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November 1, 2021

Pat Evangelista
Director of Superfund and Emergency Management Division
USEPA Region 2

John Prince
Deputy Director of Superfund & Emergency Management
USEPA Region 2

RE: Gowanus Canal Monitoring Plan

Included is the monitoring plan for the Gowanus Canal. This plan is submitted in accordance with a unilateral administrative order issued to the City of New York ("City") by the U.S. Environmental Protection Agency (USEPA) on March 29, 2021, as modified by letter dated June 29, 2021, from Pat Evangelista to Angela Licata (as modified, the "UAO").

As we briefly discussed on a Teams Meeting on October 22, 2021 with Acting Regional Administrator Walter Mugdan and Deputy Director John Prince and me, we have been working diligently to meet USEPA requirements and would like to schedule a workshop to discuss this plan and its implementation. We look forward to any input and questions on this plan.

Sincerely,

Angela Licata
Deputy Commissioner, Sustainably
New York City Department of Environmental Protection

cc: Walter Mugdan, Acting Regional Administrator, USEPA Region 2
Brian Carr, Assistant Regional Counsel, USEPA Region 2
Christos Tsiamis, Project Manager, USEPA Region 2

October 2021

Quality Assurance Project Plan for Environmental Investigations at the Gowanus Canal Superfund Site: Post Remedy Monitoring Plan

Gowanus Canal Superfund Site
Borough of Brooklyn, New York

Prepared by:
WSP USA Solutions Inc.
October 2021
Version: 01

Prepared for:
New York City Department of Environmental Protection

Document Control Number: GC005

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Figure 1: Project Organizational Chart

Figure 2: CSO Sampling Locations (RH-034 and OH-0070)

Figure 3: Proposed Sediment Sampling Locations

ATTACHMENTS

Attachment 1: Field Standard Operating Procedures

Attachment 2: Laboratory Standard Operating Procedures

Attachment 3: Field Modification Form (Template)

Attachment 4: Non-Conformance Form (Template)

Attachment 5: Example Chain of Custody (COC) for shipment of samples to laboratory

Attachment 6: Field Mobilization Protocol

QAPP Worksheet 1 and Worksheet 2
Title and Approval Page

Site/Project Name: Gowanus Canal Superfund Site / Gowanus Canal Environmental Investigations
Site Location: Borough of Brooklyn, New York
Operable Unit: Not Applicable
Docket Number: Not Applicable

Document Title: Quality Assurance Project Plan for Gowanus Canal Superfund Site: Post Remedy Monitoring Plan
Lead Organization: New York City Department of Environmental Protection (NYCDEP)
NYCDEP Contract: NYCDEP CONTRACT #CTI82620151427257 (the QAPP is prepared under this contract, but the field sampling will be implemented under a new contract with NYCDEP)

Preparer's Name and Organizational Affiliation:
Prepared by WSP USA Solutions Inc. (WSP) [formerly known as Louis Berger & Assoc., P.C. (Louis Berger)]; will be referred to WSP throughout this document
Xiulan Wang (WSP) and Ying Wang (WSP)

Preparer's Address, Telephone Number, and E-mail Address:
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Ying Wang: 420 Mount Kemble Ave, Morristown, New Jersey, 07960; (973) 407-1015; yi.wang@wsp.com

Preparation Date (Day/Month/Year): October 15, 2021

Investigative Organization's Project Manager:

_____
To Be Determined

Investigative Organization's Quality Consultant:

_____
To Be Determined

Investigative Organization's Site Quality Control Officers:

_____
To Be Determined

NYCDEP Project Manager (Approval Authority):

_____
Ron Weissbard (NYCDEP)

Note: This QAPP has been prepared under the current EEGEO15 contract. However, sampling for the field program associated with the QAPP is scheduled to start in June 2022 under a new contract with NYCDEP. The organizational chart in the QAPP will be updated with the project personnel once the new contract is registered. Before the field program, the new contractor will review the QAPP and issue a field modification if any revisions are needed.

QAPP Worksheet 1 and Worksheet 2
Title and Approval Page

1. Identify regulatory program: Comprehensive Environmental Response, Compensation, and Liability Act.
The QAPP was prepared following the requirements stated in "Uniform Federal Policy for Quality Assurance Project Plans, Optimized UFP-QAPP Worksheets" (prepared by the Intergovernmental Data Quality Task Force, March 2012).

The QAPP is also consistent with the following quality assurance guidance documents:

- "Contract Laboratory Program Guidance for Field Samplers: Sampler Guide" prepared by Office of Superfund Remediation and Technology Innovation (OSRTI), EPA/540/R-014/013, October 2014.
- "Guidance for Quality Assurance Project Plans QA/G-5" prepared by Office of Environmental Information, EPA/240/R-02/009, December 2002.
- "EPA Requirements for Quality Management Plans QA/R-2" prepared by Office of Environmental Information, EPA/240/B-01/002, March 2001.
- "EPA Requirements for Quality Assurance Project Plans QA/R-5" prepared by Office of Environmental Information, EPA/240/B-01/003, March 2001.
- "Guidance on Technical Audits and Related Assessments for Environmental Data Operations QA/G-7" prepared by Office of Environmental Information, EPA/600/R-99/080, January 2000.

2. List dates and titles of QAPP documents written for previous site work, if applicable:

Title	Approval Date
NYCDEP, October 2021. "Quality Assurance Project Plan for Environmental Investigations at the Gowanus Canal Superfund Site: Post Remedy Monitoring Plan" Version 01, October 2021, prepared by WSP, Document Control No. GC005.	This document (will be submitted in October 2021)
NYCDEP, January 2014. "Quality Assurance Project Plan for Environmental Investigations at the Gowanus Canal Superfund Site: Monitoring Plan" Version 04, January 2014, prepared by Louis Berger & Associates, PC, Document Control No. GC004. Field Modification No. 1 submitted August 2012 (addresses quality assurance sediment split sample collection) Field Modification No. 2 submitted February 2013 (describes the collection of surface water column samples) Field Modification No. 3 submitted February 2013 (describes the collection of additional VOC and TSS samples during scheduled CSO wet and dry weather events) Field Modification No. 4 submitted April 2013 (describes the reanalysis of existing surface sediment samples and suspended matter samples to measure and report supported Thorium-234) Field Modification No. 5 submitted July 2013 (describes the collection of sediment cores at the head end of Gowanus Canal to characterize the material that may be potentially sourced due to Flushing Tunnel startup) Field Modification No. 6 prepared by AECOM/HydroQual Joint Venture and submitted October 2013 (describes the collection of samples to characterize sediments that may be potentially dredged or that may be potentially exposed after dredging) Field Modification No. 7 submitted April 2014 (describes data validation of potentially dredged material sediment samples) Field Modification No.8 submitted June 2014 (describes the collection of surface water column samples to quantify baseline contaminant concentrations in Gowanus Bay/Upper New York Harbor (during flood tide when tidal water is flowing into Gowanus Canal) and Buttermilk Channel (at the point of inflow into the flushing tunnel regardless of tide), and to measure the current velocity under various tidal conditions to calibrate the hydrodynamic model. Field Modification No. 9 submitted October 2014 (describes the collection of surface water	January 2014

column samples, surface water monitoring samples, CSO and SWO discharge samples, and sediment oxygen demand samples to update the existing hydrodynamic model, and to calibrate the organic carbon or dissolved oxygen model for the Gowanus Canal.	

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

- United States Environmental Protection Agency (USEPA) has listed the Gowanus Canal Superfund Site on the National Priorities List (NPL) for hazardous waste.
- NYCDEP will be implementing this environmental investigation at the Gowanus Canal Superfund Site.
- The selected contractor will provide consulting services under the new contract to be registered with NYCDEP. The sampling for this QAPP will be implemented under the new contract with NYCDEP.
- Stakeholders include NYCDEP, USEPA, NYSDEC, and other stakeholders.

QAPP Worksheet 3 and Worksheet 5
Project Organization and QAPP Distribution

Project Organization and QAPP Distribution

QAPP Recipients	Title	Organization	Telephone Number	E-mail Address	Document Control Number
Ron Weissbard	Director, Hazardous Materials Assessment	NYCDEP	718-595-5186	RWeissbard@dep.nyc.gov	Electronic copy of Document Control No. GC005
To Be Determined	Project Manager	To Be Determined	To Be Determined	To Be Determined	Electronic copy of Document Control No. GC005
To Be Determined	Quality Consultant	To Be Determined	To Be Determined	To Be Determined	Electronic copy of Document Control No. GC005
To Be Determined	Site Quality Control Officers	To Be Determined	To Be Determined	To Be Determined	Electronic copy of Document Control No. GC005
To Be Determined	Project Scientist	To Be Determined	To Be Determined	To Be Determined	Electronic copy of Document Control No. GC005
To Be Determined	Field Team Leader	To Be Determined	To Be Determined	To Be Determined	Electronic copy of Document Control No. GC005
Field Team Members	Investigative Organization Field Team	To Be Determined	To be determined	To be determined	Electronic copy of Document Control No. GC005
To Be Determined	Investigative Organization Health and Safety Manager	To Be Determined	To Be Determined	To Be Determined	Electronic copy of Document Control No. GC005
Georgina Brooks	SGS AXYS Laboratory Project Manager	SGS AXYS Analytical Services, Ltd (SGS AXYS)	250-655-5801	Georgina.Brooks@sgs.com	Electronic copy of Document Control No. GC005
Heather Manz	Katahdin Analytical Services Laboratory Project Manager	Katahdin Analytical Services (Katahdin)	207-874-2400	hmanz@katahdinlab.com	Electronic copy of Document Control No. GC005

Project Organization and QAPP Distribution

QAPP Recipients	Title	Organization	Telephone Number	E-mail Address	Document Control Number
Patrick McLaren	SedTrend Analysis Limited Laboratory Project Manager	SedTrend Analysis Limited (SedTrend)	250-652-1334	pmclaren@sedtrend.com	Electronic copy of Document Control No. GC005
To Be Determined	Data Validator	To Be Determined	To Be Determined	To Be Determined	Electronic copy of Document Control No. GC005

Note 1: Refer to Figure 1 "Project Organizational Chart" for key project personnel, lines of authority, and lines of communication among the lead agency, prime contractors, and subcontractors.

QAPP Worksheet 4, Worksheet 7, and Worksheet 8
Personnel Qualification and Sign-Off Sheet

Personnel Qualification and Sign-Off Sheet

Name	**Project Title/Role**	**Education/Experience**	**Specialized Training and Certifications**	**Signature/Date** *Signature indicates that personnel have read and agree to implement this QAPP as written.*
Ron Weissbard	Project Manager (NYCDEP)	PE, BSE Chemical Engineering	Not applicable	
To Be Determined	Project Manager	To Be Determined	40-hour HAZWOPER CPR/First Aid	
To Be Determined	Quality Consultant	To Be Determined	40-hour HAZWOPER CPR/First Aid	
To Be Determined	Project Scientist	To Be Determined	40-hour HAZWOPER CPR/First Aid	
To Be Determined	Site Quality Control Officer	To Be Determined	40-hour HAZWOPER CPR/First Aid	
To Be Determined	Field Team Leader	To Be Determined	40-hour HAZWOPER CPR/First Aid	
To Be Determined	Organization Health and Safety Manager	To Be Determined	40-hour HAZWOPER CPR/First Aid	
Field Team Members	Field Team Members	To be determined	40-hour HAZWOPER CPR/First Aid	

Note 1: Laboratories are under contract with the selected NYCDEP contractor; by signing subcontract, laboratories must adhere to the QAPP.
Note 2: Copies of OSHA HAZWOPER certifications will be stored on file at the selected NYCDEP contractor office.

QAPP Worksheet 6
Communication Pathways

Communication Pathways

Communication Drivers	Organization	Name	Contact Information	Procedure (Timing, Pathways, Documentation etc.)
Real time modification, notifications, and approval	To Be Determined	Site Quality Control Officer and Project Manager	Refer to QAPP Worksheet 3	Real time modification to the project will require the approval of the Project Manager and Site Quality Control Officer. Modifications will be documented using the Field Modification Form (refer to Attachment 3).
Approval of amendments and field modifications to the QAPP	NYCDEP	NYCDEP Project Manager	Refer to QAPP Worksheet 3	Submit Field Modification Form (refer to Attachment 3) to NYCDEP for review and approval.
Document control	To Be Determined	Project Manager	Refer to QAPP Worksheet 3	Project Manager (or delegate) is responsible for document preparation and distribution as well as document control.
Communicating serious issue with field work	To Be Determined	Field Team Leader and Project Manager	Refer to QAPP Worksheet 3	Field team member will report serious issues on the field program to the Field Team Leader, whom will then inform the Project Manager (or delegate). Project Manager will communicate serious issues with NYCDEP and other concerned parties, and initiate corrective action.
Corrective action to address serious field work issues	To Be Determined	Site Quality Control Officer	Refer to QAPP Worksheet 3	Site Quality Control Officer will report corrective action to the Project Manager (or delegate). A Non-Conformance Quality Control Report will be issued (refer to Attachment 4), and as necessary, a Field Modification Form (refer to Attachment 3) will be submitted for approval. The Project Manager (or delegate) or Technical Lead will communicate with NYCDEP on corrective actions.
Field communication	To Be Determined	Field Team Leader	Refer to QAPP Worksheet 3	The Field Team Leader will coordinate field activities, sample collection, and sample shipment with field crew. Summarize daily activity in electronic mail format to Project Manager. Refer to Worksheets 31, 32, and 33 on reporting field activities. Field Team Leader will be responsible for providing direction to all subcontractors in the field.

Communication Pathways

Communication Drivers	Organization	Name	Contact Information	Procedure (Timing, Pathways, Documentation etc.)
Health and Safety Communication	To Be Determined	Project Safety Officer	Refer to QAPP Worksheet 3	The Field Team Leader will have a dual role and act as the Project Safety Officer. Field Team Leader/Project Safety Officer (or delegate) will conduct a daily health and safety meeting to discuss daily work activities and potential health and safety issues. Field Team Leader/Project Safety Officer (or delegate) will follow the Project Health and Safety Plan and contact the Project Health and Safety Manager and Project Manager if any health and safety issues arise. Health and Safety issues and corrective action will be documented per the project health and safety plan.
Communication with laboratories and validators	To Be Determined	Site Quality Control Officer	Refer to QAPP Worksheet 3	Site Quality Control Officer will communicate with laboratories to notify them of anticipated sample shipment and resolve any analytical issues. Site Quality Control Officer will communicate with data validators to resolve any technical issues and complete a validated electronic database of sample results. Minor and major excursions in data quality will be reported to the Quality Consultant for review.
Review of Data	To Be Determined	Site Quality Control Officer along with Quality Consultant	Refer to QAPP Worksheet 3	The Site Quality Control Officer will communicate with the Project Manager (or delegate) and Quality Consultant on any analytical issues that arise. Project Manager and Quality Consultant will be responsible for reviewing the data and providing recommendations to the Project Team.

QAPP Worksheet 9
Project Planning Session Summary

Project Name: Quality Assurance Project Plan for Gowanus Canal Superfund Site **Projected Date(s) of Sampling**: June 2022 – June 2024 **Project Managers**: Ron Weissbard (NYCDEP), Rebecca Tummon (WSP), and Chitra Prabhu (HDR)			**Site Name**: Gowanus Canal Superfund Site **Site Location**: Boroughs of Brooklyn and Queens, New York	
Date of Session: Multiple sessions (September through October 2021) Scoping Session Purpose: Coordination meeting on Gowanus Canal Post Remedy Monitoring Plan				
Name	Title/Role	Organization	Phone #	E-mail Address
Rebecca Tummon	Project Manager	WSP	212-612-7923	rebecca.tummon@wsp.com
Chitra Prabhu	Technical Lead	HDR	914-993-2011	chitra.prabhu@hdrinc.com
Ron Weissbard	Project Manager	NYCDEP	718-595-5186	RWeissbard@dep.nyc.gov

Comments/Decisions: During the coordination meeting, the Project Team discussed scope of work, types of samples that are required for the Post Remedy Monitoring Program, and field schedule. It is anticipated that field work will start around June 2022. The discussion included the following:
(1) collection of CSO outfalls from RH-034 and OH-007 quarterly for 2-year monitoring program to provide a baseline to characterize the concentrations of CSO solids that enter the Gowanus Canal;
(2) collection of sediment samples in RTA1 for in-Canal monitoring to estimate the average COPC concentrations;
(3) the analytical parameters that are required under the CSO monitoring and sediment programs;
(4) the field and data validation schedule; and
(5) the complete QAPP submittal schedule.

QAPP Worksheet 10
Problem Definition

Problem Definition (Worksheet 10)

The problem to be addressed by the project:
This plan is submitted in accordance with a unilateral administrative order issued to the City of New York ("City") by the U.S. Environmental Protection Agency (USEPA) on March 29, 2021, as modified by letter dated June 29, 2021, from Pat Evangelista to Angela Licata (as modified, the "UAO"). The UAO requires certain design, removal, and remedial action work in connection with the Gowanus Canal Superfund Site ("Site" or "Canal"). By letter dated July 14, 2021, the City notified EPA of its intention to comply with the UAO by performing the CSO design and CSO remedial action work required by the Order. In particular, the City will construct tanks to control CSO solids discharge from the two largest outfalls to the Canal – RH-034 and OH-007. In addition to the construction of these tanks, the UAO requires the City to implement a monitoring plan in an effort to understand the impact of CSO solids on the remedial target area (RTA)[1] post-remedy. The monitoring requirements as part of this UAO include the following:

1. Monitoring beginning on June 1, 2022, to establish a baseline for CSO solids loading and solids contaminant levels;
2. Periodic in-Canal monitoring beginning one year after capping is completed in RTA 1.

To meet the above requirements, the monitoring plan will quantify the levels of contaminants of potential concern (COPCs) on solids discharging from CSO outfalls RH-034 and OH-007 along with the levels of total suspended solids (TSS), suspended solids organic carbon, and suspended solids grain size distribution. Samples will be collected from these two outfalls quarterly for a period of two years starting from June 1, 2022. This sampling will provide a baseline for the characteristic of the CSO solids that enter the Canal. To quantify the solids loading to the Canal, the City will use data from this monitoring program along with modeling to develop CSO solids loading to the Canal.

The UAO also requires in-Canal monitoring to be initiated one year after the remedial action in RTA 1 has been completed. However, depending on sedimentation rates, the one-year criterion specified in the UAO may not be adequate to accumulate enough soft sediments in the Canal for monitoring. To plan for the in-Canal monitoring required by the UAO, the City will use the solids loading and grain size distribution data along with the Gowanus Canal modeling to develop an understanding of the sedimentation rates and solids transport in the Canal for various solids sources (CSO/stormwater discharges, inflow from Buttermilk Channel through the flushing tunnel, and tidal advection/dispersion from Upper New York Bay). The solids transport model will be updated to represent the Canal bathymetry after the USEPA remedy is implemented. The model will use solids loading and grain size distribution data collected from the 2022 CSO monitoring program and data collected by the City and USEPA as part of the RI/FS to represent all solids sources to the Canal. This modeling is necessary to understand the timeframe needed for the sediment bed to accumulate six inches of soft sediments for in-Canal monitoring. The timeframe estimated by the model to accumulate six inches of soft sediment will be verified using a multibeam bathymetry survey in RTA1.

Once the timeframe for collecting sediment samples has been verified, 10 sediment samples will be collected in RTA 1 to estimate the average COPC concentrations in the surface sediments after remedial action has been implemented. The average COPC concentrations in the sediments will be then compared to the project remedial goals (PRGs) proposed by the USEPA in the ROD. If the concentrations in sediment exceeds the PRG, then the contaminant

[1]EPA has divided the Canal into three Remediation Target Areas ("RTAs"). RTA 1 extends from Butler Street to 3rd Street. RTA 2 extends from 3rd Street to Hamilton Street. RTA 3 extends from Hamilton Street to the mouth of the Canal at 23rd Street.

Problem Definition (Worksheet 10)

patterns in the 10 sediment samples will be compared with the contaminant patterns on CSO solids to evaluate whether the solids from CSOs are responsible for the elevated concentrations measured in the sediments. This step is necessary given that the sediments in the RTA 1 may be influenced by cleanup activities in RTA 2 and RTA 3, and other sources. Note that for metals, the PRG proposed by the USEPA assumes that the metals are bioavailable. Therefore, if the geometric mean concentrations for metals in the soft sediments exceed the PRGs, then the simultaneously extracted metals/acid volatile sulfide (SEM/AVS) analyses will be conducted to measure the bioavailability of metals. These analyses will be submitted to USEPA to assess the need for maintenance dredging.

The environmental questions being asked:

- What are the COPC concentrations in the solids entering the Canal from CSO outfalls OH-007 and RH-034?
- What is the solids loading to the Canal from CSO outfalls OH-007 and RH-034?
- What is the COPC concentration in the surface sediments in the RTA 1 of the Canal post remedy?

A synopsis of secondary data or information from site reports:

- The CSO LTCP developed by the City for the Gowanus Canal
- RI/FS data collected by USEPA
- Data collected by the City as part of comments on the USEPA Proposed Remedial Action Plan (PRAP) for the Gowanus Canal

The possible classes of contaminants and the affected matrices:

Large Volume Samples from CSO discharges (particulate phase) will be analyzed for:

- PCB Congeners (USEPA Method 1668)
- PAHs and Alkylated PAHs (SW-846 Modified Method 8270)
- TSS (USEPA Method 160.2 modified / SM 2540D)
- Particulate Organic Carbon (POC) (Lloyd Kahn-modified)
- Grain Size Distribution (ASTM D422 or equivalent)

Large Volume Samples from CSO discharges (Whole water and dissolved phase) will be analyzed for:

- Metals (SW-6020/ SW6010)

Sediment Samples will be analyzed for

- PCB Congeners (USEPA Method 1668)
- PAHs and Alkylated PAHs (SW-846 Modified Method 8270)
- Metals (SW-6020/ SW6010)
- AVS/SEM (EPA-821/R-91-100 Draft 1991)
- Total Organic Carbon (TOC) (Lloyd Kahn-modified)
- Grain Size Distribution (ASTM D422 or equivalent)
- Percent Moisture (SM2540G)

Problem Definition (Worksheet 10)

The rationale for inclusion of chemical and nonchemical analyses:

Chemical and Physical analyses for laboratory samples:

- PCB Congeners: PCB compounds are COPCs. PCB concentrations will be quantified in the CSO samples as particulate phase and in sediment samples. PCB analysis will include reporting of 209 PCB congeners to examine PCB patterns and potentially identify unique PCB congener "fingerprints."
- PAHs and alkylated PAHs: PAH compounds are COPCs. PAH analysis will include parent PAH compounds as well as alkylated PAH compounds. PAH concentrations will be quantified in the CSO samples as particulate phase and in sediment samples.
- Metals: Lead and copper have been identified as COPCs for the Site and a PRG has been proposed by USEPA for these two metals. Lead and copper concentrations will be quantified in the CSO samples as whole water and dissolved phase samples. These metals will also be analyzed in sediment samples. Particulate phase concentrations in CSO samples will be calculated as the difference between whole water and dissolved phase concentrations.
- AVS/SEM: AVS/SEM will be used to evaluate the potential bioavailability of metals in surface sediments.
- Grain Size Distribution: Grain size distribution will be analyzed to characterize the physical properties of surface sediments and the properties of CSO solids entering the Canal.
- TSS: TSS will be measured to quantify the concentration of solids in CSO discharges on a per unit volume basis. The TSS values will be used to normalize the contaminant concentrations to a mass of contaminant per mass of suspended solids basis. The TSS analytical method will be modified to consistently utilize a 0.7um filter size, consistent with the pore size used for the contaminant suspended mass measurements.
- TOC/POC: organic carbon is a key parameter influencing the bioavailability of organic compounds. PRGs developed for organic compounds are based on the mean canal-wide surface sediment TOC concentration of 6 percent. TOC/POC will be used to normalize the chemistry data on OC-basis.
- Percent Moisture: Moisture measurement is required to convert sediment chemistry results to dry-weight basis.

QAPP Worksheet 11
Project Quality Objectives/Systematic Planning Process Statements

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

Who will use the data?
NYCDEP and other stakeholders including USEPA and NYSDEC.

What will the data be used for?

The monitoring program will be conducted in two phases. Samples from CSO outfalls RH-034 and OH-007 will be collected over a two year period beginning in June 2022 to characterize the baseline COPC concentrations in solids from these outfalls. This data will also be used to develop an estimate of baseline solids loading to the Canal from these two outfalls.

For the second phase of this monitoring program, 10 sediment samples will be collected in RTA 1 to characterize the COPC concentrations in the surface sediments after remedial action has been implemented. The COPC concentrations in sediment samples will be used to represent the average COPC concentrations in the soft sediments of RTA1 after capping. The average concentration in the sediments will be compared to their PRGs as documented in the ROD for the Site.

What type of data is needed?
A comprehensive dataset is required to accomplish the objectives of the sampling program. The following data will be collected as part of the sampling program:

- CSO samples will be analyzed for particulate phase for PCB congeners, and PAH and alkylated PAHs.
- CSO samples will be analyzed for whole water and dissolved phase for metals.
- CSO will be also analyzed for TSS, POC, and TSS grain size distribution.
- Sediment samples will be analyzed for PCB congeners, PAH and alkylated PAHs, metals, TOC, AVS/SEM and grain size distribution.

How "good" do the data need to be in order to support the environmental decision?
Data must be technically defensible and of sufficient quality to support the project study questions (refer to Worksheet 10). Field-generated data will be verified and post-processed according to the requirements in Worksheet 34. All chemical data will be validated by an independent data validator according to Worksheets 35 and 36. Refer to Worksheet 15 for a summary of analytical parameters and quantitation limits.

How much data are needed? (number of samples for each analytical group, matrix, and concentration)
Refer to Worksheet 20 for the number of field samples and quality control samples to be shipped to the laboratory.

Where, when, and how should the data be collected/generated?
Where: Gowanus Canal

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

When: Beginning June 2022
How: As per the procedures outlined in worksheet 17

Who will collect and generate the data?
Samples will be collected by the NYCDEP contractor. The samples will be analyzed for chemical and physical parameters by subcontract laboratories. Refer to Worksheet 19 for container/preservation information, Worksheet 20 for the number of samples shipped, and Worksheets 26 and 27 for shipment information.

How will the data be reported?

- Validated analytical data will be reported and submitted to the NYCDEP according to the requirements in Worksheet 29; analytical data will be compiled into a database that is consistent with USEPA Region 2 Multimedia Electronic Data Deliverable (MEDD) format (*USEPA Region 2 Electronic Data Deliverable Comprehensive Specification Manual 6.0, May 2019*). Laboratory data packages will be provided in PDF format.
- Data validation will follow requirements provided in Worksheets 34 through 36. Validation reports will be provided in PDF format.
- Field generated data (GPS points and field observation) will be verified and post-processed (refer to Worksheet 34); data will be reported electronically into a database that is consistent with USEPA Region 2 MEDD format.
- Field notes will be documented in field forms and photographs (refer to Worksheet 29). Field forms will be verified for completeness.

How will the data be archived?
Electronic data (MEDD deliverables provided in MS Excel spreadsheets containing validated data), field-generated data, PDF formatted laboratory data reports, and PDF formatted validation reports will be kept in the project files. The length of time that records will be archived will be at the discretion of NYCDEP.

QAPP Worksheet 12-1
PAH and Alkylated PAH (Sediment) Measurement Performance Criteria

Matrix: Sediment
Analytical Group or Method: PAH and Alkylated PAH / Modified SW-846 Method 8270D/ Lab SOP MLA-021
Concentration Level: Low

Data Quality Indicator (DQI)	QC Sample or Measurement Performance Activity	Measurement Performance Criteria [1]
Representativeness	Preservation	see Worksheet #19
Sensitivity	Method Blank	< QL
Accuracy	Surrogates	Recoveries within SGS AXYS SOP MLA-021 specified control limits
Accuracy	Laboratory Control Sample (LCS)	Recoveries within SGS AXYS SOP MLA-021 specified control limit
Accuracy	Matrix Spike (MS)	Recoveries within SGS AXYS SOP MLA-021 specified control limits unless sample conc. > 4x spike level (spike swamped out)
Precision	Laboratory Replicate	RPD ≤ 40% if reported values are ≥ 5x QL; otherwise use professional judgement
Accuracy	Certified Reference Material (CRM) (NIST 1944)	Reported concentrations within ±30% of certified and reference values. Applicable for values that are ≥ 3x concentration of the lowest calibration point in the ICAL.
Precision & Representativeness	Field Duplicate	RPD ≤ 50% if reported values are ≥ 5x QL; otherwise use professional judgement
Sensitivity	Equipment Blank	< QL
Representativeness	Temperature Blank	0 -6 degrees Celsius
Completeness	Data Completeness Check	>90% sample collection and >90% laboratory analysis
Comparability/Sensitivity	Data Quality Assessment	Comparable units, QL, and methods; detection limits meet project goals

1. The assigned laboratory must perform and meet all of the measurement performance criteria that assess the analytical DQIs specified in their SOPs and the analytical methods specified.
2. An equipment blank will be generated in the field by rinsing field equipment with laboratory-grade water.
3. Method modifications are listed in QAPP WS #23.

QAPP Worksheet 12-2
PCB Congeners (Sediment) Measurement Performance Criteria

Matrix: Sediment
Analytical Group or Method: PCB Congeners / USEPA Method 1668C/Lab SOP MLA-010
Concentration Level: Low

Data Quality Indicator (DQI)	QC Sample or Measurement Performance Activity	Measurement Performance Criteria [1]
Representativeness	Preservation	see Worksheet #19
Sensitivity	Method Blank	< QL
Accuracy	Surrogates	Recoveries within SGS AXYS SOP MLA-010 specified control limits
Accuracy	Laboratory Control Sample (LCS)	Recoveries within SGS AXYS SOP MLA-010 specified control limits
Precision	Laboratory Replicate	RPD ≤ 40% if reported values are ≥ 5x QL; otherwise use professional judgement
Accuracy	Certified Reference Material (CRM) (NIST 1944)	Reported concentrations within ±30% of certified and reference values. Applicable for values that are ≥ 3x concentration of the lowest calibration point in the ICAL and only applicable for non-coeluting congeners.
Precision & Representativeness	Field Duplicate	RPD ≤ 50% if reported values are ≥ 5x QL; otherwise use professional judgement
Sensitivity	Equipment Blank	< QL
Representativeness	Temperature Blank	0 -6 degrees Celsius
Completeness	Data Completeness Check	>90% sample collection and >90% laboratory analysis
Comparability/Sensitivity	Data Quality Assessment	Comparable units, QL, and methods; detection limits meet project goals

1. The assigned laboratory must perform and meet all of the measurement performance criteria that assess the analytical DQIs specified in their SOPs and the analytical methods specified.
2. An equipment blank will be generated in the field by rinsing field equipment with laboratory-grade water.

QAPP Worksheet 12-3
TAL Metals + Tin (Sediment) Measurement Performance Criteria

Matrix: Sediment
Analytical Group or Method: TAL Metals + Tin / SW-846 Methods 6010D & 6020A
Concentration Level: Low

Data Quality Indicator (DQI)	QC Sample or Measurement Performance Activity	Measurement Performance Criteria [1]
Representativeness	Preservation	see Worksheet #19
Sensitivity	Method Blank	< QL
Accuracy	Laboratory Control Sample (LCS)	vendor or lab control limits
Accuracy	Matrix Spike (MS)	Recoveries within 75-125% unless sample conc. > 4x spike level (spike swamped out)
Precision	Laboratory Replicate	RPD ≤ 25% if results ≥5xQL. Results <5xQL: absolute value of difference < ±2xQL
Precision & Representativeness	Field Duplicate	RPD ≤ 50% if reported values are ≥ 5x QL; otherwise use professional judgement
Sensitivity	Equipment Blank	< QL
Representativeness	Temperature Blank	0 -6 degrees Celsius
Completeness	Data Completeness Check	>90% sample collection and >90% laboratory analysis
Comparability/Sensitivity	Data Quality Assessment	Comparable units, QL, and methods; detection limits meet project goals

1. The assigned laboratory must perform and meet all of the measurement performance criteria that assess the analytical DQIs specified in their SOPs and the analytical methods specified.
2. An equipment blank will be generated in the field by rinsing field equipment with laboratory-grade water.

QAPP Worksheet 12-4
AVS/SEM (Sediment) Measurement Performance Criteria

Matrix: Sediment
Analytical Group or Method: Acid Volatile Sulfides & Simultaneously Extracted Metals (AVS/SEM) / USEPA Draft Method 821-R-91-100 & SW-846 Methods 6010C & 7470A
Concentration Level: Low

Data Quality Indicator (DQI)	QC Sample or Measurement Performance Activity	Measurement Performance Criteria [1]
Representativeness	Preservation	see Worksheet #19
Sensitivity	Method Blank	< QL
Accuracy	Laboratory Control Sample (LCS)	Recoveries within 80-120%
Accuracy	Matrix Spike (MS)	Recoveries within 75-125% unless sample conc. > 4x spike level (spike swamped out)
Precision	Laboratory Replicate	RPD ≤ 25% if results ≥5xQL. Results <5xQL: absolute value of difference < ±2xQL
Precision & Representativeness	Field Duplicate	RPD ≤ 50% if reported values are ≥ 5x QL; otherwise use professional judgement
Sensitivity	Equipment Blank	< QL
Representativeness	Temperature Blank	0 -6 degrees Celsius
Completeness	Data Completeness Check	>90% sample collection and >90% laboratory analysis
Comparability/Sensitivity	Data Quality Assessment	Comparable units, QL, and methods; detection limits meet project goals

1. The assigned laboratory must perform and meet all of the measurement performance criteria that assess the analytical DQIs specified in their SOPs and the analytical methods specified.
2. An equipment blank will be generated in the field by rinsing field equipment with laboratory-grade water.

QAPP Worksheet 12-5
TOC (Sediment) Measurement Performance Criteria

Matrix: Sediment
Analytical Group or Method: Total Organic Carbon (TOC) / EPA Region II Lloyd Kahn method
Concentration Level: Low

Data Quality Indicator (DQI)	QC Sample or Measurement Performance Activity	Measurement Performance Criteria [1]
Representativeness	Preservation	see Worksheet #19
Sensitivity	Method Blank	< QL
Accuracy & Precision	Laboratory Control Sample (LCS)	Recoveries within 80-120%
Accuracy	Certified Reference Material (CRM) (NIST 1944)	Reported concentration within ± 30% of certified TOC value
Accuracy	Matrix Spike (MS)	Recoveries within 75-125% unless sample conc. > 4x spike level (spike swamped out)
Precision	Laboratory Quadruplicate	%RSD ≤ 30%
Precision & Representativeness	Field Duplicate	RPD ≤ 50% if reported values are ≥ 5x QL; otherwise use professional judgement
Sensitivity	Equipment Blank	< QL
Representativeness	Temperature Blank	0 -6 degrees Celsius
Completeness	Data Completeness Check	>90% sample collection and >90% laboratory analysis
Comparability/Sensitivity	Data Quality Assessment	Comparable units, QL, and methods; detection limits meet project goals

1. The assigned laboratory must perform and meet all of the measurement performance criteria that assess the analytical DQIs specified in their SOPs and the analytical methods specified.
2. An equipment blank will be generated in the field by rinsing field equipment with laboratory-grade water.

QAPP Worksheet 12-6
Grain Size (Sediment) Measurement Performance Criteria

Matrix: Sediment
Analytical Group or Method: Grain Size Distribution / ASTM D422-63
Concentration Level: Low

Data Quality Indicator (DQI)	QC Sample or Measurement Performance Activity	Measurement Performance Criteria [1]
Precision	Laboratory Replicate	RPD ≤ 40%
Precision & Representativeness	Field Duplicate	RPD ≤ 50%
Completeness	Data Completeness Check	>90% sample collection and >90% laboratory analysis
Comparability/Sensitivity	Data Quality Assessment	Comparable units, QL, and methods; detection limits meet project goals

1. The assigned laboratory must perform and meet all of the measurement performance criteria that assess the analytical DQIs specified in their SOPs and the analytical methods specified.

QAPP Worksheet 12-7
PAH and Alkylated PAH (Suspended Particulates) Measurement Performance Criteria

Matrix: Suspended Particulate Material on Filter
Analytical Group or Method: PAH and Alkylated PAH / Modified SW-846 Method 8270D/ Lab SOP MLA-021
Concentration Level: Low

Data Quality Indicator (DQI)	QC Sample or Measurement Performance Activity	Measurement Performance Criteria [1]
Representativeness	Preservation	see Worksheet #19
Sensitivity	Method Blank	< QL
Accuracy	Surrogates	Recoveries within SGS AXYS SOP MLA-021 specified control limits
Accuracy & Precision	Laboratory Control Sample (LCS) / Laboratory Control Spike Duplicate (LCSD)	Recoveries within SGS AXYS SOP MLA-021 specified control limits RPD ≤ 40%
Accuracy	Certified Reference Material (CRM) (NIST 1944)	Reported concentrations within ±30% of certified and reference values. Applicable for values that are ≥ 3x concentration of the lowest calibration point in the ICAL.
Precision & Representativeness	Field Duplicate	RPD ≤ 50% if reported values are ≥ 5x QL; otherwise use professional judgement
Sensitivity	Equipment Blank	< QL
Representativeness	Temperature Blank	0 -6 degrees Celsius
Completeness	Data Completeness Check	>90% sample collection and >90% laboratory analysis
Comparability/Sensitivity	Data Quality Assessment	Comparable units, QL, and methods; detection limits meet project goals

1. The assigned laboratory must perform and meet all of the measurement performance criteria that assess the analytical DQIs specified in their SOPs and the analytical methods specified.
2. An equipment blank will be generated in the field by rinsing field equipment with laboratory-grade water.
3. Method modifications are listed in QAPP WS #23.

QAPP Worksheet 12-8
PCB Congeners (Suspended Particulates) Measurement Performance Criteria

Matrix: Suspended Particulate Material on Filter
Analytical Group or Method: PCB Congeners / USEPA Method 1668C/Lab SOP MLA-010
Concentration Level: Low

Data Quality Indicator (DQI)	QC Sample or Measurement Performance Activity	Measurement Performance Criteria [1]
Representativeness	Preservation	see Worksheet #19
Sensitivity	Method Blank	< QL
Accuracy	Surrogates	Recoveries within SGS AXYS SOP MLA-010 specified control limits
Accuracy & Precision	Laboratory Control Sample (LCS) / Laboratory Control Spike Duplicate (LCSD)	Recoveries within SGS AXYS SOP MLA-010 specified control limits RPD ≤ 40%
Accuracy	Certified Reference Material (CRM) (NIST 1944)	Reported concentrations within ±30% of certified and reference values. Applicable for values that are ≥ 3x concentration of the lowest calibration point in the ICAL and only applicable for non-coeluting congeners.
Precision & Representativeness	Field Duplicate	RPD ≤ 50% if reported values are ≥ 5x QL; otherwise use professional judgement
Sensitivity	Equipment Blank	< QL
Representativeness	Temperature Blank	0 -6 degrees Celsius
Completeness	Data Completeness Check	>90% sample collection and >90% laboratory analysis
Comparability/Sensitivity	Data Quality Assessment	Comparable units, QL, and methods; detection limits meet project goals

1. The assigned laboratory must perform and meet all of the measurement performance criteria that assess the analytical DQIs specified in their SOPs and the analytical methods specified.
2. An equipment blank will be generated in the field by rinsing field equipment with laboratory-grade water.

QAPP Worksheet 12-9
TAL Metals (Whole Water & Dissolved Water) Measurement Performance Criteria

Matrix: Whole Water & Dissolved Surface Water
Analytical Group or Method: TAL Metals + Tin / SW-846 Methods 6010D & 6020A
Concentration Level: Low

Data Quality Indicator (DQI)	QC Sample or Measurement Performance Activity	Measurement Performance Criteria [1]
Representativeness	Preservation	see Worksheet #19
Sensitivity	Method Blank	< QL
Sensitivity	Filter Blank (for Dissolved phase only)	< QL
Accuracy	Laboratory Control Sample (LCS)	Recoveries within 80-120% or vendor certified limits if SRM used as LCS
Accuracy	Matrix Spike (MS)	Recoveries within 75-125% unless sample conc. > 4x spike level (spike swamped out)
Precision	Laboratory Replicate or Matrix Spike Duplicate (MSD)	MSD %Rec 75-125% (unless swamped out) RPD ≤ 25% if results ≥5xQL. Results <5xQL: absolute value of difference < ±QL
Precision & Representativeness	Field Duplicate	RPD ≤ 40% if reported values are ≥ 5x QL; otherwise use professional judgement
Sensitivity	Equipment Blank	< QL
Representativeness	Temperature Blank	0 -6 degrees Celsius
Completeness	Data Completeness Check	>90% sample collection and >90% laboratory analysis
Comparability/Sensitivity	Data Quality Assessment	Comparable units, QL, and methods; detection limits meet project goals

1. The assigned laboratory must perform and meet all of the measurement performance criteria that assess the analytical DQIs specified in their SOPs and the analytical methods specified.
2. An equipment blank will be generated in the field by rinsing field equipment with laboratory-grade water.
3. Either a Laboratory Replicate or a Matrix Spike Duplicate (MSD) can be performed per analytical batch.

QAPP Worksheet 12-10
TSS (Whole Water) Measurement Performance Criteria

Matrix: Suspended Particulate Material on Filter
Analytical Group or Method: Total Suspended Solids / EPA 160.2/SM 2540D-modified
Concentration Level: Low

Data Quality Indicator (DQI)	QC Sample or Measurement Performance Activity	Measurement Performance Criteria [1]
Representativeness	Preservation	see Worksheet #19
Sensitivity	Method Blank (includes a filter)	< QL
Accuracy & Precision	Laboratory Control Sample (LCS) & Laboratory Control Sample Duplicate (LCSD)	Recoveries within 75-125% RPD ≤ 30%
Precision & Representativeness	Field Duplicate	RPD ≤ 40% if reported values are ≥ 5x QL; otherwise use professional judgement
Sensitivity	Equipment Blank	< QL
Representativeness	Temperature Blank	0 -6 degrees Celsius
Completeness	Data Completeness Check	>90% sample collection and >90% laboratory analysis
Comparability/Sensitivity	Data Quality Assessment	Comparable units, QL, and methods; detection limits meet project goals

1. The assigned laboratory must perform and meet all of the measurement performance criteria that assess the analytical DQIs specified in their SOPs and the analytical methods specified.
2. An equipment blank will be generated in the field by rinsing field equipment with laboratory-grade water.
3. Method modifications are listed in QAPP WS #23.
4. LCS/LCSD will be analyzed as measure of accuracy and precision. Laboratory replicate cannot be performed because the laboratory must use the entire 4-liter sample volume in the TSS analysis as a QAPP modification of this method.
5. Laboratory blank includes a filter as part of the standard method; consequently, a separate filter blank is not required for this parameter.

