40
8270 SIM	Fluoranthene	206-44-0	Tissue	7.8	40	ug/Kg	57-120	56-117	40
8270 SIM	Fluorene	86-73-7	Tissue	2.5	40	ug/Kg	54-118	54-110	40
8270 SIM	Hexachlorobenzene	118-74-1	Tissue	4.0	40	ug/Kg	63-112	63-112	40
8270 SIM	Hexachlorobutadiene	87-68-3	Tissue	6.2	40	ug/Kg	56-105	56-105	40
8270 SIM	Hexachloroethane	67-72-1	Tissue	12	40	ug/Kg	34-118	34-118	40
8270 SIM	Indeno(1,2,3-cd)pyrene	193-39-5	Tissue	3.7	40	ug/Kg	57-129	49-121	40
8270 SIM	Isophorone	78-59-1	Tissue	2.0	40	ug/Kg	59-109	59-109	40
8270 SIM	Naphthalene	91-20-3	Tissue	4.9	40	ug/Kg	41-111	39-94	40
8270 SIM	Nitrobenzene	98-95-3	Tissue	6.4	40	ug/Kg	60-120	60-120	40
8270 SIM	N-Nitrosodimethylamine	62-75-9	Tissue	130	200	ug/Kg	56-113	56-113	40
8270 SIM	N-Nitrosodi-n-propylamine	621-64-7	Tissue	22	40	ug/Kg	50-123	50-123	40
8270 SIM	N-Nitrosodiphenylamine	86-30-6	Tissue	3.0	40	ug/Kg	56-107	56-107	40
8270 SIM	Pentachlorophenol	87-86-5	Tissue	30	100	ug/Kg	41-105	41-105	40
8270 SIM	Phenanthrene	85-01-8	Tissue	2.7	40	ug/Kg	48-118	55-109	40
8270 SIM	Phenol	108-95-2	Tissue	45	100	ug/Kg	46-126	46-126	40
8270 SIM	Pyrene	129-00-0	Tissue	4.4	40	ug/Kg	50-114	61-100	40
8270 SIM	2,4,6-Tribromophenol (Surr.)	118-79-6	Tissue	NA	NA	%	44-125	NA	NA
8270 SIM	2-Fluorobiphenyl (Surr.)	321-60-8	Tissue	NA	NA	%	50-106	NA	NA
8270 SIM	2-Fluorophenol (Surr.)	367-12-4	Tissue	NA	NA	%	44-97	NA	NA
8270 SIM	Nitrobenzene-d5 (Surr.)	4165-60-0	Tissue	NA	NA	%	39-130	NA	NA
8270 SIM	Phenol-d6 (Surr.)	13127-88-3	Tissue	NA	NA	%	52-100	NA	NA
8270 SIM	Terphenyl-d14 (Surr.)	1718-51-0	Tissue	NA	NA	%	53-137	NA	NA

TABLE 4 cont.

8270 SIM	PBDE 100	189084-64-8	Tissue	0.024	1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 128	CASID30336	Tissue	0.014	1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 138	182677-30-1	Tissue	0.0093	1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 153	68631-49-2	Tissue	0.0095	1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 154	207122-15-4	Tissue	0.011	1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 17	147217-75-2	Tissue	0.018	1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 183	207122-16-5	Tissue	0.01	1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 190	CASID30338	Tissue	0.12	1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 203	CASID30339	Tissue	0.012	1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 206	63936-56-1	Tissue	0.017	1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 209	1163-19-5	Tissue	0.042	1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 28	41318-75-6	Tissue	0.015	1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 47	5436-43-1	Tissue	0.024	1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 66	189084-61-5	Tissue	0.046	1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 71	189084-62-6	Tissue	0.026	1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 85	182346-21-0	Tissue	0.028	1	ug/Kg	70-130	70-130	40
8270 SIM	PBDE 99	60348-60-9	Tissue	0.041	1	ug/Kg	70-130	70-130	40
8270 SIM	PDBE 47C13(Surr.)	NA	Tissue	NA	NA	%	70-130	70-130	40
8270 SIM	PDBE 99C13(Surr.)	NA	Tissue	NA	NA	%	70-130	70-130	40

a Method Detection Limits are subject to change as new MDL studies are completed.
a MDL is the smallest analyte concentration that can be demonstrated to be different from zero with 99% confidence
b The LOD is the smallest amount of a substance that must be present in a sample in order to be detected with 99% confidence.
Verification is acceptable if the response is > 3x instrument noise & ion abundance
c The LOQ is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias.

STANDARD OPERATING PROCEDURE

VOLATILE ORGANIC COMPOUNDS BY GC/MS

VOC-8260
Revision 17

Effective Date: January 15, 2011

Approved By:

K. Rasmussen	Supervisor	12/13/11 Date
Julie Gish	QA Manager	12/27/11 Date
	Laboratory Manager	12/27/11 Date

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DOCUMENT CONTROL	
NUMBER:	
Initials:	Date:

VOLATILE ORGANIC COMPOUNDS BY GC/MS

1. SCOPE AND APPLICATION

1.1. This procedure is used to determine the concentration of volatile organic compounds in water and soil using USEPA Method 8260C. This method is also applicable to TCLP ZHE leachates and may also be applicable to various types of aqueous and nonaqueous waste samples.

1.2. Attachment C lists the compounds that can be determined by this method and the achievable method reporting limits (MRLs) in water and soil. The reported MRL may be adjusted if required for specific project requirements; however, the capability of achieving other reported MRLs must be demonstrated. The Method Detection Limits (MDLs) that have been achieved are given in Attachment C, and may change slightly as MDL studies are repeated.

1.3. The nominal quantitation range for water samples is 0.5 – 80 ug/L. The nominal quantitation range for low concentration soils is 5-200 ug/kg. The nominal quantitation range for high concentration soils is 50-8000 ug/kg.

2. METHOD SUMMARY

2.1. This procedure gives gas chromatographic/mass spectrometric (GC/MS) conditions for the detection of parts per billion (ppb) levels of volatile organic compounds. A sample aliquot is injected into the gas chromatograph (GC) by either the purge and trap method or by direct injection. The compounds are separated on a fused silica capillary GC column. The compounds are detected by a mass selective detector (MSD), which gives both qualitative as well as quantitative information.

2.2. In the purge and trap process an inert gas, helium, is bubbled through the sample aliquot, at room temperature. This gas stream sweeps the volatile organic compounds out of the aqueous phase and into the gas stream - it purges the compounds out of the sample. The gas stream then passes through a sorbent column which selectively adsorbs, (traps) these compounds out of the helium. The preparation and analysis of soil samples uses procedures described in USEPA Method 5030B or 5035/5035A. After the purging sequence is done, the sorbent column (the trap) is heated and desorbed onto the GC column. The GC column separates the compounds and passes then onto the MSD for identification and quantification.

2.3. The sensitivity of this method depends on the level of background contamination (i.e. interferences) rather than on instrumental limitations. Highly contaminated waste samples will require a methanol extraction prior to analysis. This will elevate the reporting levels and may mask low levels of compounds of interest.

3. DEFINITIONS

Analysis Window - Samples are analyzed in a set referred to as "a window". The window begins with the injection of the tune verification standard. After this standard has passed the method specific criteria a 12 hour analysis window is started. Next, a calibration curve or a continuing calibration standard (CCV see below) is run. If the CCV meets the specified criteria, sample and QC analyses are run until the 12 hour time limit closes. A new window must then be opened and the sequence repeated.

Internal Standards - Internal standards are organic compounds which are similar to the analytes of interest but which are not found in the samples. The chosen internal standards are used to help calibrate the instrument's response and to compensate for slight instrument variations from injection to injection.

Independent Calibration Verification (ICV) - Verification of the ratio of instrument response to analyte amount, a calibration check, is done by analyzing for analyte standards in an appropriate solvent. ICV solutions are made from a stock solution which is different from the stock used to prepare calibration standards.

Laboratory Control Sample (LCS) - In the LCS analysis, predetermined quantities of standard solutions of all analytes are added to a blank matrix prior to sample extraction and analysis. The purpose of the LCS is to monitor analytical control for the sample batch. Percent recoveries are calculated for each of the analytes.

Matrix Spike/Duplicate Matrix Spike Analysis - In the matrix spike analysis, predetermined quantities of standard solutions of all analytes are added to a sample matrix prior to sample extraction and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Samples are split into duplicates, spiked, and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision. The concentration of the spike should be at 5 to 10 times the MRL or at levels specified by a project analysis plan.

Standard Curve - A standard curve is a curve which plots concentrations of a known analyte standard versus the instrument response to the analyte.

Surrogate - Surrogates are organic compounds which are similar to the analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples, and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

Continuing Calibration Verification Standard (CCV) - A mid-level standard injected into the instrument at specified intervals and is used to verify the initial calibration.

Method Blank (MB) - The method blank (also called continuing calibration blank) is a volume of clean reagent water analyzed on each GC/MS used for sample analysis. The purpose of the blank is to determine the levels of contamination associated with the instrumental analysis itself, particularly with regard to the carry-over of analytes from standards or highly contaminated samples into other analyses.

4. INTERFERENCES

4.1. Interferences by common laboratory extraction solvents, such as Methylene Chloride, Acetone, and Freon 113 can cause problems. The area where volatile organic analyses are performed should be free of these solvents.

4.2. Other interferences include but are not limited to impurities in the inert purge gas, dirty plumbing/purge vessels, cross contamination by highly contaminated samples to clean ones in transport and storage, and carry over from one analysis to subsequent ones.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

6. SAMPLE CONTAINERS, COLLECTION, PRESERVATIONS, AND STORAGE

6.1. Refer to procedures for methods 5030 and 5035 for sample container and collection procedures. All sample containers for volatile organic analyses should be washed with soap and water, deionized water rinsed, and baked at 105°C ± 5°C for approximately 2 hours prior to use. Alternatively, one can buy precleaned sample containers from major lab equipment suppliers. All containers should be of glass or amber glass and equipped with a screw top cap and PFTE (teflon) lined septa.

6.2. Samples collected using EPA Method 5035 should be shipped in Encore sample tubes or collected in VOA vials containing sodium bisulfate (low concentration) and/or methanol (high concentration).

6.3. Collect all samples in duplicate, triplicate when possible. Prepare the proper number of sample bottles/containers prior to the sampling event with preservatives to adjust the samples pH to <2 with 1:1 HCI (water samples).

6.4. Slowly fill sample bottles to just overflowing taking care not to flush out the preservative or to entrain air bubbles in the samples. Seal the bottles with PFTE lined septa toward the sample and invert to check for entrained air bubbles.

6.5. Experimental evidence has shown refrigeration at 4°C alone will not stop biological degradation of some aromatic volatile organics. Adjusting the pH of the replicate samples to less than two (pH <2) with 1:1 HCl (@ 2-3 drops per 40 mLs) preserves samples for 14 days after collection. Residual chlorine can also degrade some organic compounds, generating trihalomethanes (THM's).

6.6. All samples must be stored at 4 ± 2°C and must be analyzed within 14 days of collection. See SOP VOC-5035 for additional holding time information. Any free product samples to be tested do not have any set holding times but should be analyzed as soon as possible.

6.7. The analysis of 2-CVE in water by method 8260 requires the collection of an unpreserved sample. 2-Chloroetheyl Vinyl Ether is highly reactive and preservation may accelerate loss by polymerization or other rapid chemical reaction. Therefore, the accuracy of results from a preserved sample cannot be guaranteed. If a client requests 2-CVE they must collect three preserved and three unpreserved vials and the sample must be logged in for a separate 2-CVE analysis.

7. APPARATUS AND EQUIPMENT

7.1. Gas chromatograph/Mass Selective Detector Systems

7.1.1. Each GC/MS system is set up with a GC capable of cooling the GC oven/column (subambient capability optional), injection onto a capillary column, and a stainless steel jet separator at the column's detector end prior to the transfer line interfaced with the MSD. Each MSD is a HP5971, HP5972, HP5973 or HP5975 that is controlled by the HP-MSDOS Chemstation software.

Instrument ID	Cofiguration	Column
MS04	Split/splitless – capillary direct	DB-624, 60m, 0.25mm, 1.4um
MS13, MS18, MS19, MS23, MS24	Split/splitless – capillary direct	RTX-624, 20m, 0.18mm, 1um
MS12	Split/splitless – capillary direct	DB-624, 20m, 0.18mm, 1.0um

7.1.2. Instrument systems and associated test methods are listed below. Individual operating conditions for 8260 instruments are given in Attachment A.

Instrument ID	Description	Tests Performed

MS04	5890/5972	8260W, 8260S, 524.2, 624
MS13	6890/5973	8260W, 8260S, 624
MS18	5890/5973	8260W, 8260S
MS19	6890/5973	8260S, 524.2
MS23	6890/5973	624,8260W
MS24	7890/5975	8260W, 8260S
MS12	5890/5970	Screening only

7.2. Purge and Trap with Autosampler

Each volatile GC/MS analytical system uses a purge and trap to introduce the sample onto the GC column. Each purge and trap has an autosampler (A/S) attached to run multiple samples, one at a time, and run unattended for extended periods of time. Varian Archon, Tekmar ALS 2016, or equivalents are preferred for extended unattended automated analyses.

7.3. GC Columns

- Restex RTX-624 (or equivalent) 75 M x 0.53mm id fused silica column 3.0µm film thickness
- Restex RTX-624 (or equivalent) 60 M x 0.32mm id fused silica column 1.8µm film thickness
- Restex RTX-624 (or equivalent) 20 M x 0.18mm id fused silica column 1.0µm film thickness
- J&W Scientific DB-624 (or equivalent) 60 M x 0.25 mm id fused silica column 1.4µm film thickness.
- J&W Scientific DB-624 (or equivalent) 20 M x 0.18 mm id fused silica column 1.0µm film thickness.