QAPP Worksheet 12-11
POC (Suspended Particulates) Measurement Performance Criteria

Matrix: Suspended Particulate Material on Filter
Analytical Group or Method: Particulate Organic Carbon / EPA Region II Lloyd Kahn-modified
Concentration Level: Low

Data Quality Indicator (DQI)	QC Sample or Measurement Performance Activity	Measurement Performance Criteria [1]
Representativeness	Preservation	see Worksheet #19
Sensitivity	Method Blank	< QL
Sensitivity	Filter Blank	< QL
Accuracy & Precision	Laboratory Control Sample (LCS)	Recoveries within 75-125%
Accuracy	Certified Reference Material (CRM) (ERA "Nutrients in Soil" Cat. # 542)	Reported concentration within ± 30% of certified value
Accuracy	Matrix Spike (MS)	Recoveries within 75-125% unless sample conc. > 4x spike level (spike swamped out)
Precision	Matrix Spike Duplicate (MSD)	MSD %Rec 75-125% unless sample conc. > 4x spike level (spike swamped out) RPD ≤ 30% if results ≥5xQL; otherwise, otherwise absolute difference between values < ±QL
Precision	Laboratory Replicate	RPD ≤ 30% and report the individual POC results and the average of the replicates
Precision & Representativeness	Field Duplicate	RPD ≤ 40% if reported values are ≥ 5x QL; otherwise use professional judgement
Sensitivity	Equipment Blank	< QL
Representativeness	Temperature Blank	0 -6 degrees Celsius
Completeness	Data Completeness Check	>90% sample collection and >90% laboratory analysis
Comparability/Sensitivity	Data Quality Assessment	Comparable units, QL, and methods; detection limits meet project goals

1. The assigned laboratory must perform and meet all of the measurement performance criteria that assess the analytical DQIs specified in their SOPs and the analytical methods specified.

2. An equipment blank will be generated in the field by rinsing field equipment with laboratory-grade water.
3. Method modifications are listed in QAPP WS #23.

QAPP Worksheet 12-12
Grain Size (Aqueous) Measurement Performance Criteria

Matrix: Aqueous
Analytical Group or Method: Grain Size Distribution / Laser Particle Size Distribution
Concentration Level: Low

Data Quality Indicator (DQI)	QC Sample or Measurement Performance Activity	Measurement Performance Criteria [1]
Accuracy	Laboratory Control Sample (LCS)	Per SedTrend SOP for acceptance limits on accuracy of the distribution
Precision	Laboratory Replicate	RPD ≤ 40%
Precision & Representativeness	Field Duplicate	RPD ≤ 50%
Completeness	Data Completeness Check	>90% sample collection and >90% laboratory analysis
Comparability/Sensitivity	Data Quality Assessment	Comparable units, QL, and methods; detection limits meet project goals

1. The assigned laboratory must perform and meet all of the measurement performance criteria that assess the analytical DQIs specified in their SOPs and the analytical methods specified.

QAPP Worksheet 13
Secondary Data Uses and Limitations

Secondary Data Uses and Limitations

Data Type	Data Source (Originating Organization, Report Title, and Date)	How Data Will Be Used (Data uses relative to current project)	Limitations on Data Use (Factors affecting the reliability of data and limitations on data use)
Surface water data	USEPA Gowanus Canal Remedial Investigation Report (2011)	Data will be used to develop solids loading from the Gowanus bay and Buttermilk Channel and other non-CSO sources.	None (beyond the analytical uncertainty of the data).
Surface water Data	NYCDEP Data Collected as part of responses to PRAP	Data will be used to develop solids loading from the Gowanus bay and Buttermilk Channel and other non-CSO sources.	None (beyond the analytical uncertainty of the data).

QAPP Worksheet 14 and Worksheet 16
Project Tasks & Schedule

Activity	Responsible Organization	Planned Start Date	Planned Completion Date	Deliverable	Deliverable Due Date
Submit draft QAPP to NYCDEP	WSP	October 11, 2021	October 15, 2021	QAPP (draft)	October 15, 2021
Comments on draft QAPP	NYCDEP	October 15, 2021	October 22, 2021	Comments on QAPP	October 22, 2021
Submit draft QAPP to NYCDEP	WSP	October 22, 2021	October 29, 2021	QAPP (draft)	October 29, 2021
Approval of draft QAPP	NYCDEP	November 1, 2021	November 1, 2021	QAPP (draft)	November 1, 2021
Submit draft QAPP to USEPA	WSP/NYCDEP	November 1, 2021	November 2, 2021	QAPP (draft)	November 2, 2021
Comments on draft QAPP	USEPA	November 2, 2021	November 16, 2021	Comments on QAPP	November 16, 2021
Submit final QAPP to USEPA	WSP/NYCDEP	November 16, 2021	November 22, 2021	QAPP (final)	November 22, 2021
Approval of final QAPP	USEPA	November 22, 2021	December 3, 2021	QAPP (final)	December 3, 2021
Conduct field programs and samples collection	The selected contractor under a new contract with NYCDEP	June 2022	June 2024	Validated data collected as part of the sampling program.	Intermediately after June 2022 as data gets validated.
Analysis of sediment samples	The selected contractor under a new contract with NYCDEP	To be determined	To be determined	Unvalidated samples database	To be determined
Analysis of CSO samples	The selected contractor under a new contract with NYCDEP	June 2022 through June 2024	June 2022 through June 2024	Unvalidated samples database	To be determined
Validation of samples	The selected contractor under a new contract with NYCDEP	To be determined	To be determined	Validated samples database	To be determined
Evaluate field data and prepare memoranda	The selected contractor under a new contract with NYCDEP	To be determined	To be determined	Draft and Final memoranda	To be determined

QAPP Worksheet 15-1
PAH and Alkylated PAH (Sediment) Reference Limits and Evaluation Tables
Matrix: Sediment
Analytical Group: PAH and Alkylated PAH (Modified SW846 Method 8270)
Concentration Level: Low

Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
					EDL	LOQ
1-Methylchrysene	3351-28-8	ng/g dw	Not available	PQL = Lab LOQ	1.0	2.50
1-Methylnaphthalene	90-12-0	ng/g dw	Not available	PQL = Lab LOQ	1.0	2.50
1-Methylphenanthrene	832-69-9	ng/g dw	Not available	PQL = Lab LOQ	1.0	3.00
1,2-Dimethylnaphthalene	573-98-8	ng/g dw	Not available	PQL = Lab LOQ	1.0	2.50
1,2,6-Trimethylphenanthrene	30436-55-6	ng/g dw	Not available	PQL = Lab LOQ	1.0	2.92
1,4,6,7-Tetramethylnaphthalene	13764-18-6	ng/g dw	Not available	PQL = Lab LOQ	1.0	2.50
1,7-Dimethylphenanthrene	483-87-4	ng/g dw	Not available	PQL = Lab LOQ	1.0	2.50
1,7-Dimethylfluorene	442-66-0	ng/g dw	Not available	PQL = Lab LOQ	1.0	2.50
1,8-Dimethylphenanthrene	7372-87-4	ng/g dw	Not available	Not applicable	1.0	Not applicable
2-Methylanthracene	613-12-7	ng/g dw	Not available	PQL = Lab LOQ	1.0	2.50
2-Methylfluorene	2361-84-2	ng/g dw	Not available	PQL = Lab LOQ	1.0	2.50
2-Methylnaphthalene	91-57-6	ng/g dw	Not available	PQL = Lab LOQ	1.0	2.99
2-Methylphenanthrene	2531-84-2	ng/g dw	Not available	PQL = Lab LOQ	1.0	2.50
2,3,5-Trimethylnaphthalene	2245-38-7	ng/g dw	Not available	PQL = Lab LOQ	1.0	3.00
2,3,6-Trimethylnaphthalene	829-26-5	ng/g dw	Not available	PQL = Lab LOQ	1.0	2.50
2,4-Dimethyldibenzothiophene	31317-18-7	ng/g dw	Not available	Not applicable	1.0	Not applicable
2,6-Dimethylnaphthalene	581-42-0	ng/g dw	Not available	PQL = Lab LOQ	1.0	3.01
2,6-Dimethylphenanthrene	17980-16-4	ng/g dw	Not available	Not applicable	1.0	Not applicable
3-Methylfluoranthene/Benzo[a]fluorene	238-84-6	ng/g dw	Not available	PQL = Lab LOQ	1.0	2.50
3-Methylphenanthrene	832-71-3	ng/g dw	Not available	Not applicable	1.0	Not applicable
3,6-Dimethylphenanthrene	1576-67-6	ng/g dw	Not available	PQL = Lab LOQ	1.0	2.50
5,9-Dimethylchrysene	139493-40-6	ng/g dw	Not available	PQL = Lab LOQ	1.0	2.50
7-Methylbenzo[a]pyrene	63041-77-0	ng/g dw	Not available	PQL = Lab LOQ	1.0	2.50
2/3-Methyldibenzothiophenes	23MDBTHPH	ng/g dw	Not available	PQL = Lab LOQ	1.0	2.50
5/6-Methylchrysenes	56METHCHRY	ng/g dw	Not available	PQL = Lab LOQ	1.0	2.50
9/4-Methylphenanthrenes	883-20-5/832-64-4	ng/g dw	Not available	Not applicable	1.0	Not applicable

Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
					EDL	LOQ
Acenaphthene	83-32-9	ng/g dw	Not available	PQL = Lab LOQ	0.5	3.01
Acenaphthylene	208-96-8	ng/g dw	Not available	PQL = Lab LOQ	0.5	2.95
Anthracene	120-12-7	ng/g dw	Not available	PQL = Lab LOQ	0.5	3.01
Fluorene	86-73-7	ng/g dw	Not available	PQL = Lab LOQ	0.5	2.95
Naphthalene	91-20-3	ng/g dw	Not available	PQL = Lab LOQ	0.5	2.96
Phenanthrene	85-01-8	ng/g dw	Not available	PQL = Lab LOQ	0.5	3.03
Benzo[a]anthracene	56-55-3	ng/g dw	Not available	PQL = Lab LOQ	0.5	3.02
Benzo[a]pyrene	50-32-8	ng/g dw	Not available	PQL = Lab LOQ	0.5	3.00
Benzo[b]fluoranthene	205-99-2	ng/g dw	Not available	PQL = Lab LOQ	0.5	3.02
Benzo[e]pyrene	192-97-2	ng/g dw	Not available	PQL = Lab LOQ	0.5	3.00
Benzo[g,h,i]perylene	191-24-2	ng/g dw	Not available	PQL = Lab LOQ	1	2.96
Benzo[j,k]fluoranthene	207-08-9	ng/g dw	Not available	PQL = Lab LOQ	0.5	3.02
Chrysene	218-01-9	ng/g dw	Not available	PQL = Lab LOQ	0.5	3.01
Dibenzo[a,h]anthracene	53-70-3	ng/g dw	Not available	PQL = Lab LOQ	1.0	2.96
Dibenzothiophene	132-65-0	ng/g dw	Not available	PQL = Lab LOQ	1.0	3.00
Fluoranthene	206-44-0	ng/g dw	Not available	PQL = Lab LOQ	0.5	2.97
Indeno[1,2,3-cd]pyrene	193-39-5	ng/g dw	Not available	PQL = Lab LOQ	1.0	3.02
Perylene	198-55-0	ng/g dw	Not available	PQL = Lab LOQ	1.0	3.00
Pyrene	129-00-0	ng/g dw	Not available	PQL = Lab LOQ	0.5	2.97
Biphenyl	92-52-4	ng/g dw	Not available	PQL = Lab LOQ	1.0	2.50
Retene	483-65-9	ng/g dw	Not available	PQL = Lab LOQ	1.0	2.50
C1-Acenaphthenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	0.5	Not available
C1-Benzo[a]anthracenes/Chrysenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C1-Benzofluoranthenes/Benzopyrenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C1-Biphenyls	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C1-Dibenzothiophenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C1-Fluorenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C1-Naphthalenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C1-Phenanthrenes/Anthracenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C1-Fluoranthenes/Pyrenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C2-Benzo[a]anthracenes/Chrysenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available

Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
					EDL	LOQ
C2-Benzofluoranthenes/Benzopyrenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C2-Biphenyls	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C2-Dibenzothiophenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C2-Fluoranthenes/Pyrenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C2-Fluorenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C2-Naphthalenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C2-Phenanthrenes/Anthracenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C3-Benzo[a]anthracenes/Chrysenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C3-Dibenzothiophenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C3-Fluoranthenes/Pyrenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C3-Fluorenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C3-Naphthalenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C3-Phenanthrenes/Anthracenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C4-Benzo[a]anthracenes/Chrysenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C4-Dibenzothiophenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C4-Fluoranthenes/Pyrenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C4-Naphthalenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available
C4-Phenanthrenes/Anthracenes	Not available	ng/g dw	Not available	PQL = Lab LOQ	1.0	Not available

Notes:
1. Project Action Levels for PAHs and Alkylated PAHs have not been established yet for this project.
2. For this project, Project QL has been set at the achievable laboratory level of quantitation (LOQ).
3. Laboratory EDLs "Estimated Detection Limits" represent typical detection limits that can be obtained by SGS Axys Analytical Services. EDLs are based on the smallest peak that can meet all quantification criteria and has a signal-to-noise (S:N) ratio of 3:1. EDLs were based on the 10-gram (dry weight) sample size with a final extract volume of 500 µL. Sample-specific EDLs will depend on the final extract volume (which may vary depending on matrix interference), dilution factors, and the final field sample size.
4. LOQs represent typical quantitation limits that can be obtained by SGS Axys Analytical Services. The LOQ is supported by the standards in the calibration curve. LOQs were based on the 10-gram (dry weight) sample size with a final extract volume of 500 µL. Sample-specific LOQs reported by the laboratory will be dependent on the final extract volume (which may vary depending on matrix interference), dilution factors, and the final field sample size.
5. Non-detects will be reported at the sample-specific EDLs. Data will be estimated (qualified "J") between the sample-specific EDL and sample-specific LOQ.
6. Units represent mass of contaminant per mass of sediment (dry weight basis).

QAPP Worksheet 15-2
PCB Congeners (Sediment) Reference Limits and Evaluation Tables
Matrix: Sediment
Analytical Group: PCB Congeners (USEPA Method 1668 Version C)
Concentration Level: Low

Congener Number	Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
						EDL	LOQ
PCB 1	2-MoCB	2051-60-7	pg/g dw	Not available	PQL = Lab LOQ	0.1	5
PCB 2	3-MoCB	2051-61-8	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 3	4-MoCB	2051-62-9	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 4	2,2'-DiCB	13029-08-8	pg/g dw	Not available	PQL = Lab LOQ	0.2	3
PCB 5	2,3-DiCB	16605-91-7	pg/g dw	Not available	PQL = Lab LOQ	0.2	3
PCB 6	2,3'-DiCB	25569-80-6	pg/g dw	Not available	PQL = Lab LOQ	0.2	3
PCB 7	2,4-DiCB	33284-50-3	pg/g dw	Not available	PQL = Lab LOQ	0.2	3
PCB 8	2,4'-DiCB	34883-43-7	pg/g dw	Not available	PQL = Lab LOQ	0.2	5
PCB 9	2,5-DiCB	34883-39-1	pg/g dw	Not available	PQL = Lab LOQ	0.2	3
PCB 10	2,6-DiCB	33146-45-1	pg/g dw	Not available	PQL = Lab LOQ	0.2	3
PCB 11	3,3'-DiCB	2050-67-1	pg/g dw	Not available	PQL = Lab LOQ	0.2	10
PCB 12 (Coelutes with PCB 13)	3,4-DiCB	2974-92-7	pg/g dw	Not available	PQL = Lab LOQ	0.2	4
PCB 13 (Coelutes with PCB 12)	3,4'-DiCB	2974-90-5	pg/g dw	Not available	PQL = Lab LOQ	C12	C12
PCB 14	3,5-DiCB	34883-41-5	pg/g dw	Not available	PQL = Lab LOQ	0.2	3
PCB 15	4,4'-DiCB	2050-68-2	pg/g dw	Not available	PQL = Lab LOQ	0.2	4
PCB 16	2,2',3-TrCB	38444-78-9	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 17	2,2',4-TrCB	37680-66-3	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 18 (Coelutes with PCB 30)	2,2',5-TrCB	37680-65-2	pg/g dw	Not available	PQL = Lab LOQ	C30	C30
PCB 19	2,2',6-TrCB	38444-73-4	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 20 (Coelutes with PCB 28)	2,3,3'-TrCB	38444-84-7	pg/g dw	Not available	PQL = Lab LOQ	C28	C28
PCB 21 (Coelutes with PCB 33)	2,3,4-TrCB	55702-46-0	pg/g dw	Not available	PQL = Lab LOQ	0.1	3

Congener Number	Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
						EDL	LOQ
PCB 22	2,3,4'-TrCB	38444-85-8	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 23	2,3,5-TrCB	55720-44-0	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 24	2,3,6-TrCB	55702-45-9	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 25	2,3',4-TrCB	55712-37-3	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 26 (Coelutes with PCB 29)	2,3',5-TrCB	38444-81-4	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 27	2,3',6-TrCB	38444-76-7	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 28 (Coelutes with PCB 20)	2,4,4'-TrCB	7012-37-5	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 29 (Coelutes with PCB 26)	2,4,5-TrCB	15862-07-4	pg/g dw	Not available	PQL = Lab LOQ	C26	C26
PCB 30 (Coelutes with PCB 18)	2,4,6-TrCB	35693-92-6	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 31	2,4',5-TrCB	16606-02-3	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 32	2,4',6-TrCB	38444-77-8	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 33 (Coelutes with PCB 21)	2',3,4-TrCB	38444-86-9	pg/g dw	Not available	PQL = Lab LOQ	C21	C21
PCB 34	2',3,5-TrCB	37680-68-5	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 35	3,3',4-TrCB	37680-69-6	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 36	3,3',5-TrCB	38444-87-0	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 37	3,4,4'-TrCB	38444-90-5	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 38	3,4,5-TrCB	53555-66-1	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 39	3,4',5-TrCB	38444-88-1	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 40 (Coelutes with PCB 41 & 71)	2,2',3,3'-TeCB	38444-93-8	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 41 (Coelutes with PCB 40 & 71)	2,2',3,4-TeCB	52663-59-9	pg/g dw	Not available	PQL = Lab LOQ	C40	C40
PCB 42	2,2',3,4'-TeCB	36559-22-5	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 43	2,2',3,5-TeCB	70362-46-8	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 44 (Coelutes with PCB 47 & 65)	2,2',3,5'-TeCB	41464-39-5	pg/g dw	Not available	PQL = Lab LOQ	0.1	3

Congener Number	Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
						EDL	LOQ
PCB 45 (Coelutes with PCB 51)	2,2',3,6-TeCB	70362-45-7	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 46	2,2',3,6'-TeCB	41464-47-5	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 47 (Coelutes with PCB 44 & 65)	2,2',4,4'-TeCB	2437-79-8	pg/g dw	Not available	PQL = Lab LOQ	C44	C44
PCB 48	2,2',4,5-TeCB	70362-47-9	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 49 (Coelutes with PCB 69)	2,2',4,5'-TeCB	41464-40-8	pg/g dw	Not available	PQL = Lab LOQ	C69	C69
PCB 50 (Coelutes with PCB 53)	2,2',4,6-TeCB	62796-65-0	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 51 (Coelutes with PCB 45)	2,2',4,6'-TeCB	68194-04-7	pg/g dw	Not available	PQL = Lab LOQ	C45	C45
PCB 52	2,2',5,5'-TeCB	35693-99-3	pg/g dw	Not available	PQL = Lab LOQ	0.1	4
PCB 53 (Coelutes with PCB 50)	2,2',5,6'-TeCB	41464-41-9	pg/g dw	Not available	PQL = Lab LOQ	C50	C50
PCB 54	2,2',6,6'-TeCB	15968-05-5	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 55	2,3,3',4'-TeCB	74338-24-2	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 56	2,3,3',4'-TeCB	41464-43-1	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 57	2,3,3',5-TeCB	70424-67-8	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 58	2,3,3',5'-TeCB	41464-49-7	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 59 (Coelutes with PCB 62 & 75)	2,3,3',6-TeCB	74472-33-6	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 60	2,3,4,4'-TeCB	33025-41-1	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 61 (Coelutes with PCB 70, 74, & 76)	2,3,4,5-TeCB	33284-53-6	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 62 (Coelutes with PCB 59 & 75)	2,3,4,6-TeCB	54230-22-7	pg/g dw	Not available	PQL = Lab LOQ	C59	C59
PCB 63	2,3,4',5-TeCB	74472-34-7	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 64	2,3,4',6-TeCB	52663-58-8	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 65 (Coelutes with PCB 44 & 47)	2,3,5,6-TeCB	33284-54-7	pg/g dw	Not available	PQL = Lab LOQ	C44	C44

Congener Number	Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
						EDL	LOQ
PCB 66	2,3',4,4'-TeCB	32598-10-0	pg/g dw	Not available	PQL = Lab LOQ	0.1	4
PCB 67	2,3',4,5-TeCB	73575-53-8	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 68	2,3',4,5'-TeCB	73575-52-7	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 69 (Coelutes with PCB 49)	2,3',4,6-TeCB	60233-24-1	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 70 (Coelutes with PCB 61, 74, & 76)	2,3',4',5-TeCB	32598-11-1	pg/g dw	Not available	PQL = Lab LOQ	C61	C61
PCB 71 (Coelutes with PCB 40 & 41)	2,3',4',6-TeCB	41464-46-4	pg/g dw	Not available	PQL = Lab LOQ	C40	C40
PCB 72	2,3',5,5'-TeCB	41464-42-0	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 73	2,3',5',6-TeCB	74338-23-1	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 74 (Coelutes with PCB 61, 70, & 76)	2,4,4',5-TeCB	32690-93-0	pg/g dw	Not available	PQL = Lab LOQ	C61	C61
PCB 75 (Coelutes with PCB 59 & 62)	2,4,4',6-TeCB	32598-12-2	pg/g dw	Not available	PQL = Lab LOQ	C59	C59
PCB 76 (Coelutes with PCB 61, 70, & 74)	2',3,4,5-TeCB	70362-48-0	pg/g dw	Not available	PQL = Lab LOQ	C61	C61
PCB 77	3,3',4,4'-TeCB	32598-13-3	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 78	3,3',4,5-TeCB	70362-49-1	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 79	3,3',4,5'-TeCB	41464-48-6	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 80	3,3',5,5'-TeCB	33284-52-5	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 81	3,4,4',5-TeCB3	70362-50-4	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 82	2,2',3,3',4-PeCB	52663-62-4	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 83 (Coelutes with PCB 99)	2,2',3,3',5-PeCB	60145-20-2	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 84	2,2',3,3',6-PeCB	52663-60-2	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 85 (Coelutes with PCB 116 & 117)	2,2',3,4,4'-PeCB	65510-45-4	pg/g dw	Not available	PQL = Lab LOQ	C117	C117
PCB 86 (Coelutes with PCB 87, 97, 109, 119, & 125)	2,2',3,4,5-PeCB	55312-69-1	pg/g dw	Not available	PQL = Lab LOQ	C109	C109
PCB 87 (Coelutes with PCB	2,2',3,4,5'-PeCB	38380-02-8	pg/g dw	Not available	PQL = Lab LOQ	C109	C109

Congener Number	Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
						EDL	LOQ
86, 97, 109, 119, & 125)							
PCB 88 (Coelutes with PCB 91)	2,2',3,4,6-PeCB	55215-17-3	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 89	2,2',3,4,6'-PeCB	73575-57-2	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 90 (Coelutes with PCB 101 & 113)	2,2',3,4',5-PeCB	68194-07-0	pg/g dw	Not available	PQL = Lab LOQ	C113	C113
PCB 91 (Coelutes with PCB 88)	2,2',3,4',6-PeCB	68194-05-8	pg/g dw	Not available	PQL = Lab LOQ	C88	C88
PCB 92	2,2',3,5,5'-PeCB	52663-61-3	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 93 (Coelutes with PCB 95, 98, 100, & 102)	2,2',3,5,6-PeCB	73575-56-1	pg/g dw	Not available	PQL = Lab LOQ	C95	C95
PCB 94	2,2',3,5,6'-PeCB	73575-55-0	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 95 (Coelutes with PCB 93, 98, 100, & 102)	2,2',3,5',6-PeCB	38379-99-6	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 96	2,2',3,6,6'-PeCB	73575-54-9	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 97 (Coelutes with PCB 86, 87, 109, 119, & 125)	2,2',3',4,5-PeCB	41464-51-1	pg/g dw	Not available	PQL = Lab LOQ	C109	C109
PCB 98 (Coelutes with PCB 93, 95, 100, & 102)	2,2',3',4,6-PeCB	60233-25-2	pg/g dw	Not available	PQL = Lab LOQ	C95	C95
PCB 99 (Coelutes with PCB 83)	2,2',4,4',5-PeCB	38380-01-7	pg/g dw	Not available	PQL = Lab LOQ	C83	C83
PCB 100 (Coelutes with PCB 93, 95, 98, & 102)	2,2',4,4',6-PeCB	39485-83-1	pg/g dw	Not available	PQL = Lab LOQ	C95	C95
PCB 101 (Coelutes with PCB 90 & 113)	2,2',4,5,5'-PeCB	37680-73-2	pg/g dw	Not available	PQL = Lab LOQ	C113	C113
PCB 102 (Coelutes with PCB 93, 95, 98, & 100)	2,2',4,5,6'-PeCB	68194-06-9	pg/g dw	Not available	PQL = Lab LOQ	C95	C95
PCB 103	2,2',4,5',6-PeCB	60145-21-3	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 104	2,2',4,6,6'-PeCB	56558-16-8	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 105	2,3,3',4,4'-PeCB	32598-14-4	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 106	2,3,3',4,5-PeCB	70424-69-0	pg/g dw	Not available	PQL = Lab LOQ	0.1	3

Congener Number	Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
						EDL	LOQ
PCB 107	2,3,3',4',5-PeCB	70424-68-9	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 108 (Coelutes with PCB 124)	2,3,3',4,5'-PeCB	70362-41-3	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 109 (Coelutes with PCB 86, 87, 97, 119, & 125)	2,3,3',4,6-PeCB	74472-35-8	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 110 (Coelutes with PCB 115)	2,3,3',4',6-PeCB	38380-03-9	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 111	2,3,3',5,5'-PeCB	39635-32-0	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 112	2,3,3',5,6-PeCB	74472-36-9	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 113 (Coelutes with PCB 90 & 101)	2,3,3',5',6-PeCB	68194-10-5	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 114	2,3,4,4',5-PeCB	74472-37-0	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 115 (Coelutes with PCB 110)	2,3,4,4',6-PeCB	74472-38-1	pg/g dw	Not available	PQL = Lab LOQ	C110	C110
PCB 116 (Coelutes with PCB 85 & 117)	2,3,4,5,6-PeCB	18259-05-7	pg/g dw	Not available	PQL = Lab LOQ	C117	C117
PCB 117 (Coelutes with PCB 85 & 116)	2,3,4',5,6-PeCB	68194-11-6	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 118	2,3',4,4',5-PeCB	31508-00-6	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 119 (Coelutes with PCB 86, 87, 97, 109, & 125)	2,3',4,4',6-PeCB	56558-17-9	pg/g dw	Not available	PQL = Lab LOQ	C109	C109
PCB 120	2,3',4,5,5'-PeCB	68194-12-7	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 121	2,3',4,5,'6-PeCB	56558-18-0	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 122	2',3,3',4,5-PeCB	76842-07-4	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 123	2',3,4,4',5-PeCB	65510-44-3	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 124 (Coelutes with PCB 108)	2',3,4,5,5'-PeCB	70424-70-3	pg/g dw	Not available	PQL = Lab LOQ	C108	C108
PCB 125 (Coelutes with PCB 86, 87, 97, 109, &	2',3,4,5,6'-PeCB	74472-39-2	pg/g dw	Not available	PQL = Lab LOQ	C109	C109

Congener Number	Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
						EDL	LOQ
119)							
PCB 126	3,3',4,4',5-PeCB	57465-28-8	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 127	3,3',4,5,5'-PeCB	39635-33-1	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 128 (Coelutes with PCB 166)	2,2',3,3',4,4'-HxCB	38380-07-3	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 129 (Coelutes with PCB 138, 160, & 163)	2,2',3,3',4,5-HxCB	55215-18-4	pg/g dw	Not available	PQL = Lab LOQ	C138	C138
PCB 130	2,2',3,3',4,5'-HxCB	52663-66-8	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 131	2,2',3,3',4,6-HxCB	61798-70-7	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 132	2,2',3,3',4,6'-HxCB	38380-05-1	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 133	2,2',3,3',5,5'-HxCB	35694-04-3	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 134 (Coelutes with PCB 143)	2,2',3,3',5,6-HxCB	52704-70-8	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 135 (Coelutes with PCB 151 & 154)	2,2',3,3',5,6'-HxCB	52744-13-5	pg/g dw	Not available	PQL = Lab LOQ	C151	C151
PCB 136	2,2',3,3',6,6'-HxCB	38411-22-2	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 137	2,2',3,4,4',5-HxCB	35694-06-5	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 138 (Coelutes with PCB 129, 160, & 163)	2,2',3,4,4',5'-HxCB	35065-28-2	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 139 (Coelutes with PCB 140)	2,2',3,4,4',6-HxCB	56030-56-9	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 140 (Coelutes with PCB 139)	2,2',3,4,4',6'-HxCB	59291-64-4	pg/g dw	Not available	PQL = Lab LOQ	C139	C139
PCB 141	2,2',3,4,5,5'-HxCB	52712-04-6	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 142	2,2',3,4,5,6-HxCB	41411-61-4	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 143 (Coelutes with PCB 134)	2,2',3,4,5,6'-HxCB	68194-15-0	pg/g dw	Not available	PQL = Lab LOQ	C134	C134
PCB 144	2,2',3,4,5',6-HxCB	68194-14-9	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 145	2,2',3,4,6,6'-HxCB	74472-40-5	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 146	2,2',3,4',5,5'-HxCB	51908-16-8	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 147 (Coelutes with	2,2',3,4',5,6-HxCB	68194-13-8	pg/g dw	Not available	PQL = Lab LOQ	0.1	3

Congener Number	Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
						EDL	LOQ
PCB 149)							
PCB 148	2,2',3,4',5,6'-HxCB	74472-41-6	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 149 (Coelutes with PCB 147)	2,2',3,4',5',6-HxCB	38380-04-0	pg/g dw	Not available	PQL = Lab LOQ	C147	C147
PCB 150	2,2',3,4',6,6'-HxCB	68194-08-1	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 151 (Coelutes with PCB 135 & 154)	2,2',3,5,5',6-HxCB	52663-63-5	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 152	2,2',3,5,6,6'-HxCB	68194-09-2	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 153 (Coelutes with PCB 168)	2,2',4,4',5,5'-HxCB	35065-27-1	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 154 (Coelutes with PCB 135 & 151)	2,2',4,4',5',6-HxCB	60145-22-4	pg/g dw	Not available	PQL = Lab LOQ	C151	C151
PCB 155	2,2',4,4',6,6'-HxCB	33979-03-2	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 156 (Coelutes with PCB 157)	2,3,3',4,4',5-HxCB	38380-08-4	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 157 (Coelutes with PCB 156)	2,3,3',4,4',5'-HxCB	69782-90-7	pg/g dw	Not available	PQL = Lab LOQ	C156	C156
PCB 158	2,3,3',4,4',6-HxCB	74472-42-7	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 159	2,3,3',4,5,5'-HxCB	39635-35-3	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 160 (Coelutes with PCB 129, 138, & 163)	2,3,3',4,5,6-HxCB	41411-62-5	pg/g dw	Not available	PQL = Lab LOQ	C138	C138
PCB 161	2,3,3',4,5',6-HxCB	74472-43-8	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 162	2,3,3',4',5,5'-HxCB	39635-34-2	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 163 (Coelutes with PCB 129, 138, & 160)	2,3,3',4',5,6-HxCB	74472-44-9	pg/g dw	Not available	PQL = Lab LOQ	C138	C138
PCB 164	2,3,3',4',5',6-HxCB	74472-45-0	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 165	2,3,3',5,5',6-HxCB	74472-46-1	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 166 (Coelutes with PCB 128)	2,3,4,4',5,6-HxCB	41411-63-6	pg/g dw	Not available	PQL = Lab LOQ	C128	C128
PCB 167	2,3',4,4',5,5'-HxCB	52663-72-6	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 168 (Coelutes with	2,3',4,4',5',6-HxCB	59291-65-5	pg/g dw	Not available	PQL = Lab LOQ	C153	C153

Congener Number	Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
						EDL	LOQ
PCB 153)							
PCB 169	3,3',4,4',5,5'-HxCB	32774-16-6	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 170	2,2',3,3',4,4',5-HpCB	35065-30-6	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 171 (Coelutes with PCB 173)	2,2'3,3',4,4',6-HpCB	52663-71-5	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 172	2,2',3,3',4,5,5'-HpCB	52663-74-8	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 173 (Coelutes with PCB 171)	2,2',3,3',4,5,6-HpCB	68194-16-1	pg/g dw	Not available	PQL = Lab LOQ	C173	C173
PCB 174	2,2',3,3',4,5,6'-HpCB	38411-25-5	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 175	2,2',3,3',4,5',6-HpCB	40186-70-7	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 176	2,2',3,3',4,6,6'-HpCB	52663-65-7	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 177	2,2',3,3',4',5,6-HpCB	52663-70-4	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 178	2,2',3,3',5,5',6-HpCB	52663-67-9	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 179	2,2',3,3',5,6,6'-HpCB	52663-64-6	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 180 (Coelutes with PCB 193)	2,2',3,4,4',5,5'-HpCB	35065-29-3	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 181	2,2',3,4,4',5,6-HpCB	74472-47-2	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 182	2,2',3,4,4',5,6'-HpCB	60145-23-5	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 183 (Coelutes with PCB 185)	2,2',3,4,4',5',6-HpCB	52663-69-1	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 184	2,2',3,4,4',6,6'-HpCB	74472-48-3	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 185 (Coelutes with PCB 183)	2,2',3,4,5,5',6-HpCB	52712-05-7	pg/g dw	Not available	PQL = Lab LOQ	C183	C183
PCB 186	2,2',3,4,5,6,6'-HpCB	74472-49-4	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 187	2,2',3,4',5,5',6-HpCB	52663-68-0	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 188	2,2',3,4',5,6,6'-HpCB	74487-85-7	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 189	2,3,3',4,4',5,5'-HpCB	39635-31-9	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 190	2,3,3',4,4',5,6-HpCB	41411-64-7	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 191	2,3,3',4,4',5',6-HpCB	74472-50-7	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 192	2,3,3',4,5,5',6-HpCB	74472-51-8	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 193 (Coelutes with	2,3,3',4',5,5',6-HpCB	69782-91-8	pg/g dw	Not available	PQL = Lab LOQ	C180	C180

Congener Number	Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
						EDL	LOQ
PCB 180)							
PCB 194	2,2',3,3',4,4',5,5'-OcCB	35694-08-7	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 195	2,2',3,3',4,4',5,6-OcCB	52663-78-2	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 196	2,2',3,3',4,4',5,6'-OcCB	42740-50-1	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 197 (Coelutes with PCB 200)	2,2',3,3',4,4',6,6'-OcCB	33091-17-7	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 198 (Coelutes with PCB 199)	2,2',3,3',4,5,5',6-OcCB	68194-17-2	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 199 (Coelutes with PCB 198)	2,2',3,3',4,5,5',6'-OcCB	52663-75-9	pg/g dw	Not available	PQL = Lab LOQ	C198	C198
PCB 200 (Coelutes with PCB 197)	2,2',3,3',4,5,6,6'-OcCB	52663-73-7	pg/g dw	Not available	PQL = Lab LOQ	C197	C197
PCB 201	2,2',3,3',4,5',6,6'-OcCB	40186-71-8	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 202	2,2',3,3',5,5',6,6'-OcCB	2136-99-4	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 203	2,2',3,4,4',5,5',6-OcCB	52663-76-0	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 204	2,2',3,4,4',5,6,6'-OcCB	74472-52-9	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 205	2,3,3',4,4',5,5',6-OcCB	74472-53-0	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 206	2,2',3,3',4,4',5,5',6-NoCB	40186-72-9	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 207	2,2',3,3',4,4',5,6,6'-NoCB	52663-79-3	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 208	2,2',3,3',4,5,5',6,6'-NoCB	52663-77-1	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
PCB 209	Decachlorobiphenyl	2051-24-3	pg/g dw	Not available	PQL = Lab LOQ	0.1	3
Monochlorobiphenyl	Monochlorobiphenyl	27323-18-8	pg/g dw	Not available	Not available	Not available	Not available
Dichlorobiphenyl	Dichlorobiphenyl	25512-42-9	pg/g dw	Not available	Not available	Not available	Not available
Trichlorobiphenyl	Trichlorobiphenyl	25323-68-6	pg/g dw	Not available	Not available	Not available	Not available

Congener Number	Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
						EDL	LOQ
Tetrachlorobiphenyl	Tetrachlorobiphenyl	26914-33-0	pg/g dw	Not available	Not available	Not available	Not available
Pentachlorobiphenyl	Pentachlorobiphenyl	25429-29-2	pg/g dw	Not available	Not available	Not available	Not available
Hexachlorobiphenyl	Hexachlorobiphenyl	26601-64-9	pg/g dw	Not available	Not available	Not available	Not available
Heptachlorobiphenyl	Heptachlorobiphenyl	28655-71-2	pg/g dw	Not available	Not available	Not available	Not available
Octachlorobiphenyl	Octachlorobiphenyl	55722-26-4	pg/g dw	Not available	Not available	Not available	Not available
Nonachlorobiphenyl	Nonachlorobiphenyl	53742-07-7	pg/g dw	Not available	Not available	Not available	Not available
Decachlorobiphenyl	Decachlorobiphenyl	2051-24-3	pg/g dw	Not available	Not available	Not available	Not available
PCB Aroclor 1016	PCB Aroclor 1016	12674-11-2	pg/g dw	Not available	Not available	Not available	Not available
PCB Aroclor 1221	PCB Aroclor 1221	11104-28-2	pg/g dw	Not available	Not available	Not available	Not available
PCB Aroclor 1232	PCB Aroclor 1232	11141-16-5	pg/g dw	Not available	Not available	Not available	Not available
PCB Aroclor 1242	PCB Aroclor 1242	53469-21-9	pg/g dw	Not available	Not available	Not available	Not available
PCB Aroclor 1248	PCB Aroclor 1248	12672-29-6	pg/g dw	Not available	Not available	Not available	Not available
PCB Aroclor 1254	PCB Aroclor 1254	11097-69-1	pg/g dw	Not available	Not available	Not available	Not available
PCB Aroclor 1260	PCB Aroclor 1260	11096-82-5	pg/g dw	Not available	Not available	Not available	Not available
Total PCB congeners	Total PCB Congeners	1336-36-3	pg/g dw	Not available	Not available	Not available	Not available

Notes:
1. Project Action Level has not been established yet for this project.
2. For this project, Project QL has been set at the achievable laboratory level of quantitation (LOQ).
3. Homologue concentrations and total PCB congeners are calculated as the sum of all detected congeners reported by the laboratory regardless of laboratory qualification. PCB Aroclors concentrations are calculated by converting the summed concentrations of a suite of characteristic PCB congeners to concentrations using empirical factors determined from the analysis of Aroclors mixtures. PCB Congeners that are reported as Estimated Maximum Potential Concentrations (EMPCs) since the ratio of the quantitation and confirmation ions are not within theoretical limits (called non-ratioing peaks in the SGS-AXYS SOP) are not included in the calculations for Aroclors. Since these concentrations are a mathematical computation, LOQs are not applicable.
4. Laboratory EDLs "Estimated Detection Limits" represent typical detection limits that can be obtained by SGS Axys Analytical Services. EDLs are based on the smallest peak that can meet all quantification criteria and has a signal-to-noise (S:N) ratio of 2.5:1. EDLs were based on the 10-gram (dry weight) sample size with a final extract volume of 20 µL. Sample-specific EDLs will be dependent on the final extract volume (which may vary depending on matrix interference), dilution factors, and the final field sample size.
5. LOQs represent typical quantitation limits that can be obtained by SGS Axys Analytical Services. The LOQ is supported by the standards in the calibration curve. LOQs were based on the 10-gram (dry weight) sample size with a final extract volume of 20 µL. Sample-specific LOQs reported by the laboratory will be dependent on the final extract volume (which may vary depending on matrix interference), dilution factors, and the final field sample size.
6. Non-detects will be reported at the sample-specific EDLs. Data will be estimated (qualified "J") between the sample-specific EDL and sample-specific LOQ.
7. Units represent mass of contaminant per mass of sediment (dry weight basis).

8. Laboratory will report individual PCB congeners or PCB coelutions and will calculate PCB homologue, PCB Aroclors, and Total PCB congener concentrations based on individual PCB congeners. Aroclor equivalent concentrations will be calculated by converting the summed concentrations of a suite of characteristic PCB congeners to Aroclor concentrations using empirical factors determined by SGS Axys Laboratory from the analysis of commercial Aroclor standards. PCB Congeners that are reported as EMPCs are not included in the calculations for Aroclors. The PCB Aroclor conversion factors are obtained from the SGS Axys Laboratory SOP for USEPA Method 1668C. The conversion formulas are listed below:
Aroclor 1016 = The sum of PCBs 8, 18/30, 31, 28/20 concentrations multiplied by 2.7;
Aroclor 1221 = The sum of PCBs 1, 3, 8 concentrations multiplied by 1.4;
Aroclor 1232 = The sum of PCBs 1, 3, 18/30 concentrations multiplied by 3.4;
Aroclor 1242 = The sum of PCBs 8, 18/30, 31, 28/20 concentrations multiplied by 3.0;
Aroclor 1248 = The sum of PCBs 44/47/65, 49/69, 66 concentrations multiplied by 6.1;
Aroclor 1254 = The sum of PCBs 86/87/97/109/119/125, 83/99 concentrations multiplied by 8.0;
Aroclor 1260 = The sum of PCBs 183/185, 180/193, 170 concentrations multiplied by 5.0.

QAPP Worksheet 15-3
TAL Metals + Tin (Sediment) Reference Limits and Evaluation Tables
Matrix: Sediment
Analytical Group: TAL Metals + Tin (SW846 Method 6010D, and 6020A)
Concentration Level: Low

Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
					MDL	QL
Aluminum	7429-90-5	mg/kg dw	Not available	PQL = Lab QL	0.71	30
Antimony	7440-36-0	mg/kg dw	Not available	PQL = Lab QL	0.02	0.10
Arsenic	7440-38-2	mg/kg dw	Not available	PQL = Lab QL	0.15	0.50
Barium	7440-39-3	mg/kg dw	Not available	PQL = Lab QL	0.04	0.20
Beryllium	7440-41-7	mg/kg dw	Not available	PQL = Lab QL	0.00	0.10
Cadmium	7440-43-9	mg/kg dw	Not available	PQL = Lab QL	0.01	0.1
Calcium	7440-70-2	mg/kg dw	Not available	PQL = Lab QL	1.8	10
Chromium	7440-47-3	mg/kg dw	Not available	PQL = Lab QL	0.05	0.40
Cobalt	7440-48-4	mg/kg dw	Not available	PQL = Lab QL	0.01	0.10
Copper	7440-50-8	mg/kg dw	Not available	PQL = Lab QL	0.07	0.30
Iron	7439-89-6	mg/kg dw	Not available	PQL = Lab QL	1.4	10
Lead	7439-92-1	mg/kg dw	Not available	PQL = Lab QL	0.01	0.10
Magnesium	7439-95-4	mg/kg dw	Not available	PQL = Lab QL	0.68	10
Manganese	7439-96-5	mg/kg dw	Not available	PQL = Lab QL	0.04	0.20
Nickel	7440-02-0	mg/kg dw	Not available	PQL = Lab QL	0.03	0.20
Potassium	7440-09-7	mg/kg dw	Not available	PQL = Lab QL	2.90	100
Selenium	7782-49-2	mg/kg dw	Not available	PQL = Lab QL	0.04	0.50
Silver	7440-22-4	mg/kg dw	Not available	PQL = Lab QL	0.01	0.10
Sodium	7440-23-5	mg/kg dw	Not available	PQL = Lab QL	1.5	100
Thallium	7440-28-0	mg/kg dw	Not available	PQL = Lab QL	0.01	0.1
Tin	7440-31-5	mg/kg dw	Not available	PQL = Lab QL	0.06	0.50
Vanadium	7440-62-2	mg/kg dw	Not available	PQL = Lab QL	0.11	0.50
Zinc	7440-66-6	mg/kg dw	Not available	PQL = Lab QL	0.13	1.0

Notes:
1. Project Action Level has not been established yet for this project.
2. For this project, Project QL has been set at the achievable laboratory level of quantitation (LOQ).

3. Laboratory MDLs represent typical detection limits that can be obtained by Katahdin Analytical Laboratory. MDLs were based on the 1 g (dry weight) sample size with a final digestate volume of 100 ml. Sample-specific MDLs will depend on the final digestate volume (which may vary depending on matrix interference), dilution factors, and the final sample size.
4. Laboratory QLs represent typical quantitation limits that can be obtained by Katahdin Analytical Laboratory. The QL is supported by the standards in the calibration curve. QLs were based on the 1 g (dry weight) sample size with a final digestate volume of 100 ml. Sample-specific QL reported by the laboratory will depend on the final digestate volume (which may vary depending on matrix interference), dilution factors, and the final sample size.
5. Non-detects will be reported at the sample-specific quantitation limit. Data will be estimated (qualified "J") between the sample specific MDL and sample specific QL.
6. Units represent mass of contaminant per mass of sediment (dry weight basis).
7. Aluminum, calcium, iron, potassium, magnesium, and sodium will be analyzed by SW846 Method 6010D, and the remaining metals will be analyzed by SW846 Method 6020A.

QAPP Worksheet 15-4
AVS/SEM (Sediment) Reference Limits and Evaluation Tables
Matrix: Sediment
Analytical Group: AVS/SEM (EPA-821/R-91-100 and SW846 Methods 6010C & 7470A)
Concentration Level: Low

Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
					MDL	QL
Acid Volatile Sulfide	Not available	µmole/g	Not available	PQL = Lab QL	0.038	0.10
Antimony	7440-36-0	µmole/g	Not available	PQL = Lab QL	0.00019	0.00131
Cadmium	7440-43-9	µmole/g	Not available	PQL = Lab QL	0.000014	0.00089
Chromium	7440-47-3	µmole/g	Not available	PQL = Lab QL	0.00017	0.00385
Copper	7440-50-8	µmole/g	Not available	PQL = Lab QL	0.00073	0.007868
Lead	7439-92-1	µmole/g	Not available	PQL = Lab QL	0.00015	0.000483
Mercury	7439-97-6	µmole/g	Not available	PQL = Lab QL	0.000016	0.000020
Nickel	7440-02-0	µmole/g	Not available	PQL = Lab QL	0.00022	0.00341
Silver	7440-22-4	µmole/g	Not available	PQL = Lab QL	0.00010	0.00185
Zinc	7440-66-6	µmole/g	Not available	PQL = Lab QL	0.0011	0.00612

Notes:
1. Project Action Level has not been established yet for this project.
2. For this project, Project QL has been set at the achievable laboratory level of quantitation (LOQ).
3. Laboratory MDLs represent typical detection limits that can be obtained by Katahdin Analytical Laboratory. MDLs were based on the 10 g (dry weight) sample size with a final digestate volume of 40ml for AVS and 200 ml for SEM. Sample-specific MDLs will depend on the final digestate volume (which may vary depending on matrix interference), dilution factors, and the final sample size.
4. Laboratory QLs represent typical quantitation limits that can be obtained by Katahdin Analytical Laboratory. The QL is supported by the standards in the calibration curve. MDLs were based on the 10 g (dry weight) sample size with a final digestate volume of 40ml for AVS and 200 ml for SEM. Sample-specific QL reported by the laboratory will be depended on the final digestate volume (which may vary depending on matrix interference), dilution factors, and the final sample size.
5. Non-detects will be reported at the sample-specific quantitation limit. Data will be estimated (qualified "J") between the sample specific MDL and sample specific QL.
6. Units represent molar mass of contaminant per mass of sediment (dry weight basis).

QAPP Worksheet 15-5
TOC (Sediment) Reference Limits and Evaluation Tables
Matrix: Sediment
Analytical Group: TOC (Lloyd Kahn)
Concentration Level: Low

Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
					MDL	QL
Total Organic Carbon	Not available	µg/g dw	Not available	PQL = Lab QL	85	400

Notes:
1. Project Action Level has not been established yet for this project.
2. For this project, Project QL has been set at the achievable laboratory level of quantitation (LOQ).
3. Laboratory MDLs represent typical detection limits that can be obtained by Katahdin Analytical Laboratory. MDLs were based on the 0.5 g (dry weight) sample size. Sample-specific MDLs will depend on the final sample size, sample preparation and analysis factors.
4. Laboratory QL represents the typical quantitation limit that can be obtained by Katahdin Analytical Laboratory. The QL is supported by the standards in the calibration curve. QLs were based on the 0.5 g (dry weight) sample size. Sample-specific QL reported by the laboratory will be depended on the final sample size, sample preparation and analysis factors. the final extract volume (which may vary depending on matrix interference), dilution factors, and the final sample size.
5. Non-detects will be reported at the sample-specific quantitation limit. No “J” data will be reported between the sample specific MDL and sample specific QL for TOC in sediment.
6. Units represent mass of contaminant per mass of sediment (dry weight basis).

QAPP Worksheet 15-6
PAH and Alkylated PAH (Suspended Particulates) Reference Limits and Evaluation Tables
Matrix: Surface Water (suspended particulate matter retained on filter)
Analytical Group: PAH and Alkylated PAH (Modified SW846 Method 8270)
Concentration Level: Low

Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
					EDL	LOQ
1-Methylchrysene	3351-28-8	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
1-Methylnaphthalene	90-12-0	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
1-Methylphenanthrene	832-69-9	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
1,2-Dimethylnaphthalene	573-98-8	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
1,2,6-Trimethylphenanthrene	30436-55-6	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
1,4,6,7-Tetramethylnaphthalene	13764-18-6	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
1,7-Dimethylphenanthrene	483-87-4	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
1,7-Dimethylfluorene	442-66-0	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
1,8-Dimethylphenanthrene	7372-87-4	ng/L	Not available	Not applicable	1.5	3.75
2-Methylanthracene	613-12-7	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
2-Methylfluorene	2361-84-2	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
2-Methylnaphthalene	91-57-6	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
2-Methylphenanthrene	2531-84-2	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
2,3,5-Trimethylnaphthalene	2245-38-7	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
2,3,6-Trimethylnaphthalene	829-26-5	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
2,4-Dimethyldibenzothiophene	31317-18-7	ng/L	Not available	Not applicable	1.5	3.75
2,6-Dimethylnaphthalene	581-42-0	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
2,6-Dimethylphenanthrene	17980-16-4	ng/L	Not available	Not applicable	1.5	3.75
3-Methylfluoranthene/Benzo[a]fluorene	238-84-6	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
3-Methylphenanthrene	832-71-3	ng/L	Not available	Not applicable	1.5	3.75
3,6-Dimethylphenanthrene	1576-67-6	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
5,9-Dimethylchrysene	139493-40-6	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
7-Methylbenzo[a]pyrene	63041-77-0	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
2/3-Methyldibenzothiophenes	23MDBTHPH	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
5/6-Methylchrysenes	56METHCHRY	ng/L	Not available	PQL = Lab LOQ	1.5	3.75

Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
					EDL	LOQ
9/4-Methylphenanthrenes	883-20-5/832-64-4	ng/L	Not available	Not applicable	1.5	Not Available
Acenaphthene	83-32-9	ng/L	Not available	PQL = Lab LOQ	0.75	3.75
Acenaphthylene	208-96-8	ng/L	Not available	PQL = Lab LOQ	0.75	3.75
Anthracene	120-12-7	ng/L	Not available	PQL = Lab LOQ	0.75	3.75
Fluorene	86-73-7	ng/L	Not available	PQL = Lab LOQ	0.75	3.75
Naphthalene	91-20-3	ng/L	Not available	PQL = Lab LOQ	0.75	3.75
Phenanthrene	85-01-8	ng/L	Not available	PQL = Lab LOQ	0.75	3.75
Benzo[a]anthracene	56-55-3	ng/L	Not available	PQL = Lab LOQ	0.75	3.75
Benzo[a]pyrene	50-32-8	ng/L	Not available	PQL = Lab LOQ	0.75	3.75
Benzo[b]fluoranthene	205-99-2	ng/L	Not available	PQL = Lab LOQ	0.75	3.75
Benzo[e]pyrene	192-97-2	ng/L	Not available	PQL = Lab LOQ	0.75	3.75
Benzo[g,h,i]perylene	191-24-2	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
Benzo[j,k]fluoranthene	207-08-9	ng/L	Not available	PQL = Lab LOQ	0.75	3.75
Chrysene	218-01-9	ng/L	Not available	PQL = Lab LOQ	0.75	3.75
Dibenzo[a,h]anthracene	53-70-3	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
Dibenzothiophene	132-65-0	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
Fluoranthene	206-44-0	ng/L	Not available	PQL = Lab LOQ	0.75	3.75
Indeno[1,2,3-cd]pyrene	193-39-5	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
Perylene	198-55-0	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
Pyrene	129-00-0	ng/L	Not available	PQL = Lab LOQ	0.75	3.75
Biphenyl	92-52-4	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
Retene	483-65-9	ng/L	Not available	PQL = Lab LOQ	1.5	3.75
C1-Acenaphthenes	Not available	ng/L	Not available	Not applicable	0.75	Not Available
C1-Benzo[a]anthracenes/Chrysenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C1-Benzofluoranthenes/Benzopyrenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C1-Biphenyls	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C1-Dibenzothiophenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C1-Fluorenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C1-Naphthalenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C1-Phenanthrenes/Anthracenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C1-Fluoranthenes/Pyrenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available

Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
					EDL	LOQ
C2-Benzo[a]anthracenes/Chrysenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C2-Benzofluoranthenes/Benzopyrenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C2-Biphenyls	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C2-Dibenzothiophenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C2-Fluoranthenes/Pyrenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C2-Fluorenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C2-Naphthalenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C2-Phenanthrenes/Anthracenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C3-Benzo[a]anthracenes/Chrysenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C3-Dibenzothiophenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C3-Fluoranthenes/Pyrenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C3-Fluorenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C3-Naphthalenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C3-Phenanthrenes/Anthracenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C4-Benzo[a]anthracenes/Chrysenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C4-Dibenzothiophenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C4-Fluoranthenes/Pyrenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C4-Naphthalenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available
C4-Phenanthrenes/Anthracenes	Not available	ng/L	Not available	Not applicable	1.5	Not Available

Notes:

1. Project Action Level for PAH and Alkylated PAH has not been established yet for this project.
2. For this project, Project QL has been set at the achievable laboratory level of quantitation (LOQ).
3. Laboratory EDLs “Estimated Detection Limits” represent typical detection limits that can be obtained by SGS Axys Analytical Services. EDLs are based on the smallest peak that can meet all quantification criteria and has a signal-to-noise (S:N) ratio of 3:1. EDLs were based on extraction and analysis of particulate material remaining on filtering system (consisting of a 0.5 μm glass wound cartridge filter and 0.7 μm glass fiber flat filers) after 20-L of water are filtered with a final extract volume of 500 μL and a 3x extract split applied. Sample-specific EDLs are determined on the final extract volume (which may vary depending on matrix interference), dilution factors, split factor, and the final field sample size .
4. LOQs represent typical quantitation limits that can be obtained by SGS Axys Analytical Services. The LOQ is supported by the standards in the calibration curve. LOQs were based on extraction and analysis of particulate material remaining on filtering system (consisting of a 0.5 μm glass wound cartridge filter and 0.7 μm glass fiber flat filers) after 20-L of water are filtered with a final extract volume of 500 μL and a 3x extract split applied. Actual LOQs reported by the

laboratory will be dependent on the final extract volume (which may vary depending on matrix interference), dilution factors, split factor and the final field sample size.
5. Non-detects will be reported at the sample-specific EDLs. Data will be estimated (qualified "J") between the sample-specific EDL and sample-specific LOQ.
6. Unit represents mass of contaminant per liter of water filtered.

QAPP Worksheet 15-7
PCB Congeners (Suspended Particulates) Reference Limits and Evaluation Tables
Matrix: Surface Water (suspended particulate matter retained on filter)
Analytical Group: PCB Congeners (USEPA Method 1668 Version C)
Concentration Level: Low

Congener Number	Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
						EDL	LOQ
PCB 1	2-MoCB	2051-60-7	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 2	3-MoCB	2051-61-8	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 3	4-MoCB	2051-62-9	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 4	2,2'-DiCB	13029-08-8	pg/L	Not available	PQL = Lab LOQ	0.3	3
PCB 5	2,3-DiCB	16605-91-7	pg/L	Not available	PQL = Lab LOQ	0.3	3
PCB 6	2,3'-DiCB	25569-80-6	pg/L	Not available	PQL = Lab LOQ	0.3	3
PCB 7	2,4-DiCB	33284-50-3	pg/L	Not available	PQL = Lab LOQ	0.3	3
PCB 8	2,4'-DiCB	34883-43-7	pg/L	Not available	PQL = Lab LOQ	0.3	3
PCB 9	2,5-DiCB	34883-39-1	pg/L	Not available	PQL = Lab LOQ	0.3	3
PCB 10	2,6-DiCB	33146-45-1	pg/L	Not available	PQL = Lab LOQ	0.3	3
PCB 11	3,3'-DiCB	2050-67-1	pg/L	Not available	PQL = Lab LOQ	0.3	3
PCB 12 (Coelutes with PCB 13)	3,4-DiCB	2974-92-7	pg/L	Not available	PQL = Lab LOQ	0.3	3
PCB 13 (Coelutes with PCB 12)	3,4'-DiCB	2974-90-5	pg/L	Not available	PQL = Lab LOQ	C12	C12
PCB 14	3,5-DiCB	34883-41-5	pg/L	Not available	PQL = Lab LOQ	0.3	3
PCB 15	4,4'-DiCB	2050-68-2	pg/L	Not available	PQL = Lab LOQ	0.3	3
PCB 16	2,2',3-TrCB	38444-78-9	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 17	2,2',4-TrCB	37680-66-3	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 18 (Coelutes with PCB 30)	2,2',5-TrCB	37680-65-2	pg/L	Not available	PQL = Lab LOQ	C30	C30
PCB 19	2,2',6-TrCB	38444-73-4	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 20 (Coelutes with PCB 28)	2,3,3'-TrCB	38444-84-7	pg/L	Not available	PQL = Lab LOQ	C28	C28
PCB 21 (Coelutes with PCB	2,3,4-TrCB	55702-46-0	pg/L	Not available	PQL = Lab LOQ	0.15	3

Congener Number	Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
						EDL	LOQ
33)							
PCB 22	2,3,4'-TrCB	38444-85-8	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 23	2,3,5-TrCB	55720-44-0	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 24	2,3,6-TrCB	55702-45-9	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 25	2,3',4-TrCB	55712-37-3	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 26 (Coelutes with PCB 29)	2,3',5-TrCB	38444-81-4	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 27	2,3',6-TrCB	38444-76-7	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 28 (Coelutes with PCB 20)	2,4,4'-TrCB	7012-37-5	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 29 (Coelutes with PCB 26)	2,4,5-TrCB	15862-07-4	pg/L	Not available	PQL = Lab LOQ	C26	C26
PCB 30 (Coelutes with PCB 18)	2,4,6-TrCB	35693-92-6	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 31	2,4',5-TrCB	16606-02-3	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 32	2,4',6-TrCB	38444-77-8	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 33 (Coelutes with PCB 21)	2',3,4-TrCB	38444-86-9	pg/L	Not available	PQL = Lab LOQ	C21	C21
PCB 34	2',3,5-TrCB	37680-68-5	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 35	3,3',4-TrCB	37680-69-6	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 36	3,3',5-TrCB	38444-87-0	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 37	3,4,4'-TrCB	38444-90-5	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 38	3,4,5-TrCB	53555-66-1	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 39	3,4',5-TrCB	38444-88-1	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 40 (Coelutes with PCB 41 & 71)	2,2',3,3'-TeCB	38444-93-8	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 41 (Coelutes with PCB 40 & 71)	2,2',3,4-TeCB	52663-59-9	pg/L	Not available	PQL = Lab LOQ	C40	C40
PCB 42	2,2',3,4'-TeCB	36559-22-5	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 43	2,2',3,5-TeCB	70362-46-8	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 44 (Coelutes with PCB	2,2',3,5'-TeCB	41464-39-5	pg/L	Not available	PQL = Lab LOQ	0.15	3

Congener Number	Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
						EDL	LOQ
47 & 65)							
PCB 45 (Coelutes with PCB 51)	2,2',3,6-TeCB	70362-45-7	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 46	2,2',3,6'-TeCB	41464-47-5	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 47 (Coelutes with PCB 44 & 65)	2,2',4,4'-TeCB	2437-79-8	pg/L	Not available	PQL = Lab LOQ	C44	C44
PCB 48	2,2',4,5-TeCB	70362-47-9	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 49 (Coelutes with PCB 69)	2,2',4,5'-TeCB	41464-40-8	pg/L	Not available	PQL = Lab LOQ	C69	C69
PCB 50 (Coelutes with PCB 53)	2,2',4,6-TeCB	62796-65-0	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 51 (Coelutes with PCB 45)	2,2',4,6'-TeCB	68194-04-7	pg/L	Not available	PQL = Lab LOQ	C45	C45
PCB 52	2,2',5,5'-TeCB	35693-99-3	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 53 (Coelutes with PCB 50)	2,2',5,6'-TeCB	41464-41-9	pg/L	Not available	PQL = Lab LOQ	C50	C50
PCB 54	2,2',6,6'-TeCB	15968-05-5	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 55	2,3,3',4'-TeCB	74338-24-2	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 56	2,3,3',4'-TeCB	41464-43-1	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 57	2,3,3',5-TeCB	70424-67-8	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 58	2,3,3',5'-TeCB	41464-49-7	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 59 (Coelutes with PCB 62 & 75)	2,3,3',6-TeCB	74472-33-6	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 60	2,3,4,4'-TeCB	33025-41-1	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 61 (Coelutes with PCB 70, 74, & 76)	2,3,4,5-TeCB	33284-53-6	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 62 (Coelutes with PCB 59 & 75)	2,3,4,6-TeCB	54230-22-7	pg/L	Not available	PQL = Lab LOQ	C59	C59
PCB 63	2,3,4',5-TeCB	74472-34-7	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 64	2,3,4',6-TeCB	52663-58-8	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 65 (Coelutes with PCB	2,3,5,6-TeCB	33284-54-7	pg/L	Not available	PQL = Lab LOQ	C44	C44

Congener Number	Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
						EDL	LOQ
44 & 47)							
PCB 66	2,3',4,4'-TeCB	32598-10-0	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 67	2,3',4,5-TeCB	73575-53-8	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 68	2,3',4,5'-TeCB	73575-52-7	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 69 (Coelutes with PCB 49)	2,3',4,6-TeCB	60233-24-1	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 70 (Coelutes with PCB 61, 74, & 76)	2,3',4',5-TeCB	32598-11-1	pg/L	Not available	PQL = Lab LOQ	C61	C61
PCB 71 (Coelutes with PCB 40 & 41)	2,3',4',6-TeCB	41464-46-4	pg/L	Not available	PQL = Lab LOQ	C40	C40
PCB 72	2,3',5,5'-TeCB	41464-42-0	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 73	2,3',5',6-TeCB	74338-23-1	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 74 (Coelutes with PCB 61, 70, & 76)	2,4,4',5-TeCB	32690-93-0	pg/L	Not available	PQL = Lab LOQ	C61	C61
PCB 75 (Coelutes with PCB 59 & 62)	2,4,4',6-TeCB	32598-12-2	pg/L	Not available	PQL = Lab LOQ	C59	C59
PCB 76 (Coelutes with PCB 61, 70, & 74)	2',3,4,5-TeCB	70362-48-0	pg/L	Not available	PQL = Lab LOQ	C61	C61
PCB 77	3,3',4,4'-TeCB	32598-13-3	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 78	3,3',4,5-TeCB	70362-49-1	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 79	3,3',4,5'-TeCB	41464-48-6	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 80	3,3',5,5'-TeCB	33284-52-5	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 81	3,4,4',5-TeCB3	70362-50-4	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 82	2,2',3,3',4-PeCB	52663-62-4	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 83 (Coelutes with PCB 99)	2,2',3,3',5-PeCB	60145-20-2	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 84	2,2',3,3',6-PeCB	52663-60-2	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 85 (Coelutes with PCB 116 & 117)	2,2',3,4,4'-PeCB	65510-45-4	pg/L	Not available	PQL = Lab LOQ	C117	C117
PCB 86 (Coelutes with PCB 87, 97, 109, 119, & 125)	2,2',3,4,5-PeCB	55312-69-1	pg/L	Not available	PQL = Lab LOQ	C109	C109

Congener Number	Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
						EDL	LOQ
PCB 87 (Coelutes with PCB 86, 97, 109, 119, & 125)	2,2',3,4,5'-PeCB	38380-02-8	pg/L	Not available	PQL = Lab LOQ	C109	C109
PCB 88 (Coelutes with PCB 91)	2,2',3,4,6-PeCB	55215-17-3	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 89	2,2',3,4,6'-PeCB	73575-57-2	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 90 (Coelutes with PCB 101 & 113)	2,2',3,4',5-PeCB	68194-07-0	pg/L	Not available	PQL = Lab LOQ	C113	C113
PCB 91 (Coelutes with PCB 88)	2,2',3,4',6-PeCB	68194-05-8	pg/L	Not available	PQL = Lab LOQ	C88	C88
PCB 92	2,2',3,5,5'-PeCB	52663-61-3	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 93 (Coelutes with PCB 95, 98, 100, & 102)	2,2',3,5,6-PeCB	73575-56-1	pg/L	Not available	PQL = Lab LOQ	C95	C95
PCB 94	2,2',3,5,6'-PeCB	73575-55-0	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 95 (Coelutes with PCB 93, 98, 100, & 102)	2,2',3,5',6-PeCB	38379-99-6	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 96	2,2',3,6,6'-PeCB	73575-54-9	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 97 (Coelutes with PCB 86, 87, 109, 119, & 125)	2,2',3',4,5-PeCB	41464-51-1	pg/L	Not available	PQL = Lab LOQ	C109	C109
PCB 98 (Coelutes with PCB 93, 95, 100, & 102)	2,2',3',4,6-PeCB	60233-25-2	pg/L	Not available	PQL = Lab LOQ	C95	C95
PCB 99 (Coelutes with PCB 83)	2,2',4,4',5-PeCB	38380-01-7	pg/L	Not available	PQL = Lab LOQ	C83	C83
PCB 100 (Coelutes with PCB 93, 95, 98, & 102)	2,2',4,4',6-PeCB	39485-83-1	pg/L	Not available	PQL = Lab LOQ	C95	C95
PCB 101 (Coelutes with PCB 90 & 113)	2,2',4,5,5'-PeCB	37680-73-2	pg/L	Not available	PQL = Lab LOQ	C113	C113
PCB 102 (Coelutes with PCB 93, 95, 98, & 100)	2,2',4,5,6'-PeCB	68194-06-9	pg/L	Not available	PQL = Lab LOQ	C95	C95
PCB 103	2,2',4,5',6-PeCB	60145-21-3	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 104	2,2',4,6,6'-PeCB	56558-16-8	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 105	2,3,3',4,4'-PeCB	32598-14-4	pg/L	Not available	PQL = Lab LOQ	0.15	3

Congener Number	Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
						EDL	LOQ
PCB 106	2,3,3',4,5-PeCB	70424-69-0	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 107	2,3,3',4',5-PeCB	70424-68-9	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 108 (Coelutes with PCB 124)	2,3,3',4,5'-PeCB	70362-41-3	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 109 (Coelutes with PCB 86, 87, 97, 119, & 125)	2,3,3',4,6-PeCB	74472-35-8	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 110 (Coelutes with PCB 115)	2,3,3',4',6-PeCB	38380-03-9	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 111	2,3,3',5,5'-PeCB	39635-32-0	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 112	2,3,3',5,6-PeCB	74472-36-9	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 113 (Coelutes with PCB 90 & 101)	2,3,3',5',6-PeCB	68194-10-5	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 114	2,3,4,4',5-PeCB	74472-37-0	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 115 (Coelutes with PCB 110)	2,3,4,4',6-PeCB	74472-38-1	pg/L	Not available	PQL = Lab LOQ	C110	C110
PCB 116 (Coelutes with PCB 85 & 117)	2,3,4,5,6-PeCB	18259-05-7	pg/L	Not available	PQL = Lab LOQ	C117	C117
PCB 117 (Coelutes with PCB 85 & 116)	2,3,4',5,6-PeCB	68194-11-6	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 118	2,3',4,4',5-PeCB	31508-00-6	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 119 (Coelutes with PCB 86, 87, 97, 109, & 125)	2,3',4,4',6-PeCB	56558-17-9	pg/L	Not available	PQL = Lab LOQ	C109	C109
PCB 120	2,3',4,5,5'-PeCB	68194-12-7	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 121	2,3',4,5,'6-PeCB	56558-18-0	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 122	2',3,3',4,5-PeCB	76842-07-4	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 123	2',3,4,4',5-PeCB	65510-44-3	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 124 (Coelutes with PCB 108)	2',3,4,5,5'-PeCB	70424-70-3	pg/L	Not available	PQL = Lab LOQ	C108	C108
PCB 125 (Coelutes with	2',3,4,5,6'-PeCB	74472-39-2	pg/L	Not available	PQL = Lab LOQ	C109	C109

Congener Number	Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
						EDL	LOQ
PCB 86, 87, 97, 109, & 119)							
PCB 126	3,3',4,4',5-PeCB	57465-28-8	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 127	3,3',4,5,5'-PeCB	39635-33-1	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 128 (Coelutes with PCB 166)	2,2',3,3',4,4'-HxCB	38380-07-3	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 129 (Coelutes with PCB 138, 160, & 163)	2,2',3,3',4,5-HxCB	55215-18-4	pg/L	Not available	PQL = Lab LOQ	C138	C138
PCB 130	2,2',3,3',4,5'-HxCB	52663-66-8	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 131	2,2',3,3',4,6-HxCB	61798-70-7	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 132	2,2',3,3',4,6'-HxCB	38380-05-1	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 133	2,2',3,3',5,5'-HxCB	35694-04-3	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 134 (Coelutes with PCB 143)	2,2',3,3',5,6-HxCB	52704-70-8	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 135 (Coelutes with PCB 151 & 154)	2,2',3,3',5,6'-HxCB	52744-13-5	pg/L	Not available	PQL = Lab LOQ	C151	C151
PCB 136	2,2',3,3',6,6'-HxCB	38411-22-2	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 137	2,2',3,4,4',5-HxCB	35694-06-5	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 138 (Coelutes with PCB 129, 160, & 163)	2,2',3,4,4',5'-HxCB	35065-28-2	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 139 (Coelutes with PCB 140)	2,2',3,4,4',6-HxCB	56030-56-9	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 140 (Coelutes with PCB 139)	2,2',3,4,4',6'-HxCB	59291-64-4	pg/L	Not available	PQL = Lab LOQ	C139	C139
PCB 141	2,2',3,4,5,5'-HxCB	52712-04-6	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 142	2,2',3,4,5,6-HxCB	41411-61-4	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 143 (Coelutes with PCB 134)	2,2',3,4,5,6'-HxCB	68194-15-0	pg/L	Not available	PQL = Lab LOQ	C134	C134
PCB 144	2,2',3,4,5',6-HxCB	68194-14-9	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 145	2,2',3,4,6,6'-HxCB	74472-40-5	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 146	2,2',3,4',5,5'-HxCB	51908-16-8	pg/L	Not available	PQL = Lab LOQ	0.15	3

Congener Number	Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
						EDL	LOQ
PCB 147 (Coelutes with PCB 149)	2,2',3,4',5,6-HxCB	68194-13-8	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 148	2,2',3,4',5,6'-HxCB	74472-41-6	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 149 (Coelutes with PCB 147)	2,2',3,4',5',6-HxCB	38380-04-0	pg/L	Not available	PQL = Lab LOQ	C147	C147
PCB 150	2,2',3,4',6,6'-HxCB	68194-08-1	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 151 (Coelutes with PCB 135 & 154)	2,2',3,5,5',6-HxCB	52663-63-5	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 152	2,2',3,5,6,6'-HxCB	68194-09-2	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 153 (Coelutes with PCB 168)	2,2',4,4',5,5'-HxCB	35065-27-1	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 154 (Coelutes with PCB 135 & 151)	2,2',4,4',5',6-HxCB	60145-22-4	pg/L	Not available	PQL = Lab LOQ	C151	C151
PCB 155	2,2',4,4',6,6'-HxCB	33979-03-2	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 156 (Coelutes with PCB 157)	2,3,3',4,4',5-HxCB	38380-08-4	pg/L	Not available	PQL = Lab LOQ	0.15	6
PCB 157 (Coelutes with PCB 156)	2,3,3',4,4',5'-HxCB	69782-90-7	pg/L	Not available	PQL = Lab LOQ	C156	C156
PCB 158	2,3,3',4,4',6-HxCB	74472-42-7	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 159	2,3,3',4,5,5'-HxCB	39635-35-3	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 160 (Coelutes with PCB 129, 138, & 163)	2,3,3',4,5,6-HxCB	41411-62-5	pg/L	Not available	PQL = Lab LOQ	C138	C138
PCB 161	2,3,3',4,5',6-HxCB	74472-43-8	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 162	2,3,3',4',5,5'-HxCB	39635-34-2	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 163 (Coelutes with PCB 129, 138, & 160)	2,3,3',4',5,6-HxCB	74472-44-9	pg/L	Not available	PQL = Lab LOQ	C138	C138
PCB 164	2,3,3',4',5',6-HxCB	74472-45-0	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 165	2,3,3',5,5',6-HxCB	74472-46-1	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 166 (Coelutes with PCB 128)	2,3,4,4',5,6-HxCB	41411-63-6	pg/L	Not available	PQL = Lab LOQ	C128	C128
PCB 167	2,3',4,4',5,5'-HxCB	52663-72-6	pg/L	Not available	PQL = Lab LOQ	0.15	3

Congener Number	Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
						EDL	LOQ
PCB 168 (Coelutes with PCB 153)	2,3',4,4',5',6-HxCB	59291-65-5	pg/L	Not available	PQL = Lab LOQ	C153	C153
PCB 169	3,3',4,4',5,5'-HxCB	32774-16-6	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 170	2,2',3,3',4,4',5-HpCB	35065-30-6	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 171 (Coelutes with PCB 173)	2,2'3,3',4,4',6-HpCB	52663-71-5	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 172	2,2',3,3',4,5,5'-HpCB	52663-74-8	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 173 (Coelutes with PCB 171)	2,2',3,3',4,5,6-HpCB	68194-16-1	pg/L	Not available	PQL = Lab LOQ	C173	C173
PCB 174	2,2',3,3',4,5,6'-HpCB	38411-25-5	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 175	2,2',3,3',4,5',6-HpCB	40186-70-7	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 176	2,2',3,3',4,6,6'-HpCB	52663-65-7	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 177	2,2',3,3',4',5,6-HpCB	52663-70-4	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 178	2,2',3,3',5,5',6-HpCB	52663-67-9	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 179	2,2',3,3',5,6,6'-HpCB	52663-64-6	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 180 (Coelutes with PCB 193)	2,2',3,4,4',5,5'-HpCB	35065-29-3	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 181	2,2',3,4,4',5,6-HpCB	74472-47-2	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 182	2,2',3,4,4',5,6'-HpCB	60145-23-5	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 183 (Coelutes with PCB 185)	2,2',3,4,4',5',6-HpCB	52663-69-1	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 184	2,2',3,4,4',6,6'-HpCB	74472-48-3	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 185 (Coelutes with PCB 183)	2,2',3,4,5,5',6-HpCB	52712-05-7	pg/L	Not available	PQL = Lab LOQ	C183	C183
PCB 186	2,2',3,4,5,6,6'-HpCB	74472-49-4	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 187	2,2',3,4',5,5',6-HpCB	52663-68-0	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 188	2,2',3,4',5,6,6'-HpCB	74487-85-7	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 189	2,3,3',4,4',5,5'-HpCB	39635-31-9	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 190	2,3,3',4,4',5,6-HpCB	41411-64-7	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 191	2,3,3',4,4',5',6-HpCB	74472-50-7	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 192	2,3,3',4,5,5',6-HpCB	74472-51-8	pg/L	Not available	PQL = Lab LOQ	0.15	3

Congener Number	Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
						EDL	LOQ
PCB 193 (Coelutes with PCB 180)	2,3,3',4',5,5',6-HpCB	69782-91-8	pg/L	Not available	PQL = Lab LOQ	C180	C180
PCB 194	2,2',3,3',4,4',5,5'-OcCB	35694-08-7	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 195	2,2',3,3',4,4',5,6-OcCB	52663-78-2	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 196	2,2',3,3',4,4',5,6'-OcCB	42740-50-1	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 197 (Coelutes with PCB 200)	2,2',3,3',4,4',6,6'-OcCB	33091-17-7	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 198 (Coelutes with PCB 199)	2,2',3,3',4,5,5',6-OcCB	68194-17-2	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 199 (Coelutes with PCB 198)	2,2',3,3',4,5,5',6'-OcCB	52663-75-9	pg/L	Not available	PQL = Lab LOQ	C198	C198
PCB 200 (Coelutes with PCB 197)	2,2',3,3',4,5,6,6'-OcCB	52663-73-7	pg/L	Not available	PQL = Lab LOQ	C197	C197
PCB 201	2,2',3,3',4,5',6,6'-OcCB	40186-71-8	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 202	2,2',3,3',5,5',6,6'-OcCB	2136-99-4	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 203	2,2',3,4,4',5,5',6-OcCB	52663-76-0	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 204	2,2',3,4,4',5,6,6'-OcCB	74472-52-9	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 205	2,3,3',4,4',5,5',6-OcCB	74472-53-0	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 206	2,2',3,3',4,4',5,5',6-NoCB	40186-72-9	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 207	2,2',3,3',4,4',5,6,6'-NoCB	52663-79-3	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 208	2,2',3,3',4,5,5',6,6'-NoCB	52663-77-1	pg/L	Not available	PQL = Lab LOQ	0.15	3
PCB 209	Decachlorobiphenyl	2051-24-3	pg/L	Not available	PQL = Lab LOQ	0.15	3
Monochlorobiphenyl	Monochlorobiphenyl	27323-18-8	pg/L	Not available	Not available	Not available	Not available
Dichlorobiphenyl	Dichlorobiphenyl	25512-42-9	pg/L	Not available	Not available	Not available	Not available

Congener Number	Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
						EDL	LOQ
Trichlorobiphenyl	Trichlorobiphenyl	25323-68-6	pg/L	Not available	Not available	Not available	Not available
Tetrachlorobiphenyl	Tetrachlorobiphenyl	26914-33-0	pg/L	Not available	Not available	Not available	Not available
Pentachlorobiphenyl	Pentachlorobiphenyl	25429-29-2	pg/L	Not available	Not available	Not available	Not available
Hexachlorobiphenyl	Hexachlorobiphenyl	26601-64-9	pg/L	Not available	Not available	Not available	Not available
Heptachlorobiphenyl	Heptachlorobiphenyl	28655-71-2	pg/L	Not available	Not available	Not available	Not available
Octachlorobiphenyl	Octachlorobiphenyl	55722-26-4	pg/L	Not available	Not available	Not available	Not available
Nonachlorobiphenyl	Nonachlorobiphenyl	53742-07-7	pg/L	Not available	Not available	Not available	Not available
Decachlorobiphenyl	Decachlorobiphenyl	2051-24-3	pg/L	Not available	Not available	Not available	Not available
PCB Aroclor 1016	PCB Aroclor 1016	12674-11-2	pg/L	Not available	Not available	Not available	Not available
PCB Aroclor 1221	PCB Aroclor 1221	11104-28-2	pg/L	Not available	Not available	Not available	Not available
PCB Aroclor 1232	PCB Aroclor 1232	11141-16-5	pg/L	Not available	Not available	Not available	Not available
PCB Aroclor 1242	PCB Aroclor 1242	53469-21-9	pg/L	Not available	Not available	Not available	Not available
PCB Aroclor 1248	PCB Aroclor 1248	12672-29-6	pg/L	Not available	Not available	Not available	Not available
PCB Aroclor 1254	PCB Aroclor 1254	11097-69-1	pg/L	Not available	Not available	Not available	Not available
PCB Aroclor 1260	PCB Aroclor 1260	11096-82-5	pg/L	Not available	Not available	Not available	Not available
Total PCB congeners	Total PCB Congeners	1336-36-3	pg/L	Not available	Not available	Not available	Not available

Notes:

1. Project Action Level has not been established yet for this project.
2. For this project, Project QL has been set at the achievable laboratory level of quantitation (LOQ).
3. Homologue concentrations and total PCB congeners are calculated as the sum of all detected congeners reported by the laboratory regardless of laboratory qualification. PCB Aroclors concentrations are calculated by converting the summed concentrations of a suite of characteristic PCB congeners to concentrations using empirical factors determined from the analysis of Aroclors mixtures. PCB Congeners that are reported as Estimated Maximum Potential Concentrations (EMPCs) since the ratio of the quantitation and confirmation ions are not within theoretical limits (called non-ratioing peaks in the SGS-AXYS SOP) are not included in the calculations for Aroclors. Since these concentrations are a mathematical computation, LOQs are not available.
4. Laboratory EDLs “Estimated Detection Limits” represent typical detection limits that can be obtained by SGS Axys Analytical Services. EDLs are based on the smallest peak that can meet all quantification criteria and has a signal-to-noise (S:N) ratio of 2.5:1. EDLs were based on extraction and analysis of particulate material remaining on filtering system (consisting of a 0.5 μm glass wound cartridge filter and 0.7 μm glass fiber flat filers) after 20-L of water are filtered with a final extract volume of 20 μL and a 3x extract split applied. Sample-specific EDLs will depend on the final extract volume (which may vary depending on matrix interference), dilution factors, split factor and the final field sample size.
5 LOQs represent typical quantitation limits that can be obtained by SGS Axys Analytical Services. The LOQ is supported by the standards in the calibration curve. LOQs were based on extraction and analysis of particulate material remaining on filtering system (consisting of a 0.5 μm glass wound cartridge filter and

0.7 μm glass fiber flat filers) after 20-L of water are filtered with a final extract volume of 20 μL and a 3x extract split applied. Actual LOQs reported by the laboratory will be dependent on the final extract volume (which may vary depending on matrix interference), dilution factors, split factor and the final field sample size.
6. Non-detects will be reported at the sample-specific EDLs. Data will be estimated (qualified "J") between the sample-specific EDL and sample-specific LOQ.
7. Unit represents mass of contaminant per liter of water filtered.
8. Laboratory will report individual PCB congeners or PCB coelutions and will calculate PCB homologue, PCB Aroclors, and Total PCB congener concentrations based on individual PCB congeners. Aroclor equivalent concentrations will be calculated by converting the summed concentrations of a suite of characteristic PCB congeners to Aroclor concentrations using empirical factors determined by SGS Axys Laboratory from the analysis of commercial Aroclor standards. PCB Congeners that are reported EMPCs are not included in the calculations for Aroclors. The PCB Aroclor conversion factors are obtained from the SGS Axys Laboratory SOP for USEPA Method 1668C. The conversion formulas are listed below:
Aroclor 1016 = The sum of PCBs 8, 18/30, 31, 28/20 concentrations multiplied by 2.7;
Aroclor 1221 = The sum of PCBs 1, 3, 8 concentrations multiplied by 1.4;
Aroclor 1232 = The sum of PCBs 1, 3, 18/30 concentrations multiplied by 3.4;
Aroclor 1242 = The sum of PCBs 8, 18/30, 31, 28/20 concentrations multiplied by 3.0;
Aroclor 1248 = The sum of PCBs 44/47/65, 49/69, 66 concentrations multiplied by 6.1;
Aroclor 1254 = The sum of PCBs 86/87/97/109/119/125, 83/99 concentrations multiplied by 8.0;
Aroclor 1260 = The sum of PCBs 183/185, 180/193, 170 concentrations multiplied by 5.0.

QAPP Worksheet 15-8
TAL Metals + Tin (Whole Water & Dissolved Water) Reference Limits and Evaluation Tables
Matrix: Surface Water (whole water and dissolved phases)
Analytical Group: TAL Metals + Tin (SW846 Method 6010D, and 6020A)
Concentration Level: Low

Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
					MDL	QL
Aluminum	7429-90-5	µg/L	Not available	PQL = Lab QL	4.4	100
Antimony	7440-36-0	µg/L	Not available	PQL = Lab QL	0.054	1.0
Arsenic	7440-38-2	µg/L	Not available	PQL = Lab QL	2.2	5.0
Barium	7440-39-3	µg/L	Not available	PQL = Lab QL	0.27	2.0
Beryllium	7440-41-7	µg/L	Not available	PQL = Lab QL	0.034	1.0
Cadmium	7440-43-9	µg/L	Not available	PQL = Lab QL	0.030	1.0
Calcium	7440-70-2	µg/L	Not available	PQL = Lab QL	11	100
Chromium	7440-47-3	µg/L	Not available	PQL = Lab QL	0.22	4.0
Cobalt	7440-48-4	µg/L	Not available	PQL = Lab QL	0.060	1.0
Copper	7440-50-8	µg/L	Not available	PQL = Lab QL	0.18	3.0
Iron	7439-89-6	µg/L	Not available	PQL = Lab QL	5.42	100
Lead	7439-92-1	µg/L	Not available	PQL = Lab QL	0.074	1.0
Magnesium	7439-95-4	µg/L	Not available	PQL = Lab QL	7.8	100
Manganese	7439-96-5	µg/L	Not available	PQL = Lab QL	0.35	2.0
Nickel	7440-02-0	µg/L	Not available	PQL = Lab QL	0.15	2.0
Potassium	7440-09-7	µg/L	Not available	PQL = Lab QL	41	1000
Selenium	7782-49-2	µg/L	Not available	PQL = Lab QL	0.19	5.0
Silver	7440-22-4	µg/L	Not available	PQL = Lab QL	0.050	1.0
Sodium	7440-23-5	µg/L	Not available	PQL = Lab QL	24	1000
Thallium	7440-28-0	µg/L	Not available	PQL = Lab QL	0.060	1.0
Tin	7440-31-5	µg/L	Not available	PQL = Lab QL	0.15	5.0
Vanadium	7440-62-2	µg/L	Not available	PQL = Lab QL	0.51	5.0
Zinc	7440-66-6	µg/L	Not available	PQL = Lab QL	3.9	10

Notes:
1. Project Action Level has not been established yet for this project.