7.4. Each volatile GC/MS data processing station uses the most recent version of the EPA/NIST Mass Spectral Library. The current version is the NIST98k library.

7.5. Analytical balance - Capable of accurately weighing to 0.001g, Mettler PE160 or equivalent.

7.6. Syringes, Hamilton Gas-Tight in 10uL, 25uL, 100uL, 500uL, and 1000uL sizes.

7.7. Standard storage vials, screw thread with Mini-inert caps.

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1. Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination. The preparation for all laboratory prepared reagents and solutions must be documented in a laboratory logbook. Refer to the SOP

Reagent/Standards Login and Tracking (ADM-RTL) for the complete procedure and documentation requirements

8.2. Methanol, purge and trap grade or equivalent.

8.3. Reagent water, prepared from deionized water, by charcoal filtration and then purging with high purity helium that is set at 4-5psi for approximately 2 hours prior to use.

8.4. Blank soil matrix – Ottawa sand, Accustandard specialty sands.

8.5. Helium, compressed high purity grade.

8.6. BFB Tuning Verification Stock Standard – A 25000ppm stock standard is purchased (Accustandard). This stock solution is diluted in methanol to give a working standard of 50ppm.

8.7. Stock Standard Solutions

8.7.1. Commercially prepared and certified stock standards are used routinely for all the method specified analytes. All such mixtures are also routinely checked against an independent source for both analyte identification and analyte concentration. All such stock standard mixtures have expiration dates given by the manufacturer and must be replaced if the comparison with the independent check standards indicates a problem. Alternatively, stock standards may be prepared from neat chemicals. Store with minimal headspace, at -10° to -20°C and protect from light.

8.7.2. When preparing stock standards from neat chemicals accurately weigh approximately 0.1g of material and dilute with methanol to 10mL in a volumetric flask. If the purity of the neat chemical is <96% adjust the calculated concentration accordingly.

8.8. Working Standards - Prepare these standards from stock solutions. Prepare at concentrations which facilitate ease of preparation of instrument-level standards (calibration standards, etc.). Refer to Table 1 for Standard Expiration Date Guidelines. Store standards with minimal headspace in appropriately sized standard storage vials with mini-inert caps. Solutions should be checked for degradation or evaporation prior to use.

8.9. Calibration Standards

8.9.1. A minimum of five different concentration levels for all the analytes are prepared by diluting working standards into reagent water. The lowest concentration level must be at the method reporting level, or a level corresponding to a sample concentration meeting project-specific data quality objectives, with the remaining four levels defining the working linear range of the analytical system. *The permanent gas stock standards used to prepare calibration standards must not be*

more than one week old. See Attachment B for detailed instructions and forms for preparing calibration standards and ICVs.

8.9.2. The suggested levels are 0.5, 2, 10, 20 and 40 ppb for waters; and 5, 20, 50, 100, and 200 ppb for soils. All calibration solutions are made up daily.

8.10. Internal Standards and Surrogates

The surrogates recommended are Dibromofluoromethane, toluene-d_8 and 4-bromofluorobenzene. The internal standards recommended are fluorobenzene, 1,4-difluorobenzene, 1,4-dichlorobenzene-d_4 and chlorobenzene-d_5. Other internal standards and surrogates may be used, depending on the analysis requirements. All surrogates and internal standards are added to every calibration standard. The spike level for samples, blanks, and matrix spikes is 10 ug/L for waters and 50 ug/L for soils.

8.11. Spiking Solutions

8.11.1. Prepare LCS and MS spiking solutions the same as ICV solutions as listed in Attachment B for calibration solutions. Waters are typically spiked at 10 ppb and soils are typically spiked at 50 ppb.

8.11.2. Matrix spike and laboratory control spike solutions should contain the full list of analytes of interest. However, a subset may be reported.

Note: Refer to Table 1 for Standard Expiration Date Guidelines.

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument. Pertinent information (serial numbers, instrument I.D., etc.) must be in the logbook. This includes the routine maintenance described in section 9. The entry in the log must include: date of event, the initials of who performed the work, and a reference to analytical control.

9.2. Carrier gas - Inline purifiers or scubbers should be in place for all sources of carrier gas. These are selected to remove water, oxygen, and hydrocarbons. Purifiers should be changed as recommended by the supplier.

9.3. Purge and Trap /Autosamplers

9.3.1. The purge/trap system should be baked out and back-flushed daily as needed, generally prior to use on a daily basis.

9.3.2. Replace the trap monthly or sooner if performance deteriorates.

9.3.3. The heating cup temperature is checked each time the instrument is calibrated and documented on the calibration run log.

9.4. Gas Chromatograph

9.4.1. Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and "clean" (uniform, without fragmentation) by using the proper column cutting tool.

9.4.2. Over time, the column will exhibit poorer overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced. This is especially true when evident in conjunction with calibration difficulties.

9.5. Mass Spectrometer

9.5.1. Tune the MS as needed to result in consistent and acceptable performance (see section 11).

9.5.2. For units under service contract, certain maintenance is performed by instrument service staff, including pump oil changed, vacuuming boards, etc., as recommended by the manufacturer.

9.5.3. MS source cleaning should be performed as needed, depending on the performance of the unit. This may be done by the analyst or by instrument service staff.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in the SOP for Documentation of Training, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Sample Preparation

11.1.1. Water Samples

11.1.1.1. No preparation is generally required, other than dilution with reagent water to bring analytes into the upper half of the calibration range. Thus, a 10 mL sample volume is run straight from the sample vial. See the SOP for *Purge and Trap for Aqueous Samples* (VOC-5030) for details.

11.1.1.2. All water samples must be checked to have a pH $\leq$ 2 after sample analysis has taken place. Narrow range pH paper is used and the results are recorded on the injection log.

11.1.1.3. TCLP ZHE leachates are diluted 1:400 in reagent water prior to analysis. The TCLP samples and method blanks are diluted from the acidified ZHE extract; and the TCLP MS and LCS are diluted from a non-acidified extract, spiked, and poured into an HCL preserved VOA vial.

11.1.2. Soil samples are analyzed as either low concentration (direct purge) or high concentration (methanol preservation/extraction). Refer to the SOP for *Purge and Trap/Extraction for Volatile Organics in Soil and Waste Samples, Close System* (VOC-5035) for details.

11.1.2.1. For low concentration analyses, one of the sampling options given in method 5035 is to be used. Depending on the option used, follow the instructions given in the method. Typically, 1-5g is weighed out into the sample vial and 5 mL of reagent water is added. QC spikes and internal standards are then added, and the sample is purged at a temperature of 40°C° ±1°. Calibration standards, LCS, and method blanks require 5g Ottawa sand as the matrix.

11.1.2.2. In the event that low concentration analyses are specified but samples were not taken using a 5035 procedure, a portion of the sample is analyzed via direct heated purge of soil and EPA Method 5030A is cited. The analytical report should also be narrated with a statement indicating that 5030A has been deleted from SW-846. The low concentration analyses require a calibration specific to direct soil analysis.

11.1.2.3. The mid-level type is a methanol extraction method. In general, a 5g wet weight of soil is extracted with 5mL of purge-and-trap methanol in a scintillation vial. Place 5mL of purge-and-trap methanol into vial, tare, and add 5g of sample, and record the weight. Quickly cap and vortex until the sample is thoroughly mixed. A 1:100 dilution (500uL to 50mL) of this

extract is then prepared in reagent water and analyzed using the water calibration. The extract weight, volume used, and methanol lot number are recorded on the injection log (or a bench sheet).

NOTE: For soil/solid samples requiring VOA and non-VOA analyses and only one container was submitted to the lab, sample receiving will label the sample container as "VOA Analysis First" and/or attach a "VOA FIRST" tag. The VOA department will remove a sample aliquot first for their analyses. The sample should be handled as if it were a Rush analysis, so that the other non-VOA analyses will not be unduly delayed. The VOA analyst who opens the container will either break the custody seal and will initial and date it when the container was opened or sign and date the "VOA FIRST" tag. A VOA Analysis First note will also be included on the SR.

11.2. Initial Calibration

NOTE: The calibration procedure(s) and options chosen must follow the CAS protocols. Any exceptions to the calibration procedures detailed in the CAS SOP for *Calibration of Instruments for Organics Chromatographic Analyses* are described as follows:

11.2.1. BFB Tuning

11.2.1.1. Prior to calibration and sample analyses, analyze a 25ng or 50ng injection of Bromofluorobenzene (BFB). Each volatile GC/MS analytical system set up to run 8260C must meet the criteria listed in Table 2 for the injection of BFB. The analysis time for BFB is used to define the start of the 12-hour window in which all analyses must be performed. Once the instrument is tuned, all subsequent analyses of standards, samples, and QA/QC samples within the same 12-hour window must be analyzed using the identical mass spectrometer operating conditions.

11.2.1.2. Obtain the spectrum for evaluation using one of the following options:

- Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of BFB. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the BFB peak or part of any other closely eluting peak.

- Use one scan at the apex of the peak. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of BFB. The background subtraction should be designed only to eliminate column bleed or instrument background ions.

Do not subtract part of the BFB peak or part of any other closely eluting peak.

- Use the average across the entire peak up to a total of 5 scans. Peak integration must be consistent with standard operating procedure. If the peak is wider than 5 scans, the tune will consist of the peak apex scan and the two scans immediately preceding and following the apex. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of BFB. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the BFB peak or part of any other closely eluting peak.

- Use the average across the entire peak. Peak integration must be consistent with standard operating procedure. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of BFB. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the BFB peak or part of any other closely eluting peak.

11.2.1.3. Evaluate the spectrum against the criteria specified in Table 2. The criteria used must be the same for all ion abundance criteria checks associated with a given analysis. For example, initial calibration, continuing calibration(s), QC, and sample analyses for a given sample must all use the same criteria.

11.2.2. GC/MS Analytical System Initial Calibrations

11.2.2.1. Prior to conducting any sample analyses, a multi-point (5 point minimum) calibration must be run. Recommended calibration levels are 0.5, 2, 10, 20, and 40 ppb for waters, and 5, 20, 50, 100, and 200 ppb for soils. Analyze each calibration standard and tabulate the area response of the characteristic quantitation ions (Table 3) versus concentration for each compound, internal standard and surrogate. Calculate the response factors (RF) for each compound and surrogate relative to the specified internal standard by:

$$RF_x = \frac{(A_x)(C_{ISTD})}{(A_{ISTD})(C_x)}$$

Where:

A_x = Area of the characteristic quantitation ion for compound x.
A_{ISTD} = Area of the characteristic quantitation ion for the specified internal standard.
C_x = The concentration of the compound added.

C_{ISTD} = The concentration of the specified internal standard.

11.2.2.2. Calculate the mean response factor $(\overline{RF_x})$ for each analyte and surrogate from the five calibration levels. Calculate standard deviation (SD) and the percent relative standard deviations (%RSD) for each analyte from the mean with:

$$\%RSD = \frac{(SD)}{(\overline{RF_x})} 100.$$

11.2.2.3. The % RSD should be less than 20% for each compound.

11.2.2.4. If the % RSD for any compound is ≤ 20%, linearity can be assumed over the calibration range, and the relative response factor for each analyte and surrogate is used.

11.2.2.5. If the %RSD for a compound is >20%, then alternative calibration models should be used. See the SOP (SOC-CAL) *Calibration of Instruments for Organics Chromatographic Analysis* for further guidance.

11.2.2.6. The mean response factor for each target analyte should meet the minimum response factors listed in Table 5. Meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity. Due to the large number of compounds that may be analyzed by this method, some compounds will fail to meet this criterion. For these occasions, the analyte is qualified as not meeting the method recommended response factor criterion.

11.2.2.7. When instrument response does not follow a linear model, a non-linear calibration model may be used. Refer to the CAS SOP for *Calibration of Instruments for Organics Chromatographic Analysis (SOC-CAL)* for alternative curve fit guidance.

11.2.2.8. If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit and do not meet the minimum correlation coefficient (0.99) for alternate curve fits, then the chromatographic system is considered too imprecise for analysis to begin and corrective action is necessary. Further preventative maintenance may be required or the system may not be adequately *primed* for initial calibration.

11.2.3. Review of calibration curve

11.2.3.1. The calibration curve must be reviewed to ensure it represents the calibration data. This is done by re-fitting each calibration level against the true concentration of each calibration standard. The % difference between the calculated concentration verses the true concentration should be ≤ 30% for each calibration level and may not exceed 50% for any level.

11.2.3.2. Due to the large number of compounds that may be analyzed, one or more analytes may exceed 20% RSD or 0.99 COD. The initial calibration may still be acceptable if the following conditions are met:

- The % difference between the calculated concentrations verses the true concentration for each level of the initial calibration curve meets the criteria specified in section 11.2.3.1.

- In order to report non-detects, it must be demonstrated that there is adequate sensitivity to detect the failed compounds at the applicable lower quantitation limit. This is done by re-evaluating the concentrations of the calibrations standards against the calculated concentrations.

NOTE: Certain project plans that fall under the (DoD QSM) contain additional initial calibration acceptance criteria. In these cases, the analyst must refer to the project plan to know if the criteria listed in the DoD QSM or QAPP-specified criteria or EPA method calibration criteria are to be used.

11.2.4. Independent Calibration Verification

11.2.4.1. Following initial calibration, analyze an ICV standard. The ICV solution must be obtained for all analytes that are analyzed and reported. Calculate the percent difference (%D) or % Drift from the ICV true value. The acceptance limits for the ICV are ± 30% of true value.