2. For this project, Project QL has been set at the achievable laboratory level of quantitation (LOQ).
3. Laboratory MDLs represent typical detection limits that can be obtained by Katahdin Analytical Laboratory. MDLs were based on extraction and analysis of 50ml of water with a final extract volume of 50 ml. Sample-specific MDLs will depend on the final digestate volume (which may vary depending on matrix interference), dilution factors, and the final sample size.
4. Laboratory QLs represent typical quantitation limits that can be obtained by Katahdin Analytical Laboratory. The QL is supported by the standards in the calibration curve. QLs were based on extraction and analysis of 50ml of water with a final extract volume of 50 ml. Sample-specific QLs reported by the laboratory will be depended on the final digestate volume (which may vary depending on matrix interference), dilution factors, and the final sample size.
5. Non-detects will be reported at the sample-specific quantitation limit. Data will be estimated (qualified "J") between the sample specific MDL and sample specific QL.
6. Unit represents mass of contaminant per liter of water.
7. Calcium, iron, potassium, magnesium, and sodium will be analyzed by SW846 Method 6010D, and the remaining metals will be analyzed by SW846 Method 6020A.

QAPP Worksheet 15-9
TSS and POC Reference Limits and Evaluation Tables
Matrix: Surface Water and Particulate on filter
Analytical Group: TSS (EPA160.1/SM2540D-modified), DOC (SW846 Method 9060-modified), and POC (Lloyd Kahn-modified)
Concentration Level: Low

Analyte	CAS Number	Units	Project Action Level	Project QL	Achievable Laboratory Limits	
					MDL	QL
Total Suspended Solid	Not available	mg/L	Not available	PQL = Lab QL	0.19	0.625
Particulate Organic Carbon	Not available	μg/L	Not available	PQL = Lab QL	25	50

Notes:
1. Project Action Level has not been established yet for this project.
2. For this project, Project QL has been set at the achievable laboratory level of quantitation (LOQ).
3. Laboratory MDLs represent typical detection limits that can be obtained by Katahdin Analytical Laboratory. MDLs were based on a sample size of 4-Liter. Sample-specific MDLs depend on the final sample size, sample preparation and analysis factors.
4. Laboratory QLs represent typical quantitation limits that can be obtained by Katahdin Analytical Laboratory. The QL is supported by the standards in the calibration curve. QLs were based on a sample size of 4 Liter. Actual QL reported by the laboratory will depend on dilution factors, the final sample size, and laboratory preparation.
5. Non-detects will be reported at the sample-specific quantitation limit. Data will be estimated (qualified "J") between the sample specific MDL and sample specific QL.
6. For TSS, unit represents mass of suspended solids per liter of water filtered; and for POC, unit represents mass of suspended particulate organic carbon per liter of water filtered.
7. Method modifications are listed in QAPP WS #23.

QAPP Worksheet 17
Sampling Design and Rationale

Field Program and Rationale for CSO Samples

- Two CSOs, RH-034 and OH-007 (refer to Figure 2 for the proposed sampling locations), will be sampled during four wet weather events per year. *Rationale: CSO samples will be used to characterize COPC concentrations on CSO solids and estimate the solids loading discharge to Gowanus Canal*.
- CSO Samples will be collected from the combined sewer regulators, approximately one block from the discharge points, to eliminate the influence of the Canal on the samples.
- Weather conditions that would trigger a sampling event: "Wet Weather Event" will be defined as a minimum 0.1 inch/hour rainfall event. A minimum of 48 hours of dry weather (<0.1 inch of rain) is required between events. Rainfall thresholds that initiate discharge from CSO outfalls are presented in the Mobilization Protocol (Attachment 3).
- CSO samples will be collected as time-composites based on expected storm duration, to the extent possible. In an effort to capture the entire storm event, one to two time composite samples will be initiated and collected based on storm forecast length (*e.g*., a 2-hour based composite and a 4-hour based composite). At the end of the sampling event, one time composite will be shipped to the lab for analysis for each station. Selection of the time composite for lab analysis will be decided in the field based on the actual length of the storm event.
- Aliquots to CSO composite samples will be added on a regular basis (*e.g.* ,every 15 minutes over a two-hour period) so as to meet the anticipated volume requirements at the end of the expected storm duration. Sample bottles will be filled in a round-robin fashion as a means to minimize differences among sample bottles to be sent for various analyses.
- CSO samples will be analyzed for PAH (parent and alkylated), PCB Congeners, metals, POC and grain size distribution. PAH, PCB, TSS, POC and grain size distribution will be analyzed on the suspended phase (defined as particles retained on a 0.7 um filter). Metals will be analyzed in whole water and a corresponding dissolved phase sample (defined as filtrate passing through the 0.7 um filter).

Field Program and Rationale for Sediment Samples

- Surface sediment samples will be collected from 10 locations approximating a grid along the main axis of RTA 1 (refer to Figure 3 for proposed sampling locations). *Rationale: to develop an estimate of the average COPC concentration in the soft sediments after the USEPA remedy has been implemented.*
- GPS coordinates will be recorded at each sampling location.
- At each sampling location, sediment will be collected from the top 6-inch horizon using a petite Ponar dredge (or similar equipment). Details are provided in the Field SOP No. 3 (Attachment 1).
- Visual and olfactory observations and sediment texture (i.e., fine sand, medium sand, coarse sand, gravel, silt, clay, and organic/other matter such as wood chips) will be recorded for each sample.
- Sediment samples will be analyzed for PAH and alkylated PAH, PCB Congeners, metals, AVS/SEM, TOC, and grain size distribution.
- Samples will be obtained within 15 feet of each planned location (*i.e.,* within a 15-foot radius circle about the location), with a location accuracy of ± 3 feet based on the GPS.

FIELD STANDARD OPERATING PROCEDURES (provided in Attachment 1)

- SOP No. 1: Procedure to Locate Sample Points Using Global Positioning System (October 2021, Version 01)
- SOP No. 2: Procedure for Navigation and Boat Positioning (October 2021 , Version 01)

- SOP No. 3: Procedure for Collection and Processing of Surface Sediments (October 2021, Version 01)
- SOP No. 5: Procedure for the Decontamination of Sediment and Soil Equipment (October 2021021, Version 01)
- SOP No. 6: Procedure for Management and Disposal of Investigation-Derived Waste (October 2021, Version 01)
- SOP No. 7: Procedure for Collection of Field Blank and Rinse Blank Samples (October 2021, Version 01)
- SOP No. 8: Procedure to Conduct Sample Management (October 2021, Version 01)
- SOP No. 9: Procedure for Collection of Large Volume/Small Volume CSO Samples (October 2021, Version 01)
- SOP No. 10: Procedure for Decanting Samples for Grain Size Analysis (October 2021, Version 01)

QAPP Worksheet 18
Sampling Locations and Methods

Sampling Locations and Methods

Sampling Location and Sample ID	Matrix	Depth	Sampling Method	Analytical Group	Sampling SOP Reference	Rationale for Sampling Location
Sediment sampling **NYCDEP-PR-SED-XX** NYCDEP = New York City Department of Environmental Protection PR = Post Remedy Monitoring Program SED = Sediment XX = Location number ranged from 01 to 10	Sediment	0-6 inch	Ponar Dredge	PAH and alkylated PAH, PCB Congeners, TAL Metals+Tin, AVS/SEM, TOC, and Grain Size	Refer to Worksheet 21	Surface sediment samples will be collected to characterize the COPC concentrations, refer to Figure 3 for sampling locations.
CSO Outfall **NYCDEP-PR-CSO-RH-034-MMYY-W** NYDEP = New York City Department of Environmental Protection PR = Post Remedy CSO = Combined Sewer Overflow RH-034 = Location identification of sample MMYY = collection month and year W or D or S: W = Whole water; D = Dissolved phase; S = Suspended matter.	Aqueous (whole water , dissolved, and particulate phases)	Not applicable	Peristaltic pump	Particulate Phase: PAH and alkylated PAH, and PCB Congeners; Whole water and dissolved phases: TAL Metals+Tin, TSS, POC, and Grain Size	Refer to Worksheet 21	CSO samples will be collected to provide a baseline to characterize the concentrations of CSO solids that enter the Gowanus Canal refer to Figure 2 for sampling locations.

Note 1: Surface sediment field duplicate ID scheme will be labeled as follows: **NYCDEP-PR-SED-FD-01**, the equipment blank ID scheme will be labeled as follows: **NYCDEP-PR-SED-EB-01**.
Note 2: CSO sample field duplicate ID scheme per sampling event will be labeled as follows: **NYCDEP-PR-CSO-FD-01-W/D/S**, and the equipment blank ID scheme per sampling event will be labeled as follows: **NYCDEP-PR-CSO-EB-01-W/D/S**

QAPP Worksheet 19 and Worksheet 30
Sample Containers, Preservation, and Hold Time

Sample Containers, Preservation, and Hold Time

Analytical Group	Matrix	Method SOP	Container Type	Preservation	Analytical Holding Time	Assigned Laboratory	Data Package Turnaround Time
PAH and Alkylated PAH	Sediment	Modified SW846 Method 8270	4 oz amber container with Teflon-lined lid	Ship on wet ice at less than 6 degrees Celsius.	PAH: 14 days to extraction; 40 days from extraction to analysis; PCB: 1 year to extraction; extracts hold up 1 year for analysis; holding time can extends to 1 year if stored in the dark at less than minus 10 degrees Celsius.	SGS AXYS (British Columbia, Canada)	30 business days
PCB Congeners	Sediment	EPA Method 1668C					
TAL Metals + Tin	Sediment	SW846 Method 6010/6020	4 oz amber container with Teflon-lined lid	Ship on wet ice at less than 6 degrees Celsius.	All metals: 180 days to analysis;	Katahdin (Scarborough, Maine)	21 calendar days
TOC	Sediment	Lloyd Kahn			14 days to analysis, holding time can extended to 6 months if samples are frozen below minus 10 degrees Celsius		

Sample Containers, Preservation, and Hold Time

Analytical Group	Matrix	Method SOP	Container Type	Preservation	Analytical Holding Time	Assigned Laboratory	Data Package Turnaround Time
AVS/SEM	Sediment	EPA-821/R-91-100 Draft 1991	2oz glass container (no headspace)	Ship on wet ice at less than 6 degrees Celsius. Maintain samples at less than 6 degrees Celsius until shipment.	AVS: 14 days to analysis; SEM: 14 days to extract and 6 months to analysis; holding time can be extended to 28 days if samples are frozen minus 10 degrees Celsius	Katahdin (Scarborough, Maine)	21 calendar days
Grain Size	Sediment	ASTM D422 or equivalent	Minimum 200 grams	No preservation	No holding time	Katahdin (Scarborough, Maine)	21 calendar days
Grain Size	Aqueous	Laser Particle Size Distribution	Five 4-liter containers	Allow sample to settle for minimum of 5 days. Decant supernatant; ship settled solids for analysis. No preservation.	No holding time	SedTrend (British Columbia, Canada)	21 calendar days
PAH and Alkylated PAH	Aqueous (suspended particulate matter retained on filter)	Modified SW846 Method 8270	20-liter stainless steel	Field preserve with 1 gram of sodium azide, ship on wet ice at less than 6 degrees Celsius.	Filter within 7 days of sample collection. Particulate: Extract within 14 days of sample collection, extract can hold up to 1 year for analysis.	SGS AXYS (British Columbia, Canada)	30 business days
PCB Congeners		EPA Method 1668C					
TAL Metals + Tin	Aqueous (whole water)	SW846 Method 6010/6020	1-liter HDPE	Pre-preserve with HNO3 to pH <2, ship on wet ice at less than 6 degrees Celsius	Digest and analyze within 6 months.	Katahdin (Scarborough, Maine)	21 calendar days

Sample Containers, Preservation, and Hold Time

Analytical Group	Matrix	Method SOP	Container Type	Preservation	Analytical Holding Time	Assigned Laboratory	Data Package Turnaround Time
TAL Metals + Tin	Aqueous (dissolved phase)	SW846 Method 6010/6020	1-liter HDPE	Unpreserved, ship on wet ice at less than 6 degrees Celsius	Lab filter 0.7 μm within 48 hours of sample collection, and then preserve with HNO3 to pH <2. Digest and analyze within 6 months.	Katahdin (Scarborough, Maine)	21 calendar days
TSS	Solid particulate on filter	USEPA Method 160.2 / SM2540D – modified	4-liter HDPE	Unpreserved, ship on wet ice at less than 6 degrees Celsius.	Lab-filter 0.7 μm within 48 hours of collection. Analyze within 7 days of collection.	Katahdin (Scarborough, Maine)	21 calendar days
POC	Solid particulate on filter	Lloyd Kahn – modified			Lab-filter 0.7 μm within 48 hours of collection (use TSS filtered particulates for analysis). Analyze within 28 days of collection.		

Note 1: Laboratory will hold NELAC certification.
Note 2: Technical Holding Time refers to the time of sample generation to sample extraction/digestion. Technical holding time will be suspended if sample is frozen below minus 10 degrees Celsius.

QAPP Worksheet 20
Field Quality Control Sample Summary

Sediment Sampling

Matrix	Analytical Group	Estimated Number of Field Samples	Estimated Number of Field Duplicates	Estimated Number of Lab Replicates	Estimated Number of Matrix Spike	Estimated Number of Matrix Spike Duplicate	Estimated Number of Performance Evaluation Sample	Estimated Number of Equipment Blank	Estimated Total Number of Samples to Lab
Sediment	PAH and Alkylated PAH	10	1	1	1	0	1	1	15
Sediment	PCB Congeners	10	1	1	0	0	1	1	14
Sediment	TAL Metals + Tin	10	1	1	1	0	0	1	14
Sediment	AVS/SEM	10	1	1	1	0	0	1	14
Sediment	TOC	10	1	1	1	0	1	1	15
Sediment	Grain Size	10	1	1	0	0	0	0	12

Note:
1. Equipment blank will be generated in the field by rinsing the field equipment with laboratory-grade water.

CSO Sampling

Matrix	Analytical Group	Estimated Number of Field Samples	Estimated Number of Field Duplicates	Estimated Number of Equipment Blank	Estimated Total Number of Samples to Lab	QAPP Note
Aqueous	PAH and Alkylated PAH	2 per event × 4 events per year = 8 per year	1 per year	1 per year	10 per year	Lab filtration to generate one phase (suspended particulate matter retained on filter)
Aqueous	PCB Congeners	2 per event × 4 events per year = 8 per year	1 per year	1 per year	10 per year	Lab filtration to generate one phase (suspended particulate matter retained on filter)
Aqueous	TAL Metals	2 per event × 4 events per year = 8 per year	1 per year	1 per year	10 per year	Two containers are collected and shipped: one for whole water and one for dissolved phase (lab filter)
Aqueous	TSS/POC	2 per event × 4 events per year = 8 per year	1 per year	1 per year	10 per year	Lab filtration

Note 1: A total of eight CSO sampling events are planned over 2-year monitoring program, yielding 16 samples in total (2 samples per event x 8 events = 16 samples)
Note 2: Equipment blank will be generated in the field by rinsing field equipment with laboratory-grade water.
Note 3: Refer to Worksheet 12 or Worksheet 28 for type and frequency of quality control samples under each analytical method.

QAPP Worksheet 21
Field Standard Operating Procedures

Field Standard Operating Procedures

Field SOP Reference Number	Title, Revision Date, and/or Number	Originating Organization	Equipment Type	Modified for Project Work?	Comments
SOP No. 1	Procedure to Locate Sample Points Using a Global Positioning System (GPS) (Revision 01, October 2021)	NYCDEP	Refer to SOP	No	Attachment 1
SOP No. 2	Procedure for Navigation and Boat Positioning (Revision 01, October 2021)	NYCDEP	Refer to SOP	No	Attachment 1
SOP No. 3	Procedure to Collect and Process Surface Sediment (Revision 01, October 2021)	NYCDEP	Refer to SOP	No	Attachment 1
SOP No. 4	Classification of Soil and Sediment (Revision 01, October 2021)	NYCDEP	Refer to SOP	No	Attachment 1
SOP No. 5	Procedure for the Decontamination of Sediment Sampling Equipment (Revision 01, October 2021)	NYCDEP	Refer to SOP	No	Attachment 1
SOP No. 6	Procedure for Management and Disposal of Investigative-Derived Waste (Revision 01, October 2021)	NYCDEP	Refer to SOP	No	Attachment 1
SOP No. 7	Procedure for Collection of Field Blank and Rinse Blank Samples (Revision 01, October 2021)	NYCDEP	Refer to SOP	No	Attachment 1
SOP No. 8	Procedure to Conduct Sample Management (Revision 01, October 2021)	NYCDEP	Refer to SOP	No	Attachment 1
SOP No. 9	Procedure for Collection of Large Volume – Small Volume CSO Samples (Revision 01, October 2021)	NYCDEP	Refer to SOP	No	Attachment 1
SOP No. 10	Procedure for Decanting Grain Size Samples (Revision 01, October 2021)	NYCDEP	Refer to SOP	No	Attachment 1
EPA540-R-07-06	USEPA Contract Laboratory Program Guidance for Field Samplers: Sampler's Guide, EPA540-R-014-013, October 2014	USEPA	Refer to referenced document	No	To be used by the field team as a guide for collecting and preparing samples

Field Standard Operating Procedures

Field SOP Reference Number	Title, Revision Date, and/or Number	Originating Organization	Equipment Type	Modified for Project Work?	Comments
Scribe User's Guide	ERT Comprehensive Training Manual for Scribe v3.8 (or latest version)	USEPA	Refer to referenced document	No	Scribe software will be used to manage samples and create COC; however, depending on field conditions, staff can generate a hand-written COC for sample shipment.
SOP No. HW-32	Standard Operating Procedure for Implementing The National Strategy for Procuring Analytical Services for All OSWER Programs (Superfund, RCRA, Brownfields), Revision 5, March 17, 2005	USEPA	Refer to referenced document	No	To be used by the field team as a guide for collecting and preparing samples

Refer to the following attachments for additional material:
Attachment 1: Field Standard Operating Procedures
Attachment 2: Laboratory Standard Operating Procedures
Attachment 3: Field Modification Form (Template)
Attachment 4: Non-conformance Form (Template)
Attachment 5: Example Chain of Custody (COC) for shipment of samples to laboratory
Attachment 6: Field Mobilization Protocol

QAPP Worksheet 22
Field Equipment Calibration, Maintenance, Testing, and Inspection

Field Equipment Calibration, Maintenance, Testing, and Inspection

Field Equipment	Calibration Activity	Maintenance Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	SOP Reference
Hand-Held GPS (Trimble or equivalent)	None	Battery and operation check performed before each use. Confirm sufficient satellite signal before each use; otherwise document uncertainty in coordinates	Check for signs of damage to casing or sensors. Record coordinates of fixed benchmark each day as a "daily check point".	Complete "daily check point" daily	Within manufacturer allowed tolerance. Position < 1 meter Uncertainty (assuming sufficient satellite signal).	Replace battery and check user manual	SOP No. 1
Peristaltic Pump	None	Battery and operation check performed before each use. Replace tubing between stations	Check for signs of damage	Check before each use	Flow rate ±10 percent of targeted flow	Replace battery, check user manual, and check tubing in pump	SOP No. 9

GPS

- Field crew will refer to SOP No. 01 and the GPS's operator manual.
- Quality control will be accomplished by using the GPS to navigate to known waypoints and by viewing plotted GPS field data.
- The GPS will be set to use accuracy-based logging. The GPS will not record data unless the satellites provide a minimum accuracy (user selectable value; 1 meter is the target value and will be adjusted if necessary depending on field conditions).

Peristaltic Pump

- Field crew will refer to the peristaltic pump's operator manual for troubleshooting and usage information.
- Equipment calibration is not necessary.

QAPP Worksheet 23
Analytical Standard Operating Procedures

Analytical SOP References Table (Worksheet 23)

Analytical SOP Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Assigned Laboratory	Modified for Project Work?
Modified SW846 Method 8270	Modified SW846 Method 8270 Laboratory SOP MLA-021 (Revision 12.07, October 2020) "Analytical Method for the Determination of Polycyclic Aromatic Hydrocarbons (PAH), Alkylated Polycyclic Aromatic Hydrocarbons, and Alkanes"	Definitive	Sediment, Aqueous, and Suspended particulate matter retained on filter / PAH and Alkylated PAH	HRGC/LRMS	SGS AXYS (British Columbia, Canada)	No
USEPA Method 1668C	USEPA Method 1668C Laboratory SOP MLA-010 (Revision 12.06, December 2020) "Analytical Method for the Determination of 209 PCB Congeners"	Definitive	Sediment, Aqueous, and Suspended particulate matter retained on filter / PCB Congeners	HRGC/HRMS	SGS AXYS (British Columbia, Canada)	No
SW846 Method 6010/6020	SW846 Method 6010/6020 Laboratory SOP CA-638 (Revision 02, June 2020) "Trace Metals Analysis by ICP-AES using USEPA Method 6010D" Laboratory SOP CA-627 (Revision 14, June 2020) "Trace Metals Analysis by ICP-MS using USEPA Method 6020"	Definitive	Sediment and Aqueous / TAL Metals	ICP-AES and ICP-MS	Katahdin (Scarborough, Maine)	No
SW846 Method 3050	SW846 Method 3050 Laboratory SOP CA-605 (Revision 10, June 2020) "Acid Digestion of Solid Samples by USEPA Method 3050 for Metals Analysis by ICP-AES, ICP-MS"	Definitive	Sediment / TAL Metals (preparation)	Not Applicable	Katahdin (Scarborough, Maine)	No

Analytical SOP References Table (Worksheet 23)

Analytical SOP Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Assigned Laboratory	Modified for Project Work?
SW846 Method 3010	SW846 Method 3010 Laboratory SOP CA-604 (Revision 09, January 2019) "Acid Digestion of Aqueous Samples by EPA Method 3010 for ICP and ICP-MS Analysis of Total or Dissolved Metals"	Definitive	Aqueous / TAL Metals (preparation)	Not Applicable	Katahdin (Scarborough, Maine)	No
EPA Draft Method 821-R-91-100	EPA Draft Method 821-R-91-100 Laboratory SOP CA-738 (Revision 4, January 2019) "Determination of Acid Volatile Sulfide and Simultaneously Extractable Metals in Sediments"	Definitive	Sediment / AVS-SEM	AVS: Colorimetric SEM: ICP-AES	Katahdin (Scarborough, Maine)	No
Lloyd Kahn	Lloyd Kahn Laboratory SOP CA-741 (Revision 09, September 2020) "Determination of Total Organic Carbon in Solids using the EPA Region II Method Lloyd Kahn and SW846 9060 MOD"	Definitive	Sediment / TOC Solid particulate on filter / POC	Combustion	Katahdin (Scarborough, Maine)	Yes for POC: see notes below
USEPA Method 160.2 / SM2540D	USEPA Method 160.2 / SM2540D Laboratory SOP CA-720 (Revision 09, June 2020) "Total Suspended Solids (Non-filterable Residue) by EPA Method 160.2 and Standard Method 2540D"	Definitive	Solid particulate on filter / TSS	Gravimetric	Katahdin (Scarborough, Maine)	Yes for TSS preparation: see notes below
Grain Size Distribution (ASTM D422)	ASTM D422 Laboratory SOP CA-551 (Revision 03, June 2020) "Grain Size Analysis"	Definitive	Sediment / Grain Size	Mechanical sieve and hydrometer	Katahdin (Scarborough, Maine)	No
Grain Size-SedTrend	Laboratory Standard Operating Procedure for Particle Size Analyses (Revision 4, June 2018)	Definitive	Aqueous / Grain Size	Mechanical sieve/laser size distribution	SedTrend (British Columbia, Canada)	No

Analytical SOP References Table (Worksheet 23)

Analytical SOP Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Assigned Laboratory	Modified for Project Work?
SM2540G	SM2540G Laboratory SOP CA-717 (Revision 11, February 2019) "Total Solids/Total Volatile Solids Determination in Solid Matrices"	Definitive	Sediment / Percent Moisture	Balance	Katahdin (Scarborough, Maine)	No
SW846 Method 7470A	SW846 Method 7470A Laboratory SOP CA-615-13 (Revision 13, February 2021) "Digestion and Analysis of Aqueous Samples for Mercury by USEPA Method 7470"	Definitive	Sediment / SEM-Mercury	CVAA	Katahdin (Scarborough, Maine)	No

Laboratory will analyze samples using the most recent SOP version (refer to laboratory data package for specific SOP version used to analyze field samples). Laboratory SOPs are available upon request.

Sediment Sampling Project Notes

Sample Handling

- Upon laboratory receipt, allow the sample to settle, remove as much standing water as practical using either centrifugation, siphoning, or decanting.
- After removal of standing water, homogenize the sediment thoroughly and use the resultant sediment for sample extraction prior to analysis.

Analyses Project Notes

- Laboratory should consider percent moisture of sediment samples before sub-aliquot sample mass for analyses and try to achieve project defined reporting limits.
- PCB Aroclors and homologue data will be provided by the laboratory as calculated concentrations using empirical formulas based on the PCB congener concentrations. PCB Congeners that are reported as Estimated Maximum Potential Concentrations (EMPCs) since the ratio of the quantitation and confirmation ions are not within theoretical limits (called non-ratioing peaks in the SGS-AXYS SOP) are not included in the calculations for Aroclors.
- PAH and PCB data reported should be recovery corrected using isotope dilution internal standard quantification.
- Metals analysis will include 23 TAL metals plus Tin (refer to Worksheet 15 for the list of metals). Aluminum, calcium, iron, magnesium, potassium, and sodium will be analyzed by SW846 Method 6010D; and the remaining metals will be analyzed by SW846 Method 6020A.
- AVS/SEM samples will be maintained as frozen before shipment. Upon laboratory receipt, laboratory should freeze samples as soon as possible and maintain frozen until analysis.
- TOC: laboratory must report results for all 4 replicates and the average when quadruplicate analyses are performed.
- TOC: Report data to 3 significant figures for values >QL.

- Percent moisture (or total solids) will be measured in sediments to convert chemical results to units on a dry-weight basis and to further characterize surface sediment samples.
- Grain Size: Grain size distribution will be characterized with sieving for particle size large then 75 μm and hydrometer for particle size smaller than 75 μm.

Sieve Number	Sieve Opening	Classification
Not applicable	75mm	Gravel: Retained on No. 4
Not applicable	50mm	
Not applicable	37.5mm	
Not applicable	25.0mm	
Not applicable	19.0mm	
Not applicable	6.3mm	
No. 4	4.75mm	
No. 10	2.00mm	Coarse Sand: Pass No. 4 and retained on No. 10
No. 20	0.850mm	Medium Sand: Pass No. 10 and retained on No. 40
No. 40	0.425mm	
No. 60	0.250mm	Fine Sand: Pass No. 40 and retained on No. 200
No. 80	0.180mm	
No. 100	0.150mm	
No. 200	0.075mm	
Hydrometer	0.005mm	Silt: Hydrometer 0.074mm to 0.005mm
Hydrometer	<0.005mm	Clay: Hydrometer <0.005mm

Surface Water Sampling Project Notes

PAH (parent and alkylated) and PCB Congeners Notes for Large Volume Aqueous Samples

- Laboratory shall decontaminate and solvent-clean with dichloromethane all bottle ware and filters (cartridge housing and flat filters) prior to collection and analysis of field samples. Laboratory shall decontaminate filtration system before filtration of each field sample and laboratory control sample.
- Laboratory shall filter the 20-liter sample to create the aqueous (dissolved)-phase sample and the solids (suspended particulate matter on filter) sample. Laboratory shall filter sample within 7 days of collection and refer to Worksheet 19 for maximum extract holding time.
- PCB Aroclor data (along with PCB Homologues) will be provided by the laboratory as calculated concentrations (empirical formula based on the PCB congener concentrations).
- Solids (Suspended Particulate Matter on Filter) Phase: The suspended solids will be captured on a 10 cm × 5 cm, 0.5 µm wound glass fiber filter (part number: G0.5A4A from General Filtration, Ontario, Canada) and on 142 mm diameter, 0.7 µm glass fiber flat filter(s) (Whatman: Model 1825-142). For

high TSS samples, solids in the cartridge housing and all of the flat filters will be extracted together using a Dean-Starke apparatus (with toluene) for PAH (parent and alkylated) and PCB Congeners. For low TSS samples, laboratory will be allowed to filter the samples with glass fiber flat filters and extract the filters with Soxhlet extraction (using dichloromethane). Visual assessment of the flow rate of the water through the system will determine when filters need to be changed. Volume at the time of filter change will be recorded by laboratory. Note that after the filters are extracted, the extract will be split one third for PAH, one third for PCB Congeners, and one third for archive. Additional cleanup may be conducted using column chromatography Biobeads.

- Data Reporting:
    - Laboratory will quantify the volume of the sample based on weight. The sample size will be used to calculate the final analyte concentration.
    - Solid-(suspended particulate matter on filter) phase concentration shall be "recovery corrected" and reported by the laboratory as mass of contaminant per liter of water filtered.

Metals Notes for Aqueous Samples

- Field crew will ship two 1-liter plastic containers for metals analysis (one container will be preserved and the other will be unpreserved). The preserved metals containers will be analyzed as an aqueous (whole water) sample for metals concentrations. The unpreserved metals container will be filtered and analyzed as an aqueous (dissolved) sample for metals concentrations.
- Laboratory shall filter metals and mercury samples using a 0.7 µm glass fiber flat filter (Fisher brand: SA1J078H00).
- Laboratory shall filter and preserve sample within 48 hours of collection (refer to Worksheet 19).
- Field duplicate samples will also be used to assess field precision.
- A filter blank and laboratory blank (method blank) will be submitted per SDG to assess potential impacts from the laboratory filtration process.
- Metals analyses will include TAL metals (including mercury) plus tin. Method 6010D (ICP-AES) will be used for the metals: calcium, iron, magnesium, potassium, and sodium. All other metals will be analyzed via method 6020A (ICP-MS).

TSS/POC Notes for Aqueous Samples

- Laboratory will use the Combustion Method (using methods listed above) to measure POC.
- TSS and POC will be measured from a single sample container of 4 liters (refer to Worksheet 19 for preservation and holding time).
- Project-Specific Method Modifications for TSS/POC:
    - Laboratory shall filter the entire 4-L sample through tared 0.7 µm glass fiber flat filter(s) (Fisher brand: SA1J078H00or equivalent). Multiple filters may be required for each sample.
    - If solids remain in the sample container, laboratory shall use the filtrate to rinse the sample bottle to remove solids (not distilled or laboratory water).
    - After filtration of the entire sample is complete, the laboratory will wash the filter with 3 successive 100-Ml laboratory deionized water washes prior to drying and weighing the filter for TSS.
    - The suspended particulates on the filters will be analyzed for POC via drying, grinding (ball-mill), and homogenizing the filter+particulates for analysis. HCl is added to remove inorganic carbon prior to combustion. The final POC value reported will be the average of 2 analytical replicate measurements (lab will also report the individual replicates).
- Data Reporting:
    - TSS: Laboratory shall use a spreadsheet to document the volume of water filtered through each flat filter. The final suspended solids value

reported in the Electronic data deliverable (EDD) and laboratory data package will be the total solids mass divided by the total volume of water filtered in units of mg/L.

Grain Size Project Notes for Aqueous Samples

- Laboratory shall not chemically pre-treat samples without prior authorization from client.
- Prior to every analysis, the laser (Mastersizer 2000) automatically aligns the laser beam, and a background measurement of the suspension medium is recorded.
- Samples are introduced into the dispersion unit by wet sieving through a 1 mm mesh, eliminating possible blockage of the pumping mechanism by particles that are too large. Disaggregation of the sample is achieved by both mechanical stirring and mild ultrasonic dispersion in the sample-handling unit. If material remains on the 1-mm sieve, then the weight percent for each of the coarse sizes (-2.0Ø to 0.5Ø, which is equivalent to 4.0 mm to 0.7 mm) is obtained by dry sieving at 0.5Ø intervals. The overlap in sizes between the laser and sieve data allow the two distributions to be merged into one complete grain-size distribution.
- Reported grain size distribution will cover a range of 0.02 µm to 4 mm.

QAPP Worksheet 24 and Worksheet 25
Analytical Instrument Calibration

Analytical Instrument Calibration Table (Worksheet 24)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
HRGC/LRMS PAH and Alkylated PAH	Mass Resolution using PFTBA Initial calibration (ICAL) minimum 5-pt; Calibration verification (CCV)	Mass Resolution: daily ICAL: prior to sample analysis and when CCVs fail; ICV: immediately after ICAL; CCV: initially and every 12 hours with closing sequence CCV	Mass Resolution: per SOP Instrument Sensitivity: S:N ≥ 3:1 for 10 pg of acenaphthene and dibenzo(a,h)85nthracene ICAL: %RSD ≤ 20% (35% for targets with non-labeled analog) CCV: %D ≤ ± 25%; opening and closing sequence CCV RRFs RPD ≤ 40%	Perform instrument maintenance; re-prepare and/or reanalyze calibration standards; re-analyze samples, as necessary	Analyst or SGS Axys Lab Supervisor	USEPA SW-846 Modified 8270 for HRGC/LRMS Laboratory SOP MLA-021

Analytical Instrument Calibration Table (Worksheet 24)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
HRGC/HRMS PCB Congeners	Mass Resolution using PFK Initial calibration (ICAL) minimum 5-pt Calibration verification (CCV)	Mass Resolution: at the beginning and end of each shift ICAL: prior to sample analysis and when CCVs fail CCV: at the beginning of each 12-hr shift	Mass Resolution: meet criteria per Table 7 of Method 1668C Instrument Sensitivity: S:N ≥ 10:1 for calibration standards Ion Ratios for all Congeners within ±15% of theoretical values in CS 0.5 ICAL: %RSD ≤ 20% CCV: %D ≤ ± 25% (35% for several labeled surrogates as defined in SOP MLA-010))	Perform instrument maintenance; re-prepare and/or reanalyze calibration standards; re-analyze samples, as necessary	Analyst or SGS Axys Lab Supervisor	USEPA Method 1668 Laboratory SOP MLA-010
ICP-MS Metals	Tune Initial calibration (ICAL) minimum 4-pt Independent calibration verification (ICV) Calibration verification (CCV)	Tune: daily prior to ICAL ICAL: prior to sample analysis and when CCVs fail ICV: immediately after ICAL CCV: prior to sample analysis; repeated every 10 samples	Tune: criteria per Method 6020 ICAL: correlation coefficient r2 ≥ 0.998 ICV & CCV: %D ≤ ± 10%	Perform instrument maintenance; re-prepare and/or reanalyze calibration standards; re-analyze samples, as necessary	Analyst or Katahdin Lab Supervisor	USEPA SW-846 Method 6020 Laboratory SOP CA-627

Analytical Instrument Calibration Table (Worksheet 24)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
ICP-AES Metals	Initial calibration (ICAL) 2-pt (cal std + blank) Independent calibration verification (ICV) Calibration verification (CCV)	ICAL: prior to sample analysis and when CCVs fail ICV: immediately after ICAL CCV: prior to sample analysis; repeated every 10 samples	ICAL: correlation coefficient r2 ≥ 0.998 ICV & CCV: %D ≤ ± 10%	Perform instrument maintenance; re-prepare and/or reanalyze calibration standards; re-analyze samples, as necessary	Analyst or Katahdin Lab Supervisor	USEPA SW-846 Method 6010 Laboratory SOP CA-608
Colorimetric AVS	Initial calibration (ICAL) minimum 5-pt Independent calibration verification (ICV) Calibration verification (CCV)	ICAL: prior to sample analysis and when CCVs fail ICV: immediately after ICAL CCV: prior to sample analysis; repeated every 10 samples	ICAL: correlation coefficient r2 ≥ 0.995 ICV & CCV: %D ≤ ± 20%	Perform instrument maintenance, as needed; re-prepare and/or reanalyze calibration standards; re-analyze samples, as necessary	Analyst or Katahdin Lab Supervisor	USEPA Draft Method EPA 821/R-91-100 Laboratory SOP CA-738
CVAA Mercury	Initial calibration (ICAL) 5-pt Calibration verification (CCV)	ICAL: prior to sample analysis and when CCVs fail ICV: immediately after ICAL CCV: prior to sample analysis; repeated every 10 samples	ICAL: correlation coefficient r2 $\geq$ 0.995 ICV/CCV: %D $\leq \pm$ 10%	Perform instrument maintenance, as needed; re-prepare and/or reanalyze calibration standards; re-analyze samples, as necessary	Analyst or Katahdin Lab Supervisor	USEPA SW-846 Method 7470 Laboratory SOP CA-615

Analytical Instrument Calibration Table (Worksheet 24)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
Combustion/IR TOC POC	Initial calibration (ICAL) Independent calibration verification (ICV) Calibration verification (CCV)	ICAL: prior to sample analysis and when CCVs fail ICV: immediately after ICAL CCV: prior to sample analysis; repeated every 10 samples	ICAL: correlation coefficient $r^2 \geq 0.995$ ICV & CCV: %D ≤ ± 20%	Perform instrument maintenance, as needed; re-prepare and/or reanalyze calibration standards; re-analyze samples, as necessary	Analyst or Katahdin Lab Supervisor	USEPA Region 2 Lloyd Kahn Method & Gustafsson et al., 1997 Laboratory SOP CA-741
Gravimetric Balance TSS	Balance verification with three weights (5.0 grams, 2.0 grams, and 0.2 gram weights).	Daily	Within ± 0.0002g of true value	Perform instrument maintenance, as needed; re- calibrate; re-analyze samples, as necessary	Analyst or Katahdin Lab Supervisor	EPA Method 160.2 Laboratory SOP CA-720
Balance	Refer to manual for calibration of gravimetric balance	ICAL: prior to sample analysis. Minimum of 1-point calibration curve.	ICAL: ±0.01 grams	Investigate and identify problem. Recalibrate and reanalyze affected samples.	Analyst or Katahdin Lab Supervisor	ASTM D422
Mechanical Sieve Shaker	Refer to manual for mechanical sieve shaker	ICAL: prior to sample analysis.	ICAL: minimum 10,000 rpm	Investigate and identify problem. Recalibrate and reanalyze affected samples.	Analyst or Katahdin Lab Supervisor	ASTM D422

Analytical Instrument Calibration Table (Worksheet 24)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
Hydrometer in Sedimentation Cylinder	Refer to manual for hydrometer	ICAL: prior to sample analysis. Set up a control hydrometer prior to sample analysis	Temperature (±0.5°C) and meniscus (±0.0005)correction in Control Hydrometer. Recorded Time: ±20 sec	Investigate and identify problem. Recalibrate and reanalyze affected samples.	Analyst or Katahdin Lab Supervisor	ASTM D422
Mechanical Sieve/Laser Size Distribution	Calibrate as required in SOP	The system is tested for accuracy and proper function for every 300 analyses	Calibrate Dv10, Dv50, and Dv90 within manufacture tolerate limit for Mastersizer 2000 optical unit and sample handing unit	Investigate and identify problem. Recalibrate and reanalyze affected samples.	Analyst or SedTrend Lab Supervisor	Laboratory Standard Operating Procedure for Particle Size Analyses (Revision 4, June 2018)

QAPP Worksheet 26 and Worksheet 27
Sample Handling, Custody, and Disposal

Sample Handling, Custody, and Disposal

Activity	Organization and Responsible Person for Activity	SOP Reference Number	Comments
Sample labeling and custody seal	The investigative organization field team	SOP No. 08	Refer to Note 1
COC completion	The investigative organization field team	SOP No. 08	Refer to Note 1
Cooler packaging	The investigative organization field team	SOP No. 08	Refer to Attachment 1
Shipping coordination	The investigative organization field team and laboratory sample custodian	Refer to Worksheet 6 for communication pathways	Refer to Worksheet 6 for communication pathways
Sample receipt, inspection, and login	Assigned laboratory	Refer to Worksheet 23 for analytical method	Sample receipt, inspection, and login will be conducted per Laboratory Quality Manual. Copy of login will be sent to the investigative organization (NYCDEP contractor).
Sample custody and storage	Assigned laboratory	Refer to Worksheet 23 for analytical method	Sample custody and storage will be conducted per Laboratory Quality Manual. Copy of signed COC will be sent to the investigative organization (NYCDEP contractor).
Sample disposal	Assigned laboratory	Refer to Worksheet 23 for analytical method	At least 90 days for disposal

Note 1: SCRIBE software will be used to manage samples and create COC; however, depending on field conditions, staff can generate a hand-written COC for sample shipment.

Sample Identification System

All samples collected from the Site must be identified with a sample label, secured with custody seal, and tracked on the COC. Indelible ink will be used to complete sample labels (if needed). Sample labels will require (at a minimum) the following information for each sample container (refer to Worksheet 18 for sample ID scheme):

1. Project Name
2. Sample ID
3. Sample Matrix
4. Analytical Group
5. Sample Date and Time
6. Preservation

Chain of Custody Procedure

Sample custody procedures ensure the timely, correct, and complete analysis of each sample for all parameters requested. A sample is considered to be in someone's custody if it:

- Is in his/her possession.
- Is in his/her view, after being in his/her possession.
- Is in his/her possession and has been placed in a secured location.
- Is in a designated secure area.

Sample custody documentation provides a written record of sample collection and analysis. The sample custody procedures provide for specific identification of samples associated with an exact location, the recording of pertinent information associated with the sample (including time of sample collection and any preservation techniques), and a COC record that serves as physical evidence of sample custody. Scribe software will be used to generate COC, create sample labels, and manage tracking of samples. If needed, handwritten COCs will be generated; indelible ink will be used to complete sample labels (if needed). Custody procedures will be similar to the procedures outlined in *USEPA CLP Guidance for Field Samplers* (USEPA, 2014). The COC documentation system provides the means to individually identify, track, and monitor each sample from the time of collection through final data reporting. The following information (at a minimum) will be included on the COC forms. All COC forms must be signed in ink:

- Project Name
- Signature of sampler
- Sample ID
- Date and Time of collection
- Sample matrix
- Analyses required
- Preservation (if applicable).
- Air express/shipper's bill of lading identification numbers

The COC form serves as an official communication to the laboratory detailing the particular analyses required for each sample. The COC record will accompany the samples from the time of sampling through all transfers of custody. It will be kept on file at the laboratory where samples are analyzed and archived. An electronic or hard-copy of each COC should be also made and kept in the project electronic-file directory. The sample management team completes a COC record to accompany each shipment from the field to the laboratory. The completed COC is placed in a zip-lock bag and taped to the inside cover of the sample shipping container. If there is more than one container in a shipment, copies of the COC form will be placed in each container. Each container is then sealed with signed custody seals and custody is transferred to the laboratory.

Field Sample Handling, Custody Procedures, and Shipment

Field records provide a means of recording information for each field activity performed at the Site. COC procedures document pertinent sampling data and all transfers of custody until the samples reach the analytical laboratory. The sample packaging and shipment procedures summarized below will ensure that the samples arrive at the laboratory with the COC intact. Refer to SOP No. 08 in Attachment 1 for sample management information. Worksheet 19 lists the specific

sample preservation requirements for each test method. The general responsibilities of the field team are listed below:

- The field team is personally responsible for the care and custody of the samples until they are transferred to the sample management team until they are properly dispatched. As few people as possible should handle the samples.
- The investigative organization field team is responsible for entering the proper information on the field forms, including all pertinent information such as sample identification number, date and time of sample collection, type of analysis, and description of sample location. The information entered on the field form will be used to generate a COC.
- All sample containers will be labeled with the project identification, sample number, matrix, and type of analysis required.
- The samples will be properly preserved, bagged, and packed into coolers. The original COC form will be placed into the lead cooler and will be shipped to the laboratory.
- The sample management team of the investigative organization will review all samples to determine whether proper custody procedures were followed.

The custody of samples must be maintained from the time of sampling through shipment and relinquishment to the laboratory. Instructions for transferring custody are given below:

- All samples are accompanied by a COC. When transferring custody of samples, the individuals relinquishing and receiving will sign, date, and note the time on the COC. This form documents sample custody transfer from the sample management team, through the shipper, to the analytical laboratory. Since a common carrier will usually not accept responsibility for handling COC forms, the name of the carrier is entered under "Received by," the bill-of-lading number is recorded in the comments section, and the COC form is placed in a zip-lock plastic bag and taped to the inside lid of the lead shipping cooler. Copies of the COC form will be placed in each additional cooler in a shipment.
- Samples will be packaged for shipment and either picked up at the Site by the laboratory or dispatched to the appropriate laboratory via standard delivery service. SOP No. 08 in Attachment 1 contain the proper sample packaging techniques. A separate COC record must accompany each shipment. Shipping containers will be sealed for shipment to the laboratory. Two custody seals will be applied to each cooler to document that the container was properly sealed and to determine if the container was tampered with during shipment. The signed custody seals will be placed on the coolers in such a manner that the custody seal would be broken if the cooler were opened (*i.e.*, diagonally opposite corners of the cooler lid).
- The original COC will accompany the shipment. A copy (electronic or hardcopy) will be retained at the investigative organization offices.
- If the samples are sent by common carrier or air freight, proper documentation must be maintained. For example, the bill of lading must be retained by the Field Team Leader.

Laboratory Sample Receipt

A designated sample custodian accepts custody of the samples and verifies that the information on the sample labels matches the information on the COC. The sample custodian will document any discrepancies and will sign and date all appropriate receiving documents. The sample custodian will also document the condition of the samples upon receipt at the laboratory. Once the samples have been accepted by the laboratory, checked and logged in, they must be maintained in accordance with laboratory custody and security requirements. To ensure traceability of samples while in the possession of the laboratory, a method for sample identification that has been documented in a laboratory SOP will be used to assign sample numbers. The following stages of analysis must be documented by the laboratory: Sample Extraction/Preparation, Sample Analysis, Data Reduction, and Data Reporting. Laboratory personnel are responsible for the custody of samples until they are returned to the sample custodian.

Laboratory Custody Procedures

The laboratory custody procedures will be equivalent to those described in the latest edition of the laboratory custody SOPs, including:

- A designated sample custodian accepts custody of the samples and verifies that the information on the sample labels matches the information on the COC. The sample custodian will document any discrepancies and will sign and date all appropriate receiving documents. The sample custodian will also document the condition of the samples upon receipt at the laboratory.
- Once the samples have been accepted by the laboratory, checked and logged in, they must be maintained in accordance with laboratory custody and security requirements.
- To ensure traceability of samples while in the possession of the laboratory, a method for sample identification that has been documented in a laboratory SOP will be used to assign sample numbers.
- The following stages of analysis must be documented by the laboratory:
    - Sample Extraction/Preparation.
    - Sample Analysis.
    - Data Reduction.
    - Data Reporting.
- Laboratory personnel are responsible for the custody of samples until they are returned to the sample custodian.
- When sample analyses and quality assurance checks have been completed in the laboratory, the used portion of the sample must be stored or disposed of in accordance with the protocols specified in the Laboratory Quality Manual. Identifying labels, data sheets, COCs, and laboratory records will be retained until analyses and quality assurance checks are completed in accordance with the protocols specified in the Laboratory Quality Manual.

Final Evidence Files

In the final phase of sample custody, the COC records and sample analysis request form copies are archived in their respective project files. Laboratory custody forms, sample preparation and analysis logbooks, and data packages will become part of the laboratory final evidence file. Other relevant documentation including records, reports, and correspondence, logs, pictures, and data review reports will be archived by the investigative organization (NYCDEP contractor).

QAPP Worksheet 28-1
PAH and Alkylated PAH (Sediment) Analytical Quality Control and Corrective Action

Matrix: Sediment
Analytical Group: PAH and Alkylated PAH
Analytical Method/SOP: Modified Method 8270D / Lab SOP MLA-021

QC Sample	Number / Frequency	Method / SOP Acceptance Criteria	Corrective Action	Title / Position of person responsible for corrective action	Project-Specific MPC
Method Blank	1 per prep batch of up to 20 samples	Results < QL	Report field samples that are < QL or 10x blank contamination if blank > QL; otherwise reanalyze affected samples and add laboratory qualifiers as needed.	Laboratory Analyst	Results < QL
Equipment Blank	1 per 20 field samples	QAPP	Investigate source of potential field contamination and qualify data according to Worksheet 36.	Project Manager or Site QC Officer and Data Validator	Results < QL
Surrogates	added to all field and QC samples	SGS AXYS SOP MLA-021 specified control limits	Investigate and resolve problem, reanalyze affected samples.	Laboratory Analyst	Recoveries within SGS AXYS SOP MLA-021 specified control limits
Laboratory Control Sample (LCS)	1 per prep batch of up to 20 samples	SGS AXYS SOP MLA-021 specified control limits	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	Recoveries within SGS AXYS SOP MLA-021 specified control limits
Matrix Spike (MS)	1 per prep batch of up to 20 samples	SGS AXYS SOP MLA-021 specified control limits	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	Recoveries within SGS AXYS SOP MLA-021 specified control limits unless sample conc. > 4x spike level (spike swamped out)
Laboratory Replicate	1 per prep batch of up to 20 samples	RPD ≤ 40%	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	RPD ≤ 40% if reported values are ≥ 5x QL; otherwise use professional judgement

QC Sample	Number / Frequency	Method / SOP Acceptance Criteria	Corrective Action	Title / Position of person responsible for corrective action	Project-Specific MPC
Certified Reference Material (NIST 1944)	1 per prep batch of up to 20 samples	QAPP	Investigate and resolve problem, reanalyze batch if laboratory fails measurement performance criteria, and add laboratory qualifiers as needed.	Laboratory Analyst	Reported concentrations within ±30% of certified and reference values. Applicable for values that are ≥ 3x concentration of the lowest calibration point in the ICAL.
Field Duplicate	1 per 20 field samples	QAPP	Data validation will qualify data according to Worksheet 36.	Project Manager or Site QC Officer and data validator	RPD ≤ 50% if reported values are ≥ 5x QL; otherwise use professional judgement
Temperature Blank	1 per cooler shipment	QAPP	Continue to analyze sample; validator will qualify data according to Worksheet 36.	Laboratory and data validator	0 - 6 degrees Celsius

QAPP Worksheet 28-2
PCB Congeners (Sediment) Analytical Quality Control and Corrective Action

Matrix: Sediment
Analytical Group: PCB Congeners
Analytical Method/SOP: USEPA Method 1668C/Lab SOP MLA-010

QC Sample	Number / Frequency	Method / SOP Acceptance Criteria	Corrective Action	Title / Position of person responsible for corrective action	Project-Specific MPC
Method Blank	1 per prep batch of up to 20 samples	Results < QL	Report field samples that are < QL or 10x blank contamination if blank > QL; otherwise reanalyze affected samples and add laboratory qualifiers as needed.	Laboratory Analyst	Results < QL
Equipment Blank	1 per 20 field samples	QAPP	Investigate source of potential field contamination and qualify data according to Worksheet 36.	Project Manager or Site QC Officer and Data Validator	Results < QL
Surrogates	added to all field and QC samples	SGS AXYS SOP MLA-010 specified control limits	Investigate and resolve problem, reanalyze affected samples.	Laboratory Analyst	Recoveries within SGS AXYS SOP MLA-010 specified control limits
Laboratory Control Sample (LCS)	1 per prep batch of up to 20 samples	SGS AXYS SOP MLA-010 specified control limits	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	Recoveries within SGS AXYS SOP MLA-010 specified control limits
Laboratory Replicate	1 per prep batch of up to 20 samples	RPD ≤ 40%	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	RPD ≤ 40% if reported values are ≥ 5x QL; otherwise use professional judgement
Certified Reference Material (NIST 1944)	1 per prep batch of up to 20 samples	QAPP	Investigate and resolve problem, reanalyze batch if laboratory fails measurement performance criteria, and add laboratory qualifiers as needed.	Laboratory Analyst	Reported concentrations within ±30% of certified and reference values. Applicable for values that are ≥ 3x concentration of the lowest calibration point in the ICAL and only applicable to non-coeluting congeners

Matrix: Sediment
Analytical Group: PCB Congeners
Analytical Method/SOP: USEPA Method 1668C/Lab SOP MLA-010

QC Sample	Number / Frequency	Method / SOP Acceptance Criteria	Corrective Action	Title / Position of person responsible for corrective action	Project-Specific MPC
Field Duplicate	1 per 20 field samples	QAPP	Data validation will qualify data according to Worksheet 36.	Project Manager or Site QC Officer and data validator	RPD ≤ 50% if reported values are ≥ 5x QL; otherwise use professional judgement
Temperature Blank	1 per cooler shipment	QAPP	Continue to analyze sample; validator will qualify data according to Worksheet 36.	Laboratory and data validator	0 - 6 degrees Celsius

QAPP Worksheet 28-3
TAL Metals + Tin (Sediment) Analytical Quality Control and Corrective Action

Matrix: Sediment
Analytical Group: TAL Metals + Tin
Analytical Method/SOP: SW-846 Methods 6010D & 6020A

QC Sample	Number / Frequency	Method / SOP Acceptance Criteria	Corrective Action	Title / Position of person responsible for corrective action	Project-Specific MPC
Method Blank	1 per prep batch of up to 20 samples	Results < QL	Report field samples that are < QL or 10x blank contamination if blank > QL; otherwise reanalyze affected samples and add laboratory qualifiers as needed.	Laboratory Analyst	Results < QL
Equipment Blank	1 per 20 field samples	QAPP	Investigate source of potential field contamination and qualify data according to Worksheet 36.	Project Manager or Site QC Officer and Data Validator	Results < QL
Laboratory Control Sample (LCS)	1 per prep batch of up to 20 samples	Katahdin SOP specified control limits	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	%Rec 80-120% or vendor certified limits if SRM used as LCS
Matrix Spike (MS)	1 per prep batch of up to 20 samples	%Rec 75-125%	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	%Rec 75-125% unless sample conc. > 4x spike level (spike swamped out)
Laboratory Replicate	1 per prep batch of up to 20 samples	RPD ≤ 25%	Investigate and resolve problem, reanalyze affected samples, and add laboratory qualifiers as needed.	Laboratory Analyst	RPD ≤ 25% if reported values are ≥ 5x QL; Results <5xQL: absolute value of difference < ±2xQL
Field Duplicate	1 per 20 field samples	QAPP	Data validation will qualify data according to Worksheet 36.	Project Manager or Site QC Officer and data validator	RPD ≤ 50% if reported values are ≥ 5x QL; otherwise use professional judgement

Matrix: Sediment
Analytical Group: TAL Metals + Tin
Analytical Method/SOP: SW-846 Methods 6010D & 6020A

QC Sample	Number / Frequency	Method / SOP Acceptance Criteria	Corrective Action	Title / Position of person responsible for corrective action	Project-Specific MPC
Temperature Blank	1 per cooler shipment	QAPP	Continue to analyze sample; validator will qualify data according to Worksheet 36.	Laboratory and data validator	0 - 6 degrees Celsius

QAPP Worksheet 28-4
AVS/SEM (Sediment) Analytical Quality Control and Corrective Action

Matrix: Sediment
Analytical Group: Acid Volatile Sulfides & Simultaneously Extracted Metals (AVS/SEM)
Analytical Method/SOP: USEPA Draft Method 821-R-91-100 & SW-846 Methods 6010C & 7470A

QC Sample	Number / Frequency	Method / SOP Acceptance Criteria	Corrective Action	Title / Position of person responsible for corrective action	Project-Specific MPC
Method Blank	1 per prep batch of up to 20 samples	Results < QL	Report field samples that are < QL or 10x blank contamination if blank > QL; otherwise reanalyze affected samples and add laboratory qualifiers as needed.	Laboratory Analyst	Results < QL
Equipment Blank	1 per 20 field samples	QAPP	Investigate source of potential field contamination and qualify data according to Worksheet 36.	Project Manager or Site QC Officer and Data Validator	Results < QL
Laboratory Control Sample (LCS)	1 per prep batch of up to 20 samples	Katahdin SOP specified control limits	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	%Rec 80-120%
Matrix Spike (MS)	1 per prep batch of up to 20 samples	%Rec 75-125%	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	%Rec 75-125% unless sample conc. > 4x spike level (spike swamped out)
Laboratory Replicate	1 per prep batch of up to 20 samples	RPD $\leq$ 25%	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	RPD ≤ 25% if reported values are≥ 5x QL; Results <5xQL: absolute value of difference < ±2xQL
Field Duplicate	1 per 20 field samples	QAPP	Data validation will qualify data according to Worksheet 36.	Project Manager or Site QC Officer and data validator	RPD ≤ 50% if reported values are ≥ 5x QL; otherwise use professional judgement

Matrix: Sediment
Analytical Group: Acid Volatile Sulfides & Simultaneously Extracted Metals (AVS/SEM)
Analytical Method/SOP: USEPA Draft Method 821-R-91-100 & SW-846 Methods 6010C & 7470A

QC Sample	Number / Frequency	Method / SOP Acceptance Criteria	Corrective Action	Title / Position of person responsible for corrective action	Project-Specific MPC
Temperature Blank	1 per cooler shipment	QAPP	Continue to analyze sample; validator will qualify data according to Worksheet 36.	Laboratory and data validator	0 - 6 degrees Celsius

QAPP Worksheet 28-5
TOC (Sediment) Analytical Quality Control and Corrective Action

Matrix: Sediment
Analytical Group: Total Organic Carbon (TOC)
Analytical Method/SOP: EPA Region II Lloyd Kahn method

QC Sample	Number / Frequency	Method / SOP Acceptance Criteria	Corrective Action	Title / Position of person responsible for corrective action	Project-Specific MPC
Method Blank	1 per prep batch of up to 20 samples	Results < QL	Report field samples that are < QL or 10x blank contamination if blank > QL; otherwise reanalyze affected samples and add laboratory qualifiers as needed.	Laboratory Analyst	Results < QL
Equipment Blank	1 per 20 field samples	QAPP	Investigate source of potential field contamination and qualify data according to Worksheet 36.	Project Manager or Site QC Officer and Data Validator	Results < QL
Laboratory Control Sample (LCS)	1 per prep batch of up to 20 samples	%Rec 80-120%	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	%Rec 80-120%
Matrix Spike (MS)	1 per prep batch of up to 20 samples	%Rec 75-125%	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	%Rec 75-125% unless sample conc. > 4x spike level (spike swamped out)
Laboratory Quadruplicate	1 per prep batch of up to 20 samples	%RSD ≤ 30%	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	%RSD ≤ 30%
Certified Reference Material (NIST 1944)	1 per prep batch of up to 20 samples	QAPP	Investigate and resolve problem, reanalyze batch if laboratory fails measurement performance criteria, and add laboratory qualifiers as needed.	Laboratory Analyst	Reported concentrations within ±30% of certified TOC value

Matrix: Sediment
Analytical Group: Total Organic Carbon (TOC)
Analytical Method/SOP: EPA Region II Lloyd Kahn method

QC Sample	Number / Frequency	Method / SOP Acceptance Criteria	Corrective Action	Title / Position of person responsible for corrective action	Project-Specific MPC
Field Duplicate	1 per 20 field samples	QAPP	Data validation will qualify data according to Worksheet 36.	Project Manager or Site QC Officer and data validator	RPD ≤ 50% if reported values are ≥ 5x QL; otherwise use professional judgement
Temperature Blank	1 per cooler shipment	QAPP	Continue to analyze sample; validator will qualify data according to Worksheet 36.	Laboratory and data validator	0 - 6 degrees Celsius

QAPP Worksheet 28-6
Grain Size (Sediment) Analytical Quality Control and Corrective Action

Matrix: Sediment
Analytical Group: Grain Size Distribution
Analytical Method/SOP: ASTM D422-63

QC Sample	Number / Frequency	Method / SOP Acceptance Criteria	Corrective Action	Title / Position of person responsible for corrective action	Project-Specific MPC
Laboratory Replicate	1 per prep batch of up to 20 samples	RPD ≤ 40%	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	RPD ≤ 40%
Field Duplicate	1 per 20 field samples	QAPP	Continue to analyze samples and narrate issue.	Project Manager or Site QC Officer	RPD ≤ 50%

QAPP Worksheet 28-7
PAH and Alkylated PAH (Suspended Particulates) Analytical Quality Control and Corrective Action

Matrix: Suspended Particulate Material on Filter
Analytical Group: PAH and Alkylated PAH
Analytical Method/SOP: Modified Method 8270D / Lab SOP MLA-021

QC Sample	Number / Frequency	Method / SOP Acceptance Criteria	Corrective Action	Title / Position of person responsible for corrective action	Project-Specific MPC
Method Blank	1 per prep batch of up to 20 samples	Results < QL	Report field samples that are < QL or 10x blank contamination if blank > QL; otherwise reanalyze affected samples and add laboratory qualifiers as needed.	Laboratory Analyst	Results < QL
Equipment Blank	1 every 4 events	QAPP	Investigate source of potential field contamination and qualify data according to Worksheet 36.	Project Manager or Site QC Officer and Data Validator	Results < QL
Surrogates	added to all field and QC samples	SGS AXYS SOP MLA-021 specified control limits	Investigate and resolve problem, reanalyze affected samples.	Laboratory Analyst	Recoveries within SGS AXYS SOP MLA-021 specified control limits
Laboratory Control Sample (LCS)	1 per prep batch of up to 20 samples	SGS AXYS SOP MLA-021 specified control limits	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	Recoveries within SGS AXYS SOP MLA-021 specified control limits
Laboratory Control Sample Duplicate (LCSD)	1 per prep batch of up to 20 samples	SGS AXYS SOP MLA-021 specified control limits LCS/LCSD RPD ≤ 40%	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	Recoveries within SGS AXYS SOP MLA-021 specified control limits LCS/LCSD RPD ≤ 40%
Certified Reference Material (NIST 1944)	first SDG of the monitoring year (or 1 per 20 samples)	QAPP	Investigate and resolve problem, reanalyze batch if laboratory fails measurement performance criteria, and add laboratory qualifiers as needed.	Laboratory Analyst	Reported concentrations within ±30% of certified and reference values. Applicable for values that are ≥ 3x concentration of the lowest calibration point in the ICAL.

Matrix: Suspended Particulate Material on Filter
Analytical Group: PAH and Alkylated PAH
Analytical Method/SOP: Modified Method 8270D / Lab SOP MLA-021

QC Sample	Number / Frequency	Method / SOP Acceptance Criteria	Corrective Action	Title / Position of person responsible for corrective action	Project-Specific MPC
Field Duplicate	1 every 4 events	QAPP	Investigate and resolve problem, reanalyze batch if laboratory fails measurement performance criteria, and add laboratory qualifiers as needed.	Project Manager or Site QC Officer and data validator	RPD ≤ 50% if reported values are ≥ 5x QL; otherwise use professional judgement
Temperature Blank	1 per cooler shipment	QAPP	Continue to analyze sample; validator will qualify data according to Worksheet 36.	Laboratory and data validator	0 - 6 degrees Celsius

QAPP Worksheet 28-8
PCB Congeners (Suspended Particulates) Analytical Quality Control and Corrective Action

Matrix: Particulate Material on Filter
Analytical Group: PCB Congeners
Analytical Method/SOP: USEPA Method 1668C/Lab SOP MLA-010

QC Sample	Number / Frequency	Method / SOP Acceptance Criteria	Corrective Action	Title / Position of person responsible for corrective action	Project-Specific MPC
Method Blank	1 per prep batch of up to 20 samples	Results < QL	Report field samples that are < QL or 10x blank contamination if blank > QL; otherwise reanalyze affected samples and add laboratory qualifiers as needed.	Laboratory Analyst	Results < QL
Equipment Blank	1 every 4 events	QAPP	Investigate source of potential field contamination and qualify data according to Worksheet 36.	Project Manager or Site QC Officer and Data Validator	Results < QL
Surrogates	added to all field and QC samples	SGS AXYS SOP MLA-010 specified control limits	Investigate and resolve problem, reanalyze affected samples.	Laboratory Analyst	Recoveries within SGS AXYS SOP MLA-010 specified control limits
Laboratory Control Sample (LCS)	1 per prep batch of up to 20 samples	SGS AXYS SOP MLA-010 specified control limits	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	Recoveries within SGS AXYS SOP MLA-010 specified control limits
Laboratory Control Sample Duplicate (LCSD)	1 per prep batch of up to 20 samples	SGS AXYS SOP MLA-010 specified control limits LCS/LCSD RPD ≤ 40%	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	Recoveries within SGS AXYS SOP MLA-010 specified control limits LCS/LCSD RPD ≤ 40%
Certified Reference Material (NIST 1944)	first SDG of the monitoring year (or 1 per 20 samples)	QAPP	Investigate and resolve problem, reanalyze batch if laboratory fails measurement performance criteria, and add laboratory qualifiers as needed.	Laboratory Analyst	Reported concentrations within ±30% of certified and reference values. Applicable for values that are ≥ 3x concentration of the lowest calibration point in the ICAL and only applicable to non-

Matrix: Particulate Material on Filter
Analytical Group: PCB Congeners
Analytical Method/SOP: USEPA Method 1668C/Lab SOP MLA-010

QC Sample	Number / Frequency	Method / SOP Acceptance Criteria	Corrective Action	Title / Position of person responsible for corrective action	Project-Specific MPC
					coeluting congeners.
Field Duplicate	1 every 4 events	QAPP	Data validation will qualify data according to Worksheet 36.	Project Manager or Site QC Officer and data validator	RPD ≤ 50% if reported values are ≥ 5x QL; otherwise use professional judgement
Temperature Blank	1 per cooler shipment	QAPP	Continue to analyze sample; validator will qualify data according to Worksheet 36.	Laboratory and data validator	0 - 6 degrees Celsius

QAPP Worksheet 28-9
TAL Metals + Tin (Whole Water & Dissolved Water) Analytical Quality Control and Corrective Action

Matrix: Whole Water & Dissolved Water
Analytical Group: TAL Metals + Tin
Analytical Method/SOP: SW-846 Methods 6010D & 6020A

QC Sample	Number / Frequency	Method / SOP Acceptance Criteria	Corrective Action	Title / Position of person responsible for corrective action	Project-Specific MPC
Method Blank	1 per prep batch of up to 20 samples	Results < QL	Report field samples that are < QL or 10x blank contamination if blank > QL; otherwise reanalyze affected samples and add laboratory qualifiers as needed.	Laboratory Analyst	Results < QL
Filter Blank (for Dissolved phase only)	1 per prep batch of up to 20 samples	Results < QL	Narrate if Result > QL.	Laboratory Analyst	Results < QL
Equipment Blank	1 every 4 events	QAPP	Investigate source of potential field contamination and qualify data according to Worksheet 36.	Project Manager or Site QC Officer and Data Validator	Results < QL
Laboratory Control Sample (LCS)	1 per prep batch of up to 20 samples	Katahdin SOP specified control limits	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	%Rec 80-120% or vendor certified limits if SRM used as LCS
Matrix Spike (MS)	1 per prep batch of up to 20 samples	%Rec 75-125%	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	%Rec 75-125% unless sample conc. > 4x spike level (spike swamped out)
Laboratory Replicate or Matrix Spike Duplicate (MSD)	1 per prep batch of up to 20 samples	%Rec 75-125% RPD $\leq$ 25%	Investigate and resolve problem, reanalyze affected samples, and add laboratory qualifiers as needed.	Laboratory Analyst	MSD %Rec 75-125% (unless swamped out) RPD ≤ 25% if reported values are ≥ 5x QL; Results <5xQL: absolute value of difference < ±QL
Field Duplicate	1 every 4 events	QAPP	Data validation will qualify data according to Worksheet 36.	Project Manager or Site QC	RPD ≤ 40% if reported values are ≥ 5x QL; otherwise use

Matrix: Whole Water & Dissolved Water
Analytical Group: TAL Metals + Tin
Analytical Method/SOP: SW-846 Methods 6010D & 6020A

QC Sample	Number / Frequency	Method / SOP Acceptance Criteria	Corrective Action	Title / Position of person responsible for corrective action	Project-Specific MPC
				Officer and data validator	professional judgement
Temperature Blank	1 per cooler shipment	QAPP	Continue to analyze sample; validator will qualify data according to Worksheet 36.	Laboratory and data validator	0 - 6 degrees Celsius

QAPP Worksheet 28-10
TSS (Whole Water) Analytical Quality Control and Corrective Action

Matrix: Suspended Particulate Material on Filter
Analytical Group: Total Suspended Solids (TSS)
Analytical Method/SOP: EPA 160.2/SM 2540D-modified

QC Sample	Number / Frequency	Method / SOP Acceptance Criteria	Corrective Action	Title / Position of person responsible for corrective action	Project-Specific MPC
Method Blank (includes a filter)	1 per prep batch of up to 20 samples	Results < QL	Report field samples that are < QL or 10x blank contamination if blank > QL; otherwise reanalyze affected samples and add laboratory qualifiers as needed.	Laboratory Analyst	Results < QL
Equipment Blank	1 every 4 events	QAPP	Investigate source of potential field contamination and qualify data according to Worksheet 36.	Project Manager or Site QC Officer and Data Validator	Results < QL
Laboratory Control Sample (LCS)	1 per prep batch of up to 20 samples	%Rec 75-125%	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	%Rec 75-125%
Laboratory Control Sample Duplicate (LCSD)	1 per prep batch of up to 20 samples	%Rec 75-125% RPD ≤ 30%	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	%Rec 75-125% RPD ≤ 30%
Field Duplicate	1 every 4 events	QAPP	Data validation will qualify data according to Worksheet 36.	Project Manager or Site QC Officer and data validator	RPD ≤ 40% if reported values are ≥ 5x QL; otherwise use professional judgement
Temperature Blank	1 per cooler shipment	QAPP	Continue to analyze sample; validator will qualify data according to Worksheet 36.	Laboratory and data validator	0 - 6 degrees Celsius

QAPP Worksheet 28-11
POC (Suspended Particulates) Analytical Quality Control and Corrective Action

Matrix: Suspended Particulate Material on Filter
Analytical Group: Particulate Organic Carbon (POC)
Analytical Method/SOP: EPA Region II Lloyd Kahn modified

QC Sample	Number / Frequency	Method / SOP Acceptance Criteria	Corrective Action	Title / Position of person responsible for corrective action	Project-Specific MPC
Method Blank	1 per prep batch of up to 20 samples	Results < QL	Report field samples that are < QL or 10x blank contamination if blank > QL; otherwise reanalyze affected samples and add laboratory qualifiers as needed.	Laboratory Analyst	Results < QL
Filter Blank	1 per prep batch of up to 20 samples	Results < QL	Narrate if Result > QL.	Laboratory Analyst	Results < QL
Equipment Blank	1 every 4 events	QAPP	Investigate source of potential field contamination and qualify data according to Worksheet 36.	Project Manager or Site QC Officer and Data Validator	Results < QL
Laboratory Control Sample (LCS)	1 per prep batch of up to 20 samples	%Rec 75-125%	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	%Rec 75-125%
Matrix Spike (MS)	1 per prep batch of up to 20 samples	%Rec 75-125%	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	%Rec 75-125% unless sample conc. > 4x spike level (spike swamped out)
Matrix Spike Duplicate (MSD)	Not required but if performed 1 per prep batch of up to 20 samples	%Rec 75-125% RPD ≤ 30%	Investigate and resolve problem, reanalyze affected samples, and add laboratory qualifiers as needed.	Laboratory Analyst	MSD %Rec 75-125% unless sample conc. > 4x spike level (unless spike swamped out) RPD ≤ 30% if reported values are ≥ 5x QL; Results <5xQL: absolute value of difference < ±QL
Laboratory Replicate	1 per prep batch of up to 20 samples	RPD ≤ 30%	Investigate and resolve problem, reanalyze affected samples, and	Laboratory Analyst	RPD ≤ 30% and report the individual POC results and the

Matrix: Suspended Particulate Material on Filter
Analytical Group: Particulate Organic Carbon (POC)
Analytical Method/SOP: EPA Region II Lloyd Kahn modified

QC Sample	Number / Frequency	Method / SOP Acceptance Criteria	Corrective Action	Title / Position of person responsible for corrective action	Project-Specific MPC
			add laboratory qualifiers as needed.		average of the replicates
CRM (ERA "Nutrients in Soil" Cat. # 542)	1 every 4 events	± 30% of certified value	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	± 30% of certified value
Field Duplicate	1 every 4 events	QAPP	Data validation will qualify data according to Worksheet 36.	Project Manager or Site QC Officer and data validator	RPD ≤ 40% if reported values are ≥ 5x QL; otherwise use professional judgement
Temperature Blank	1 per cooler shipment	QAPP	Continue to analyze sample; validator will qualify data according to Worksheet 36.	Laboratory and data validator	0 - 6 degrees Celsius

QAPP Worksheet 28-12
Grain Size (Aqueous) Analytical Quality Control and Corrective Action

Matrix: Aqueous
Analytical Group: Grain Size
Analytical Method/SOP: SedTrend Lab SOP (Laser Particle Size distribution)

QC Sample	Number / Frequency	Method / SOP Acceptance Criteria	Corrective Action	Title / Position of person responsible for corrective action	Project-Specific MPC
Laboratory Control Sample (LCS)	1 per analytical batch	Per SedTrend SOP for acceptance limits on accuracy of the distribution	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	Per SedTrend SOP for acceptance limits on accuracy of the distribution
Laboratory Replicate	1 per analytical batch	RPD ≤ 40%	Investigate and resolve problem, reanalyze affected samples, add laboratory qualifiers as needed, and narrate issue.	Laboratory Analyst	RPD ≤ 40%
Field Duplicate	1 every 4 events	QAPP	Continue to analyze samples and narrate issue.	Project Manager or Site QC Officer	RPD ≤ 50%

QAPP Worksheet 29
Project Documents and Records

Record	Responsible Person for Record	Description and Verification of Record	Off-Site Storage Location
Field notes and observation	Field sampling team	Field team will document observations in field notes and photographs. Hardcopies of field notes will be saved in the project files; electronic scan of field notes and photographs will be provided to the NYCDEP. Refer to Worksheet 6 for communication pathways and reporting of non-conformances to the NYCDEP.	Investigative organization (NYCDEP contractor) project directory
COCs	Sample management officer	Refer to Worksheet 26 and Worksheet 27 on COC, sample handling, and transfer of custody.	Investigative organization (NYCDEP contractor) project directory
Airway bills	Sample management officer	The investigative organization will maintain a record of tracking number and airway bills used to ship samples.	Investigative organization (NYCDEP contractor) project directory
Field measurements	Site QC officer (or delegate)	Field measurements will be recorded in the field forms or field notebook, and the data will be formatted per USEPA Region 2 electronic data deliverable format. Field data will be verified by the Investigative organization (NYCDEP contractor), and the files will be saved in the project directory.	Field-generated data will be formatted in USEPA MEDD format
EDD and laboratory data packages	Site QC officer (or delegate)	Electronic data deliverables (EDD) and laboratory data packages will be prepared by laboratory and submitted to the Investigative organization (NYCDEP contractor). Laboratory data packages will satisfy the requirements of Level 4 package. Compiled database will be verified by the Investigative organization (NYCDEP contractor) using USEPA EDP Checker. Files will be saved in the project directory.	Compiled electronic database in USEPA MEDD format
Data Validation Report	Site QC officer (or delegate)	Refer to Worksheet 36 for validation guidance. Data validation reports will be reviewed and verified by the Investigative organization (NYCDEP contractor), and the files will be saved in project directory. PDF copies of the validation reports will be provided to the NYCDEP.	Investigative organization (NYCDEP contractor) project directory

Note: Worksheet 29 provides a record of all documents and records that will be generated for the project. This information will support data completeness and data integrity as well as allowing ease of retrieval data.

Project Document Control System

Document Control No. GC005
Electronic copies of Document Control No. GC005 will be distributed according to Worksheet 3

Project documents will be controlled by the investigative organization Project Manager (refer to Worksheet 6 for communication pathways), who will maintain and distribute hardcopies and electronic copies of the project documents and any amendments. Electronic copies of project information will be maintained in the project electronic-file directory and the project database.

Data Recording

Data for this project will be collected by handwritten entries onto field notebooks. The field notebooks will be bound and the pages will be consecutively numbered. Sufficient information will be recorded in the field notebooks to permit reconstruction of site characterization activities conducted. Field notebooks will be kept in the possession of the appropriate field personnel, or in a secure place when not being utilized during field work. Upon completion of the field activities, field notebooks will become part of the final project evidence file. Errors in field notebooks will be corrected by crossing out the error and entering the correct information or data. An error discovered on a document will be corrected by the individual responsible for the entry, as possible. Entries recorded in field notebooks will be made in black, waterproof ink and include, but not be limited to, the following information:

- Author, date, and times of arrival at and departure from the work site;
- Description of the field activity and summary of daily tasks;
- Sample collection method;
- Number and volume of sample(s) collected;
- Details of the sampling location, including a sketch map illustrating the sampling location;
- Field observations;
- Description of photographs collected;
- Types of field instruments used and purpose of use, including calibration methods and results;
- Field measurements made;
- Sample identification number(s); and
- Sample documentation information.

As appropriate, field-generated data will be transcribed into an electronic database. SCRIBE software will be used to generate COC, create sample labels, and manage tracking of samples. However, depending on field conditions, staff can generate a hand-written COC for sample shipment. Computer-generated data associated with laboratory analyses will be generated by subcontract laboratory. Database will be provided in USEPA Region 2 MEDD format.

Data Quality Assurance Checks

The Investigative organization (NYCDEP contractor) Project Manager or Quality Control Officer will monitor the progress of sample collection to verify that samples are collected as planned. The
progress of sample collection and processing will be monitored through documentation of the samples collected each day.

The laboratory will have a formal in-house Quality Assurance Plan to which it adheres and implements as part of daily operations. Data generation processes will be reviewed and modified to meet objectives, if necessary. A formalized data generation procedure will be utilized. Each analyst must have previously demonstrated, through the laboratory quality assurance program, their requisite skills.

Laboratory Data Transmittal

Laboratory data are managed by the laboratory's internal management system, beginning with sample check-in on the sample-receiving data terminal. Laboratory data packages for each sample delivery group (SDG) will be delivered for data validation. EDDs will be formatted to be consistent with the USEPA Region 2 MEDD format.

Data Storage and Retrieval

COCs will be stored in the custody of the Project Manager for the duration of the project. Laboratory data packages and validation reports submitted to the investigative organization will be stored in the custody of the Site Quality Officer. Raw data and electronic media of all field samples, including quality control samples and blanks, will be archived from the date of generation and will be kept by the laboratory. Hard copies of project files will be archived off-site at a secure facility and retained until the end of the contract. Data will be transferred to the NYCDEP upon completion of the project. Retrieval of data by others will be at the discretion of the NYCDEP. The length of time that records will be archived will be at the discretion of the NYCDEP.

QAPP Worksheet 31, Worksheet 32, and Worksheet 33
Assessments and Corrective Action

Assessments and Corrective Action

Assessment Type	Person Responsible for Assessment	Frequency	Assessment Document	Person Responsible for Responding	Response Document	Person Responsible for Implementing Corrective Action	Person Responsible for Monitoring Corrective Action
Internal Laboratory Audit (refer to details discussed below)	Laboratory	Per Laboratory Quality Manual	Per Laboratory Quality Manual for assessment.	Subcontract laboratory	Per Laboratory Quality Manual for response.	Subcontract laboratory; per Laboratory Quality Manual	Subcontract laboratory; per Laboratory Quality Manual
Quality control reports of any non-conformance (refer to details discussed below)	Site Quality Control Officer	As needed	Non-conformance Form (refer to Attachment 4). Refer to Worksheet 6 for communication pathway for reporting a non-conformance.	NYCDEP	Non-Conformance Form (refer to Attachment 4)	Site Quality Control Officer	Project Manager will monitor that corrective action has been implemented and reported to the NYCDEP following implementation of corrective action
Field Health and Safety Audit	Field Safety Officer	As needed, audits will be scheduled if a non-conformance arises.	Non-Conformance Form (refer to Attachment 4)	Project Manager	Non-Conformance Form (refer to Attachment 4)	Field Safety Officer	Project Team. Audit report and all incidents will be communicated to the NYCDEP
Technical System Audits	Site Quality Control Officer or Project Manager	As needed, audits will be scheduled if a non-conformance arises.	Non-Conformance Form (refer to Attachment 4)	Project Manager	Non-Conformance Form (refer to Attachment 4)	Site Quality Control Officer or Project Manager or delegate	Project Manager. Audit report and all non-conformances will be communicated to the NYCDEP

Assessments and Corrective Action

Assessment Type	Person Responsible for Assessment	Frequency	Assessment Document	Person Responsible for Responding	Response Document	Person Responsible for Implementing Corrective Action	Person Responsible for Monitoring Corrective Action
Memoranda	Investigative organization Project Manager	Per report submittal	Memoranda	NYCDEP	Memoranda including data evaluation	Investigative organization (NYCDEP contractor)	NYCDEP will approve memoranda following confirmation of revisions.

Non-Conformance

A non-conformance is defined as an identified or suspected deficiency or discrepancy with regard to an approved document (*e.g.,* improper sampling procedures, improper instrument calibration, calculation, computer program); or an item where the quality of the end product itself or subsequent activities using the document or item would be affected by the deficiency; or an activity that is not conducted in accordance with the established plans or procedures.

Any field member engaged in project work that discovers or suspects a non-conformance is responsible for initiating a non-conformance report to the Site Quality Control Officer or designee. The Site Quality Control Officer will evaluate each non-conformance report, and the Site Quality Control Officer will provide a disposition, which describes the actions to be taken.

The Project Manager will verify that no further project work dependent on the nonconforming item or activity is performed until approval is obtained and the non-conformance is properly addressed. If the non-conformance is related to material, the Project Manager shall be responsible for making or identifying, with the non-conformance report number, the nonconforming item (if practical) and indicating that it is nonconforming and is not to be used. A copy of each non-conformance report will be included in the project file. Copies of all non-conformances shall be maintained by the Site Quality Control Officer or designee.

Assessment and Oversight

To monitor the capability and performance of the field activities, a field health and safety audit and technical systems audit will be performed. The Health and Safety Officer or Field Team Leader will review the field program to ensure compliance with the Health and Safety Plan. Project Quality Control Officer will review field program to ensure compliance with the QAPP. Subcontract laboratories are responsible to follow their internal quality control manual for auditing purposes. Performance audits of laboratories are conducted to measure the accuracy of the measurement systems.

Field Corrective Actions

At the end of each sampling day, the sampling team is to report any problems requiring corrective action that were encountered during the day. Corrective action will be undertaken when a non-conforming condition is identified. A non-conforming condition occurs when QA objectives for precision, accuracy, completeness, representativeness, or comparability are not met, or when procedural practices or other conditions are not acceptable. A report is to be filed that documents the problems encountered and the corrective action implemented. A Stop-Work Order may be issued by the Project Quality Control Officer or designee, following notification to the Project Manager, if corrective action does not adequately address a problem, or if no resolution can be reached.

Internal Laboratory Audits

As part of its quality assurance program, the Laboratory Quality Assurance Manager will conduct periodic checks and audits of the analytical systems to ensure that the systems are working properly, and personnel are adhering to established procedures and documenting the required information. These checks and audits will also assist in determining or detecting where problems are occurring. In addition to conducting internal reviews and audits, as part of its established quality assurance program, the laboratory is required to take part in regularly scheduled performance evaluation and laboratory audits from state and federal agencies for applicable tests. Each laboratory selected to support this program must maintain current state or federal certifications, as appropriate.

If a particular laboratory analysis is deemed "out of control," corrective action will be taken by the laboratory to maintain continued data quality. Each laboratory must adhere to their in-house corrective action policy. The coordinator of the laboratory's analytical section will be responsible for initiating laboratory corrective action when necessary.

Laboratory Corrective Actions

If a particular laboratory analysis is deemed "out of control," corrective action will be taken by the laboratory to maintain continued data quality. The laboratory must adhere to their in-house corrective action policy. The coordinator of the laboratory's analytical section will be responsible for initiating laboratory corrective actions, when necessary.