Note: For DoD QSM projects, the criterion is within ± 20% of the expected value for all project analytes.

11.2.4.2. If a second source standard is not available or is cost prohibitive (such as certain non-routine analytes), then an independently prepared solution (prepared by analyst other than analyst preparing initial calibration standards) may be used as the ICV and must meet the criteria above.

11.2.4.3. After the multi-point calibration has passed all of the above criteria, and the Independent Calibration Verification has been performed, samples can be analyzed. The calibration curve mid-point standard may serve as the CCV for

the opening set of samples within the same 12-hour window as the initial calibration.

11.3. Daily GC/MS Calibration Verification - CCV

11.3.1. The start of a 12-hour analysis window requires a check of the instrument tune via an injection of 25ng or 50 ng of BFB. Refer to section 11.2.1.2 for the procedure. If the criteria found in Table 3 are met, then a check of the initial calibration curve is done. If the first analysis of the BFB fails, inspect the instrument for malfunction and perform maintenance as necessary. A second BFB tune verification may then be performed. If the second run also fails, it may be necessary to retune and recalibrate the system.

11.3.2. After the tuning criteria have been verified, the initial calibration must be checked and verified by analyzing a midrange calibration standard. The 10 ppb level for waters and 50 ppb level for soils is recommended. For water, CCVs are prepared by adding 10µl of the 50 ppm 8260 working standard and 5µl of the 2000 ppm ketone mix into 50 mL reagent water and a 10 mL aliquot is purged. For soil, CCVs are prepared by adding 25µl of the 100ppm (nominal) working standard into 50 mL reagent water, and a 5 mL aliquot is purged.

11.3.3. If the percent difference or percent drift for a compound is less than or equal to 20%, then the initial calibration for that compound is assumed to be valid. Due to the large number of compounds that may be analyzed by this method, some compounds may fail to meet the ≤ 20% criteria. If no more than 20% of the compounds, included in the initial calibration, differ from their true concentration by 40%, the initial calibration is valid and no corrective action is necessary.

11.3.4. In cases where compounds fail, they may still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit.

11.3.5. Non-detected analytes can be reported from analyses when a CCV exhibit a positive bias (i.e., outside the upper control limit), no further documentation is required.

11.3.6. For situations when the CCV fails to meet the criterion in section 11.3.3, and a confirmed detection exceed the MRL, the sample must be reanalyzed to ensure accurate quantification. If it is not possible to reanalyze the sample, the result must be reported as an estimated value.

11.3.7. If the tune criteria and the continuing calibration criteria are met, then the retention times of all compounds, surrogates, and internal standards are checked against the initial calibration. If the retention time for any internal standard changes by more than 30 seconds from the retention time from the mid-point standard of the most recent

initial calibration, the system must be inspected for malfunctions and corrections must be made, as required.

11.3.8. If the area for any of the internal standards changes by a factor of 2 (-50% to +100%) from the area from the mid-point standard of the most recent initial calibration, corrections must be made to the system.

11.3.9. Quantitation of all compounds will be based on the initial calibration.

11.4. GC/MS Analysis

11.4.1. Evaluate GC/MS screen and make proper dilutions.

11.4.2. Prepare samples as described in section 11. Use the same operating conditions as were used for initial calibration.

11.4.3. If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, extract dilution must take place.

11.5. Identification of Analytes

11.5.1. The MSD data system software identifies a sample component by first finding and identifying the surrogate and internal standards. After they have been integrated, the extracted ion chromatogram is searched for all calibrated analytes.

11.5.2. The qualitative identification of each compound determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum is generated from analysis of a calibration standard and is updated with each initial calibration.

11.5.3. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met.

11.5.3.1. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

11.5.3.2. The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

11.5.3.3. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

11.5.4. Table 3 lists characteristic ions as given in Method 8260C. If there is no peak found for an analyte in the expected retention time window and the mass spectrum does not match according to the method criteria, then the analyte is "not found". Print out spectra for all confirmed hits.

11.6. The analyst reviews all analyses to confirm (or correct) all data system qualitative interpretations.

11.7. If results are to be reported on a dry weight basis, determine the dry weight of a separate aliquot of the sample, using the SOP for Total Solids.

12. QA/QC REQUIREMENTS

Note: The analyst should refer to the CAS *SOP for Sample Batches* and the CAS *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation.*

12.1. Initial Precision and Recovery Validation

The accuracy and precision of the procedure must be validated before analysis of samples begins, or whenever significant changes to the procedures have been made or when an analyst is new to the procedure. To do this, analyze four water sample spikes, calculate the average recovery and standard deviation, and evaluate as described in EPA SW-846. The concentration of the analytes to be spiked should be in the working calibration range. Initial Demonstration of Cabability studies must be performed as part of analyst training. Copies of the studies are maintained in the lab and in the analyst's training file.

12.2. Method Detection Limits, LOD and LOQ

12.2.1. Method detection limit (MDL), Limits of Detection (LOD) and Limits of Quantification (LOQ) must be determined before analysis of samples can begin. Refer to the SOP for the Performing Method Detection Limit Studies and Estimation of Limits of Detection and Quantitation.

12.2.2. Calculate the average concentration found (x) in µg/mL, and the standard deviation of the concentrations (s) in µg/mL for each analyte. Calculate the MDL for each analyte.

12.3. Ongoing QC Samples required are described in the CAS-Kelso Quality Assurance Manual and in the *SOP for Sample Batches*. For every sample batch, at least one method blank and

one LCS, and one MS/DMS pair must be run for each matrix. Analytical windows must have at least one base (primary) sample analysis to require a MS/DMS pair.

12.3.1. Method blank - A method blank is analyzed with every batch of 20 or fewer samples to demonstrate that there are no method interferences. The method blank must also be run every 12-hour window to demonstrate that interferences from the analytical and preparation steps minimized. No target analytes should be detected above the MRL in the method blank. For some project specific needs, additional requirements or exceptions may be given.

Note: **For DoD projects** – The Method Blank will be considered contaminated if:

- The concentration of any target analyte in the blank exceeds ½ the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).
- The concentration of any common laboratory contaminant in the blank exceeds the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).
- The blank result otherwise affects the samples results as per the test method requirements or the project-specific objectives.

12.3.2. A lab control sample (LCS) must be prepared and analyzed with every batch, not to exceed 20 samples. The LCS is prepared by spiking a blank with the matrix spike solution, and going through the entire preparation and analysis. Calculate percent recovery (%R) as follows:

$$\%R = X/TV \times 100$$

Where X =Concentration of the analyte recovered
TV =True value of amount spiked

Acceptance criteria for lab control samples are listed in Attachment C. The accuracy of the analysis is controlled on a subset of target analytes. If the project analyte list is fewer than 20 analytes, all are considered control analytes. For DoD projects all project target analytes are considered control analytes. Analytes which are used for control analytes are listed in Table 4. Project-specific acceptance limits may supersede those listed in this SOP. If the lab control sample (LCS) fails acceptance limits for any of the control compounds, any associated sample data is rejected and corrective action must be taken. This may include evaluation of the sample preparation, analytical system, and calibration; and may require re-extraction, re-analysis, and/or recalibration and re-analysis.

12.3.3. A matrix spike/duplicate matrix spike (MS/DMS) must be prepared and analyzed with every batch of 20 or fewer samples. The MS is prepared by spiking a sample aliquot with the matrix spike solution, and going through the entire preparation and analysis. Calculate percent recovery (%R) as follows:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where: X = Measured concentration of the spiked sample aliquot
X1 = Measured concentration of unspiked sample aliquot
TV = True value (theoretical concentration) of amount spiked

Calculate Relative Percent Difference (RPD) as:

$$RPD = \frac{|R1 - R2|}{(R1 + R2)/2} \times 100$$

Where: R1 = measured concentration of the first sample aliquot
R2 = measured concentration of the second sample aliquot

Evaluate the recovery and RPD using the QC acceptance criteria in Attachment C or project-specific acceptance limits*. If the MS/DMS recovery is out of acceptance limits for reasons other than matrix effects, corrective action must be taken.

Note: for DoD project, a matrix spike is required with each batch of samples. Recovery acceptance limits are the same as LCS acceptance limits.

12.3.4. Calculate and evaluate the surrogate recovery using the QC acceptance criteria in Attachment C. If surrogate recovery is outside acceptance criteria, the sample data must be closely evaluated for possible matrix interferences. If none are present, then corrective action must be identified.

12.4. Acceptance Criteria

12.4.1. The acceptance criteria for tuning verification, initial, and continuing calibration verification have been outlined above in Section 11.

12.4.2. Current acceptance criteria for matrix spikes, LCSs and surrogates are listed in Attachment C. The acceptance criteria listed are current criteria, but are subject to change as control limits are updated.

12.5. Corrective action requirements have been outlined above in Section 11. Also, the corrective action requirements of any project-specific project plan should be used when applicable.

12.6. Additional QA/QC measures include control charting of QC sample results.

13. DATA REDUCTION, REVIEW, AND REPORTING

13.1. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 50% of the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. The resolution should be verified on the midpoint concentration of the initial calibration as well as the laboratory designated continuing calibration verification level if closely eluting isomers are to be reported.

13.2. Calculations

13.2.1. The GC/MS data stations, in current use, all use the H-P RTE Integrator to generate the raw data used to calculate the standards $\overline{RF_x}$ values, the sample amounts, and the spike values. The software does three passes through each data file. The first two identify and integrate each internal standard and surrogate. The third pass uses the time-drift information from the first two passes to search for all method analytes in the proper retention times and with the proper characteristic quantitation ions. The results for a water sample are calculated as follows when $\overline{RF_x}$ is used:

$$A_x = \frac{(Resp_x)(Amt_{ISTD})}{(Resp_{ISTD})(\overline{RF_x})}$$

Where: A_x = the amount, in ppb, of the analytes in the sample;
$Resp_x$ = the peak area of the analytes of interest;
$Resp_{ISTD}$ = the peak area of the associated internal standard;
Amt_{ISTD} = the amount, in ppb, of internal standard added

$\overline{RF_x}$ = the average response from the five-point for the analytes of interest.

13.2.2. The results for low concentration soil work are calculated by taking the normal print out, in ppb, (see the water results outlined above) and correcting for the total, dry soil sample actually purged:

$$(A_x) = \frac{(5\ grams)}{(ASW_t\ gr)(\%\ Solids)} = A_x\ Low\text{-}Level\ Soil$$

Where: A_x = the amount, in ppb, from the data system; five grams is the nominal amount of soil that is heated and purged;
ASW_t = the actual soil wet weight, in grams, that is purged; and
% Solids the correction factor for dry weight.

13.2.3. Results for a high concentration soil samples (methanol extracts) are calculated as follows:

$$(A_x) = \frac{(Dilution)(V_{EXTR})}{(ASW_t)(\%\ Solids)} = A_x\ High\text{-}Level\ Soil\ Amt.$$

Where: A_x = the data station results, in ppb;
Dilution = the dilution of the extract.
ASW_t = the actual wet weight of soil extracted; and
% Solids = the dry soil correction.
V_{EXTR} = the methanol extract volume (mL)*

* The water contained in the native sample is accounted for when determining the final extract volume. The final volume of the methanol extract is the total volume of the methanol/water mixture. Calculate the final volume as follows:

$$Final Volume\ Methanol/Water = mL\ of\ solvent + \left(\frac{\%Moisture\ x\ Sample\ Wt.(g)}{100}\right)$$

13.3. Data Review

Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the *SOP for Laboratory Data Review Process* for details.

13.4. Reporting

13.4.1. Reports are generated using the STEALTH Data Reporting System which compiles the SMO login information and Enviroquant data. This compilation is then transferred to a file, which STEALTH uses to generate a report. The forms generated may be CAS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13.4.2. Alternatively, Excel templates located in R:\VOA\forms may be used to prepare reports from hard-copy data. The analyst should choose the appropriate form and QC pages to correspond to required tier level. The detected analytes, surrogate and matrix spikes are then transferred, by hand, to the templates.

14. CONTENGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this (and other applicable) SOP(s). Also, refer to the *SOP for Corrective Action* for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the SOP for Report Generation or in project-specific requirements.

15. METHOD PERFORMANCE

This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

The method detection limit (MDL) is established using the procedure described in the SOP for *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation* (ADM-MDL). Method Reporting Limits are established for this method based on this SOP.

16. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible, and within method requirements. Standards are prepared in volumes consistent with laboratory use in order to minimize the volume of expired standards to be disposed of. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

17. WASTE MANAGEMENT

17.1. The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

17.2. This method uses non-halogenated solvents and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and disposed of in accordance with Federal and State regulations.

18. TRAINING

18.1. Training Outline

18.1.1. Review literature by reading references. Review the EPA methodology and any applicable state-specific methods. Review the SOP. Also review the MSDS for methanol.

18.1.2. Observe the procedure as performed by an experienced analyst at least three times.

18.1.3. Assist in the procedure under the guidance of an experienced analyst for a period of three months. During this training process, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

18.1.4. Following the three-month training period the analyst is expected to complete an initial demonstration of capability study (IDC) for solid samples by direct, solid samples by extraction, and water samples. Summaries of the IDC are reviewed and signed by the technical director and forwarded to the employee's training file.

18.1.4.1. Perform IDC studies by preparing and analyzing four replicate laboratory control samples spiked at a level of 10-20 times the MRL. Calculate average percent recovery and relative standard deviation for the four replicate analyses. Refer to Method 8000C and 8260C for analysis and evaluation guidelines.