QAPP Worksheet 34
Data Verification and Validation Inputs

Data Verification and Validation Inputs

Item	Description	Verification of Completeness	Validation of Conformance to Specification
Planning Document 1	QAPP (refer to Worksheet 16 for schedule)	Verified by investigative organization (NYCDEP contractor)	Approved by NYCDEP
Planning Document 2	Field Standard Operating Procedures (refer to Attachment 1)	Verified by investigative organization (NYCDEP contractor)	Approved by NYCDEP
Planning Document 3	Laboratory procurement (refer to Attachment 2)	Verified by investigative organization (NYCDEP contractor)	None required
Field Records 1	Field Modification Forms (refer to Attachment 3)	Verified by investigative organization (NYCDEP contractor)	Approved by NYCDEP
Field Records 2	Non-conformance Report (refer to Attachment 4)	Verified by investigative organization (NYCDEP contractor)	Reviewed by NYCDEP
Field Records 3	COC for samples (refer to Attachment 5)	Verified by investigative organization (NYCDEP contractor)	None required
Field Records 4	Airway bills	Verified by investigative organization (NYCDEP contractor)	None required
Field Records 5	Field measurements	Verified by investigative organization (NYCDEP contractor)	None required
Analytical Data Package 1	Laboratory generated EDD	Verified by laboratory	Reviewed by validator
Analytical Data Package 2	Level 4 Laboratory data package	Verified by laboratory	Reviewed by validator
Analytical Data Package 3	Data validation report	Verified by validator	Reviewed by investigative organization (NYCDEP contractor)

Data Verification and Validation Inputs

Item	Description	Verification of Completeness	Validation of Conformance to Specification
Analytical Data Package 4	Compile (validated) database	Verified with EDP checker	Reviewed by investigative organization (NYCDEP contractor)
Reporting	Memoranda (data summary evaluation)	Verified by investigative organization (NYCDEP contractor)	Approved by NYCDEP

QAPP Worksheet 35
Data Verification Procedures

Data Verification Procedures

Records Reviewed	Process Description	Responsible Person
COC	COC for field samples will be reviewed initially by the Site Quality Control Officer for complete and correct information. During validation, the COC will be verified against laboratory receipt and report to examine traceability of data. The investigative organization will maintain records of sample receipt and laboratory login information.	investigative organization (NYCDEP contractor) / Data Validator
Field notes, photographs, and measurements	Record field activities and collection of field measurements. The investigative organization will maintain records of field notes and photographs. Field measurements (water quality measurements) will be transcribed into an electronic database that conforms to the USEPA Region 2 MEDD format.	investigative organization (NYCDEP contractor)
Field equipment calibration	Field equipment will be maintained and calibrated per Worksheet 22. All maintenance and calibration will be documented in field notes.	investigative organization (NYCDEP contractor)
Laboratory EDD and data package	Laboratory data reports (Level 4) and EDDs will be verified by the laboratory performing the work for completeness and technical accuracy prior to release.	Laboratory Project Manager
Corrective Action	Corrective action will be implemented and verified for completeness. Corrective actions must also be communicated to appropriate staff. Refer to Worksheet 33 for Corrective Action Report requirements.	investigative organization (NYCDEP contractor)
Field Modification	Verify completeness of QAPP Modification Form documenting the modification to the QAPP.	investigative organization (NYCDEP contractor)
Validation of laboratory data reports	Data validation according to Worksheet 36. Data validation will confirm that holding times and sample preservations as stated in the QAPP were adhered to by the field team and laboratory. Data validation will consider measurement performance criteria for precision and accuracy and evaluate quantitation limits. Validation will be summarized in the validation report.	Data Validator
Compile (validated) electronic database	Verify that entry of qualifiers into the database (and data validation reports) were correct and complete. Verify that compiled (validated) electronic database is complete and conforms to USEPA Region 2 MEDD format.	investigative organization (NYCDEP contractor)

Data Verification Procedures

Records Reviewed	Process Description	Responsible Person
Deviations from SOP and project documents	Determine impacts of any deviation from method and the project plan.	Project Team

QAPP Worksheet 36
Data Validation Procedures

Data Validation Procedures

Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
Sediment	PAH and Alkylated PAH, PCB Congeners, TAL Metals, and TOC	Low	Stage 4 validation for the first SDG and Stage 2A validation for the remaining SDGs USEPA Region 2 Validation guidance National Functional Guidelines	To be determined
Sediment	Grain Size	Low	Stage 1 data verification	investigative organization (NYCDEP contractor)
Aqueous	PAH and Alkylated PAH, PCB Congeners, TAL Metals, TSS and POC	Low	Stage 4 validation for the first SDG and Stage 2A validation for the remaining SDGs USEPA Region 2 Validation guidance National Functional Guidelines	To be determined
Aqueous	Grain Size	Low	Stage 1 data verification	investigative organization (NYCDEP contractor)

Note 1: Data validation is not required for Grain Size.

The laboratory will perform a data verification of chemical data according to their laboratory quality control manual prior to submittal of EDD and laboratory data packages. A full validation (Stage 4) will be performed on the first SDG of sediment samples and the first SDG of CSO samples. The remaining samples will be subject to limited validation (Stage 2A). The Stage 2A validation will be based on information provided by the laboratory on their quality control forms. No raw data or instrument-specific quality control review will occur during the Stage 2A validation. No calculations for quality control samples will be performed, except for performance evaluation recoveries and field duplicate precision, as needed. The Stage 2A validation will provide a review of key laboratory quality control elements. If a high frequency of measurement performance issues is observed, the issue will be investigated and an additional validation effort may be implemented. At a minimum, the following data elements will be reviewed during the Stage 2A validation (as applicable to the parameter group):

- Agreement of analyses conducted with COC requests
- Holding times and sample preservation
- Laboratory blanks/equipment blanks/ field blanks
- Field duplicate relative percent differences
- Quantitation limits and sample results
- Surrogate recoveries

- LCS/LCSD (or equivalent) recoveries
- MS/MSD recoveries
- Laboratory duplicate / Matrix duplicate relative percent differences
- Performance evaluation recoveries
- Method-specific quality control results (if tabulated in laboratory QC summary forms)
- Evaluation of laboratory qualifiers applied to the dataset
- Evaluation of issues documented in the laboratory narrative
- Completeness check (so that full validation could be performed at a later date, if necessary)

The data validator will perform a validation according to "USEPA Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use" (OSWER No. 9200.1-85, EPA 540-R-08-005, January 13, 2009). The validation will use measurement performance criteria stated in the laboratory SOP and QAPP to assess quality and performance of surrogates, laboratory control samples, matrix spikes, and laboratory duplicates. Validation will use measurement performance criteria stated in the applicable analytical method to assess quality and performance of calibration. Validation guidance documents include:

- PAH and Alkylated PAH: USEPA Region II, Hazardous Waste Support Branch, SOP HW-22 "Validating Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry SW-846 Method 8270D." Revision 05, January 2011.
- PCB Congeners: USEPA Region II, Validation Standard Operating Procedure for "EPA Method 1668, Revision A, August 2003, Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS", SOP HW-46, Revision 1, September 2008.
- Metals: USEPA Region II, USEPA Hazardous Waste Support Section, ICP-AES Data Validation, SOP HW-3a, ISM02.2, Rev. 0 (July 2015); ICP-MS Data Validation, SOP HW-2b, ISM02.2, Rev. 0 (July 2015); and Mercury Validation, SOP HW-2c, ISM02.2, Rev. 0 (July 2015).
- Inorganics: USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review, Office of Superfund Remediation and Technology Innovation (OSRTI), OLEM 9240.1-66, EPA-540-R-20-006, November 2020.
- Organics: USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review, OSRTI, OLEM 9240.0-51, EPA-540-R-20-005, November 2020.

Blank action will be applied according to the following criteria:

Blank Type	Blank Result	Blank Action Level (BAL)	Sample Result	Action for IDM	Action for non-IDM (Organic & Inorganic Targets)
Method Blank, Net-Blank, ICB, CCB, and other instrument-associated blanks	Detect	5x BAL = 5 x Blank Detect ***for Isotope Dilution Methods (IDM)***	Non-detect	No Action	NA
			< QL and < 5x BAL	Negate (UJ) at level found in Sample	NA
			≥ QL and ≤ 5x BAL	Negate (U) at level found in Sample	NA

			> QL and > 5x BAL	No Action	NA
Method Blank, Net-Blank, ICB, CCB, and other instrument-associated blanks	Detect	1x BAL = 1 x Blank Detect 3x BAL = 3 x Blank Detect ***for non-Isotope Dilution Methods (non-IDM) - Organic & Inorganic***	Non-detect	NA	No Action
			< QL and < 1x BAL	NA	Negate (UJ) at level found in Sample
			≥ QL and ≤ 1x BAL	NA	Negate (U) at level found in Sample
			1x BAL < sample result ≤ 3x BAL	NA	Estimate (J+) at level found in Sample
			> QL and > 3x BAL	NA	No Action

Notes:
1. IDM = Isotope Dilution Method.
2. "< QL" results are flagged "J" from the lab.

A Performance evaluation (PE) sample, represented by a NIST certified reference material (CRM), will be analyzed in each analytical batch. Any excursions observed in the PE sample will result in qualification in the entire analytical batch. The laboratory will use the measurement performance criteria to determine if a reanalysis of the SDG is required. Validators will consider the CRM (PE) sample results along with other accuracy QC samples in the SDG. If the laboratory passes the QAPP CRM (PE) criteria, then no further validation action will be required. If the laboratory fails the QAPP CRM (PE) criteria, and a reanalysis of the batch was not performed, then the validator will use professional judgment to assess potential effects on the associated field data, which may include taking action to flag field data in the SDG. CRM (PE) samples will be analyzed per direction in Worksheet 12.

Once data validation is completed, a data validation report will be generated. The report will contain information regarding the parameters that are qualified, the reason for the qualification, and the direction of the bias (only for parameters qualified as estimated), when possible. Based upon the quality assurance review of the analytical data, specific codes (data qualifiers or "flags") will be placed next to results to provide an indication of the quantitative and qualitative reliability of the results. The data qualifier codes in the National Function Guidelines are being used for this project. Qualifiers assigned by laboratories will be defined by each laboratory in their data package and will be superseded by the data validator's qualifiers. Non-detect concentrations will be defined as "U" or "UJ" flagged results. "J" flagged results will be considered estimated but usable data. The final deliverable will be an EDD, which is formatted according to the USEPA Region 2 MEDD guidelines.

QAPP Worksheet 37
Data Usability Assessment

Data Usability Assessment

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:

- Precision: Precision is the measure of agreement among repeated measurements of the same property under identical or similar conditions. Results of field duplicates and laboratory replicates will be evaluated based on the measurement performance criteria presented on Worksheet 12. Any conclusions about the precision of the analyses or sample collection techniques will be drawn and any limitations on the use of the data will be described.
- Accuracy/Bias: Accuracy is a measure of agreement of a measurement to a known value (includes a combination of random error and systematic error components of sampling and analytical operations). Bias is the systematic or persistent distortion of a measurement process that causes errors in one direction. Results of laboratory blanks, filter blanks, equipment blanks, LCS, MS, surrogates, and internal standards will be evaluated based on the measurement performance criteria presented on Worksheet 12. Any conclusions about the accuracy/bias of the analyses based on contamination will be drawn and any limitations on the use of the data will be described.
- Sensitivity: Sensitivity is the capability of a method or instrument to discriminate between measurement responses representing different levels of the target analyte. All analytical results reported will be evaluated to determine if adequate sensitivity was achieved. The results for each analyte will be cross-checked against the quantitation limits presented in Worksheet 15. Results for analytes that do not meet project quantitation limit criteria will be summarized. Any conclusions about the sensitivity of the analyses will be drawn and any limitations on the use of the data will be described.
- Representativeness: Representativeness is a qualitative term that expresses how well the data characterizes a population. Representativeness is achieved through adherence to sampling and analytical procedures described in the QAPP; compliance with sample handling and usage of custody seals; and compliance with stipulated sample holding times and preservation. After evaluation of relative compliance with specified procedures and holding times, conclusions about data representativeness will be drawn and any limitations on the use of data will be described.
- Comparability: Comparability is a qualitative term that expresses the measure of confidence that one data set can be compared to another and can be combined for decision-making purposes. Data comparability will be assessed through evaluation of achieved sample specific reporting limits, units of measure, and adherence to specified analytical methodologies and field/sample collection standard operating procedures specified in the QAPP. After the evaluations are completed, conclusions about data comparability will be drawn and any limitations on the use of data will be described.
- Completeness: Completeness is a measure of the amount of valid data needed to be obtained from a measurement system. The field completeness will be calculated by the ratio of the number of samples received in acceptable condition by the laboratories to the number of samples planned to be collected as specified in this document.

Data Usability Assessment

Describe the evaluative procedures used to assess overall measurement error associated with the project:

Chemistry analytical data: The laboratory will perform a data verification of chemical data according to their laboratory quality control manual prior to submittal of EDD and laboratory data packages to the investigative organization (NYCDEP contractor). Validation will be conducted in accordance with the protocols outlined on Worksheet 36. The data validator will identify any qualifications and bias (if known) on the data. The validator will apply qualifiers and comments on the usability of the data. Once the data validation report is received, it will be reviewed by the Site Quality Control Officer. Any quality issues with the validation will be discussed with the NYCDEP.

Field measurements: The investigative organization (NYCDEP contractor) will perform a data verification of field records and formatted in the USEPA Region 2 MEDD format.

Identify the personnel responsible for performing the usability assessment:

Data usability assessment will not be performed at the time. However, the data validation reports will be reviewed by the Site Quality Control Officer to address if there are any quality issues associated with the data. The data usability will be verified by reviewing of the data validation reports, and any rejected results should not be used for data evaluation.

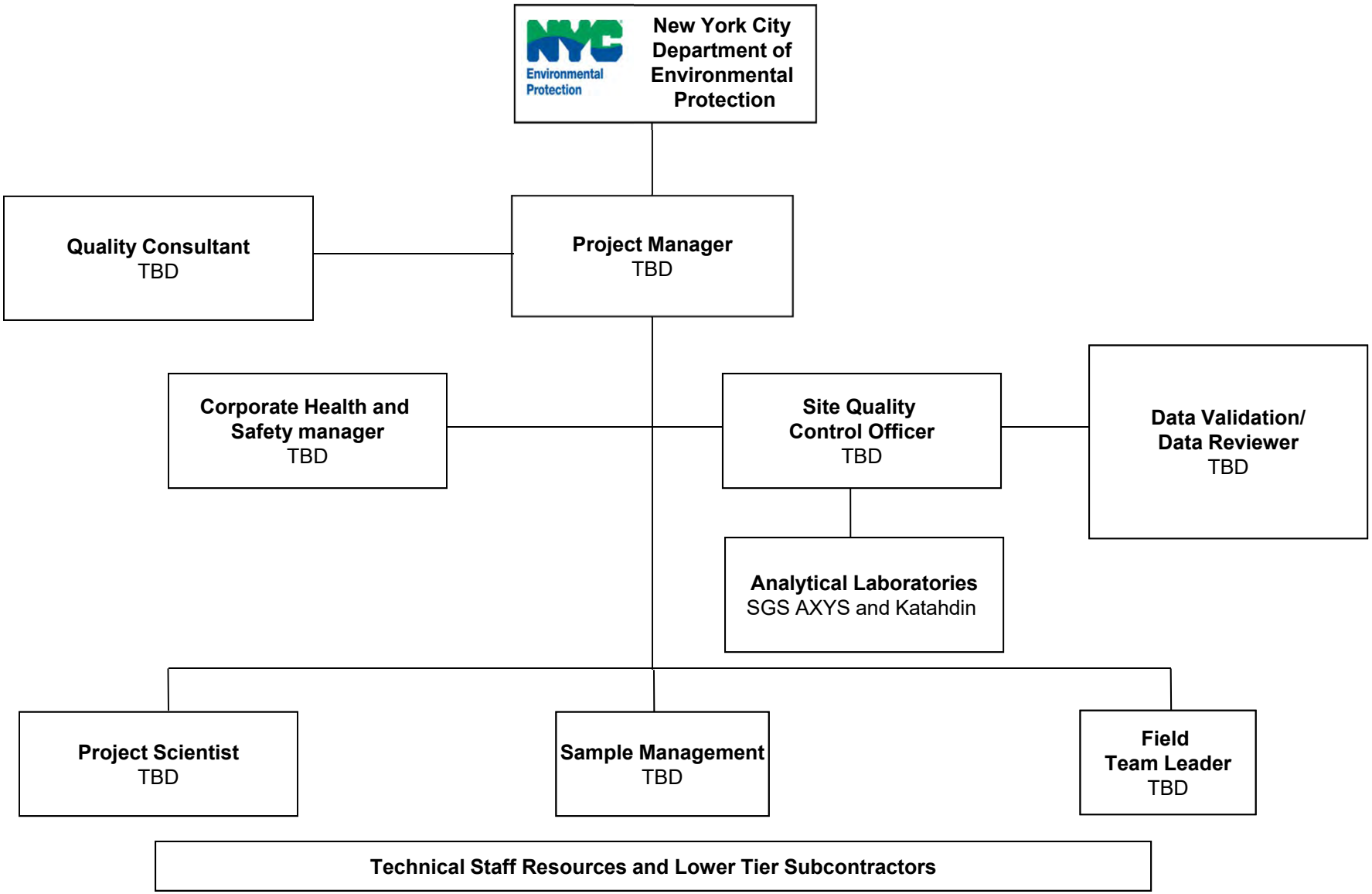
5-Step Data Usability Assessment: The data usability assessment is performed at the conclusion of data collection activities, using the outputs from data verification and data validation. It is the data interpretation phase, which involves a qualitative and quantitative evaluation of environmental data to determine if the project data are the correct type, quality, and quantity to support the project decisions.

Step 1: Review the project's objectives and sampling design	Project Team will review the environmental questions and project decision points stated in Worksheet 10, data quality objectives stated in Worksheet 11, and the sample design/rationale stated in Worksheet 17. Project Team will review key outputs defined during the planning stages to verify that they are still applicable and any field modifications. Project Team will review the sampling design for consistency with the stated objectives.
Step 2: Review the data verification and data validation outputs	• An independent auditor will complete a data validation based on the QAPP, laboratory SOPs, and validation guidelines stated in Worksheet 36. The investigative organization (NYCDEP contractor) will then back-check to verify placement of validation qualifiers and qualification reason in the electronic database (compared to the data validation report). • The investigative organization (NYCDEP contractor) will enter field measurements into the database as well as field notes (specifically those activities that occurred in the field or during shipment that may impact data quality or representativeness of the samples).
Step 3: Verify the assumptions of the selected statistical method	Statistical methods are not currently proposed.
Step 4: Implement the statistical method	Statistical methods are not currently proposed.

Data Usability Assessment

Step 5: Document data usability and draw conclusions	Data usability assessment will not be performed at the time. However, the data validation reports will be reviewed by the Site Quality Control Officer to address if there are any quality issues associated with the data. The data usability will be verified by reviewing of the data validation reports, and any rejected results should not be used for data evaluation.

FIGURES



TBD: To be determined. The QAPP is prepared under the contract with WSP. The program is scheduled to start in June 2022 under a new contract with NYCDEP. The project personnel and subcontractors will be updated under the new contract.

	Project Organizational Chart **Gowanus Canal Superfund Site**	**Figure 1**

RH-034: Butler St. (NYCDEP Property)

Note: a site visit is needed to confirm the sampling location

OH-007: 2nd Ave. at 5th St.

NYC Environmental Protection	CSO Sampling Locations (RH-034 and OH-007)	Figure 2

NYC Environmental Protection

Proposed Sediment Sampling Locations

Figure 3

ATTACHMENTS

Attachment 1

Field Standard Operating Procedures

Procedure to Collect and Process Sample Points Using a Global Positioning System (GPS)

I. Introduction

This standard operating procedure (SOP) provides guidance for setting up a Trimble Geo 7X GPS for point collection, locating a targeted sampling point, recording GPS points, differentially correcting the collected data, and exporting the corrected data to a usable format in Excel or ArcGIS.

II. Definitions

GPS: The GPS is a satellite-based positioning system, operated and controlled by the U.S. Department of Defense. The GPS includes 24 satellites and can be used by anyone who has a GPS receiver. The GPS receiver is used for position determination, navigation, and survey tasks on land, sea, and in the air. The method of utilizing GPS varies with each application and the type of GPS equipment used. Operating methods range from low precision, code phase systems to highly accurate, carrier phase systems that facilitate on-the-fly measurements, also known as real-time kinematic surveying (RTK). A hand-held GPS receiver with sub-meter horizontal accuracy will be used at the Site to capture the coordinates of sample locations.

Differential Correction: Differential correction is a class of techniques for improving the accuracy of GPS positioning by comparing measurements taken by two or more receivers. The stationary receiver (or "base station") continuously records its fixed position over a control point. The difference between the base station's actual location and its calculated location is a measure of the positioning error affecting that receiver at that location at each given moment. The base station calculates the correction needed to eliminate the error in the position calculated at that moment from GPS signals. The correction is later applied to the position calculated by the mobile receiver at the same instant.

III. Equipment and Supplies

1. Hand-held GPS unit (Trimble Geo7X or equivalent model)
2. USB cord for GPS
3. Field notebook
4. Field maps of site and target locations
5. GPS Pathfinder Software

IV. Setup

Fully charge GPS unit using provided charging cable. Once charged, power up the unit by pressing the large green button below the screen area. Press the start menu button in the lower left hand corner and select the “TerraSync” icon to open the GPS menus. For a key to the GPS menu components listed in this SOP refer to Figure 1.

Adjusting Settings

Settings may be changed by the user depending on preference or project specifications. These steps will outline how to adjust the settings to ensure real-time correction is enabled, ensure that data points are collected with a horizontal accuracy limit, change the units to feet and miles, and change the coordinate system to Lat/Long and decimal degrees. These steps should be followed prior to collecting points with the GPS to ensure desired settings are selected.

Enable Real-Time Correction

Real time correction is critical to achieving sub-meter accuracy with the Trimble 7X series.

1. Select "Setup" in the Menu drop down, and select the "Real-time Settings" button.
2. Select the "Choice 1:" drop down menu and select "Integrated SBAS".
3. Select "Done" at the bottom of the screen.

Enable Horizontal Accuracy Logging

Horizontal accuracy settings are critical when collecting points at sub-meter accuracy. Accuracy based logging allows the GPS to only record points when the accuracy is within a specified range. These settings will pause point collection if the accuracy changes to an out-of-range value while collecting the point and will resume collecting point features once the accuracy has returned to within the specified range. The range specified for this project is less than 3 feet.

1. Select "Setup" in the Menu drop down, and select the "Logging Settings" button.
2. Select the "Wrench Symbol" next to "Accuracy Settings, Horizontal – Post-processed".
3. Ensure "Accuracy Value For Display/Logging:" is set to "Horizontal" and the tab below it to "In the Field".
4. Ensure "Use Accuracy-based Logging:" is set to "Yes".
5. Scroll down and change "Required Accuracy:" to "1 m" or "3 ft.".
6. Select "Done". While collecting a point, if the accuracy exceeds this selected value, the logging will automatically pause until reception improves accuracy to below this selected value again.

Adjust Units

It may be easier for field staff and contractors to use units they are more familiar with in order to navigate to a location. The following steps describe how to adjust the units for all menus in the GPS.

1. Select "Setup" in the Menu drop down, and select the "Units" button.
2. Select the "Distance Units:" drop down, and select "Feet".
3. Adjust the additional fields as needed:
    - Area Units
    - Velocity Units
    - Angle Units
    - Lat/Long Format (DD.dddd is preferred)
4. Select "Done".

Adjust Coordinate System

These steps will outline how to adjust the coordinate system to Lat/Long in decimal degrees, however, any coordinate system can be selected with this method.

1. Select "Setup" in the Menu drop down, and select the "Coordinate System" button.
2. In the "System:" drop down, select "Latitude/Longitude".
3. In the "Datum:" drop down, select "WGS 1984".
4. Scroll down, and in the "Coordinate Units:" drop-down, select "DD.dddd".
5. Select "Done" at the bottom of the screen.

Importing a Batch Waypoint File into GPS

1. First setup the target coordinates in a text file with the location, y-coordinate, and x-coordinate separated by commas, in that order. Do not include a header at the top of the file. Note what coordinate system the data is in. Lat/Long is preferred.
2. Open GPS Pathfinder Office software and create a new project folder when prompted to do so.

3. Under “Options” select “Coordinate System” and change the selection so that it matches the coordinate system of the target coordinates.
4. Under “Options” select “Style Of Display” and change the selection so that it matches the format of the target coordinates.
5. Under “File” select “Waypoints” and “ASCII Import.” Select “Browse” and open the target coordinates text file.
6. While still in the import menu, click on the dot under “'Coordinate system of input file:” and click on “Change” and ensure that the coordinate system matches that of the target coordinates file.
7. While still in the input menu, select the arrow next to “Input file format:” and select “DNE.” “DNE” stands for “Description, Northing, Easting” which is the order the target coordinate data should be in. The “Start import at row:” selection should have “1” as the value, as the import will start at row 1, because the file should not have a header.
8. Select “OK” and “OK” again upon successful import, and select “close”. The points should show up on the map display.
9. Select “Utilities” and then “Data Transfer”. Save the file if prompted.
10. From the top left select the GPS 7X device, and click on the green button to the right of this drop-down menu. The top right of the menu will display if the unit is connected.
11. Select the tab “Send”. Click on the “Add” button and select “Waypoints”.
12. Select the saved Waypoint file and select “Open”.
13. Select “Transfer all” in the Data Transfer menu. A message will confirm that it was a successful transfer.
14. Save and close out of the menus and GPS Pathfinder.

Add a single Waypoint file to GPS
1. Select the “Nav” menu, and select “Waypoints” from the Sub-menu.
2. If there is not an existing waypoint file, select “New”, create a “File Name:”, and select “OK”. If there is an existing file, make sure it is open, and select “Options”, and “New”.
3. Enter a name for the waypoint in “Name:”, and enter the coordinates in “North:” and East:” and an elevation (use 0 if unknown) in “Elevation.”Select “OK”. The new waypoint will now be added to the list of existing waypoints in the “Waypoint” sub-menu.

V. Field Procedure

Receiving Satellites
Typically, the GPS will automatically start connecting to satellites, however, if this does not occur, follow the steps below. Note that satellite reception and real-time correction may be affected by surrounding structures, limiting the accuracy achievable in a certain location.
1. Select the Menu tab and select “Setup”.
2. Select the Sub-menu tab and select “Connect to GNSS”. The satellite icon on the top will start to blink as satellites are connected. Additionally, the real-time correction icon will blink as well until it is connected to, which is required for submeter accuracy.
3. Navigate to the “Status” menu to view connected satellites.
4. Wait until the required accuracy is achieved. This may take up to 10 minutes.

Creating a New Data File
1. Select the Menu tab and select “Data”.

2. Select the Sub-menu tab and select "New". This will create a new data file for storing new data points for up to 1 week. After a week there will be a prompt to open a new data file.
3. Enter the program name under "File Name", and select "Create" at the bottom. Enter the antenna height if needed, and select "OK".

Navigating to a Location

1. Use the procedure outlined above in Setup to add waypoints to the GPS.
2. To view all waypoints on the map, select the Menu tab and select "Map". If waypoints are not shown, select "Layers" and then select "Waypoints".
3. Select "Options" and "Zoom Extents" to show all waypoints. The map will show all waypoints marked as flags with a box around them, and the GPS location as a simple X.
4. To select a waypoint to navigate to, go to the "Nav" menu.
5. Select "Waypoints" in the Sub-menu, scroll down, and double click on the target waypoint. A bold flag symbol should come up next to the waypoint. This waypoint will now be marked as a bold flag with a box around it in the "Map" menu.
6. Select "Navigate" in the Sub-menu to show distance and bearing to the point from the current location. The "Map" menu can be used as well to show an overhead view of the moving GPS user, the direction, and the marked waypoint.

Collecting a Point

1. Select the Menu tab and select "Data".
2. Select the Sub-menu tab and select "Collect". Ensure that the Data File being written to is open and is the correct file. Data cannot be collected without an open Data File.
3. When ready to collect a data point, select "Create" at the bottom of the screen. The number of points collected will start to accumulate on the top right of the screen. A minimum of 60 points should be collected. Ensure that the accuracy is within the desired limit as determined during setup (typically <3 ft.).
4. While the points are being collected, enter the location ID into the comments section.
5. When the ID is entered, and 60 points have been logged, select "Done" at the bottom of the screen, and the point will be stored.

Checking Collected Point Coordinates and Distance to Waypoint

1. After collecting a point, open the "Map" menu.
2. Select "Options", then select "Zoom Extents".
3. In the cursor Sub-menu select the magnifying glass. Zoom into the target waypoint and the collected point, shown as an X on the screen.
4. Change the magnifying glass back to the cursor.
5. Select the collected point on the map with the cursor. The point ID and coordinates will show up.
6. Write down the coordinates and associated ID into the field notebook.
7. To determine the distance of the collected point to the waypoint, change the cursor again to the measuring stick.
8. Select the waypoint and then select the collected point, and the distance will be shown on the screen.

Troubleshooting

When experiencing an issue with instrumentation in the field, it is best to call customer support at the company the equipment was rented from. Common suppliers and their contact information is listed below:

Eco-Rental Solutions
Monday – Friday, 8:00am-5:00pm
Toll-free: 866-843-2165
Local: 914-400-0324

Pine Environmental
Monday – Friday, 8:00am-6:00pm
Phone: 609-371-9663

VI. Differential Correction and Data Export

Differential correction is a critical post-processing measure for improving the accuracy of the collected GPS points. The procedure for differential correction and export is as follows:

1. Connect the Trimble unit to the computer with the USB cord and open GPS Pathfinder Office. Open the project folder associated with the data when prompted, or create a new project folder.
2. Select "Utilities" and "Data Transfer".
3. From the top left select the Trimble 7X device, and click on the green button to the right of this drop-down menu. The top right of the menu will display if the unit is connected.
4. Select the "Receive" tab. Select "Add" and select and open the data file in the GPS folder.
5. Select "Transfer All". This will save the raw GPS data as a .SSF file to your project folder.
6. Once the files have transferred, close out of the "Data Transfer" menu.
7. Go to "File" and "Open", and select the transferred .SSF file, and select "Open".
8. Select "Utilities" and "Differential Correction" to open the wizard. Select the .SSF file and hit "Next".
9. Select "Next" for the following two menus to apply the default settings.
10. In the fourth menu, select "Select…" under "Base Provider Search".
11. Select the provider that has the highest "Integrity Index", and select "OK". Select "Next" in the wizard menu.
12. Select "Start" on the last wizard menu, and the correction will process. This may take up to 10 minutes or longer. Once the correction is done processing, select "Close".
13. Go to "File" and "Open", and select the differentially corrected .COR file, and select "Open".
14. Select "Utilities" and "Export". Ensure the selected file is correct.
15. Under "Choose an Export Setup", select "Sample ESRI Shapefile Setup", and ensure the GIS Coordinate System matches that of the file. Ignore the prompt, and select "Yes". Select "Close" once the file is done exporting.
16. The shapefile will be located in the export folder location. It can then be brought into ArcGIS as a shapefile and with the "Calculate Geometry" function, the coordinates can be generated.

VII. Quality Control

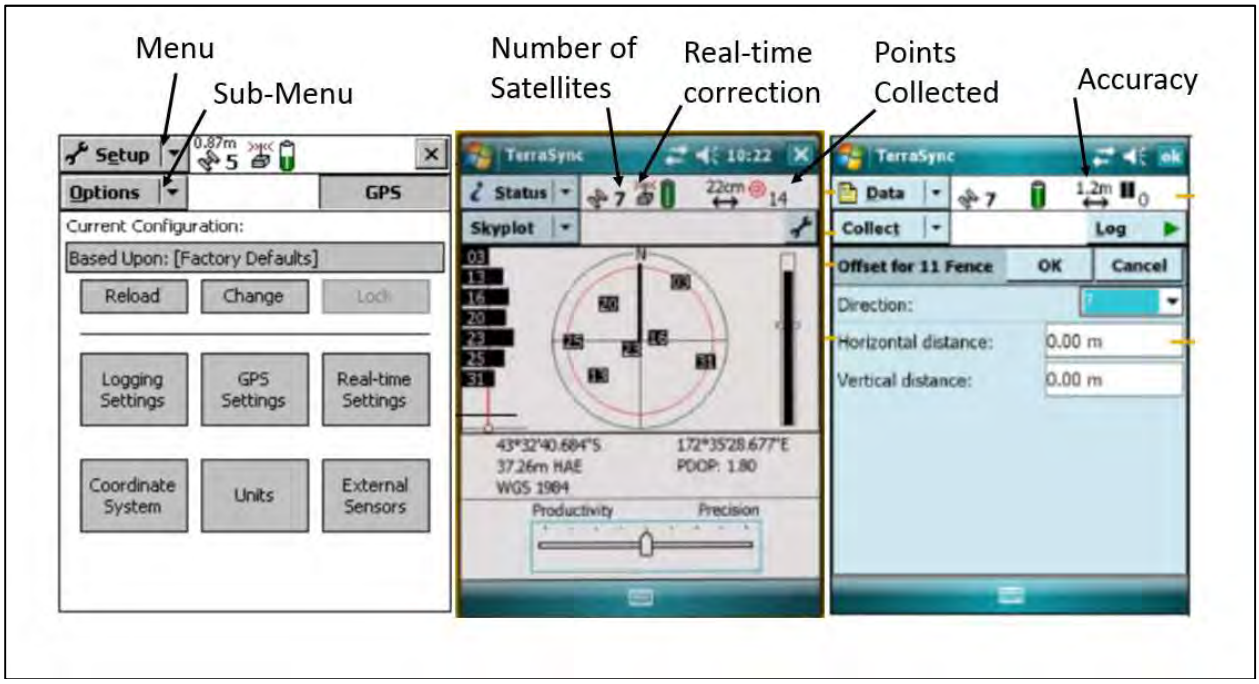
The GPS has quality control features that are built into the system. The system will not allow measurements to be taken if there are not enough satellites available to provide accurate readings, if the satellite geometry is not conducive to the survey, and for other reasons. The system maintains quality control records during a survey that contain information about the quality of the GPS position, including the number of available satellites, satellite geometry, and horizontal and vertical precision levels. These records can be accessed when the data is post processed in order to assure that the necessary quality standards are being achieved.

VIII. References

Trimble Geo 7X handheld User Guide. Trimble, November 2013.

The Nature of Geographic Information, 22. Differential Correction. Penn State College of Earth and Mineral Sciences, Accessed: November 2017

Figure 1: GPS 7x Menus



Procedure for Navigation and Boat Positioning

I. Introduction

This Standard Operating Procedure (SOP) describes the methods to be used for positioning boats for sampling in Gowanus Canal. The judgement of the vessel captain should be prioritized in regard to boat use, access, and safety.

II. Equipment and Supplies

The following is a list of equipment that may be necessary to carry out the procedures contained in this SOP. Additional equipment may be required, depending on field conditions.

- Approved documents including Quality Assurance Project Plan (QAPP) and Health and Safety Plan (HASP).
- Daily float plan and listing stations to be sampled, target station coordinates, access points along the Study Area, and sample transfer/transport locations. Include a copy for the captain.
- Personal protective equipment (PPE) as required by the HASP, including personal floatation devices (PFDs).
- Sampling vessel equipped with necessary Global Positioning System (GPS) navigation, communication, and safety equipment.
- Handheld radios, as necessary.
- Handheld GPS (refer to SOP No. 1 for procedures).
- Boat spuds and/or anchors to stabilize boat on station.

III. Procedure

The navigation plan and required equipment should be reviewed with the vessel captain prior to starting work each day. Before navigating to a target location discuss with the captain the best method for anchoring, spudding, or tying off near a location, and how adjustments will be made to achieve the target position accuracy. Discuss how communication will occur when navigating to a location, and if hand signals or radios will be used. If a location is not feasible to get to due to access, safety concerns, or other conditions, discuss with the project manager on moving the location elsewhere. All staff on board should alert the captain immediately to any hazards such as debris or shallow water. Additionally, any buys should be avoided, as these may mark out underwater debris or other entities sample locations. Final positioning should be performed in a way to minimize sediment suspension to the extent possible. This includes keeping the engine in low gear, spudding slowly, and setting anchors at least 30 feet from the sampling location.

Navigation

These steps describe general methods for using a GPS to guide the vessel captain to a location.

1. Confirm with the captain which location is being targeted.
2. Select this location waypoint in the GPS (See SOP No. 1).
3. The navigator (personnel with GPS) should stand at a location on the vessel that is safe, allows communication with the captain, and is as close as possible to the sampling location on the vessel.
4. The navigator should provide constant updates to the captain via verbal commands, hand signals, or radio commands on bearing and distance to the target location based on the GPS readings. The captain may mark the sample point on the vessel GPS as a reference for positioning.
5. Once the vessel is secured on location, the navigator should verify that the distance to the target location from the exact sampling point on the vessel is within the target sampling distance.

6. The navigator should direct the captain on any adjustments necessary to get within the target sampling distance.

Anchoring

This is the typical method for anchoring on location by a vessel captain, with assistance from the navigator. The captain may set one or more anchors to maintain position. Vessel deckhands should perform anchoring work. At no time should Louis Berger personnel assist with anchors as they have not been trained to do so.

1. Follow steps 1 through 4 in "GPS Use" above prior to beginning anchoring. The navigator should provide constant updates on bearing and distance, as the captain may need to know this information to drop the anchor at a specific time.
2. The captain will direct the vessel at least 30 feet past the target location before dropping the first anchor.
3. The deckhand will ensure the anchor is secured in the sediment. If anchors are dislodged at any time, they may need to be reset.
4. The captain may be able to keep the vessel on position with one anchor, depending on the current.
5. If additional anchors are required, the captain will move the vessel to the appropriate distance while the other anchor(s) is secured, and drop this anchor, before repositioning to the sample location.
6. Small adjustments can be made by pulling in on the anchor lines to get closer to the sample location, or if an alternate sample attempt is needed.
7. Spuds may be used in conjunction with anchors. See the following section on spudding.

Spudding

This is the typical method for spudding on location by a vessel captain, with assistance from the navigator. The captain will usually deploy at least two spuds to maintain position. Vessel deckhands should perform spudding work. At no time should Louis Berger personnel assist with spudding as they have not been trained to do so. Always confirm that spuds are at least 50 feet from any underground utilities through dig safe, existing maps, and shoreline mark-outs.

1. Follow steps 1 through 4 in "GPS Use" above prior to beginning anchoring. The navigator should provide constant updates on bearing and distance, as the captain may need to know this information to drop the spuds at a specific time.
2. The captain will direct the vessel straight at the target location.
3. Once the vessel is close enough to the target location, the deckhand will begin lowering the spud, allowing it to sink into the sediment as deep as possible from its own weight.
4. The vessel should be directly in line with the sample location. The captain can then pivot the vessel off the one spud until the sampling area on the vessel is directly over the sample point. The vessel can be pivoted again if needed for an alternate sample attempt.
5. The second spud can then be lowered as the captain maintains position.
6. Anchors or tie offs may be used with spuds as well to maintain position.

Tying Off

In some cases, the sampling point may be close enough to the shoreline, bulkheads, or structures such as pilings, so that the vessel can be tied to these surfaces to keep it in place over the sampling location. The vessel usually can be tied off from any side and maintain position easily. The captain should ensure that the surface is stable

and safe to tie off to, and that there are no posted signs prohibiting vessels. Tying off can be used in conjunction with spuds.

Recordkeeping

Once on location the navigator is responsible for collecting the GPS point(s) for the sample location (see SOP No. 1). GPS coordinates should be written down in field notes as well as a backup measure.

IV. Vertical Elevation Measurement Procedures

1 At each sampling station where elevation data is required, the water depth (from the top of the water level surface to the top of sediment surface) will be determined using a weighted measuring tape. This depth should be verified by comparing it to the boat depth finder.

2 The date and time of the measurement will be recorded on the appropriate field form.

V. Quality Assurance/Quality Control

GPS system performance checks using built-in accuracy measurements and ground truths will be performed during the course of the sampling event to confirm GPS accuracy. It is the responsibility of the Field Team Leader to periodically check to ensure that the procedures are in conformance with those stated in this SOP.

Procedure for Collection and Processing of Surface Sediments

I. Introduction

This standard operating procedure (SOP) describes the equipment and methods to be used to conduct surface sediment grab sampling at the Gowanus Canal Superfund Site. For this project, surface sediment is considered to range from 0 to 15 cm (0 to 6 inches) in depth. Collection of surface sediment is to be accomplished with a remotely activated device (dredge) and a deployment system. This technique consists of lowering a sampling dredge to the sediment surface using a rope or cable in conjunction with a davit (or by hand if using a lightweight sampler) or an extended handle. When the mechanism is activated, the device will entrap a sediment sample in spring-loaded or lever-operated jaws.

1. An Ekman dredge sampler is a lightweight device with spring activated jaws. It is used to collect moderately consolidated, fine textured sediment. Ekman samplers are used to collect undisturbed surface sediments and can preserve the quiescent sediment surface/water interface.
2. A Ponar sampler is a heavyweight device with weighted jaws. It is used to collect consolidated fine to coarse textured sediment. Ponar samplers are used when sediment sampling activities may disturb the depositional setting of the sediment bed during its deployment.

II. Equipment and Supplies

The following equipment will be used to conduct sediment sampling:

1. Sampling dredge (*e.g.,* Ekman and/or Ponar) and materials are described in the individual sections below.
2. Personal protective equipment (PPE) - including steel toe boots (if aboard a boat), waders (if in a stream), Tyvek coveralls, disposable boot covers, and disposable gloves. (Refer to HASP requirements for full PPE requirements).
3. Log sheets to record all field collected data.
4. Dedicated, plastic syringes to subsample retrieved sediment grabs for AVS/SEM analyses.

III. Guidelines

Using GPS in accordance with SOPs Nos. 1 and 2, maneuver the sampling vessel to within 20 ft. (maximum distance unless otherwise approved) of the pre-programmed target coordinates for each sediment sample location, and stabilize the vessel as much as possible. Similarly locate the sampling position at stream or tributary locations.

Confirm the location by examining the site sampling location map and landmarks. If necessary, conduct sediment probing to confirm sediment texture before attempting sample collection. Collect sample using Ekman or Ponar dredge in accordance with the following guidelines.

a) Ponar Dredge Guidelines:

1. Attach a sturdy nylon rope or steel cable to the ring provided on top of the dredge.
2. Arrange the Ponar dredge with the jaws in the open position, set the trip bar so the sampler remains open when lifted from the top. If the dredge is so equipped, place the spring loaded pin into the aligned holes in the trip bar.
3. Slowly lower the sampler to a point approximately two inches above the sediment.
4. Drop the sampler to the sediment. Slack on the line will release the trip bar or spring loaded pin; pull up sharply on the line to close the dredge.
5. Raise the dredge to the surface and slowly decant any free liquid through the screens on top of the dredge. Care should be taken to retain the fine sediment fraction during this operation.
6. Open the dredge and transfer the sediment to a stainless steel, plastic or other appropriate composition (e.g., Teflon) container. Ensure that non-dedicated containers have been adequately decontaminated. If necessary, continue to collect additional sediment until sufficient material has been secured to fulfill analytical requirements. Thoroughly homogenize the sediment, and then transfer sediment to sample containers appropriate for the analyses requested. Samples for volatile organic analysis must be collected directly from the bucket before homogenization to minimize volatilization of contaminants.

b) Ekman Dredge Guidelines (if used):

1. Install and secure the polycarbonate Ekman liner (if required).
2. Attach a sturdy nylon rope or stainless steel cable through the hole on the top of the bracket, or secure the extension handle to the bracket with machine bolts.
3. Cock the Ekman: Attach springs to both sides of the jaws. Fix the jaws so that they are in open position by placing trip cables over the release studs. Ensure that the hinged doors on the dredge top are free to open.
4. Lower the sampler to a point 4 to 6 inches above the sediment surface.
5. If the sampler is on a rope or cable, drop the sampler to the sediment. If an extension handle is used the sampler will be pushed into the sediment approximately 4 inches to prevent overfilling of the dredge.
6. Trigger the jaw release mechanism by lowering a messenger down the line, or by depressing the button on the upper end of the extension handle.
7. Raise the sampler evenly as jerking motions or excessive speed may disturb the sample. Slowly decant any free liquid through the top of the sampler. Care should be taken to retain the fine sediment fraction during this procedure.
8. Open the sampler jaws and transfer the sample into a stainless steel, plastic or other container by re-cocking the Ekman while it is resting on the container. Care should be taken to minimize the loss of fine sediments from the container. The liner, if used, will retain the shape of the sample inside of the Ekman and facilitate removal of sample in the field when it is positioned on a holder designed to accept the sample and liner. If necessary, continue to collect additional sediment grabs until sufficient material has been secured to fulfill analytical requirements. Thoroughly homogenize and then transfer sediment to sample containers appropriate for the analyses requested. Samples for volatile organic analysis must be collected directly from the sample while still in the Ekman dredge, before removal and homogenization, to minimize volatilization of contaminants.

c) AVS/SEM Sediment Sample Collection Guidelines:
Field Guidelines:

1. A dedicated syringe will be used to procure aliquots of sediment from the grab sampler prior to any homogenization or compositing of the grab sediment. The field scientist will plunge the syringe into the sediment in several locations within each grab, each time extruding the aliquot into the lab-provided 2-oz jar. When the jar is almost full (leave small but adequate head-space to allow for expansion during freezing), the sample collection will be complete for that particular grab sample.
2. Using a dedicated syringe, the field sampler will repeat the above process on the subsequent grab sample recovered from the location, filling a 2nd 2-oz sample jar for AVS/SEM sediment as described above.
3. The field scientist will repeat Step 2 as needed, if more than 2 discrete surface sediment grabs are collected from a single location.
4. Sediments will be kept cold on ice in coolers on board the boat and then frozen in the 2-oz jars immediately after the sampling team returns to shore, using dry ice or a freezer. Once frozen, they will be packed in coolers on wet ice and sent via courier or shipped to Lab, to arrive frozen.
5. It is important to note on the chain-of-custody how many jars were collected for AVS/SEM at each location so that the lab knows exactly how many jars of sediment they need to composite. Each jar will be labeled with the identical sample identification for the location.