18.1.4.2. For applicable tests, IDC studies are be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

18.2. Training is documented following the SOP for *Documentation of Training*.

NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

19. REFERENCES

19.1. CAS SOP for Purge and Trap for Aqueous Samples (VOC-5030).

19.2. CAS SOP for Purge and Trap Extraction for Volatile Organics in Soil and Waste Samples, Closed System (VOC-5035).

19.3. *Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique*, U.S. EPA, SW-846, Method 8260C, August 2006.

19.4. *Purge and Trap*, U.S. EPA, SW-846, Final Updates I and III, Methods 5030A (Rev. 1), 5030B (Rev. 2), and 5030C, May 2003.

19.5. *Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste*, U.S. EPA, SW-846, Final Update III, Method 5035, Revision 0, December 1996; and Method 5035A, July 2002.

20. METHOD MODIFICATIONS

20.1. For water samples, a purge volume of 10mL is used, whereas the method (section 7.5.5) states 5 mL or 25 mL. The use of a 10 mL volume ensures sensitivity for "5 mL" type analyses *and,* on the analytical systems in use, meets the sensitivity goals of a 25 mL purge volume analysis. Also, the use of 10 mL rather than 25 mL decreases the negative effects of water being introduced into the P/T-GC-MS system.

20.2. 11.2.3.1 Reference method recommends recalculation of low point only and that should be ± 30%. This SOP state each point is refit and each point should be with ± 30% but may not exceed ± 50%.

20.3. 11.3.3 No limit defined in reference method, so lab assigned a limit of 40% based on CLP protocols.

21. CHANGES SINCE THE LAST REVISION

21.1. Sec 8.1 added

21.2. Sec 9.1 updated

21.3. Removed 11.2.3.3 and added clarification to Note in 11.2.3.2 on what calibration criteria is used for DoD ELAP projects.

21.4. Sec 11.3.4 is new

21.5. Sec 11.4 is new

21.6. Sec 11.5 completely re-written

21.7. Sec 11.3.8 – separated from previous 11.3.7 to be more easily identified as a requirement.

21.8. Sec 13.1 is new

TABLE 1

Standard Expiration Date Guidelines

Standard	Expiration time
Neat Chemicals	Expiration date 3 years from date opened, or supplier's assigned date.
Stock Standards (unopened ampules, commercially prepared or lab prepared)	Supplier's assigned date, or 1 year if no expiration date provided.
Opened ampules and working standards	
• concentration $\geq$ 5000 ppm	6 month expiration date.
• concentration 1000 - 5000 ppm	2 month expiration date.
• concentration 200 - 1000 ppm	1 month expiration date.
• concentration < 200 ppm	7 day expiration date.
Internal Standard Solutions	One month expiration date.

Note: The analyst performing specific analytical procedures should use judgement and take into consideration the solution reactivity, volatility, and concentration when using standards to prepare calibration curves. Certain standards, depending on use and storage, may have shorter usable life than described in these guidelines.

TABLE 2

4-Bromofluorobenzene Characteristic Ion Abundance Criteria

Mass	Ion Abundance Criteria *
50	15-40% of mass 95
75	30-60% of mass 95
95	Base peak, 100% relative abundance
96	5-9% of mass 95
173	< 2% of mass 174
174	> 50% of mass 95
175	5-9% of mass 174
176	95 -101% of mass 174
177	5-9% of mass 176
Reference:	EPA 8260C

*Manufacturer Ion abundance criteria may be used

TABLE 3
Characteristic Masses (m/z) for Purgeable Organic Compounds

Acetone	58	43
Acetonitrile	41	40, 39
Acrolein	56	55, 58
Acrylonitrile	53	52, 51
Allyl alcohol	57	58, 39
Allyl chloride	76	41, 39, 78
Benzene	78	-
Benzyl chloride	91	126, 65, 128
Bromoacetone	136	43, 138, 93, 95
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
iso-Butanol	74	43
n-Butanol	56	41
2-Butanone	72	43
n-Butylbenzene	91	92, 134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91, 134
Carbon disulfide	76	78
Carbon tetrachloride	117	119
Chloral hydrate	82	44, 84, 86, 111
Chloroacetonitrile	48	75
Chlorobenzene	112	77, 114
1-Chlorobutane	56	49
Chlorodibromomethane	129	208, 206
Chloroethane	64	66
2-Chloroethanol	49	44, 43, 51, 80
Bis(2-chloroethyl) sulfide	109	111, 158, 160
2-Chloroethyl vinyl ether	63	65, 106
Chloroform	83	85
Chloromethane	50	52
Chloroprene	53	88, 90, 51
3-Chloropropionitrile	54	49, 89, 91
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-chloropropane	75	155, 157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174
1,2-Dichlorobenzene	146	111, 148
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148

TABLE 3
Characteristic Masses (m/z) for Purgeable Organic Compounds (continued)

cis-1,4-Dichloro-2-butene	75	53, 77, 124, 89
trans-1,4-Dichloro-2-butene	53	88, 75
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 98
trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,3-Dichloro-2-propanol	79	43, 81, 49
1,1-Dichloropropene	75	110, 77
cis-1,3-Dichloropropene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39
1,2,3,4-Diepoxybutane	55	57, 56
Diethyl ether	74	45, 59
1,4-Dioxane	88	58, 43, 57
Epichlorohydrin	57	49, 62, 51
Ethanol	31	45, 27, 46
Ethyl acetate	88	43, 45, 61
Ethylbenzene	91	106
Ethylene oxide	44	43, 42
Ethyl methacrylate	69	41, 99, 86, 114
Hexachlorobutadiene	225	223, 227
Hexachloroethane	201	166, 199, 203
2-Hexanone	43	58, 57, 100
2-Hydroxypropionitrile	44	43, 42, 53
Iodomethane	142	127, 141
Isobutyl alcohol	43	41, 42, 74
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134, 91
Malononitrile	66	39, 65, 38
Methacrylonitrile	41	67, 39, 52, 66
Methyl acrylate	55	85
Methyl-t-butyl ether	73	57
Methylene chloride	84	86, 49
Methyl ethyl ketone	72	43
Methyl iodide	142	127, 141
Methyl methacrylate	69	41, 100, 39
4-Methyl-2-pentanone	100	43, 58, 85
Naphthalene	128	-

TABLE 3

Characteristic Masses (m/z) for Purgeable Organic Compounds (continued)

Nitrobenzene	123	51, 77
2-Nitropropane	46	-
2-Picoline	93	66, 92, 78
Pentachloroethane	167	130, 132, 165, 169
Propargyl alcohol	55	39, 38, 53
b-Propiolactone	42	43, 44
Propionitrile (ethyl cyanide)	54	52, 55, 40
n-Propylamine	59	41, 39
n-Propylbenzene	91	120
Pyridine	79	52
Styrene	104	78
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129, 131, 166
Toluene	92	91
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132
Trichlorofluoromethane	151	101, 153
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
Surrogates:		
1,2-Dichloroethane-d4	65	67, 51
4-Bromofluorobenzene	95	174, 176
Dibromofluoromethane	113	111, 192
Toluene-d8	98	99, 70
Internal Standards:		
1,4-Difluorobenzene	114	63, 88
Fluorobenzene	96	77, 70, 50
1,4-Dichlorobenzene-d4	152	115, 150
Chlorobenzene-d5	117	119, 82

TABLE 4

Control Analytes for Non-DoD Projects

1,1-Dichloroethene
Benzene
Trichloroethene
Toluene
Chlorobenzene
1,2-Dichlorobenzene
Naphthalene
1,1,2-Trichloroethane
2-Chlorotoluene
2-Hexanone
Carbon Tetrachloride
Vinyl Chloride
Ethylbenzene
Chloroform
Bromodichloromethane
1,2,3-Trichloropropane

TABLE 5
RECOMMENDED MINIMUM RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION VERIFICATION

	Response Factor (RF)
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl chloride	0.100
Bromomethane	0.100
Chloroethane	0.100
Trichlorofluoromethane	0.100
1,1-Dichloroethene	0.100
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100
Acetone	0.010*
Carbon disulfide	0.100
Methyl Acetate	0.100
Methylene chloride	0.100
trans-1,2-Dichloroethene	0.100
cis-1,2-Dichloroethene	0.100
Methyl tert-Butyl Ether	0.100
1,1-Dichloroethane	0.200
2-Butanone	0.010*
Chloroform	0.200
1,1,1-Trichloroethane	0.100
Cyclohexane	0.100
Carbon tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
Trichloroethene	0.200
Methylcyclohexane	0.100
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
cis-1,3-Dichloropropene	0.200
trans-1,3-Dichloropropene	0.100
4-Methyl-2-pentanone	0.010*
Toluene	0.400
1,1,2-Trichloroethane	0.100
Tetrachloroethene	0.200
2-Hexanone	0.015*

TABLE 5 (cont.)
RECOMMENDED MINIMUM RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION VERIFICATION

	Response Factor (RF)
Dibromochloromethane	0.100
1,2-Dibromoethane	0.100
Chlorobenzene	0.500
Ethylbenzene	0.100
meta-/para-Xylene	0.100
ortho-Xylene	0.300
Styrene	0.300
Bromoform	0.100
Isopropylbenzene	0.100
1,1,2,2-Tetrachloroethane	0.300
1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
1,2-Dibromo-3-chloropropane	0.025*
1,2,4-Trichlorobenzene	0.200
Any other analyte not included in this table	0.100

* These analytes have poor purging efficiencies. Response factors based upon USEPA CLP guidance and laboratory performance after system maintenance.