IV. Sample Preservation, Containers, Handling and Storage

Cooling to 4°C on wet ice is generally the recommended approach for sample preservation, supplemented by the appropriate holding time for the analyses requested, or as per the QAPP. Wide mouth glass containers with Teflon lined caps are utilized for sediment samples. The necessary sample volume will be determined as a function of the analytical requirements and specified in the QAPP. If composite sampling techniques or multiple grabs are employed, equal portions of sediment from each location will be deposited into a stainless steel stock pot, plastic, or other appropriate composition (*e.g.*, Teflon) containers. The sediment is homogenized thoroughly to obtain a composite representative of the area sampled.

The composite sediment sample is then transferred to a labeled container(s) of appropriate size and construction for the analyses requested. Transfer of sediment is accomplished with a stainless steel or plastic lab spoon or equivalent.

All sampling devices should be decontaminated, and subsequently wrapped in aluminum foil. The sampling device should remain wrapped until it is needed.

V. Decontamination

Refer to SOP No. 05 for equipment decontamination procedures.

VI. Site Clean-Up

Excess sediment not included in the sample, decontamination waste, and used PPE should be managed according to SOP No. 06.

VII. Health and Safety

When sampling from water bodies, physical hazards must be identified, and adequate precautions must be taken to ensure the safety of the sampling team. The team member collecting the samples should stay away from the edge of the water body, where bank failure may cause loss of balance. When collecting samples near the edge of water bodies, personnel must wear a lifeline. All sampling personnel must wear personal flotation devices (life vests). If sampling from a boat, appropriate protective measures must be implemented as per the Health and Safety Plan (HASP). All activities are to be conducted in accordance with the HASP.

VIII. Record Keeping and Quality Control

There are no specific quality assurance activities which apply to the implementation of these procedures. However, the following general procedures apply:

- Data must be documented on field data sheets or within site logbooks.
- Instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment calibration activities must occur prior to sampling/operation and they must be documented.
- Descriptions of any deviations and the reason for deviations from the site QAPP or this SOP should be noted in the field notebook, as necessary. In addition, the logbook should track pertinent sample collection information such as: sample date/time, personnel, weather conditions, and sample identification information. Samples taken from areas with visible staining or other indications of non-homogeneous conditions should be noted.
- Field personnel will collect the proper type and quantity of quality control samples as prescribed in the QAPP.

Classification of Soil and Sediment

I. Introduction

This standard operating procedure (SOP) describes the process that will be used to classify sediment and soil core and grab samples collected during investigation activities at Gowanus Canal Superfund Site.

II. Definitions

Definitions are referenced from the ASTM D2488-00: Standard Practice for Description and Identification of Soils (Visual-Manual Procedure). Please refer to ASTM D2488-00 for more detailed definitions.

Clay: fine-grained soil with putty-like properties (plasticity) when containing water. If the soil is dry then it becomes hard, strong, and solid. Has a plasticity index equal to or greater than 4.

Gravel: particles of rock that are larger than 4.75-mm but smaller than 75-mm in size. Coarse gravel is characterized by particles 19-mm to 75-mm in size, and fine gravel is characterized by particles 4.75-mm to 19-mm in size.

Organic Clay: clay with organic content high enough to influence soil properties.

Organic Silt: silt with organic content high enough to influence soil properties.

Peat: dark brown to black decomposing vegetative matter with organic odor and spongy consistency.

Sand: particles of rock between 75-µm and 4.75-mm. Coarse sand is characterized by particles ranging from 2-mm to 4.75-mm in size, medium sand is characterized by particles ranging from 425-µm to 2-mm in size, and fine sand is characterized by particles ranging from 75-µm to 425-µm in size.

Silt: a fine-grained soil (<75 µm) with little or no plasticity. When soil is dry it has little or no strength.

III. Equipment and Supplies

1. Geotechnical Gauge- typically contains size classification, with actual sand grains from coarse to silt, roundness chart, percent composition chart, unified soil classification system, common soil colors, soil and sand compacting classifications.

2. Environmental Sample Core Log: use to record core characterization (Attachment 1)
3. Key to Core Data Logs- guide to core descriptions and symbology (Attachment 2)
4. Small hand lens.
5. Small spatula or spoon.
6. Engineering Tape Measure
7. Appropriate PPE including at a minimum; nitrile gloves, safety glasses or goggles, Tyvek suit or apron, and Tyvek or rubber booties.
8. Scale

IV. Guidelines

1. Core Logging: To describe the sediment cores an Environmental Sample Core Log (Attachment 1), a Geotechnical Gauge, and the Key to Core Data Log (Attachment 2) are required. Sample classification requirements stipulated in the Quality Assurance Project Plan (QAPP) and Field Sampling Plan (FSP) will be performed.

    - Measure the total length of the core and record it on the Environmental Sample Core Log. Describe the core(s) in the diagram provided on the Environmental Sample Core Log.

    - For each horizon note the color, size (segment length), texture, lithology, and odor. Also note other distinguishing characteristics such as shell hash, detritus, or presence of an organic sheen. Use Attachment 2 as a reference for additional data collection requirements. Note not all categories apply to all samples. These are a guide to the information that can be provided by a core description. Specific descriptions of each category are listed further along in this document. If a photographic record is to be obtained, the core ID should be present in every frame as well as an RGB color indicator and a measuring tape for size reference.

2. **Group Symbols:** Soils can be identified by assigning a group symbol. Flow charts representing these group symbols can be seen in Attachment 4 (fine-grained soils) and Attachment 5 (coarse-grained soils).
3. **Lithology:** The description or physical characterization of the soil such as clay, silt, or sand.
4. **Dilatancy:** the structural change of the soil from stress and pressure over time. This can be tested by shaking and squeezing a small round ball of the sample. The ASTM criteria are listed below and details on how to test the sample are listed in the ASTM D2488 report. (This will not be performed)

- None: No visible change in the specimen
- Slow: Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing.
- Rapid: Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing.

5. **Type:** a more descriptive means of describing the soil characters such as soil size and composition.
6. **Color:** a very important aspect of the core description and soil identification. Any changes in color should be noted on the sample log along with the location of the color boundary. Use a Munsell Soil Color Chart (can be on Geotechnical Gauge) to acquire uniform descriptions.
7. **Consistency:** using the symbols on the Key describe the consistency as hard, firm, soft or very soft. Note this is not applicable to soils with large amounts of gravel. The ASTM criteria are as follows:
    - Very soft: Thumb will penetrate soil more than 1 in
    - Soft: Thumb will penetrate soil about 1 in
    - Firm: Thumb will indent soil about ¼ in
    - Hard: Thumb will not indent soil but readily indented with thumbnail
    - Very Hard: Thumbnail will not indent soil
8. **Cementation:** used on intact coarse-grained soils. The three options are weak, moderate and strong. The ASTM criteria are as follows:
    - Weak: crumbles or breaks with handling or little finger pressure
    - Moderate: Crumbles or breaks with considerable finger pressure
    - Strong: will not crumble or break with finger pressure
9. **Structure:** physical layout of the core. Below are descriptions of the ASTM criteria noted on the "Key to Core Data Logs" (Attachment 2).
    - Homogeneous: Same color and appearance throughout
    - Stratified: Alternating layers of varying material or color with layers at least 6 mm thick; note thickness
    - Laminated: Alternating layers of varying material or color with the layers less than 6 mm thick; note thickness

- Mottled (Lensed): Inclusion of small pockets of different soils, such as small lenses of sand scattered through a mass of clay; note thickness

10. **HCl Reaction:** drop small amounts of HCl on the soil to see the reaction. Use ASTM criteria of none, weak, or strong. (This will not be performed)
    - None: no visible reaction
    - Weak: some reaction, with bubbles forming slowly
    - Strong: Violent reaction, with bubbles forming immediately
11. **Maximum particle size:** note the largest particle size seen in the sample. The "Key to Core Data Logs" has the appropriate abbreviations and descriptions. More details can be found in the ASTM D 2488 paper.
12. **Odor:** Note any organic or non-organic odors that may be released when opening the core. Many soils have a strong, distinctive odor of decaying vegetation. It is important to also note any chemical or petroleum odors.
13. **Samples:** Used to identify sample ID. Also, this column can be used to denote where the core may have been split to acquire numerous samples.
14. **Toughness**: the amount of pressure need to roll a sample of the soil into a $1/8^{th}$ inch diameter (plastic limit) thread and the strength of the thread. The ASTM criteria are listed below. (This will not be performed.)
    - Low: Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft.
    - Medium: Medium pressure is required to roll the thread to near the plastic limit. The thread and the lump have medium stiffness.
    - High: Considerable pressure is required to roll the thread to near the plastic limit. The thread and the lump have very high stiffness.
15. **Plasticity:** using the information gathered in the toughness test use the ASTM criteria to rate the plasticity of the sample. (This will not be performed.)
    - Non-plastic: A 1/8 in. thread cannot be rolled at any water content.
    - Low: The thread can barely be rolled and the lump cannot be formed when drier than the plastic limit.
    - Medium: The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be re-rolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit.

V. References

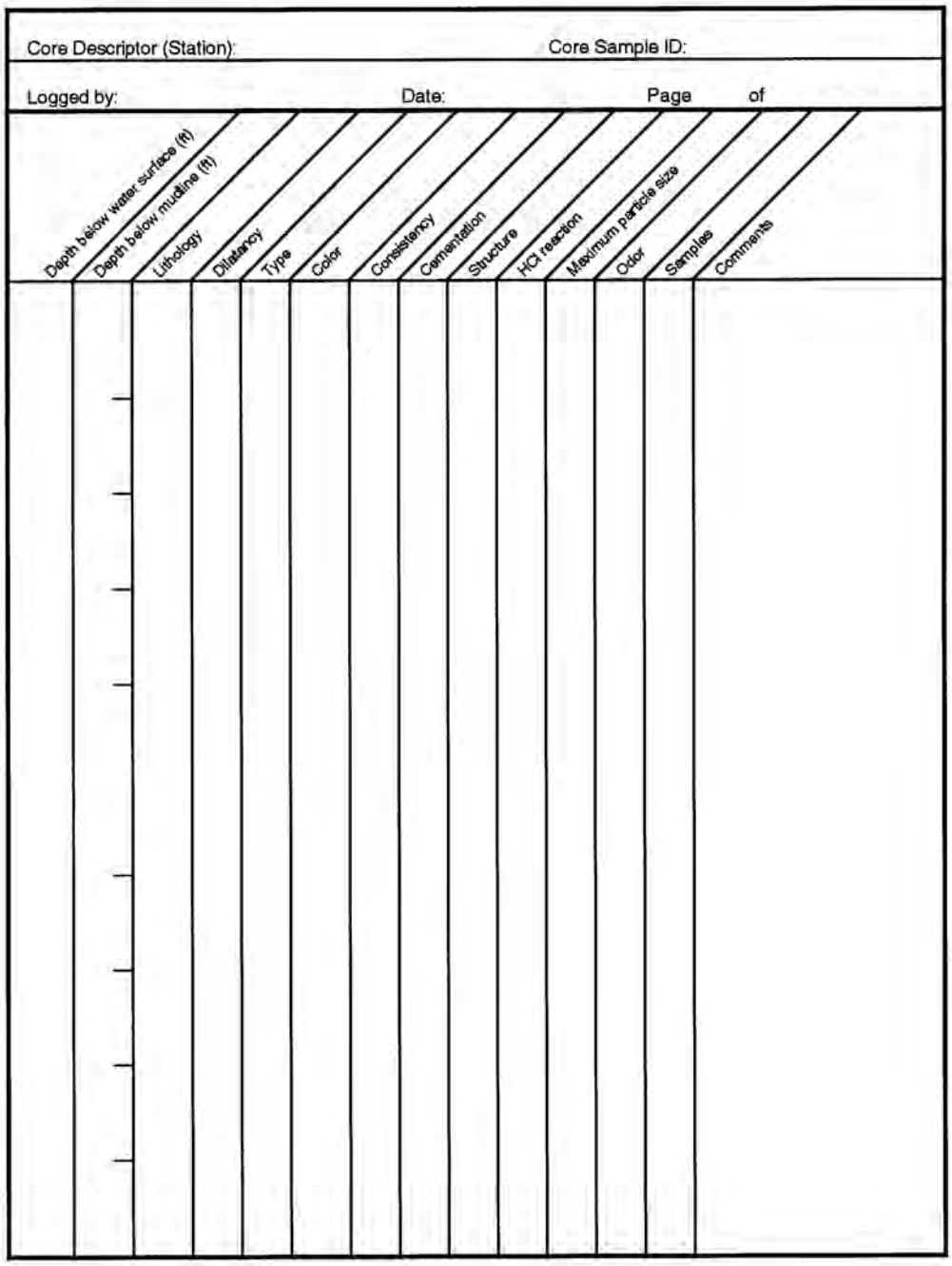
ASTM International Designation: D 2488-00, Standard Practice for Description and Identification of Soils (Visual-Manual Procedure). Edition 2000

VI. Attachments

1. Blank Environmental Sample Core Log Form
2. Key to Core Data Logs
3. Example of Completed Environmental Sample Core Log Form
4. Group Symbol Flow Chart for Fine-grained Soils
5. Group Symbol Flow Chart for Coarse-grained Soils

Attachment 1

Environmental Sample Core Log



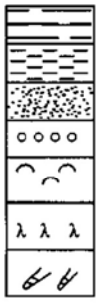
Reviewed by_____ Date _____

Attachment 2

Key to Core Data Logs

KEY TO CORE DATA LOGS

LITHOLOGY



TYPE

GW	Well-graded gravels, gravel-sand mixtures
GP	Poorly-graded gravels, gravel-sand mixtures
GM	Silty gravels, gravel-sand-silt mixtures
GC	Clayey gravels, gravel-sand-clay mixtures
SW	Well-graded sands, gravelly sands
SP	Poorly graded sands, gravelly sands
SM	Silty sands, sand-silt mixtures
SC	Clayey sands, sand-clay mixtures
ML	Silts and very fine sands, silty or clayey fine sands, or clayey silts, with slight plasticity
CL	Clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays
MH	Silts or fine sandy silts with moderate plasticity
CH	Clays of high plasticity, fat clays

COLOR

Selected from Munsell Soil Color Chart

CLAY/SILT CHARACTERISTICS

DILATANCY
N = None
S = Slow
R = Rapid

TOUGHNESS
L = Low
M = Medium
H = High

CONSISTENCY
Penetration of thumb:
<0.25 cm = hard (H)
0.25 – 2.0 cm = firm (F)
2.0 – 4.0 cm = soft (S)
>4.0 cm = very soft (VS)

CEMENTATION
N = Not cemented
W = Weakly cemented
M = Moderately cemented
S = Strongly cemented

STRUCTURE
H = Homogeneous
S = Stratified
L = Laminated
M = Mottled

HCl REACTION
N= None
W = Weak
S = Strong

MAXIMUM PARTICLE SIZE
SC = Small Cobble
CP = Coarse Pebble
MP = Medium Pebble
SP = Small Pebble
CS = Coarse Sand
MS = Medium Sand
FS = Fine Sand
VFS = Very Fine Sand
Z = Silt

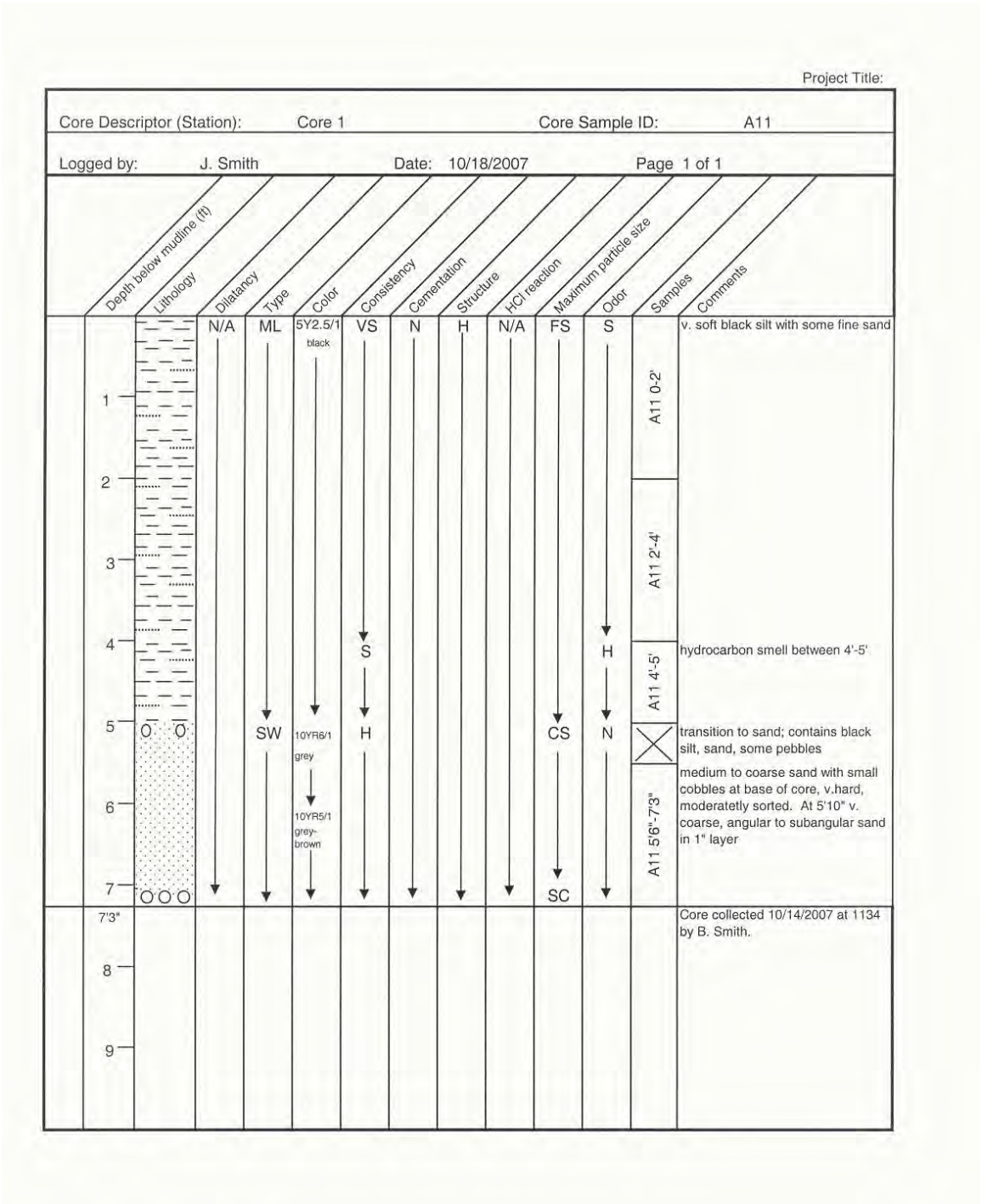
SA = Sub-angular
VA = Very angular

ODOR
N = None
S = Sulfide; HS = Hydrogen sulfide
P = Petroleum

PLASTICITY
N = None
L = Low
M = Medium
H = High

Attachment 3

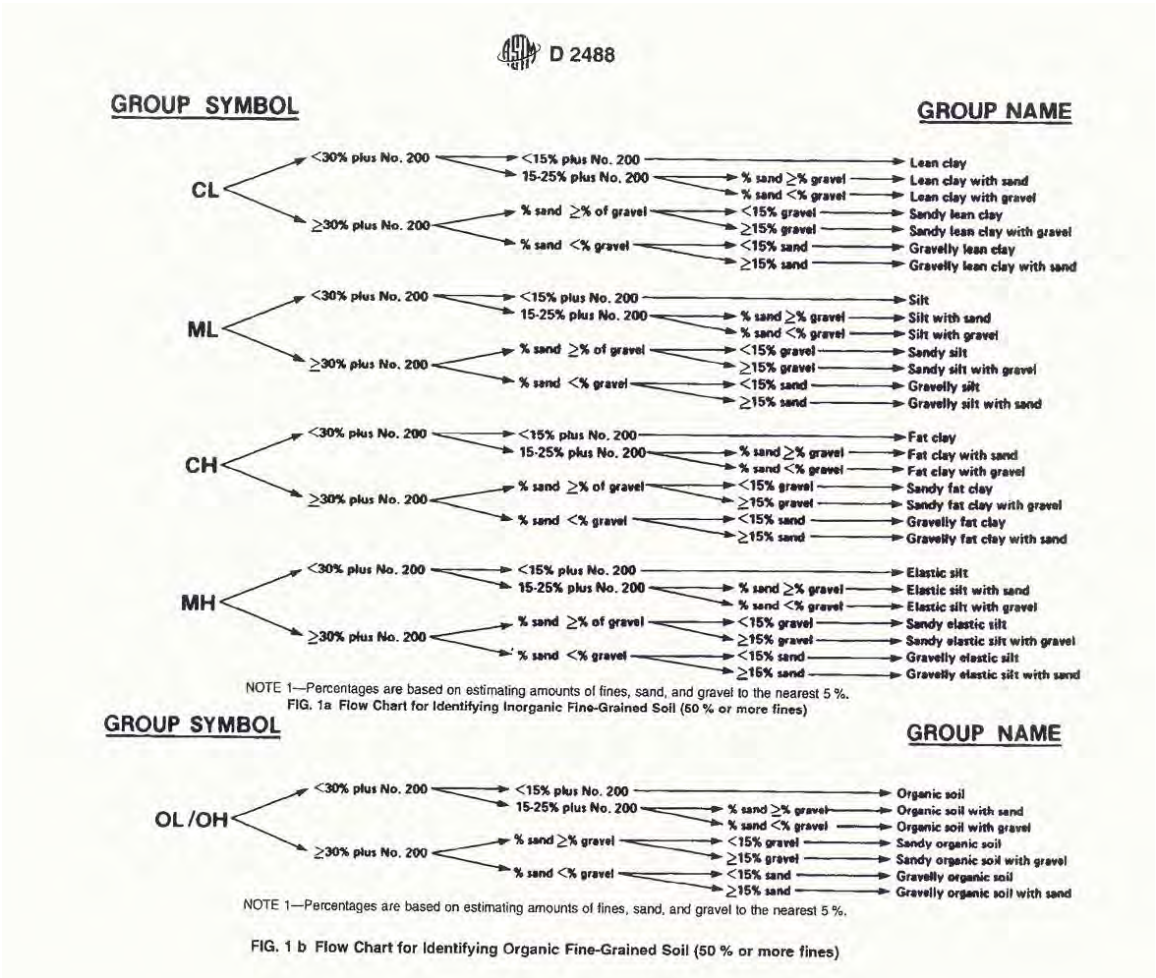
Completed Environmental Sample Core Log Form



Attachment 4

Group Symbols

Flow Chart for Fine-grained Soils



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1a Flow Chart for Identifying Inorganic Fine-Grained Soil (50 % or more fines)

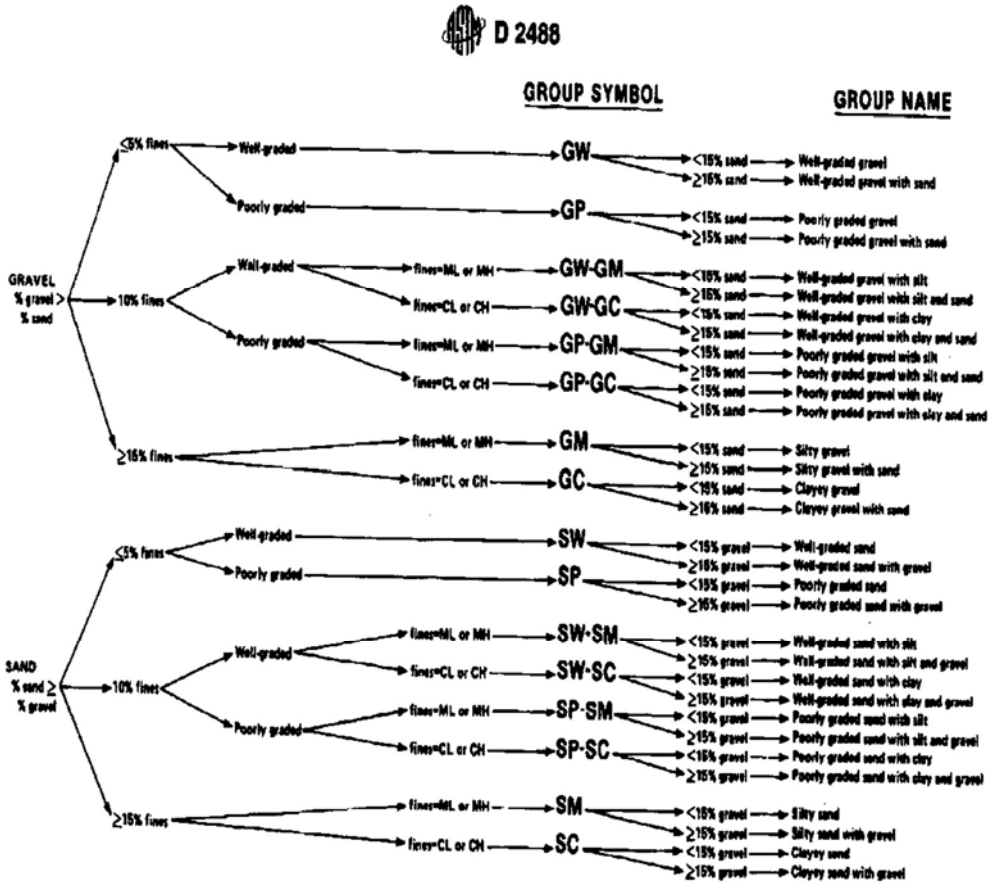
NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1 b Flow Chart for Identifying Organic Fine-Grained Soil (50 % or more fines)

Attachment 5

Group Symbols

Flow Chart for Coarse-grained Soils



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 2 Flow Chart for Identifying Coarse-Grained Soils (less than 50 % fines)

Procedure for the Decontamination of Sediment and Soil Sampling Equipment

I. Introduction

This standard operating procedure (SOP) describes the methods used to decontaminate non-dedicated equipment utilized during soil and sediment sampling. Refer to SOP No. 6 for managing and storing investigation-derived waste.

II. Equipment and Supplies

The following equipment will be used to decontaminate equipment and tools used to collect sediment and soil samples:

1. Tap or bottled water for initial cleaning and rinsing of equipment.
2. De-ionized water for final rinsing of equipment after tap water or solvent rinse.
3. Laboratory-grade non-phosphate detergent (e.g., Luminox® or Liquinox®)
4. Laboratory-grade cleaning reagents, as needed (e.g., acetone, nitric acid)
5. Portable steam cleaner, if applicable.
6. Personal protective equipment (PPE) - including disposable gloves (nitrile preferred), safety glasses (goggles preferred), disposable wipes, eye wash system, first aid kit, and waterproof outerwear (if necessary).
7. Re-sealable buckets and disposable trays approved for waste collection and transportation.
8. Squirt bottles for water, alcohol, and solvents.
9. Brushes for cleaning equipment.
10. Field notebooks, pens, pencils, and digital camera to document decontamination procedures.

III. Guidelines

All non-dedicated equipment used to collect and process environmental samples for laboratory analysis must be cleaned between sample locations to avoid cross-contamination. The following equipment may be used to collect sediment grab samples and will require decontamination:

1. Sediment Grab Sampler (e.g., Ponar, Van Veen, Smith McIntire, or Ekman- type) used for surface sediment collection.

2. Liners used to line the grab samplers during sample collection, if applicable.

3. Stainless steel scoops, spoons, bowls, stock pots, and other equipment that comes into contact with the sample, which are used for homogenization.

Split spoons and thin-walled tubes must undergo the sequential decontamination process described below.

No hand to mouth contact is permitted during decontamination operations. The decontamination area should be sectioned off to prevent personnel without the appropriate PPE from entering.

IV. Procedure for Decontamination of Equipment Used for Chemical Samples

Note that all decontamination fluids will be placed into a re-sealable container and managed per SOP No. 6 "Management and Disposal of Investigation-Derived Waste". Non-aqueous waste solutions will be separated from aqueous waste solutions.

Follow the procedure below for decontamination of non-dedicated soil and sediment sampling equipment used for the collection of chemical samples. Note that a separate solvent rinse is required for this decontamination process. These are general guidelines and the procedures may be altered depending on specific program needs and equipment to be decontaminated. For example, polycarbonate (Lexan) liners to be used in Ekman dredges will not receive nitric acid or solvent rinses as these solutions can damage the liners.

1. Place item to be decontaminated over an appropriately sized tray to capture rinse water.
2. Wash each item with bottled water to remove mud, dirt, or other visually present material.
3. Scrub the item with a brush and soapy water. Alternatively, a steam cleaner may be used on metal items, with soap and high pressure hot water.
4. Rinse the item with bottled water to remove all residual soap.
5. Over a separate container for solvent wastes, rinse the item with nitric acid using squirt bottle. Consult with project chemists to determine if this step is required for the specific sampling program.
6. Rinse the item with deionized water using squirt bottle.
7. Rinse the item with solvent using squirt bottle. Allow to air dry. Consult with project chemists to determine if this step is required for the specific sampling program.
8. Using clean gloves to prevent recontamination, wrap the item in aluminum foil to protect it until it is used.
9. Store the newly decontaminated equipment in a clean area until use.
10. Capture and store decontamination liquids in containers with re-sealable lids. All wash water and solvent rinse wastes will be containerized in re-sealable buckets approved for waste collection and consolidation. Separate buckets will be used for aqueous and non-aqueous rinses. Refer to SOP No. 6 *"Management and Disposal of Investigation-Derived Waste"* for handling and storing waste on-site.

V. References

American Society for Testing and Materials (ASTM), 1994. Standard Practice for Decontamination of Field Equipment Used at Nonradioactive Waste Sites. Designation: D 5088 – 90.

United States. Environmental Protection Agency. Science and Ecosystem Support Division. Field Equipment Cleaning and Decontamination. Epa.gov, November 2007. Web.

Management and Disposal of Investigation-Derived Waste

I. Introduction

This standard operating procedure (SOP) describes the methods used to manage, store, and dispose of investigation-derived waste (IDW) produced during remedial investigation activities at the Gowanus Canal Superfund Site. The procedures are meant to address waste generated from the collection of sediment, soil, surface water, and groundwater and any resulting contaminated PPE, equipment, and decontamination waste.

II. Definitions

FTL: Field Team Leader, head of field operations.

IDW: Investigation-Derived Waste.

PPE: Personal Protective Equipment.

SOP: Standard Operating Procedure.

AOC: Area of Contamination, "non-discrete land area on or in which there is generally dispersed contamination".

RCRA: Resource Conservation and Recovery Act.

TCLP: Toxicity Characteristic Leaching Procedure.

III. Equipment and Supplies

The purchase, maintenance, and use of the supplies and equipment listed below are the responsibility of the FTL. The following equipment and supplies will be used to collect and dispose of investigation-derived waste:

1. Waste Storage and Disposal Containers
    1. 55-gallon UN rated drums will be used for the storage and disposal of solid and non-solvent liquid wastes.
    2. 6 mil, 55-gallon plastic liners will be used in drums as additional protection.
    3. All drums will be stored on top of spill platforms capable of containing a minimum of 15 gallons per 55-gallon drum.
2. Transferring Equipment
    1. Plastic safety funnels with brass or plastic screens and vents.
    2. 5-gallon buckets with screw on lids.
    3. Hand pump/siphon with Teflon or Tygon tubing.
    4. Tools: screwdriver, hammer, drum plug wrench, and pliers.
    5. Drum dolly.

3. PPE
    1. Disposable Tyvek coveralls or apron.
    2. Safety glasses.
    3. Face shield.
    4. Disposable plastic gloves (nitrile, butyl rubber, or Viton).
    5. Shoe covers (rubber or Tyvek).
4. Spill Cleanup Equipment and Supplies
    1. Spill absorbent (Vermiculite or Speedidry™).
    2. Broom, foxtail and dustpan.
    3. 6 mil plastic sheeting.
    4. Traffic cones.
    5. Sandbags.
    6. Shovel.
    7. Paper towels.
    8. Manual drum pump (same as pump in 'Item 2. Transferring Equipment').
5. Labels and Logs**:** A supply of labels and log sheets that are referred to in this SOP are to be kept on site in an easily accessible location. Additional logs will be obtained from the FTL.
6. Digital camera to document IDW management.

IV. Guidelines

Best management practices for waste disposal will be employed at all times. Waste will be minimized whenever possible, and separate waste streams will be implemented. Existing data sets can be used to profile anticipated material to be collected. If existing data is insufficient to characterize waste, then a composite sample of the waste will be analyzed prior to disposal in order to generate a sufficient profile. Waste streams will consist of the following:

A. Nonhazardous material with chemical parameters below the TCLP limits and less than 2 ppm of PCBs.

B. Nonhazardous material with chemical parameters below the TCLP limits and between 2 and 50 ppm of PCBs.

C. Hazardous material exceeding TCLP limits or 50 ppm of PCBs.

Due to site constraints and limitations associated with transporting waste, the primary option for storing IDW on the site is to keep it on the vessel being used for sampling for the duration of the program. The material is not being transported outside of the AOC, and would therefore not trigger RCRA regulations, until it is taken off site for disposal. Vessels such as barges

would have sufficient space for a designated storage area with spill containment. If a smaller vessel is being used as well, and it cannot accommodate a 55-gallon drum, then consolidation on the larger vessel would occur with the use of 5-gallon buckets. Programs typically do not last longer than 90 days, however, this will be the maximum amount of time waste will be kept on site before a certified disposal company is contracted to remove the material.

As a contingency, if on site storage on a vessel is not possible for an extended period of time, daily pickups can be contracted through a certified disposal company.

Drums will be stored on top of spill pallets in a secure location away from regular foot traffic and heavy machinery. Nonhazardous drums will be labeled with "Nonhazardous" sign, date, and company name and contact information. Hazardous drums, if generated, will have labels including name, address, telephone number, product identifier, signal word, hazard statement, precautionary statement, and pictograms.

Upon conclusion of the program, the vessel contractor will transport the drums on board back to their facility, where a disposal contractor will meet them the same day to pick up the material and bring it to an approved facility based on the waste profile.

Sediment/Soil

Sediment processing will occur on the sampling vessel. Waste will consist of remaining processed material, rejected samples, equipment decontamination fluids, grossly contaminated PPE, and disposable equipment. Decontamination fluids including solvents (used sparingly, if at all) and detergent wastes will be collected separately during decontamination. Minute volumes of solvent waste will be set aside to evaporate, and detergent and water waste will be disposed of in the drum matching the waste profile of the material cleaned from the equipment.

55-gallon drums with plastic liners will be used for sediment. On smaller vessels with restricted space, lined, 5-gallon buckets will be used, and on a daily basis will be consolidated in the appropriate 55-gallon drums. Transfer will occur as follows:

1. Don all appropriate PPE, at a minimum; safety glasses, nitrile gloves, and a Tyvek apron.
2. Place the sediment and liner in a clear garbage bag.
3. Unscrew the drum bolt from the lid of the correct drum based on the characterized material (A, B, or C, above). Remove the band and lid from the drum.
4. Slowly place the bag of material into the drum to prevent splashing or tearing of the bag. If transferring material from a 5-gallon bucket, tie the liner in the bucket before placing the bagged material into the drum.
5. Place the lid securely on the drum and screw the drum bolt until tight.
6. Record in the waste log the date, time, type of material, sample location, and signature.

Grossly contaminated PPE will be disposed of in the appropriate drum as well.

Groundwater

Groundwater waste will be collected in 5-gallon buckets with screw on lids during sampling and then transferred into a designated 55-gallon drum at the end of each day. The waste streams

listed above (A through C) will be used for groundwater as well based on existing site data. Transfer will occur as follows:

1. Don all appropriate PPE, at a minimum; safety glasses, nitrile gloves, and a Tyvek apron.
2. Stage the buckets to be transferred into the drum.
3. Unscrew the drum bolt from the lid of the correct drum based on the characterized material (A, B, or C, above). Remove the band and lid from the drum.
4. Ensure the drum has a liner on the inside. Push aside the liner and use clips if necessary to keep it open during the transfer.
5. Use a siphon pump to transfer the liquids into the drum.
6. Wipe away any remaining solids with paper towels and dispose of these in the drum.
7. Place the lid securely on the drum and screw the drum bolt until tight.
8. Record in the waste log the date, time, type of material, sample location, and signature.

Grossly contaminated PPE will be disposed of in the appropriate drum as well.

Surface Water

It is not anticipated that any IDW would be generated from surface water sampling. Any excess surface water sampled will be returned to the creek the day it was collected.

CSO/MS4

It is not anticipated that any IDW would be generated from CSO/MS4 sampling. Any excess liquids sampled will be returned to the sewer the day of collection.

V. References

United States. U.S. Environmental Protection Agency (USEPA). Science and Ecosystem Support Division. *Operating Procedure: Management of Investigation Derived Waste*. By Art Masters. N.p., 3 July 2014. Web. <https://www.epa.gov/sites/production/files/2015-06/documents/Management-of-IDW.pdf>.

United States. Environmental Protection Agency. Office of Emergency and Remedial Response. *Management of Investigation-Derived Wastes During Site Inspections.* Epa.gov, May 1991. Web.

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Procedure for Collection of Field Blank and Rinse Blank Samples

I. Introduction

This guideline provides procedures on the collection of field blank and rinse blank samples. Field and rinse blanks are QA/QC samples intended to provide control over the collection of environmental measurements and information on subsequent validation, review, and interpretation of generated data.

II. Definition

Rinse blanks are also called equipment blanks or rinsate blanks. Rinse blanks are rinse water samples taken from clean sample containers and sampling equipment to determine if residual contaminants are present in the equipment prior to sampling. Field blanks consist of a sample of analyte free water poured into the container in the field, preserved and shipped to the laboratory with field samples. If contamination is present in blank samples, the decontamination procedure or source of equipment must be modified to eliminate non-sample contamination. Frequency of blanks will be based upon the requirements in the QAPP, but typically should be collected at a rate of one per 20 environmental samples or per decontamination event, whichever is more frequent but no more than one per day.

The following is the definition of equipment blank from the USEPA publication EPA-505-B-04-900A, Uniform Federal Policy for Quality Assurance Project Plans, March 2005.

Equipment blanks – A sample of water free of measurable contaminants poured over or through decontaminated field sampling equipment that is considered ready to collect or process an additional sample. The purpose of this blank is to assess the adequacy of the decontamination process.

III. Safety

Wear safety glasses (or goggles) and gloves when working with and collecting the blank water samples. Also, conform to other safety practices in accordance with the project HASP. Perform the procedure with a helper if possible. Clean up all spilled water and sample preservatives immediately.

IV. Supplies

1. Sample bottles and labels
2. Laboratory grade water
3. Sampling equipment subject to blank sample collection
4. PPE including gloves, safety glasses and/or goggles. Refer to project HASP for full safety requirements.
5. Paper towels

V. Procedure for Collection of Field Blank (for aqueous sampling equipment)

1. The field blank must be collected in a designated clean area of the site or field office. Spread out clean plastic sheeting on a suitable work surface. Have paper towels handy to wipe up accidental spills.

2. Stage the laboratory bottles, laboratory grade water, and appropriate sampling equipment in the designated area. Prepare the appropriate sampling equipment. For aqueous sampling, a peristaltic pump is typically used. Install a clean section of silastic pump head tubing and attach clean sections of the sample collection tubing (e.g., Teflon-lined poly tubing) to the inlet and outlet ends of the pump head tubing.

3. Insert the intake end of the tubing into a bottle of laboratory water. Turn the pump on and allow the tubing to be purged with the laboratory water for at least 2 minutes prior to capturing the pumped water in laboratory bottles. Capture the purge water in a 5-gallon bucket or other suitable container for disposal.

4. After purging is completed, continue to pump laboratory grade water through the tubing. Collect the pumped water consistent in the appropriate laboratory bottles in a round-robin fashion, filling each bottle approximately ¼ full at a time, repeating until all bottles are filled.

5. If samples are being shipped to multiple laboratories, use the respective laboratory's water to fill their sample containers. If multiple laboratory water sources are used, purging must be performed again when switching between the water supplies.

6. Transfer the filled field blank bottles to the sample manager for sample management.

7. Clean up any spills and discard any disposable equipment used (e.g., pump tubing) as appropriate.

VI. Procedure for Collection of Rinse Blank

1. The rinse blank must be collected in a designated clean area of the site or field office. Spread out clean plastic sheeting on a suitable work surface. Have paper towels handy to wipe up accidental spills.

2. Stage the laboratory bottles, laboratory grade water, and appropriate decontaminated sampling equipment in the designated area. All items used to collect and process solid matrix samples, including, but not limited to, ponar dredge, stainless steel bowls or pots, stainless steel or disposable plastic spoons, spatulas, etc. must be included in the rinse blank.

3. Pour laboratory water over the surfaces of the equipment that are expected to contact sample during collection and processing. Capture this rinse water in a suitable container used during sampling, such as a stainless-steel bowl or pot.

4. Transfer the rinse water from the bowl or pot to the appropriate laboratory sample bottles. Fill the bottles in a round-robin fashion, ¼ at a time, until full. Multiple rounds of equipment rinses and transfer to bottles may be necessary to fill the sample bottles.

5. If samples are being shipped to multiple laboratories, use the respective laboratory's water to fill their sample containers. If multiple laboratory water sources are used, rinses must be performed with each laboratory's water to generate their respective rinse blank samples.

6. Transfer the filled field blank bottles to the sample manager for sample management.

7. Clean up any spills and discard any disposable equipment used (e.g., plastic spoons, foil trays, etc.) as appropriate.

Procedure to Conduct Sample Management

I. Introduction

This guideline is to provide reference information on sample management procedures.

II. Definitions

Target Compound List (TCL). This is a list of organic compounds typically analyzed for by the CLP. The list is broken into three subdivisions: volatiles, semi-volatiles, and pesticide/PCBs.

Target Analyte List (TAL). This is a list of inorganic parameters typically analyzed for by the CLP. Parameters on this list include heavy metals and cyanide.

Trip Blanks. Trip blanks are used to check for sample contamination originating from sample transport and shipping, as well as from site conditions and cross-contamination. Trip blanks are necessary when aqueous environmental samples are collected for volatile organic analysis.

Field Blanks. Field blanks (or equipment blank) are used to check the efficacy of sampling equipment decontamination procedures. Field blanks are collected for each type of non-dedicated sampling equipment used onsite. Typically, demonstrated analyte-free water is poured over the equipment and collected into containers and analyzed for the analytes of concern.