ATTACHMENTS

Attachment A
Instrument Operating Parameters

Attachment B
Detailed Standard Preparation Instructions

Attachment C

Data Quality Objectives

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Attachment C

CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	LODb	LOQc	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)	DOD QSM (LCS %Rec.)	DOD QSM (% RPD)
8260	1,1,1,2-Tetrachloroethane	630-20-6	Soil-low	0.11	5.0	0.4	5.0	ug/kg	71-119	16-131	40	75-125	30
8260	1,1,1-Trichloroethane (TCA)	71-55-6	Soil-low	0.11	5.0	0.4	5.0	ug/kg	59-146	26-144	40	70-135	30
8260	1,1,2,2-Tetrachloroethane	79-34-5	Soil-low	0.13	5.0	0.5	5.0	ug/kg	60-128	10-153	40	55-130	30
8260	1,1,2-Trichloroethane	79-00-5	Soil-low	0.15	5.0	0.5	5.0	ug/kg	72-118	35-130	40	60-125	30
8260	1,1-Dichloroethane	75-34-3	Soil-low	0.12	5.0	0.4	5.0	ug/kg	59-137	31-135	40	75-125	30
8260	1,1-Dichloroethene	75-35-4	Soil-low	0.25	5.0	0.5	5.0	ug/kg	64-152	31-153	40	65-135	30
8260	1,1-Dichloropropene	563-58-6	Soil-low	0.13	5.0	0.5	5.0	ug/kg	62-142	25-143	40	70-135	30
8260	1,2,3-Trichlorobenzene	87-61-6	Soil-low	0.19	20	0.5	20	ug/kg	52-138	10-118	40	60-135	30
8260	1,2,3-Trichloropropane	96-18-4	Soil-low	0.45	5.0	1.4	5.0	ug/kg	53-134	23-149	40	65-130	30
8260	1,2,4-Trichlorobenzene	120-82-1	Soil-low	0.13	20	0.5	20	ug/kg	65-132	10-121	40	65-130	30
8260	1,2,4-Trimethylbenzene	95-63-6	Soil-low	0.054	20	0.2	20	ug/kg	55-140	10-142	40	65-135	30
8260	1,2-Dibromo-3-chloropropane	96-12-8	Soil-low	0.4	20	1.4	20	ug/kg	55-127	10-146	40	40-135	30
8260	1,2-Dibromoethane (EDB)	106-93-4	Soil-low	0.094	20	0.3	20	ug/kg	71-116	26-131	40	70-125	30
8260	1,2-Dichlorobenzene	95-50-1	Soil-low	0.077	5.0	0.3	5.0	ug/kg	67-124	10-124	40	75-120	30
8260	1,2-Dichloroethane (EDC)	107-06-2	Soil-low	0.07	5.0	0.2	5.0	ug/kg	65-121	32-134	40	70-135	30
8260	1,2-Dichloropropane	78-87-5	Soil-low	0.13	5.0	0.5	5.0	ug/kg	71-121	31-132	40	70-120	30
8260	1,3,5-Trimethylbenzene	108-67-8	Soil-low	0.092	20	0.3	20	ug/kg	66-132	10-160	40	65-135	30
8260	1,3-Dichlorobenzene	541-73-1	Soil-low	0.094	5.0	0.3	5.0	ug/kg	69-128	10-126	40	70-125	30
8260	1,3-Dichloropropane	142-28-9	Soil-low	0.12	5.0	0.4	5.0	ug/kg	72-118	32-133	40	75-125	30
8260	1,4-Dichlorobenzene	106-46-7	Soil-low	0.086	5.0	0.3	5.0	ug/kg	69-125	10-123	40	70-125	30
8260	1,4-Dioxane	123-91-1	Soil-low	14	250	20	250	ug/kg	10-172	10-172	40	-	30
8260	1-Chlorohexane	544-10-5	Soil-low	0.11	5.0	0.4	5.0	ug/kg	62-136	62-136	40	-	30
8260	2,2-Dichloropropane	594-20-7	Soil-low	0.098	5.0	0.3	5.0	ug/kg	50-138	34-140	40	65-135	30
8260	2-Butanone (MEK)	78-93-3	Soil-low	0.9	20	1	20	ug/kg	54-116	27-113	40	30-160	30
8260	2-Chloroethyl Vinyl Ether	110-75-8	Soil-low	0.43	10	1	10	ug/kg	63-130	10-139	40	-	30
8260	2-Chlorotoluene	95-49-8	Soil-low	0.12	20	0.4	20	ug/kg	66-129	10-140	40	70-130	30
8260	2-Hexanone	591-78-6	Soil-low	0.93	20	2	20	ug/kg	67-121	15-162	40	45-145	30
8260	2-Nitropropane	79-46-9	Soil-low	1.1	20	2.3	20	ug/kg	10-142	39-128	40	-	30
8260	3-chloro-1-propene	107-05-1	Soil-low	0.54	10	1	10	ug/kg					
8260	4-Chlorotoluene	106-43-4	Soil-low	0.088	20	0.4	20	ug/kg	65-129	10-134	40	75-125	30
8260	4-Isopropyltoluene	99-87-6	Soil-low	0.064	20	0.2	20	ug/kg	61-132	10-126	40	75-135	30
8260	4-Methyl-2-pentanone (MIBK)	108-10-1	Soil-low	1.8	20	1	20	ug/kg	69-126	30-129	40	45-145	30
8260	Acetone	67-64-1	Soil-low	2.9	20	4	20	ug/kg	32-135	18-117	40	20-160	30
8260	Acetonitrile	75-05-8	Soil-low	23	100	28	100	ug/kg	38-128	38-128	40	-	30
8260	Acrolein	107-02-8	Soil-low	1.7	100	5	100	ug/kg	10-218	10-140	40	-	30
8260	Acrylonitrile	107-13-1	Soil-low	0.43	20	1	20	ug/kg	18-179	42-163	40	-	30
8260	Benzene	71-43-2	Soil-low	0.054	5.0	0.2	5.0	ug/kg	68-122	30-137	40	75-125	30
8260	Bromobenzene	108-86-1	Soil-low	0.088	5.0	0.3	5.0	ug/kg	71-124	13-134	40	65-120	30
8260	Bromochloromethane	74-97-5	Soil-low	0.24	5.0	0.5	5.0	ug/kg	65-131	34-132	40	70-125	30
8260	Bromodichloromethane	75-27-4	Soil-low	0.16	5.0	0.5	5.0	ug/kg	61-143	14-146	40	70-130	30
8260	Bromoform	75-25-2	Soil-low	0.14	5.0	0.5	5.0	ug/kg	62-134	10-139	40	55-135	30
8260	Bromomethane	74-83-9	Soil-low	0.2	5.0	0.5	5.0	ug/kg	22-180	10-160	40	30-160	30
8260	Carbon Disulfide	75-15-0	Soil-low	0.092	5.0	0.3	5.0	ug/kg	55-141	18-140	40	45-160	30
8260	Carbon Tetrachloride	56-23-5	Soil-low	0.094	5.0	0.3	5.0	ug/kg	51-135	10-144	40	65-135	30
8260	Chlorobenzene	108-90-7	Soil-low	0.065	5.0	0.2	5.0	ug/kg	70-116	15-124	40	75-125	30
8260	Chloroethane	75-00-3	Soil-low	0.74	5.0	1	5.0	ug/kg	51-122	15-149	40	40-155	30
8260	Chloroform	67-66-3	Soil-low	0.11	5.0	0.4	5.0	ug/kg	61-137	43-133	40	70-125	30
8260	Chloromethane	74-87-3	Soil-low	0.18	5.0	0.5	5.0	ug/kg	37-146	30-133	40	50-130	30
8260	Chloroprene	126-99-8	Soil-low	0.35	20	2	20	ug/kg	10-167	55-142	40	-	30
8260	cis-1,2-Dichloroethene	156-59-2	Soil-low	0.12	5.0	0.4	5.0	ug/kg	62-138	32-137	40	65-125	30
8260	cis-1,3-Dichloropropene	10061-01-5	Soil-low	0.13	5.0	0.5	5.0	ug/kg	58-138	20-132	40	70-125	30
8260	cis-1,4-Dichloro-2-butene	1476-11-5	Soil-low	1.2	20	4	20	ug/kg	10-175	54-132	40	-	30
8260	Cyclohexane	110-82-7	Soil-low	0.32	5.0			ug/kg	70-130	70-130	40	-	30

CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	LODb	LOQc	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)	DOD QSM (LCS %Rec.)	DOD QSM (% RPD)
8260	Dibromochloromethane	124-48-1	Soil-low	0.18	5.0	0.5	5.0	ug/kg	69-120	21-132	40	-	30
8260	Dibromomethane	74-95-3	Soil-low	0.28	5.0	0.5	5.0	ug/kg	68-125	41-127	40	75-130	30
8260	Dichlorodifluoromethane	75-71-8	Soil-low	0.12	5.0	0.4	5.0	ug/kg	38-160	14-158	40	35-135	30
8260	Dichlorofluoromethane	75-43-4	Soil-low	0.15		0.5	0.5						
8260	Diethylether	60-29-7	Soil-low	0.12		0.4							
8260	Diisopropyl Ether	108-20-3	Soil-low	0.11	10	0.4	10	ug/kg	55-158	55-158	40	-	30
8260	Ethyl Acetate	141-78-6	Soil-low	1.2	20			ug/kg	61-117	10-180	40	-	30
8260	Ethyl Methacrylate	97-63-2	Soil-low	0.14	20	0.5	20	ug/kg	10-149	51-129	40	-	30
8260	Ethylbenzene	100-41-4	Soil-low	0.094	5.0	0.3	5.0	ug/kg	70-118	13-128	40	75-125	30
8260	Ethylene Oxide	75-21-8	Soil-low	12	100			ug/kg	29-158	29-158	40	-	30
8260	Hexachlorobutadiene	87-68-3	Soil-low	0.4	20	0.8	20	ug/kg	54-140	10-114	40	55-140	30
8260	Iodomethane (Methyl Iodide)	74-88-4	Soil-low	0.21	20	0.8	20	ug/kg	33-160	21-122	40	-	30
8260	Isobutanol	78-83-1	Soil-low	8.6	200	20	200	ug/kg	21-128	47-118	40	-	30
8260	Isopropylbenzene	98-82-8	Soil-low	0.081	20	0.3	20	ug/kg	67-133	10-153	40	75-130	30
8260	m,p-Xylenes	179601-23-	Soil-low	0.1	5.0	0.4	5.0	ug/kg	69-127	10-139	40	80-125	30
8260	Methacrylonitrile	126-98-7	Soil-low	0.44	20	1.5	20	ug/kg	15-140	47-120	40	-	30
8260	Methyl Acetate	79-20-9	Soil-low	0.59	5.0			ug/kg	42-121	10-172	40	-	30
8260	Methyl Methacrylate	80-62-6	Soil-low	0.39	20	1	20	ug/kg	10-147	46-125	40	-	30
8260	Methyl tert-Butyl Ether	1634-04-4	Soil-low	0.12	5.0	0.4	5.0	ug/kg	66-118	44-116	40	-	30
8260	Methylcyclohexane	108-87-2	Soil-low	0.21	5.0			ug/kg	70-130	70-130	40	-	30
8260	Methylene Chloride	75-09-2	Soil-low	0.16	10	0.5	10	ug/kg	65-122	36-123	40	55-140	30
8260	Naphthalene	91-20-3	Soil-low	0.13	20	0.5	20	ug/kg	54-134	10-127	40	40-125	30
8260	n-Butylbenzene	104-51-8	Soil-low	0.069	20	0.2	20	ug/kg	53-139	10-125	40	65-140	30
8260	n-Hexane	110-54-3	Soil-low	0.14	10	0.5	10	ug/kg	38-173	27-186	40	-	30
8260	n-Octane	111-65-9	Soil-low	4	10			ug/kg	49-138			-	30
8260	n-Propylbenzene	103-65-1	Soil-low	0.13	20	0.5	20	ug/kg	57-143	10-145	40	65-135	30
8260	o-Xylene	95-47-6	Soil-low	0.081	5.0	0.3	5.0	ug/kg	69-124	10-146	40	75-125	30
8260	Propionitrile	107-12-0	Soil-low	0.8	20	2.8	20	ug/kg	16-138	47-120	40	-	30
8260	Propylene Oxide	75-56-9	Soil-low	10	50			ug/kg				-	30
8260	sec-Butylbenzene	135-98-8	Soil-low	0.074	20	0.2	20	ug/kg	55-146	10-141	40	65-130	30
8260	Styrene	100-42-5	Soil-low	0.14	5.0	0.5	5.0	ug/kg	62-135	10-130	40	75-125	30
8260	tert-Amyl Methyl Ether	994-05-8	Soil-low	0.2	10	0.5	10	ug/kg	64-148	64-148	40	-	30
8260	tert-Butyl Alcohol	75-65-0	Soil-low	3.9	50	10	50	ug/kg	35-141	64-148	40	-	30
8260	tert-Butyl Ethyl Ether	637-92-3	Soil-low	0.065	10	0.2	10	ug/kg	58-152	58-152	40	-	30
8260	tert-Butylbenzene	98-06-6	Soil-low	0.14	20	0.5	20	ug/kg	58-152	10-152	40	65-130	30
8260	Tetrachloroethene (PCE)	127-18-4	Soil-low	0.16	5.0	0.5	5.0	ug/kg	66-126	10-132	40	65-140	30
8260	Toluene	108-88-3	Soil-low	0.15	5.0	0.5	5.0	ug/kg	75-117	24-142	40	70-125	30
8260	trans-1,2-Dichloroethene	156-60-5	Soil-low	0.12	5.0	0.4	5.0	ug/kg	63-127	29-139	40	65-135	30
8260	trans-1,3-Dichloropropene	10061-02-6	Soil-low	0.11	5.0	0.4	5.0	ug/kg	63-121	19-125	40	65-125	30
8260	trans-1,4-Dichloro-2-butene	110-57-6	Soil-low	0.43	20	1.4	20	ug/kg	26-204	10-179	40	-	30
8260	Trichloroethene (TCE)	79-01-6	Soil-low	0.15	5.0	0.5	5.0	ug/kg	67-126	18-145	40	75-125	30
8260	Trichlorofluoromethane	75-69-4	Soil-low	0.085	5.0	0.3	5.0	ug/kg	51-140	20-137	40	25-185	30
8260	Trichlorotrifluoroethane	76-13-1	Soil-low	0.24	5.0	0.4	5.0	ug/kg	53-135	24-144	40	-	30
8260	Vinyl Acetate	108-05-4	Soil-low	0.31	20	1	20	ug/kg	45-158	31686	40	-	30
8260	Vinyl Chloride	75-01-4	Soil-low	0.18	5.0	0.5	5.0	ug/kg	54-127	31-140	40	60-125	30
8260	1,2-Dichloroethane-D4 (Surr.)	17060-07-0	Soil-low	NA	NA	NA	NA	%	71-119	NA	NA	-	30
8260	4-Bromofluorobenzene (Surr.)	460-00-4	Soil-low	NA	NA	NA	NA	%	77-124	NA	NA	60-125	30
8260	Dibromofluoromethane (Surr.)	1868-53-7	Soil-low	NA	NA	NA	NA	%	83-128	NA	NA	-	30
8260	Toluene-D8 (Surr.)	2037-26-5	Soil-low	NA	NA	NA	NA	%	83-135	NA	NA	85-115	30

a Method Detection Limits are subject to cbange as new MDL studies are completed.

a MDL is the smallest analyte concentration that can be demonstrated to be different from zero with 99% confidence

CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	LODb	LOQc	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)	DOD QSM (LCS %Rec.)	DOD QSM (% RPD)
8260	1,1,1,2-Tetrachloroethane	630-20-6	Soil-mid	0.0081	.05	0.04	.05	mg/kg	71-120	66-117	40	75-125	30
8260	1,1,1-Trichloroethane (TCA)	71-55-6	Soil-mid	0.0032	.05	0.03	.05	mg/kg	61-136	61-118	40	70-135	30
8260	1,1,2,2-Tetrachloroethane	79-34-5	Soil-mid	0.0071	.05	0.05	.05	mg/kg	63-127	57-133	40	55-130	30
8260	1,1,2-Trichloroethane	79-00-5	Soil-mid	0.01	.05	0.05	.05	mg/kg	73-118	74-113	40	60-125	30
8260	1,1-Dichloroethane	75-34-3	Soil-mid	0.0087	.05	0.03	.05	mg/kg	70-124	72-114	40	75-125	30
8260	1,1-Dichloroethene	75-35-4	Soil-mid	0.02	.05	0.03	.05	mg/kg	67-141	61-132	40	65-135	30
8260	1,1-Dichloropropene	563-58-6	Soil-mid	0.019	.05	0.03	.05	mg/kg	65-130	63-119	40	70-135	30
8260	1,2,3-Trichlorobenzene	87-61-6	Soil-mid	0.007	0.2	0.04	0.2	mg/kg	49-149	60-115	40	60-135	30
8260	1,2,3-Trichloropropane	96-18-4	Soil-mid	0.021	.05	0.08	.05	mg/kg	76-128	72-138	40	65-130	30
8260	1,2,4-Trichlorobenzene	120-82-1	Soil-mid	0.0048	0.2	0.03	0.2	mg/kg	55-134	50-146	40	65-130	30
8260	1,2,4-Trimethylbenzene	95-63-6	Soil-mid	0.0064	0.2	0.02	0.2	mg/kg	69-123	65-133	40	65-135	30
8260	1,2-Dibromo-3-chloropropane	96-12-8	Soil-mid	0.033	0.2	0.08	0.2	mg/kg	46-132	40-150	40	40-135	30
8260	1,2-Dibromoethane (EDB)	106-93-4	Soil-mid	0.0073	0.2	0.04	0.2	mg/kg	70-122	71-114	40	70-125	30
8260	1,2-Dichlorobenzene	95-50-1	Soil-mid	0.0053	.05	0.04	.05	mg/kg	75-114	75-109	40	75-120	30
8260	1,2-Dichloroethane (EDC)	107-06-2	Soil-mid	0.0054	.05	0.03	.05	mg/kg	70-126	68-117	40	70-135	30
8260	1,2-Dichloropropane	78-87-5	Soil-mid	0.0082	.05	0.03	.05	mg/kg	73-121	78-114	40	70-120	30
8260	1,3,5-Trichlorobenzene	108-70-3	Soil-mid	0.009	0.2	0.03	0.2	mg/kg	10-154	10-154	40	-	30
8260	1,3,5-Trimethylbenzene	108-67-8	Soil-mid	0.0083	0.2	0.03	0.2	mg/kg	68-129	64-131	40	65-135	30
8260	1,3-Dichlorobenzene	541-73-1	Soil-mid	0.0059	.05	0.04	.05	mg/kg	69-117	74-110	40	70-125	30
8260	1,3-Dichloropropane	142-28-9	Soil-mid	0.0087	.05	0.05	.05	mg/kg	70-121	74-114	40	75-125	30
8260	1,4-Dichlorobenzene	106-46-7	Soil-mid	0.0075	.05	0.04	.05	mg/kg	69-114	74-110	40	70-125	30
8260	1,4-Dioxane	123-91-1	Soil-mid	2.4	25	4	10	mg/kg	31-184	12-187	40	-	30
8260	1-Chlorohexane	544-10-5	Soil-mid	0.018	.05	0.02	0.05	mg/kg	46-136	51-135	40	-	30
8260	2,2-Dichloropropane	594-20-7	Soil-mid	0.019	.05	0.02	.05	mg/kg	49-136	54-134	40	65-135	30
8260	2-Butanone (MEK)	78-93-3	Soil-mid	0.21	2.0	0.4	2.0	mg/kg	65-139	77-140	40	30-160	30
8260	2-Chloroethyl Vinyl Ether	110-75-8	Soil-mid	0.043	0.5	0.1	0.5	mg/kg	54-140	34-131	40	-	30
8260	2-Chlorotoluene	95-49-8	Soil-mid	0.0062	0.2	0.04	0.2	mg/kg	68-127	70-131	40	70-130	30
8260	2-Hexanone	591-78-6	Soil-mid	0.19	2.0	1	2.0	mg/kg	54-123	58-118	40	45-145	30
8260	2-Nitropropane	79-46-9	Soil-mid	0.32	0.5	0.2	0.5	mg/kg	16-146	11-123	40	-	30
8260	3-Chloro-1-propene	107-05-1	Soil-mid	0.038	0.5	0.03	0.5	mg/kg	39-120	17-132	40	-	30
8260	4-Chlorotoluene	106-43-4	Soil-mid	0.0082	0.2	0.05	0.2	mg/kg	66-121	69-123	40	75-125	30
8260	4-Isopropyltoluene	99-87-6	Soil-mid	0.0069	0.2	0.02	0.2	mg/kg	68-127	67-131	40	75-135	30
8260	4-Methyl-2-pentanone (MIBK)	108-10-1	Soil-mid	0.34	2.0	1	2.0	mg/kg	73-126	71-108	40	45-145	30
8260	Acetone	67-64-1	Soil-mid	0.24	2.0	1	2.0	mg/kg	47-142	70-135	40	20-160	30
8260	Acetonitrile	75-05-8	Soil-mid	1.3	5	2	5	mg/kg	41-124	70-130	40	-	30
8260	Acrolein	107-02-8	Soil-mid	0.17	2.0	0.5	2	mg/kg	23-145	10-241	40	-	30
8260	Acrylonitrile	107-13-1	Soil-mid	0.032	2.0	0.08	2	mg/kg	58-195	10-152	40	-	30
8260	Benzene	71-43-2	Soil-mid	0.0068	.05	0.02	0.05	mg/kg	70-134	66-123	40	75-125	30
8260	Bromobenzene	108-86-1	Soil-mid	0.01	0.2	0.04	0.2	mg/kg	66-118	78-111	40	65-120	30
8260	Bromochloromethane	74-97-5	Soil-mid	0.0096	.05	0.05	.05	mg/kg	73-117	75-111	40	70-125	30
8260	Bromodichloromethane	75-27-4	Soil-mid	0.0087	.05	0.03	.05	mg/kg	76-128	63-129	40	70-130	30
8260	Bromoform	75-25-2	Soil-mid	0.0096	.05	0.05	.05	mg/kg	54-141	43-149	40	55-135	30
8260	Bromomethane	74-83-9	Soil-mid	0.019	.05	0.03	.05	mg/kg	29-171	14-139	40	30-160	30
8260	Carbon Disulfide	75-15-0	Soil-mid	0.015	.05	0.04	.05	mg/kg	48-140	48-130	40	45-160	30
8260	Carbon Tetrachloride	56-23-5	Soil-mid	0.021	.05	0.03	.05	mg/kg	56-125	56-125	40	65-135	30
8260	Chlorobenzene	108-90-7	Soil-mid	0.0063	.05	0.04	.05	mg/kg	72-115	70-115	40	75-125	30
8260	Chloroethane	75-00-3	Soil-mid	0.015	.05	0.05	.05	mg/kg	53-134	36-126	40	40-155	30
8260	Chloroform	67-66-3	Soil-mid	0.009	.05	0.02	.05	mg/kg	73-125	72-125	40	70-125	30
8260	Chloromethane	74-87-3	Soil-mid	0.0096	.05	0.02	.05	mg/kg	50-121	43-126	40	50-130	30
8260	Chloroprene	126-99-8	Soil-mid	0.066	1.0	0.8	1.0	mg/kg	30-167	10-168	40	-	30
8260	cis-1,2-Dichloroethene	156-59-2	Soil-mid	0.011	.05	0.02	.05	mg/kg	77-124	78-119	40	65-125	30
8260	cis-1,3-Dichloropropene	10061-01-5	Soil-mid	0.0089	.05	0.05	.05	mg/kg	57-132	51-133	40	70-125	30
8260	cis-1,4-Dichloro-2-butene	1476-11-5	Soil-mid	0.14	1.0	0.4	1.0	mg/kg	28-175	68-155	40	-	30

CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	LODb	LOQc	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)	DOD QSM (LCS %Rec.)	DOD QSM (% RPD)
8260	Cyclohexane	110-82-7	Soil-mid	0.036	0.1			mg/kg	64-138	64-138	40	-	30
8260	Dibromochloromethane	124-48-1	Soil-mid	0.0068	.05	0.05	.05	mg/kg	64-126	53-132	40	65-130	30
8260	Dibromomethane	74-95-3	Soil-mid	0.017	.05	0.05	.05	mg/kg	75-124	72-114	40	75-130	30
8260	Dichlorodifluoromethane	75-71-8	Soil-mid	0.021	.05	0.05	.05	mg/kg	21-143	20-132	40	35-135	30
8260	Dichlorofluoromethane (CFC 21)	75-43-4	Soil-mid	0.016	.05	0.04	.05	mg/kg	22-153	42-137	40	-	30
8260	Diethylether	60-29-7	Soil-mid					mg/kg	39-143		40		30
8260	Ethyl Acetate	141-78-6	Soil-mid	0.1	0.5			mg/kg	56-119	10-200	40	-	30
8260	Ethyl Ether	60-29-7	Soil-mid	0.0055	0.1	0.02	0.1	mg/kg	39-143	10-159	40	-	30
8260	Ethyl Methacrylate	97-63-2	Soil-mid	0.01	0.2	0.05	0.5	mg/kg	42-146	22-129	40	-	30
8260	Ethylbenzene	100-41-4	Soil-mid	0.0045	.05	0.02	.05	mg/kg	72-121	68-121	40	75-125	30
8260	Hexachlorobutadiene	87-68-3	Soil-mid	0.016	0.2	0.04	0.2	mg/kg	52-136	51-142	40	55-140	30
8260	Iodomethane	74-88-4	Soil-mid	0.065	0.5	0.04	0.5	mg/kg	44-166	10-188	40	-	30
8260	Isobutyl Alcohol	78-83-1	Soil-mid	0.9	50	2	10	mg/kg	25-140	31-151	40	-	30
8260	Isopropylbenzene	98-82-8	Soil-mid	0.0093	0.2	0.03	0.2	mg/kg	68-121	65-138	40	75-130	30
8260	m,p-Xylenes	179601-23-	Soil-mid	0.0093	.05	0.02	.05	mg/kg	74-124	66-122	40	80-125	30
8260	Methacrylonitrile	126-98-7	Soil-mid	0.035	0.5	0.08	0.5	mg/kg	48-122	24-118	40	-	30
8260	Methyl Acetate	79-20-9	Soil-mid	0.038	0.1			mg/kg	22-158	22-158	40	-	30
8260	Methyl Methacrylate	80-62-6	Soil-mid	0.016	0.5	0.08	0.5	mg/kg	46-127	21-123	40	-	30
8260	Methyl tert-Butyl Ether	1634-04-4	Soil-mid	0.015	.05	0.02	.05	mg/kg	64-126	66-117	40	-	30
8260	Methylcyclohexane	108-87-2	Soil-mid	0.033	0.1			mg/kg	60-139	60-139	40	-	30
8260	Methylene Chloride	75-09-2	Soil-mid	0.0071	0.2	0.05	0.2	mg/kg	69-121	68-126	40	55-140	30
8260	Naphthalene	91-20-3	Soil-mid	0.0068	0.2	0.03	0.2	mg/kg	48-144	60-151	40	40-125	30
8260	n-Butylbenzene	104-51-8	Soil-mid	0.0074	0.2	0.05	0.2	mg/kg	52-142	57-139	40	65-140	30
8260	n-Heptane	142-82-5	Soil-mid	0.1	0.1			mg/kg	70-130	70-130	40	-	30
8260	n-Hexane	110-54-3	Soil-mid	0.028	0.1	0.05	0.1	mg/kg	42-225	73-119	40	-	30
8260	n-Octane	111-65-9	Soil-mid	0.23	0.1	0.05	0.5	mg/kg	48-121	48-121	40	-	30
8260	n-Propylbenzene	103-65-1	Soil-mid	0.0093	0.2	0.2	0.2	mg/kg	61-134	65-126	40	65-135	30
8260	o-Xylene	95-47-6	Soil-mid	0.0068	.05	0.02	.05	mg/kg	73-123	73-120	40	75-125	30
8260	Propionitrile	107-12-0	Soil-mid	0.11	0.5	0.4	0.5	mg/kg	48-122	30-111	40	-	30
8260	sec-Butylbenzene	135-98-8	Soil-mid	0.0078	0.2	0.02	0.2	mg/kg	57-132	61-134	40	65-130	30
8260	Styrene	100-42-5	Soil-mid	0.0045	.05	0.04	.05	mg/kg	77-122	64-141	40	75-125	30
8260	tert-Amyl Methyl Ether	994-05-8	Soil-mid					mg/kg	63-133		40		
8260	tert-Butyl Alcohol	75-65-0	Soil-mid	0.28	2.0	1	2.0	mg/kg	41-163	52-122	40	65-130	30
8260	tert-Butyl Ethyl Ether	637-92-3	Soil-mid	0.0099	0.05	0.02	0.1	mg/kg	63-123	12-155	40	-	30
8260	tert-Butylbenzene	98-06-6	Soil-mid	0.01	0.2	0.02	0.2	mg/kg	65-131	71-131	40	-	30
8260	Tetrachloroethene (PCE)	127-18-4	Soil-mid	0.015	.05	0.02	.05	mg/kg	65-126	64-124	40	65-140	30
8260	Tetrahydrofuran	109-99-9	Soil-mid	0.5	1.0	0.2	0.5	mg/kg	33-169	48-125	40	-	30
8260	Toluene	108-88-3	Soil-mid	0.0085	.05	0.02	.05	mg/kg	74-118	67-123	40	70-125	30
8260	trans-1,2-Dichloroethene	156-60-5	Soil-mid	0.009	.05	0.03	.05	mg/kg	76-128	69-115	40	65-135	30
8260	trans-1,3-Dichloropropene	10061-02-6	Soil-mid	0.0091	.05	0.02	.05	mg/kg	55-129	55-112	40	65-125	30
8260	trans-1,4-Dichloro-2-butene	110-57-6	Soil-mid	0.034	1.0	0.1	1	mg/kg	59-188	79-130	40	-	30
8260	Trichloroethene (TCE)	79-01-6	Soil-mid	0.013	.05	0.04	.05	mg/kg	69-126	62-119	40	75-125	30
8260	Trichlorofluoromethane	75-69-4	Soil-mid	0.023	.05	0.04	.05	mg/kg	42-119	45-117	40	25-185	30
8260	Trichlorotrifluoroethane	76-13-1	Soil-mid	0.26	.05	0.05	.05	mg/kg	55-131	50-121	40	-	30
8260	Vinyl Acetate	108-05-4	Soil-mid	0.37	0.5	0.1	0.5	mg/kg	51-156	10-177	40	-	30
8260	Vinyl Chloride	75-01-4	Soil-mid	0.019	.05	0.02	.05	mg/kg	53-125	49-127	40	60-125	30
8260	1,2-Dichloroethane-D4(Surr)	17060-07-0	Soil-mid	NA	NA	NA	NA	%	37-155	NA	NA	-	30
8260	4-Bromofluorobenzene(Surr)	460-00-4	Soil-mid	NA	NA	NA	NA	%	58-136	NA	NA	85-120	30
8260	Dibromofluoromethane(Surr)	1868-53-7	Soil-mid	NA	NA	NA	NA	%	55-132	NA	NA	-	30
8260	Toluene-D8 (Surr.)	2037-26-5	Soil-mid	NA	NA	NA	NA	%	81-131	NA	NA	85-115	30

a Method Detection Limits are subject to change as new MDL studies are completed.

CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	LODb	LOQc	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)	DOD QSM (LCS %Rec.)	DOD QSM (% RPD)
8260	1,1,1,2-Tetrachloroethane	630-20-6	Water	0.047	0.5	0.2	0.5	ug/L	66-124	67-127	30	80-130	30
8260	1,1,1-Trichloroethane (TCA)	71-55-6	Water	0.050	0.5	0.1	0.5	ug/L	59-136	57-151	30	65-130	30
8260	1,1,2,2-Tetrachloroethane	79-34-5	Water	0.064	0.5	0.2	0.5	ug/L	70-127	72-129	30	65-130	30
8260	1,1,2-Trichloroethane	79-00-5	Water	0.061	0.5	0.3	0.5	ug/L	74-118	74-124	30	75-125	30
8260	1,1-Dichloroethane	75-34-3	Water	0.042	0.5	0.15	0.5	ug/L	66-129	69-141	30	70-135	30
8260	1,1-Dichloroethene	75-35-4	Water	0.10	0.5	0.2	0.5	ug/L	66-129	59-171	30	70-130	30
8260	1,1-Dichloropropene	563-58-6	Water	0.051	0.5	0.1	0.5	ug/L	59-134	61-148	30	75-130	30
8260	1,2,3-Trichlorobenzene	87-61-6	Water	0.10	2	0.2	2	ug/L	68-120	57-137	30	55-140	30
8260	1,2,3-Trichloropropane	96-18-4	Water	0.14	0.5	0.4	0.5	ug/L	69-123	71-127	30	75-125	30
8260	1,2,4-Trichlorobenzene	120-82-1	Water	0.13	2	0.2	2	ug/L	58-126	57-133	30	65-135	30
8260	1,2,4-Trimethylbenzene	95-63-6	Water	0.037	2	0.2	2	ug/L	63-122	61-132	30	75-130	30
8260	1,2-Dibromo-3-chloropropane	96-12-8	Water	0.22	2	0.5	2	ug/L	55-132	59-133	30	50-130	30
8260	1,2-Dibromoethane (EDB)	106-93-4	Water	0.084	2	0.2	2	ug/L	74-118	73-122	30	80-120	30
8260	1,2-Dichlorobenzene	95-50-1	Water	0.044	0.5	0.2	0.5	ug/L	72-115	72-119	30	70-120	30
8260	1,2-Dichloroethane (EDC)	107-06-2	Water	0.073	0.5	0.2	0.5	ug/L	56-142	56-141	30	70-130	30
8260	1,2-Dichloropropane	78-87-5	Water	0.042	0.5	0.2	0.5	ug/L	67-126	63-131	30	75-125	30
8260	1,3,5-Trichlorobenzene	108-70-3	Water	0.10	5	0.2	5	ug/L	63-118	58-118	30	-	30
8260	1,3,5-Trimethylbenzene	108-67-8	Water	0.042	2	0.2	2	ug/L	62-126	60-136	30	75-130	30
8260	1,3-Dichlorobenzene	541-73-1	Water	0.041	0.5	0.2	0.5	ug/L	70-116	70-121	30	75-125	30
8260	1,3-Dichloropropane	142-28-9	Water	0.032	0.5	0.2	0.5	ug/L	75-116	74-121	30	75-125	30
8260	1,4-Dichlorobenzene	106-46-7	Water	0.054	0.5	0.2	0.5	ug/L	73-115	72-121	30	75-125	30
8260	1,4-Dioxane	123-91-1	Water	13	100	40	100	ug/L	67-160	64-145	30	-	30
8260	1-Chlorohexane	544-10-5	Water	0.057	0.5	0.1	0.5	ug/L	50-118	50-118	30	-	30
8260	2,2-Dichloropropane	594-20-7	Water	0.050	0.5	0.2	0.5	ug/L	37-145	39-161	30	70-135	30
8260	2-Butanone (MEK)	78-93-3	Water	3.8	20	4	20	ug/L	71-149	65-147	30	30-150	30
8260	2-Chloroethyl Vinyl Ether	110-75-8	Water	0.19	5	0.2	5	ug/L	61-126	10-150	30	-	30
8260	2-Chlorotoluene	95-49-8	Water	0.035	2	2.7	2	ug/L	55-131	55-139	30	75-125	30
8260	2-Hexanone	591-78-6	Water	2.9	20	10	20	ug/L	59-131	53-132	30	55-130	30
8260	2-Nitropropane	79-46-9	Water	0.91	5	10	5	ug/L	10-160	10-160	30	-	30
8260	3-Chloro-1-propene	107-05-1	Water	0.19	5	0.2	5	ug/L	42-147	70-151	30	-	30
8260	4-Chlorotoluene	106-43-4	Water	0.025	2	0.13	2	ug/L	66-121	57-138	30	75-130	30
8260	4-Isopropyltoluene	99-87-6	Water	0.044	2	0.51	2	ug/L	61-128	57-141	30	75-130	30
8260	4-Methyl-2-pentanone (MIBK)	108-10-1	Water	3.0	20	2.6	20	ug/L	64-134	64-139	30	60-135	30
8260	Acetone	67-64-1	Water	2.5	20	10	20	ug/L	68-135	68-134	30	40-140	30
8260	Acetonitrile	75-05-8	Water	7.3	50	20	50	ug/L	69-132	77-127	30	-	30
8260	Acrolein	107-02-8	Water	2.0	20	2	20	ug/L	42-118	14-180	30	-	30
8260	Acrylonitrile	107-13-1	Water	0.31	5	4	5	ug/L	65-129	73-131	30	-	30
8260	Benzene	71-43-2	Water	0.045	0.5	0.1	0.5	ug/L	69-124	63-144	30	80-120	30
8260	Bromobenzene	108-86-1	Water	0.027	2	0.2	2	ug/L	72-116	72-122	30	75-125	30
8260	Bromochloromethane	74-97-5	Water	0.091	0.5	0.2	0.5	ug/L	75-131	73-135	30	65-130	30
8260	Bromodichloromethane	75-27-4	Water	0.036	0.5	0.2	0.5	ug/L	63-129	61-134	30	75-120	30
8260	Bromoform	75-25-2	Water	0.080	0.5	0.2	0.5	ug/L	52-144	54-140	30	70-130	30
8260	Bromomethane	74-83-9	Water	0.072	0.5	0.2	0.5	ug/L	35-113	36-127	30	30-145	30
8260	Carbon Disulfide	75-15-0	Water	0.045	0.5	0.2	0.5	ug/L	46-144	52-156	30	35-160	30
8260	Carbon Tetrachloride	56-23-5	Water	0.068	0.5	0.2	0.5	ug/L	55-140	53-161	30	65-140	30
8260	Chlorobenzene	108-90-7	Water	0.045	0.5	0.2	0.5	ug/L	72-116	69-126	30	80-120	30
8260	Chloroethane	75-00-3	Water	0.13	0.5	0.2	0.5	ug/L	58-134	56-147	30	60-135	30
8260	Chloroform	67-66-3	Water	0.042	0.5	0.1	0.5	ug/L	70-129	64-133	30	65-135	30
8260	Chloromethane	74-87-3	Water	0.053	0.5	0.1	0.5	ug/L	34-130	49-127	30	40-125	30
8260	Chloroprene	126-99-8	Water	0.15	10	4	10	ug/L	43-146	67-126	30	-	30
8260	cis-1,2-Dichloroethene	156-59-2	Water	0.045	0.5	0.2	0.5	ug/L	71-118	61-139	30	70-125	30
8260	cis-1,3-Dichloropropene	10061-01-5	Water	0.038	0.5	0.2	0.5	ug/L	62-132	66-134	30	70-130	30
8260	cis-1,4-Dichloro-2-butene	1476-11-5	Water	0.84	10	4	10	ug/L	26-171	49-172	30	-	30

CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	LODb	LOQc	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)	DOD QSM (LCS %Rec.)	DOD QSM (% RPD)
8260	Cyclohexane	110-82-7	Water	0.36	1			ug/L	70-130	70-130	30	-	30
8260	Dibromochloromethane	124-48-1	Water	0.057	0.5	0.5	0.5	ug/L	67-126	68-125	30	60-135	30
8260	Dibromomethane	74-95-3	Water	0.089	0.5	0.5	0.5	ug/L	69-128	68-132	30	75-125	30
8260	Dichlorodifluoromethane	75-71-8	Water	0.083	0.5	0.2	0.5	ug/L	32-124	29-133	30	30-155	30
8260	Dichlorofluoromethane (CFC 21)	75-43-4	Water	0.053	0.5	0.2	0.5	ug/L	54-140	79-135	30	-	30
8260	Diisopropyl Ether	108-20-3	Water	0.046	2	0.1	2	ug/L	62-123	50-134	30	-	30
8260	Ethyl Acetate	141-78-6	Water	0.81	5			ug/L	59-117	70-130	30	-	30
8260	Ethyl Ether	60-29-7	Water	0.069	1	0.1	1	ug/L	54-137	31-141	30	-	30
8260	Ethyl Methacrylate	97-63-2	Water	0.11	5	0.2	5	ug/L	48-143	63-134	30	-	30
8260	Ethylbenzene	100-41-4	Water	0.042	0.5	0.1	0.5	ug/L	67-121	66-136	30	75-125	30
8260	Ethylene Oxide	75-21-8	Water	1.1	10			ug/L	70-130	70-130	30	-	30
8260	Hexachlorobutadiene	87-68-3	Water	0.19	2	0.2	2	ug/L	57-119	60-132	30	50-140	30
8260	Iodomethane	74-88-4	Water	0.27	5	0.4	5	ug/L	51-164	65-155	30	-	30
8260	Isobutyl Alcohol	78-83-1	Water	12	100	20	100	ug/L	36-142	27-182	30	-	30
8260	Isopropylbenzene	98-82-8	Water	0.031	2	0.2	2	ug/L	67-129	58-144	30	75-125	30
8260	m,p-Xylenes	179601-23-	Water	0.078	0.5	0.1	0.5	ug/L	69-121	67-135	30	75-130	30
8260	Methacrylonitrile	126-98-7	Water	0.25	5	0.8	5	ug/L	47-136	68-129	30	-	30
8260	Methyl Acetate	79-20-9	Water	0.38	1			ug/L	70-130	70-130	30	-	30
8260	Methyl Methacrylate	80-62-6	Water	0.18	5	0.5	5	ug/L	46-138	61-143	30	-	30
8260	Methyl tert-Butyl Ether	1634-04-4	Water	0.070	0.5	0.2	0.5	ug/L	54-126	54-126	30	65-125	30
8260	Methylcyclohexane	108-87-2	Water	0.33	1	0.2	1	ug/L	70-130	65-154	30	-	30
8260	Mehtylcyclopentane	96-37-7	Water					ug/L		65-154	30		
8260	Methylene Chloride	75-09-2	Water	0.23	2	0.2	2	ug/L	71-122	70-133	30	55-140	30
8260	Naphthalene	91-20-3	Water	0.10	2	0.2	2	ug/L	64-126	52-147	30	55-140	30
8260	n-Butylbenzene	104-51-8	Water	0.056	2	0.5	2	ug/L	55-130	52-144	30	70-135	30
8260	n-Heptane	142-82-5	Water	0.28	2			ug/L		99-162			
8260	n-Hexane	110-54-3	Water	0.29	1	0.5	1	ug/L	54-160	54-160	30	-	30
8260	n-Octane	111-65-9	Water	0.33	5	0.5	5	ug/L	64-138	43-157	30	-	30
8260	n-Propylbenzene	103-65-1	Water	0.037	2	1	2	ug/L	61-124	55-144	30	70-130	30
8260	o-Xylene	95-47-6	Water	0.037	0.5	0.1	0.5	ug/L	71-119	67-127	30	80-120	30
8260	Propionitrile	107-12-0	Water	1.1	5	4	5	ug/L	46-137	72-122	30	-	30
8260	sec-Butylbenzene	135-98-8	Water	0.036	2	0.1	2	ug/L	59-128	56-142	30	70-125	30
8260	Styrene	100-42-5	Water	0.039	0.5	0.2	0.5	ug/L	74-121	66-131	30	65-135	30
8260	tert-Amyl Methyl Ether	994-05-8	Water	0.078	2	0.2	2	ug/L	58-138	47-162	30	-	30
8260	tert-Butyl Alcohol	75-65-0	Water	4	20	10	20	ug/L	38-138	49-142	30	-	30
8260	tert-Butyl Ethyl Ether	637-92-3	Water	0.027	2	0.1	2	ug/L	60-123	52-130	30	-	30
8260	tert-Butylbenzene	98-06-6	Water	0.038	2	0.1	2	ug/L	61-127	59-139	30	70-130	30
8260	Tetrachloroethene (PCE)	127-18-4	Water	0.077	0.5	0.2	0.5	ug/L	62-126	61-131	30	45-150	30
8260	Tetrahydrofuran	109-99-9	Water	0.89	5	2.5	5	ug/L	31-157	71-153	30	-	30
8260	Toluene	108-88-3	Water	0.048	0.5	0.1	0.5	ug/L	69-124	71-136	30	75-120	30
8260	trans-1,2-Dichloroethene	156-60-5	Water	0.048	0.5	0.2	0.5	ug/L	67-125	65-143	30	60-140	30
8260	trans-1,3-Dichloropropene	10061-02-6	Water	0.041	0.5	0.2	0.5	ug/L	59-125	56-127	30	55-140	30
8260	trans-1,4-Dichloro-2-butene	110-57-6	Water	0.20	10	1	10	ug/L	46-170	63-157	30	-	30
8260	Trichloroethene (TCE)	79-01-6	Water	0.061	0.5	0.2	0.5	ug/L	67-128	53-139	30	70-125	30
8260	Trichlorofluoromethane	75-69-4	Water	0.086	0.5	0.2	0.5	ug/L	52-141	45-124	30	60-140	30
8260	Trichlorotrifluoroethane	76-13-1	Water	0.079	0.5	0.5	0.5	ug/L	56-141	56-161	30	-	30
8260	Vinyl Acetate	108-05-4	Water	0.91	5	1	5	ug/L	44-156	69-148	30	-	30
8260	Vinyl Chloride	75-01-4	Water	0.071	0.5	0.2	0.5	ug/L	55-123	49-136	30	50-145	30
8260	1,2-Dichloroethane-D4 (Surr.)	17060-07-0	Water	NA	NA	NA	NA	%	59-127	NA	NA	70-120	30
8260	4-Bromofluorobenzene (Surr.)	460-00-4	Water	NA	NA	NA	NA	%	68-117	NA	NA	75-120	30
8260	Dibromofluoromethane (Surr.)	1868-53-7	Water	NA	NA	NA	NA	%	73-122	NA	NA	85-115	30
8260	Toluene-D8 (Surr.)	2037-26-5	Water	NA	NA	NA	NA	%	78-129	NA	NA	85-120	30

CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	LODb	LOQc	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)	DOD QSM (LCS %Rec.)	DOD QSM (% RPD)

a MDL is the smallest analyte concentration that can be demonstrated to be different from zero with 99% confidence

b The LOD is the smallest amount of a substance that must be present in a sample in order to be detected with 99% confidence. Verification is acceptable if the response is > 3x instrument noise. Verification is acceptable if the response is > 3x instrument noise & ion abundance

c The LOQ is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias.

APPENDIX D

Responses to Comments

Alaska Department of Environmental Conservation (ADEC)
Contaminated Sites Program
Document Reviewed: Draft August 2013 Northeast Cape Removal Action Supplemental QAPP
Commenter: Curtis Dunkin-ADEC **Date Submitted:** 3 September 2013; ADEC Reviewed RTCs on 6 September 2013

#	Page #	Section	ADEC Comment	Response
1.	O-1	Proj. Overview	Revise the last sentence of the first paragraph and other similar statements throughout the document. The additional sampling proposed during this field effort is intended to support the first five-year review, not the remedial actions at the site. The document should state and clarify that this site-specific sampling is intended to only facilitate the first five-year review associated with those sites where the sampling is occurring. Also, the document should state and clarify that this sampling is not conclusive and that future sampling may be necessary for these and other sites. ADEC's understanding was that the mobilization by Jacobs to NEC was also going to involve site inspections at each of the Areas of Concern(AOCs) or sites which were identified in the DD and which are still active (not cleanup complete). This document should discuss and clarify this if those activities are planned as part of this mobilization effort. Third paragraph on this page, see first part of this comment above re: general statements about the proposed sampling supporting the five-year review activities.	The document will be revised to state this site-specific sampling is intended to facilitate the first five-year review associated with those sites where the sampling is occurring. The report on sampling results may address recommendations regarding potential future sampling needs. The first five year review may also contain recommendations for future five-year reviews, which could include sampling. **ADEC-Accepted 5 September 2013** This is a sampling work plan. The site inspections will be completed during the same mobilization as the sampling, and in accordance with EPA 540-R-01-007 guidance. **ADEC-Accepted 5 September 2013; however the work plan should state and clarify this.**
2.	O-2	Org. of Suppl.	Re: the statement in the seventh bullet on this page and similar statements throughout the document, any changes should be discussed and clarified – not just those which are considered 'significant'. Similarly in Table 1, what defines 'minor change'?	The text of bullet seven will be revised as follows: "Only those worksheets that are revised are included in this supplement." **ADEC-Accepted 5 September 2013** Additionally, the header of Table 1 will be updated as follows: "Worksheet Excluded (no change)" **ADEC-Accepted 5 September 2013**
3.	O-5	Field Obj.	Groundwater sample collection should be added for both the sites 7 and 9 landfills.	The field objectives will be updated to add one groundwater grab sample at Site 7 and Site 9 landfills. **ADEC-Accepted 5 September 2013**

#	Page #	Section	ADEC Comment	Response
4.	O-6	GW Grab Sampl.	Needs to include groundwater samples proposed by ADEC at site 7. Revise elsewhere in document when discussing groundwater sampling. Revise statements referring to the 6- and 24-inch intervals. The groundwater drive points should be advanced to either groundwater and/or refusal, whichever is encountered first. ADEC suggests that groundwater drive points be retried at least at one other location in the event that no groundwater is initially encountered in the first drive. The objective for the other attempted location should be to better characterize the presence/absence of groundwater at the site. In the event no groundwater is encountered and/or sampled, ADEC suggests that at least one of the unused groundwater samples be utilized to collect sediment from one of the surface water sampling locations. Sediment should be analyzed for the same analytes and should be included in the QAPP sections.	Groundwater drive points will be advanced to either groundwater and/or refusal, whichever is encountered first. Permafrost is anticipated at 2 feet below ground surface. Given time constraints, especially the short-term availability of the camp, only one drive point location will be attempted at Site 7, and one at Site 9. If a groundwater sample(s) is (are) not collected because the volume of water is insufficient, sediment will not be collected because sediment samples are out of the scope of the project. **ADEC-Accepted 5 September 2013. Please note that ADEC's suggestions were intended to maximize the usefulness of this mobilization and to minimize potential data gaps that could impact the five-year review protectiveness determinations. It is not an unreasonable request to conduct two more shallow drive points (which are anticipated to not be deeper than two feet) considering that the majority of this field effort is the actual mobilization and demobilization to and from the site.**
5.	O-7	GW Grab Sampl.	Any/all of the water that is obtained from the groundwater drive point should be retained and utilized for the sample (if enough to meet the minimum sample volume requirement) in the event the well goes dry during purging.	The following text will be added to the first paragraph of page Overview-7: "Any groundwater present in low-producing well points will be added to sample containers in a one-time attempt to obtain samples prior to discontinuing groundwater grab sampling." **ADEC-Accepted 5 September 2013**
6.	O-8	Waste Mngmt.	Table 2: What IDW is estimated to generate 1 cubic yard of waste as indicated?	It is anticipated only one trash bag of waste will be produced. The two instances of "1 cy" will be replaced with "1 Trash Bag" on Table 2. **ADEC-Accepted 5 September 2013**
7.	O-9	References	Include ADEC's Sampling Guidance.	The following will be added to the reference section: "ADEC (Alaska Department of Environmental Conservation). 2010 (May). Draft Field Sampling Guidance. Division of Spill Prevention and Response. Contaminated Sites Program" **ADEC-Accepted 5 September 2013**

#	Page #	Section	ADEC Comment	Response
			QAPP Worksheets	
8.	9-1	Worksheet #9	Change Curtis Dunkin's title to project manager. Project Name: Note previous comments re: reference to this specific supplemental QAPP; this work plan is for site-specific sampling to support the five-year review associated only with those specific sites. Revise references to 'North East Cape' to 'Northeast Cape'.	All instances of "Regulatory" with regard to project manager will be deleted. **ADEC-Accepted 5 September 2013**
9.	10-1	Worksheet # 10	The Environmental Questions…: Revise/clarify the reference to 'shallow groundwater' here and throughout the rest of the document. While it is anticipated that groundwater (if present) will be shallow in the areas adjacent to the landfills, the hydrogeology at sites 9 and 7 has not been thoroughly characterized. Suggest further revision of the above statement (and elsewhere in document where applicable) since it is not known whether specific contaminants of concern exist within and/or may be migrating from the landfills. Suggest revising 'chemicals of concern' to contaminants of concern'.	The term "shallow" will be removed in reference to groundwater. **ADEC-Accepted 5 September 2013** The Environmental Question will be reworded as follows: "Using ADEC 18 AAC 70 and 18 AAC 75 (Table C) criteria, are contaminants of potential concern present downgradient from landfills (Site 7 Landfill and Site 9 Landfill) in surface water and groundwater?" **ADEC-Accepted 5 September 2013** 'Chemicals of concern' will be replaced with 'contaminants of concern' **ADEC-Accepted 5 September 2013**
10.	15-7	Worksheet # 15	It should be noted that the evaluation criteria listed as the project action limits is the drinking water criteria. The applicable criteria for surface water are actually those for chronic freshwater organisms; which are significantly lower than the human drinking water standards.	Noted. **ADEC-Accepted 5 September 2013** The project action limits are cited directly as they appeared in the Final Northeast Cape HTRW Remedial Actions Work Plan. **ADEC-Accepted 5 September 2013**
11.	17-1	Worksheet # 17	Sampling Design and Rationale: last statement of this section (and other statements throughout the document where applicable) should be revised to clarify that the resulting data will assist the five-year review for the specific sites where sampling is being conducted as part of this effort. It is not currently known whether this data will 'provide sufficient information'. Second bullet of this section; add the groundwater grab samples at site 7 as previously commented above.	The text of page 17-1 will be updated as follows: "The resulting data obtained from the Jacobs sampling effort at three sites will provide information for incorporation into the first NE Cape five-year review." **ADEC-Accepted 5 September 2013** The second and third bullets will be updated to include one groundwater grab sample at Site 7 and Site 9 Landfills. **ADEC-Accepted 5 September 2013**
12.	17-2	Table 17-1	Add Site 7 groundwater grab samples as previously commented above.	Table 17-1 will be updated to include groundwater grab samples at Site 7 and Site 9 Landfills. **ADEC-Accepted 5 September 2013**

#	Page #	Section	ADEC Comment	Response
13.	18-1	Worksheet # 18	Depth: revise to at groundwater table or refusal, whichever is encountered first. Rationale for Sampling Location: Add site 7 groundwater samples.	Depth will be revised as follows: "Groundwater table or refusal" Site 7 was added to the Rationale for Sampling Location. **ADEC-Accepted 5 September 2013**
14.	19-1	Worksheet # 19	Analytical Group: replace methods AK101 etc. with the respective COCs Analytical/Preparation Method: revise to include and differentiate between the Jacobs SOP and the ADEC-approved analytical method (i.e. either include both or add a new column w/ the ADEC-approved method.	AK101 will be replaced with GRO AK102/103 will be replaced with DRO/RRO **ADEC-Accepted 5 September 2013** The table header will be updated on Worksheet #19 and #23 to state "Laboratory SOP Reference" **ADEC-Accepted 5 September 2013** The ADEC approved test method will be added to Worksheet #23. **ADEC-Accepted 5 September 2013**
15.	20-1	Worksheet # 20	Revise to include the site 7 groundwater samples as previously commented above. Analytical Group: please see comment # 14 above. Field Duplicates: why are there three field duplicate samples proposed for 6 primary samples for each of the COC analytical groups?	Worksheet #20 will be updated to include all project samples. **ADEC-Accepted 5 September 2013** The analytical group field will be updated per comment 15. **ADEC-Accepted 5 September 2013** The frequency of field duplicates (1 per 10) and MS/MSDS (1 set per 20) will be updated to be consistent with the ADEC draft sampling guidance. **ADEC-Accepted 5 September 2013**
16.		Figure A-2	ADEC's understanding is that Savoonga is approximately 60 or more miles from Northeast Cape. This should be verified and revised if necessary.	The distance on Figure A-2 will be revised to reflect a distance of approximately 60 miles. **ADEC-Accepted 5 September 2013**
17.		Figure A-3	All figures in the report for this effort should include previous surface and groundwater sampling locations, dates, and any exceedances indicated via analytical results. Legend should be revised to: 1) state 'proposed 2013 sampling location' for each matrix; 2) notate surface water features in blue; 3) notate AOCs such as the site 7 landfill boundary. The figure should be revised to adequately depict these features. Figure will need to be revised per groundwater grab samples at site 7 as commented above.	Draft and final sampling reports will be submitted. The reports will include recent site data and show sample locations and results. **ADEC-Accepted 5 September 2013; ADEC interprets 'recent' to mean all previous/available surface and groundwater sampling locations and results associated with the respective site that are determined to be useful for incorporation into the five-year review.** All figure legends will be updated to reflect 2013 proposed sampling location. **ADEC-Accepted 5 September 2013** Blue outlines will be added to surface water features. **ADEC-Accepted 5 September 2013**

#	Page #	Section	ADEC Comment	Response
18.		Figure A-4	Please see comments in # 17 above. The site 9 landfill boundary needs to be included on the figure along with other requested revisions in comment # 17 above. The surface and groundwater sample locations (one each) in the far western/southwestern edge of the AOC do not appear consistent with locations proposed and agreed to by ADEC and the ACOE.	The Site 9 landfill boundary will be added to Figure A-4 along with the other requests noted in comment 17. **ADEC-Accepted 5 September 2013**
19.		Figure A-5	Please label the spring and call out other site features within the view of this figure. Indicate the surface water flow direction(s) and depict known surface water features in blue. Please see similar comments on other figures above re: legend notations.	The spring will be labeled and other site features will be added to Figure A-5 along with the other requests noted on comment 17. **ADEC-Accepted 5 September 2013**
End of ADEC Comments				