Field Duplicate. These are two separate samples collected at the same sampling point. Field duplicates are used to determine field sampling precision and representativeness and are collected at a set frequency for each analyte group.

Matrix Spike/Matrix Spike Duplicate/Matrix Duplicates (MS/MSD). This is the process by which standard mixes of various organic TCL or inorganic TAL compounds are added to field samples prior to extraction or digestion. The percent recoveries of the MS and MSD analyses are used to evaluate the matrix effect of the sample upon the accuracy of the analytical methodology. The relative percent difference (RPD) between the MS and MSD results is an indicator of analytical precision based on each sample matrix. Generally, triple volume is required for aqueous samples for sample. MS, and MSD analyses. These samples are collected in the field at a frequency of at least 5 percent per matrix/concentration. Extra volume is also required for solid samples. Frequency of MS/MSD collection and volume requirements are detailed in each program-specific QAPP.

III. Guidelines

The purpose of sample management is to assure that all samples collected during this site investigation are accounted for when the project is completed. The sample management officer is also responsible for assuring that the proper quality assurance/quality control (QA/QC) samples are collected. These purposes are achieved by adhering to the following procedures.

1) Laboratory Coordination

Subcontractor laboratory(ies) will be procured for the project to conduct analysis of parameters listed in the QAPP. Weekly contact must be maintained with these laboratories to inform them of upcoming sampling.

2) Preparing the Sample Containers

a) The laboratory will purchase certified clean sample containers from an approved supplier. Copies of these certifications will be brought to the site while sampling and then kept in site files for future reference.

b) Each bottle used to collect a sample must be identified by a supplier and lot number to ensure that it is permanently associated with the sample collected in that particular container. This procedure also applies to containers used to carry demonstrated analyte-free water to be used for blank preparation. This is to ensure that for all samples collected, the specific sample bottles used can be traced to the sample container contractor, and QC certification paperwork and custody records are applicable to their identifying lot numbers.

3) QA/QC Samples

a) Field Blanks
   i. Field blanks are collected for each type of equipment used to collect samples.
   ii. Field blanks will be collected at a frequency described in the QAPP. Decontaminated equipment must be properly stored in an area and in a manner that will prevent cross contamination.
   iii. Field blanks typically consist of pouring demonstrated analyte-free water over clean equipment and collecting it into sample containers to be analyzed for the analytes of concern. Different sample equipment and sampling methodology may require variations in field blank collection methods.
   iv. Field blanks are preserved, packaged, and shipped in the same manner as low concentration aqueous environmental samples.

b) Field Duplicates
   i. Samples for field duplicate (FD) analysis are collected in the field, for each matrix sampled at a frequency as described in QAPP.
   ii. Sufficient quantity of matrix must be collected from the same sample location to fill a duplicate set of sample containers. The duplicate volume is shipped to the laboratory under a separate sample ID ("blind" FD).
   iii. For soil/sediment samples the volatile organic fraction is collected as collocated grab samples, while the non-volatile fraction is homogenized prior to collection.

c) Matrix Spike/Matrix Spike Duplicate (MS/MSD) & Matrix Spike/Matrix Duplicate (MS/MD)
   i. The designation of a sample for MS/MSD analysis for organics and MS/MD (MD is equivalent to LD, lab duplicate) analysis for inorganics is required for 1 in 20 environmental samples per concentration/matrix. For this program MS/MSD/MD analysis will be conducted at the frequency described in the QAPP.
   ii. Three times the total volume is necessary for collection of aqueous MS/MSD or MS/MD samples for most parameters (see QAPP for specific requirements). Extra volume is also required for the solid samples.
   iii. MS/MSD and MS/MD samples are noted as such on the chain of custody (COC).

4) Sample Documentation, Packaging, and Shipping Procedures

One or more of the field personnel will be designated as the sample management officer(s). The sample management officer will bear the ultimate responsibility for the documentation, packaging, and shipping of the samples. These procedures are outlined below.

a) Documentation/Chain of Custody

For documentation purposes, the field team will enter information about each sample into the field logbook as they collect the sample. As this information is entered into the logbook, it is also transmitted to the sample bottle label(s). The minimum information recorded includes the following:

- Sample number (sample ID)
- Sample date and time of collection
- Preservative(s) used
- Analyses required

i) Once all of the samples are grouped in association for shipment to a specific laboratory, the COC can be filled out. The sample labels are then covered with clear tape, if necessary. The sample labels will contain the following information:

- Louis Berger-designated sample number
- The month, day, and year the sample was collected
- The type of analysis requested
- The type of preservation performed in the field.

b) Packaging and Shipping Samples

i) Make sure the caps on the sample bottles are tightly sealed. Wipe down the outside of all of the sample bottles.

ii) Preserve the samples according to requirements in the QAPP.

iii) The custody seal must be applied to sample containers in such a manner as to reveal if the container was opened during transit. Note: Septum vials should not be covered over the top.

iv) Place containers in individual zip-lock bags. VOA vials for the same sample may be placed in one bag. Eliminate extra air space from the bag before sealing. EnCore® device used for VOC soil samples comes in its own ziplock bag and this bag will be used. Clean-hands mercury requires special handling and packaging in its own ziplock bag (see field sampling SOP for details).

v) Prepare the shipping container (usually a cooler). The cooler will be prepared so that no leakage can occur during shipping. All valves on the cooler will be securely duct taped, both inside and outside the cooler, and the cooler will be lined with either plastic or a large garbage bag. Only coolers that conform to the general design requirements in 49 CFR 173.410 will be used for shipment.

vi) Put 1-2 inches of packing material in the bottom of the coolers, and then place the samples into the garbage bag in the cooler.

vii) Surround the sample bottles with bags of ice (only the samples that need to be cooled – refer to the QAPP requirements for sample preservation). The ice will not be kept in its original bag but will be repacked into ziplock bags and double bagged to prevent leakage in case the inner bag tears. Use enough ice to ensure that the proper temperature (2-6°C) is maintained during transport. Place a temperature blank (40-mL vial filled with DI water and labeled as "temperature blank") into the cooler.

viii) Place packing material over and around the sample bottles. Sufficient packing material must be used so the bottles will not move or break during transport.

ix) Once the samples are packed, the plastic or garbage bag will be closed and securely tied/taped.

x) Prior to shipment, the relinquished by and received by sections of the COC form will be filled in. Generally, the shipper (courier) will not sign the COC. Therefore, the courier's name is filled in by the sample management officer. The original COC form will then be placed in a ziplock bag and taped to the inside of one of the lead cooler(s); one copy of the COC form(s) will be placed in a ziplock bag(s) and placed in the other cooler(s). One copy of the COC form will be retained by the sample management officer.

xi) Close the cooler and seal with strapping tape. If visibly dirty, the outside of the cooler will be wiped down. Apply signed and dated custody seals to the cooler. Place two custody seals diagonally across from each other where the cooler lid meets the cooler. The custody seals will be applied in such a manner as to reveal if the cooler was opened during transit.

xii) An address label will be placed on the outside of each cooler. The label will be covered with clear tape. If more than one cooler is being sent to one destination, each cooler will be appropriately labeled as 1 of X, 2 of X, *etc.* The airbill will be attached to one of the coolers. Usually, the samples will be sent via overnight carrier for next day delivery. This should be confirmed with the Field Team Leader.

xiii) The laboratory will be notified of the shipment before 9 a.m. ET on the day after shipping.

xiv) The sample management officer or designee will forward a draft COC to the laboratory prior to receipt of the samples and will perform a review of the laboratory log-in of the samples to ensure that it is consistent with the COC and QAPP QC requirements.

Note: Some samples may have very short holding times. In some limited instances, the samples may be either hand delivered to a laboratory or picked up by the laboratory's courier service.

Collection of Large Volume/Small Volume CSO Samples

I. Introduction

This procedure describes the methods for collecting whole water samples from combined sewer overflows (CSO) using a peristaltic pump for manual sampling. The collected samples will then be submitted to the selected laboratory for the filtration of suspended solids with subsequent analysis of the whole water, solids and dissolved phase. These analyses are specified in the QAPP. Refer to the QAPP for this aspect of the project to determine if modified clean-hands techniques are required for the samples collected. Collection into an intermediate container for filling of the sample containers being sent to the laboratory will not be permitted, with the exception of grain size samples, which, prior to shipment, are decanted and placed into a final sample container, as described below.

II. Equipment and Supplies

The following equipment will be used to conduct CSO sampling:

1. **Peristaltic Pump** with Silastic (platinum cured silicone flexible tubing) pump-head tubing connected to Teflon-lined polyethylene sample tubing will be used to obtain the water sample from a CSO source. This will allow for the water sample to be collected without contacting any internal pump mechanisms. Each Teflon-lined sample tube will be fitted with an ISCO 3/8 inch diameter stainless-steel strainer to help prevent clogging of the intake line. The utility of the peristaltic pump is limited to a maximum of approximately 20 to 26 feet of lift. The pump will be battery operated, with spare batteries on-hand for long duration sampling. If necessary, it may also be equipped with alligator clips to connect to a vehicle battery or deep cycle marine battery brought to the site. In some rare instances a generator may be used to power a 110 Volt Alternating Current (VAC) pump; however this is not currently planned for the project.

2. **Personnel Protective Equipment (PPE)** - including ASTM approved steel toe boots (or ASTM approved safety toe boots), safety glasses, high visibility reflective traffic vests and disposable nitrile gloves (refer to HASP for full PPE requirements), as needed. Hard hats will be worn to increase visibility of the work crews to pedestrians and traffic in the area and are required when working at these locations.

3. **Pre-Cleaned Samples Containers.** Samples for metals analyses will be collected in pre-cleaned 1 liter (L) plastic bottles. Samples for mercury analyses will be collected in 1 L “clean hands” prepared containers. Samples for organics analysis will be collected in pre-cleaned 20 L stainless steel containers, and, samples for TSS will be collected in a pre-cleaned 4 L plastic bottles.

III. Guidelines for CSO Sampling

1. Prior to mobilization to the site for CSO sampling antecedent dry weather conditions of 48 hours of 0.1 inch or less of rainfall will need to be met before sampling may begin.

2. For wet weather sampling: Prior to setting up the pumps at the overflow or outfall locations, the tide charts for Gowanus Canal will be referenced to determine what position the tide gates might be in (i.e., open or closed) at each CSO location during the course of the proposed storm/sampling event. Sampling will only be conducted when there is an observed flow over the regulator weir, or over the "bench", and through the tide gates to the Canal.

   Note: The time that a storm arrives at the site with actual rainfall may be very different than the forecast time of arrvial at mobilization. Therefore, field crews will wait for the "go-ahead" from the field team leader (FTL) or project manager (PM) before deploying the sampling equipment through the open manhole into the waste stream. This will prevent having to dispose of contaminated sampling lines which were never used to collect a sample, which can be costly in time and materials.

3. The observation manhole downstream of the tide gate and the manhole showing the weir or bench overflow will be opened and inspected for active flow. It is required that two persons, each using a manhole hook to carefully slide the manhole cover back perform this operation. Follow the safe procedure for opening manholes found in the HASP Section 3.2.1 and the Task Hazard Analysis table of the HASP. Place the safety gate around any open manhole with the chain closed. Install all safety equipment as required for this location, including cones and "Caution" tape to warn pedestrians and prevent accidental access to the work areas.

4. During wet weather conditions, the tide gate at CSO locations will be inspected to make sure that it is open and that there is flow exiting to the receiving body, if possible. In the field, this will be done by inspecting for flow at the manhole downstream of the tide gate or at the tide gate itself. Due to the heaviness of the covers over these locations, covers (typically square) will only be opened once and safety gates will be installed around the openings. Once the sampling is completed the covers will be replaced. Other safety items such as cones or caution tape may be used in addition to the safety gates to divert pedestrians safely away. Electronic tide gate sensors may also be used to monitor the gates in order to determine whether a CSO event is actually occurring, if so equipped. At the present time there are no real-time tide gate inclinometers installed for our use.

   Note: CSO samples will not be collected from waters that are restricted by the tide gates and have backed up within the sewerage system. Only actively discharging CSO water will be collected, and within the elevations in the manhole which have been

predetermined and agreed to by NYCDEP and EPA. If this changes, notification from the PM is required. Record this change in the field notes or send a confirmation text from the field documenting this request to these individuals and the FTL.

5. Manholes used for sampling from or for observing regulator weir or bench overflow will be marked in the field prior to sampling. A GPS may eventually be used to identify where the sampling location is, if necessary.

6. When sampling is to proceed: Use clean-hands protocols, as necessary, attach the peristaltic pump head Silastic tubing to the pump if not already so equipped, and attach the plastic tubing clips to prevent the pump head tubing from traveling through the sampler (The clampdown mechanism should prevent this however clips may be used as a backup. Zip ties can be used in place of clips if necessary). Do the same for the ISCO 6712 samplers; however, use the ISCO tubing equipped with the blue alignment bands. Plastic tubing clips are not necessary for the samplers using the blue banded pump head tubing.

7. The Teflon-lined sample tubes, equipped with the 3/8 inch ISCO SS Strainers, will have been pre-assembled to a dedicated sampling pole for each location. Pre-assembly of the sampling pole will include attaching a tube at the predetermined sampling depth at a given location. These locations have been agreed to by the NYCDEP and EPA and will not be changed in the field unless directed to do so and documented by NYCDEP and EPA.

   Prior to mobilization, dedicated poles with multiple sampling tubes will be prepared for each sampling location. They will be prepared generally as follows: Attach the sample lines securely to the dedicated extendable pole, or non-extending pole dedicated for use at the specific location, using zip ties. The midpoint of the strainer will be set at the pre-determined elevation. A zip tie will be affixed to the Teflon-lined tube over the location where the strainer connects prior to affixing it to the pole. Zip ties should be spaced no more than 1 foot apart in the area of the tubing which will be in the flowing water. The zip ties are to be made snug but should not collapse the sample tubing. Position the clasp for the zip ties between the tubing sections in a manner to minimize the possibility of snagging debris when in the waste stream. Clip off any excess zip tie material to prevent debris from collecting on it when placed in the flowing water. Upon completion, the tubing will be neatly spread around the round metal deployment tube with the zip ties securely holding the tubes in place. If the discharge stream appears to be strong additional zip ties may be added. It is important to keep the area near the intakes of the tubing ends clean until deployment into the manhole. Use plastic where needed.

8. Deployment of the sampling equipment into the manhole: For the CSO locations, await the go-ahead before inserting the pole (Prior to deployment check to make sure that the proper dedicated pole is on site by measuring the intake intervals). Once the go-ahead

is received insert the pole with the attached intake lines into the water source (e.g., CSO). Make certain that two people do this while avoiding overhead hazards and avoiding contact with any NYCDEP monitoring equipment already in the manhole. It is important the bottom of the pole be placed in the deepest part of the chamber. This depth has been measured, as well as the bench height so that the intake tubes (for three elevations) will be positioned as planned in the waste stream. The dedicated pole with the sample tubing attached should be affixed to the safety gate which will be installed around the manhole opening once the cover has been removed. Zip ties or rope may be used to attach the pole to the safety gate.

Note: Once deployed observe the level of the water in the manhole as compared to the intake screens. A piece of surveyor flagging tape may be tied to the pole at different levels to aid in determining where the water level is as it rises and falls throughout the rain event. Contact the FTL or DPM during the sampling if it is observed that the bench height has been reached and there is a discharge through the tide gate however the "Bottom" sample tubing intakes are not in the water. It is unlikely that this will occur, however adjustments may need to be made.

9. Prior to or during sampling, place ice around the sample bottles/containers which are organized in plastic totes or buckets (20 L canisters). Sample bottles in the plastic totes or buckets consist of three different volume sizes (1 L, 4 L and 20 L). Refer to the QAPP for additional information on the volume sizes for different analytes. Ice is not required for the 4 L glass grain size samples. Note: Place covers on the jars prior to pouring ice around the containers.

10. Before any samples are taken, the flow rate through the Peristaltic pump needs to be measured. A sampling guide as shown in Figure 1 and table below will be provided to the sampling team. This guide provides the sample aliquot volume per bottle size per sampling time interval for a given duration of storm event. All sampling intervals provided as a guide to the sampling team are adjusted to 40 mL/s, the typical flow rate for the peristaltic pumps. Using the 1 L graduated cylinder, have one sampler record 10 seconds as the other sampler fills the container. Adjust the pump speed as necessary to pump 400 mL in 10 seconds. If the pump speed cannot be adjusted, recalculate the sampling intervals using the measured pump speed. This process should be repeated every half hour before taking a sample to ensure that the flow rate is still 40 mL/s or the previously measured rate, as flow can be affected by changing water levels, and clogging in the tubing lines.

Table: Example Sample Guide for filling Sample Bottles

Bottle Set	Sampling Period (hrs)	Number of Intervals	Bottle Volume (L)					
			1		4		20	
			Volume Increment (mL)	APPROX Pump Interval (sec)	Volume Increment (mL)	APPROX Pump Interval (sec)	Volume Increment (mL)	APPROX Pump Interval (sec)
A	2	9	110	*2.8*	420	*10.5*	2,220	*56*
B	4	17	60	*1.5*	220	*5.5*	1,180	*30*
C	6	25	40	*1.0*	150	*3.8*	800	*20*

11. To begin sampling start the pump, purge the line for at least a minute to purge the line volume four to five times and proceed to fill the sample containers. Samples will be collected as a time composite for the duration of the storm. Time composite samples are aliquots taken over multiple intervals throughout established time periods resulting in full containers at the end of those time periods (e.g. 17 aliquots, one taken every 15 minutes, over 4 hours). **Note**: When the pump is stopped between each of the timed samples the line must be fully purged for at least one minute or the equivalent of four to five volumes when it is restarted prior to collecting the next portion of water into the sample containers.

12. Record the start time for the sampling event.

13. Guidance for filling the jars, and the aliquots required will be provided prior to the sampling. Three sets (A,B and C) of sample jars or containers representing three different storm overflow periods are typically filled so that the chances of obtaining a satisfactory sample volume for each analyte type is almost guaranteed. During a given time interval, all the bottles for all three sets will be filled by the sampler using the sampling guide provided to the team. Note that the three sets will be filled sequentially, all bottles in set A will be filled first, followed by the bottles in set B and finally all the bottles in set C. Only one time-composite sample for the actual storm duration will be analyzed. The selection of the sample to be sent to the laboratory will be made by the DPM prior to leaving the sampling location. The samples not selected will be poured back into the manhole pending approval from NYCDEP, or they will be disposed of back at the treatment facility during Sample Management.

    Note: To track the progress of filling the sample containers, the 1 L graduated cylinder will be used as one of the “sample containers”, receiving the same aliquot filling as the 1 L bottles. Crews will assess the filling of the cylinder as sampling progresses.

14. When the jars become almost full, or the event has ended, turn off the pump. Do not overfill the containers. Essentially the first full bottle for each sample integration bottle set stops the sampling for that bottle set. Record the end time for the sampling event.

15. Remove the sampling equipment from the open manhole. Be mindful of traffic and overhead lines when removing the sampling equipment.

16. Safely replace the manhole cover as found in HASP Section 3.2.1 and the Task Hazard Analysis table of the HASP.

17. In the sample management area, label and pack the samples on ice as needed and ship samples to the laboratory. See SOP 08 Procedure to Conduct Sample Management.

18. Some of the parts for the manual sampling devices, specifically the tubing train, are dedicated and require replacement prior to sampling at each location for each sampling event.

19. Quality Control Samples: Duplicate samples, as needed, will be collected using a separate pump assembly with identical flow rates and tubing apparatus. MS/MSD samples will be collected along with the normal sampling process using the same tubing.

20. Parts of the sampling setup, specifically the tubing apparatus (pole, screens, tubing lengths), are dedicated and require replacement prior to the next sampling event at each location.

21. The sample collection tote and pump will be decontaminated between sampling events.

22. At the end of sampling at the sampling location: clean-up and demobilize the sampling location; decontaminate all equipment before putting in storage by wiping with Clorox wipes; discard used equipment and PPE in the trash. Remaining excess sample brought to the sample management area will be poured into the sanitary or combined sewer system.

Title: Decanting Samples for Grain Size Analysis

I. Introduction

This procedure describes the methods for decanting water samples collected from combined sewer overflows (CSOs), stormwater runoff locations (MS4), and at other locations that may be included during future tasks. Once the collected sample has been decanted the remaining material, consisting of grit, suspended solids and other particles in water will then be submitted to the selected laboratory for subsequent grain size analysis. These analyses are specified in the QAPP.

Decanting is a term used in wine making which describes the process of pouring from one container into another without disturbing the settled material in the first container. The process of decanting CSO waste is more difficult, since the suspended material which has been allowed to settle is often very light and disturbs too easily when being poured. Therefore, although the term "decant" is used in this document, the process is actually one of syphoning.

II. Equipment and Supplies

The following equipment will be used to decant the original sample, which has been collected in up to five 1-gallon clear glass jars:

- Tygon tubing, or other transparent flexible tubing (approximate ¼" to 3/8" ID)
- In-line tubing valve or clamp, for tubing up to 0.45" (http://www.usplastic.com/catalog/item.aspx?itemid=31257&catid=858)
- 12" nylon bottle brush
- Plastic sheeting
- 5-gallon collection bucket
- PPE—Safety glasses, nitrile gloves, and face mask
- Waterproof field camera
- 1-gallon plastic jar for sample compositing

III. Guidelines for Decanting/Syphoning the Collected Sample

- At the completion of a sampling event, the sample obtained for grain size shall be transported to an appropriate field facility where the particles are allowed settle out.
- Verify to ensure that each 1-gallon jar in the sampling tote to be decanted has the sampling location, sampling date, event number, and any additional information (split, duplicate, etc.) already written on the cap with a permanent marker. This information must be verified prior to decanting.

- The sample should not be moved during this period. This resting period should be a minimum of 8 hours, however 24 hours to 7 days might be required if the sample has light fine material floating in it. Allowing the sample to settle more than a week, if necessary, should be discussed with the Project Chemist or Project Manager.

Decanting, for these purposes, is performed by syphoning off the clearer water from above the settled particles. These settled particles are what are being gathered for analysis, in addition to any particles adhering to the walls of the sample jar.

IV. Procedure for Decanting/Syphoning the Sample

Prior to decanting the sample, the materials and sample must be carefully carried and placed onto a prepared table that is covered with plastic sheeting. Care must be taken when transporting the sample in order to avoid agitating solids that have settled.

- All the grain size jars representing a particular sample shall be decanted together. All supplies shall be gathered and prepared. These shall include:
  - Up to five 1-gallon wide mouth glass jars containing the sample to be decanted
  - The Tygon tubing used to syphon the water from above the settled material. Prepare the tubing as follows:
    - Cut the tubing to sufficient length to reach from the bottom of the glass jar which is on the table, out its top and then extend to the bottom of the 5-gallon collection bucket which is placed on the floor next to the table
    - Affix a clamp, if used, to control the flow from through the syphon
- CAUTION: Wear the appropriate PPE—Safety glasses and nitrile gloves at a minimum; a face mask is optional
- NOTE: Before opening the sample jars, ensure that all jars have the same markings on the lids.
- Record sample information on the Grain Size Decanting Form

Open all sample jars from only one location at a time. Fill the tubing with tap or distilled water and clamp/pinch the tubing near the discharge end

- Carefully insert the tubing into the jar about 1 inch below the water level
- Place the discharge end in the collection bucket
- Open the clamp, or un-pinch the line, and allow the syphon action to transfer the water into the collection bucket
- Slow the syphoning action if particles appear to be drawn into the tube or if there is floating matter in the water which might otherwise be drawn into the tube
- Continue carefully syphoning off the water, moving the tubing down as the water level drops

- As the bottom of the jar is approached and solids appear to be drawn near the tube, stop syphoning
- Continue syphoning each of the remaining containers in a similar fashion
- NOTE: The final combined sample being shipped to the laboratory will be in a 1-gallon plastic container. Therefore the material remaining in each of the five jars after syphoning cannot be more than 20% of one gallon. Typically, approximately one-half gallon is shipped to the laboratory.
- Combine the remaining material from each of the five jars into one plastic jar as follows:
  - Stir the material in the jar and pour all the solids into the plastic sample jar
  - Using the nylon bottle brush to scrub off any particulates which have adhered to the sides of the jar and pour into the plastic sample jar. If some water is needed to release this material, take it from the collection bucket. Pour the scrubbed off material into the plastic jar.
  - Continue this process for each of the glass jars
  - Finally, if necessary, rinse the brush into the plastic jar using a small amount of water from the collection bucket
- Label the cap of the plastic sample jar, including: the sampling location, sampling date, event number, and any additional information (split, duplicate, etc.) and give the sample to the Sample Management personnel
- NOTE: Duplicate samples are not labeled with the sampling location, only with the sampling date and the word “Duplicate”. The lab should not know the parent sampling location.

V. Photo Collection During Decanting/Syphoning Process

Photos of each sample should be taken in a manner that allows the person processing the photos to easily identify which samples the photos came from. The simplest way to do this is to include a jar lid with sample information on it in each photo. The following photos should be taken:

- Before decanting: a photo of all settled jars at the same time
- Before decanting: a close-up photo of one of the settled jars
- After decanting and before combining: a photo of all settled jars at the same time
- After decanting and before combining: a close-up photo of one of the settled jars
- A final photo of the plastic jar containing all the decanted, composited grain size samples

VI. Field Data Reporting and Grain Size Sample Handling

- Deliver all final samples and all recorded field data to the Sample Management (SM) Personnel. This includes:

- Labeling the plastic jar containing the decanted material from a single location including date of collection and sampling event, Hand all samples for each location to the SM personnel. Duplicates and splits are filtered as unique samples and labeled as such.
- Deliver the Grain Size Decanting Forms for each sampling location to the SM personnel. This data must include:
  - The sample location ID
  - If the samples were duplicates or splits. If they are duplicates, which location ID they are duplicates of
  - The number of containers decanted
  - Date of sampling and date of decanting
  - Anything notable such as "only three jars decanted"

VII. Cleanup and Preparation for Next Event

Cleanup and preparation for the next event shall consist of the following:

- Rinse out all used tubing using the hose and allow to air dry.
- Clean and save any tubing clamps
- Scrub all glass jars and lids with the nylon bottle brush, rinse and allow to air dry. Make sure that all lids have the Teflon liner and that the liner has been inserted the right way. There should not be water behind the liner.
- Reload the totes for the next event after the jars have dried. Make sure to place cardboard between the jars so that they don't break when they are being transported to or from sampling locations.

Gowanus Canal Grain Size Decanting Form

Personnel Present:			**Date and Time:**		
Location	**Sampling Date**	**Sample Number/ Time**	**Date Decanted**	**Number of Jars**	**Notes**

Gowanus Canal Grain Size Decanting Form

Observations/Comments:	
Staff Name (Printed):	**Agency:**
Staff Signature:	**Date and Time:**

Attachment 2

Laboratory Standard Operating Procedures

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SUMMARY OF SGS AXYS METHOD MLA-021 REV. 12 VER. 07:

ANALYTICAL METHOD FOR THE DETERMINATION OF POLYCYCLIC AROMATIC HYDROCARBONS (PAH), ALKYLATED POLYCYCLIC AROMATIC HYDROCARBONS, AND ALKANES

SGS AXYS Method MLA-021 describes the determination of concentrations of PAHs, alkylated PAHs and alkanes in solid (sediment, soil, ash), tissue (including blood), aqueous, XAD-2 column (resin and filters), air, and oil samples and in solvent extracts such as those produced by dialysis of SPMD (Semipermeable Membrane Device) samplers.

The method may be used for analysis of samples where USEPA Methods 1625B or 8270C/D have been requested **provided the modifications described in this document are permitted by contract**.

This summary document covers only the analysis of PAHs and alkylated PAHs.

Target Analytes

PAHs and alkylated PAHs - determined by multi-point calibration [2]

List 1 Compounds

Naphthalene [1]	Benzofluoranthenes
Acenaphthylene	Benzo[e]pyrene
Acenaphthene	Benzo[a]pyrene
Fluorene	Perylene
Phenanthrene	Dibenz[a,h]anthracene
Anthracene	Indeno[1,2,3-cd]pyrene
Fluoranthene	Benzo[ghi]perylene
Pyrene	Dibenzothiophene
Benzo[a]anthracene	2-Methylnaphthalene [1]
Chrysene	2,6-Dimethylnaphthalene [1]
Benzo[b]fluoranthene	2,3,5-Trimethylnaphthalene [1]
Benzo[j+k]fluoranthenes	1-Methylphenanthrene

List 2 Compounds

1-Methylnaphthalene [1]	C1-Phenanthrenes/Anthracenes
C1-Naphthalenes [1]	1,7-Dimethylphenanthrene
1,2-Dimethylnaphthalene [1]	1,8-Dimethylphenanthrene
C2-Naphthalenes [1]	2,6-Dimethylphenanthrene
2,3,6-Trimethylnaphthalene [1]	3,6-Dimethylphenanthrene
C3-Naphthalenes [1]	C2-Phenanthrenes/Anthracenes
1,4,6,7-Tetramethylnaphthalene [1]	1,2,6-Trimethylphenanthrene
C4-Naphthalenes [1]	C3-Phenanthrenes/Anthracenes
2-Methylphenanthrene	Retene

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3-Methylphenanthrene
9/4-Methylphenanthrenes
2-Methylanthracene
C4-Phenanthrenes/Anthracenes
Biphenyl

List 3 Compounds

C1-Biphenyls
C2-Biphenyls
C1-Acenaphthenes
2-Methylfluorene
C1-Fluorenes
1,7-Dimethylfluorene
C2-Fluorenes
C3-Fluorenes
2/3-Methyldibenzothiophenes
C1-Dibenzothiophenes
2,4-Dimethyldibenzothiophene
4,6-Dimethyldibenzothiophene
C2-Dibenzothiophenes
C3-Dibenzothiophenes
C4-Dibenzothiophenes
3-Methylfluoranthene/Benzo[a]fluorene
C1-Fluoranthenes/Pyrenes
C2-Fluoranthenes/Pyrenes
C3-Fluoranthenes/Pyrenes
C4-Fluoranthenes/Pyrenes
1-Methylchrysene
5/6-Methylchrysenes
C1-Benzo[a]anthracenes/Chrysenes
5,9-Dimethylchrysene
C2-Benzo[a]anthracenes/Chrysenes
C3-Benzo[a]anthracenes/Chrysenes
C4-Benzo[a]anthracenes/Chrysenes
7-Methylbenzo[a]pyrene
C1-Benzofluoranthenes/Benzopyrenes
C2-Benzofluoranthenes/Benzopyrenes

[1] Maximum value when determined in XAD-2 adsorbent.

[2] Optionally calibration may be performed by single-point bracketing calibration.

1.0 EXTRACTION AND CLEANUP PROCEDURES

All samples are spiked with deuterated surrogate standards prior to extraction and extracted as per the table below. Optional extraction procedures are shown within parentheses.

Sample Extraction

Matrix	Extraction
Aqueous	Sample with ≤1% suspended solids - Liquid-liquid extraction with dichloromethane. Sample with >1% suspended solids - sample is centrifuged prior to extraction and the particulate fraction separately extracted by Soxhlet extraction with dichloromethane. The two extracts are then combined. (Optional: the supernatant can be added to the extraction solvent for the particulate)
Solid (sediment, soil, sludge, particles on filter paper)	Soxhlet extraction with dichloromethane (Optional: Base digestion and liquid-liquid extraction with hexane)

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Matrix	Extraction
Solid (ash)	Soxhlet extraction with toluene
Solid (fly ash)	Sonication with hydrochloric acid and filtering. Liquid-liquid extraction of filtrate using dichloromethane, Soxhlet extraction of particulate using toluene:acetone 80:20. The two extracts are combined.
Tissue	Soxhlet extraction with dichloromethane. (Optional: Base digestion and liquid-liquid extraction with pentane)
Whole blood/serum/plasma	Liquid-liquid extraction with ethanol:hexane:saturated ammonium sulphate.
XAD-2 column and filter	XAD-2 columns and filters are usually co-extracted for multiple analyses (For example: PCB, pesticides) and the resulting extracts are split with a portion being used for PAH analysis
Ambient air (PUF and filter)	The PUF and filter(s) are Soxhlet extracted together using dichloromethane
Stationary Source Air Samples (Stack Gas sample trains)	The filter is sonicated with dilute hydrochloride acid and filtered. Equipment rinsates are collected, filtered, dried and/or extracted depending on sampling conditions.
SPMD samples	SPMD tubes are dialyzed with hexane.

After extraction the extracts are routinely cleaned up using the following procedures:

- column chromatography on silica
- gel permeation (Biobeads) column chromatography
- treatment with activated copper (except tissues)

The extracts may be cleaned up further, as necessary, using some or all of the following procedures:

- washing with base
- column chromatography on Biobeads
- column chromatography on alumina

2.0 INSTRUMENTATION

Instrumental analysis is performed by low-resolution mass spectrometry (LRMS) using an RTX-5 capillary GC column. The LRMS is operated at a unit mass resolution in the electron impact (EI) ionization mode using multiple ion detection (MID) acquiring at least one characteristic ion for each target analyte and surrogate standard.

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Benzo[b]fluoranthene Correction to Remove Surrogate Interference

At low concentrations native benzo[b]fluoranthene results are affected by an interference from the surrogate standard D_{12}-benzo[k]fluoranthene. D_{12}-benzo[k]fluoranthene has the same chromatographic retention time as benzo[b]fluoranthene and produces an artifactual response in the quantifying ion channel m/z 252. To remedy this interference the estimated peak area contribution from D_{12}-benzo[k]fluoranthene is subtracted from the benzo[b]fluoranthene peak area before quantification.

The peak area of d_{12}-benzo[b]fluoranthene in ion channel *m/z* 252 of the procedural blank is divided by the area of the same compound in ion channel *m/z* 264 to calculate the ion abundance ratio, usually around 0.003. (D_{12}-benzo[b]fluoranthene is used rather than d_{12}-benzo[k]fluoranthene because it doesn't coelute with any known native compound.) The corrected peak area of the native benzo[b]fluoranthene is then calculated as:

$$A^{\text{corr.}}_{\text{benzo}[b]\text{fluoranthene}(252)} = A^{\text{sample}}_{\text{benzo}[b]\text{fluoranthene }(252)} - A^{\text{sample}}_{\text{d12-benzo}[k]\text{fluoranthene}(264)} \left(\frac{A^{\text{blank}}_{\text{d12-benzo}[b]\text{fluoranthene}(252)}}{A^{\text{blank}}_{\text{d12-benzo}[b]\text{fluoranthene}(264)}} \right)$$

Where $A^{\text{corr.}}_{\text{benzo}[b]\text{fluoranthene}(252)}$ = Corrected peak area for benzo[b]fluoranthene in the sample

$A^{\text{sample}}_{\text{benzo}[b]\text{fluoranthene }(252)}$= Measured peak area for benzo[b]fluoranthene in the sample

$A^{\text{sample}}_{\text{d12-benzo}[k]\text{fluoranthene}(264)}$= Peak area for d_{12}-benzo[k]fluoranthene in the sample

$A^{\text{blank}}_{\text{d12-benzo}[b]\text{fluoranthene}(252)}$= Area for d_{12}-benzo[b]fluoranthene (m/z 252) in the blank

$A^{\text{blank}}_{\text{d12-benzo}[b]\text{fluoranthene}(264)}$= Area for d_{12}-benzo[b]fluoranthene (m/z 264) in the blank

This correction is performed by the instrument quantification software and the LIMS software.

3.0 CALIBRATION

Initial calibration is performed using a five point calibration series of solutions that encompass the working concentration range. Initial calibration solutions contain the suite of labelled surrogate and recovery standards and authentic target PAHs listed as "PAHs and alkylated PAHs determined by multi-point calibration". Calibration procedures use the mean RRFs determined from the initial calibration to calculate analyte concentrations. Calibration is verified at least once every 12 hours by analysis of a mid-level calibration solution.

Upon the request of the client single-point bracketing calibration protocols may be followed. Bracketing calibration procedures use mean RRFs from the analysis of the mid-level calibration solution to calculate analyte concentration.

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Concentration of PAHs/Alkylated PAHs Calibration Standard Solutions

TARGET ANALYTE	Level A (Sens. Std) (ng/mL)	Concentration of Calibration Standard Solutions (ng/mL)					Conc. of Native Std (Low Level) (ng/mL)	Conc. of Native Std (High Level) (ng/mL)
		Level B	Level C	Level D	Level E (Mid-level)	Level F		
Acenaphthene	10	50	100	500	2000	5000	2000	20 000
Acenaphthylene	10	50	100	500	2000	5000	2000	20 000
Anthracene	10	50	100	500	2000	5000	2000	20 000
Benz[a]anthracene	10	50	100	500	2000	5000	2000	20 000
Benzo[b]fluoranthene	10	50	100	500	2000	5000	2000	20 000
Benzo[k]fluoranthene	10	50	100	500	2000	5000	2000	20 000
Benzo[ghi]perylene	10	50	100	500	2000	5000	2000	20 000
Benzo[a]pyrene	10	50	100	500	2000	5000	2000	20 000
Benzo[e]pyrene	10	50	100	500	2000	5000	2000	20 000
Biphenyl	10	50	100	500	2000	5000	2000	20 000
Chrysene	10	50	100	500	2000	5000	2000	20 000
Dibenzo[ah]anthracene	10	50	100	500	2000	5000	2000	20 000
2,6-Dimethylnaphthalene	10	50	100	500	2000	5000	2000	20 000
Fluoranthene	10	50	100	500	2000	5000	2000	20 000
Fluorene	10	50	100	500	2000	5000	2000	20 000
Indeno[1,2,3-cd]pyrene	10	50	100	500	2000	5000	2000	20 000
1-Methylnaphthalene	10	50	100	500	2000	5000	2000	20 000
2-Methylnaphthalene	10	50	100	500	2000	5000	2000	20 000
1-Methylphenanthrene	10	50	100	500	2000	5000	2000	20 000
Naphthalene	10	50	100	500	2000	5000	2000	20 000
Perylene	10	50	100	500	2000	5000	2000	20 000
Phenanthrene	10	50	100	500	2000	5000	2000	20 000
Pyrene	10	50	100	500	2000	5000	2000	20 000
2,3,5-Trimethylnaphthalene	10	50	100	500	2000	5000	2000	20 000
Dibenzothiophene	10	50	100	500	2000	5000	2000	20 000
3,6-Dimethylphenanthrene	10	50	100	500	2000	5000	2000	20 000
Retene	10	50	100	500	2000	5000	2000	20 000
2-Methylanthracene	10	50	100	500	2000	5000	2000	20 000
1,2-Dimethylnaphthalene	10	50	100	500	2000	5000	2000	20 000
2-Methylphenanthrene	10	50	100	500	2000	5000	2000	20 000
1,2,6-Trimethylphenanthrene	10	50	100	500	2000	5000	2000	20 000
2,3,6-Trimethylnaphthalene	10	50	100	500	2000	5000	2000	20 000
1,7-Dimethylphenanthrene	10	50	100	500	2000	5000	2000	20 000
1,4,6,7-Tetramethylnaphthalene	10	50	100	500	2000	5000	2000	20 000
2-Methylfluorene	10	50	100	500	2000	5000	2000	20 000
1,7-Dimethylfluorene	10	50	100	500	2000	5000	2000	20 000
2-Methyldibenzothiophene	10	50	100	500	2000	5000	2000	20 000
2,4-Dimethyldibenzothiophene	10	50	100	500	2000	5000		
4,6-Dimethyldibenzothiophene	10	50	100	500	2000	5000	2000	20 000
5,9-Dimethylchrysene	10	50	100	500	2000	5000	2000	20 000
7-Methylbenzo[a]pyrene	10	50	100	500	2000	5000	2000	20 000
3-Methylfluoranthene	10	50	100	500	2000	5000	2000	20 000
6-Methylchrysene	10	50	100	500	2000	5000	2000	20 000
1-Methylchrysene	10	50	100	500	2000	5000	2000	20 000

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LABELLED SURROGATE STANDARDS	Level A (Sens. Std) (ng/mL)	Concentration of Calibration Standard Solutions					Conc. of Surrogate Std (Low Level) (ng/mL)	Conc. of Surrogate Std (High Level) (ng/mL)
		Level B	Level C	Level D	Level E	Level F		
d_8-Naphthalene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{10}-2-Methylnaphthalene	2000	2000	2000	2000	2000	2000	2000	20 000
d_8-Acenaphthylene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{10}-Phenanthrene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{10}-Fluoranthene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Benz[a]anthracene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Chrysene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-2,6-Dimethylnaphthalene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Benzo[b]fluoranthene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Benzo[k]fluoranthene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Benzo[a]pyrene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Perylene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Indeno[1,2,3-cd]pyrene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{14}-Dibenzo[ah]anthracene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Benzo[ghi]perylene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{10}-Biphenyl	2000	2000	2000	2000	2000	2000	2000	20 000
d_8-Dibenzothiophene	2000	2000	2000	2000	2000	2000	2000	20 000
LABELLED RECOVERY STANDARDS							**Conc. of Recovery Std (ng/mL)**	
d_{10}-Acenaphthene	2000	2000	2000	2000	2000	2000	20 000	
d_{10}-Pyrene	2000	2000	2000	2000	2000	2000	20 000	
d_{12}-Benzo[e]pyrene	2000	2000	2000	2000	2000	2000	20 000	

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4.0 QUANTIFICATION PROCEDURES

Concentrations of target PAHs are calculated using the isotope dilution method of quantification. Compounds are quantified by comparing the area of the quantification ion to that of the corresponding deuterium-labelled standard and correcting for response factors. Response factors are determined daily using authentic PAHs. Calculations are carried out using HP EnviroQuant and Prolab MS-Extended for targeting and quantification.

$$\text{Concentration of Target (ng/g or ng/L)} = \left(\frac{\text{area of Target}}{\text{area of Surr Std}}\right) \times \left(\frac{\text{weight of Surr Std (ng)}}{\text{RRF}}\right) \times \left(\frac{1}{\text{weight of sample (g or L)}}\right)$$

where $\text{RRF} = \left(\frac{\text{area of Target}}{\text{area of Surr Std}}\right) \times \left(\frac{\text{concentration of Surr Std}}{\text{concentration of Target}}\right)$

and the Surr Std is either the surrogate or the internal standard

4.1 Reporting Limits

Concentrations and detection limits for the target PAHs are reported. Typical reporting units for all data are ng/g, ng/L, or ng/sample. Concentrations for solids are reported on a dry weight basis. Concentrations in tissues (including blood/serum/plasma) are reported on a wet weight basis and/or on a lipid weight basis when requested. Concentrations in aqueous are reported on a volume basis. Concentrations in XAD-2 resin, filters and stack gas samples are reported on a per sample basis or a per volume basis. Concentrations in particulate filters are reported on a per sample basis.

The following are commonly requested reporting limits:

Sample Specific Detection Limit or Sample Detection Limit (SDL) – determined individually for every sample analysis run by converting the area equivalent of 3.0 times (2.5 times for EPA 1600 series methods) the estimated chromatographic noise height to a concentration in the same manner that target peak responses are converted to final concentrations. The SDL accounts for any effect of matrix on the detection system and for recovery achieved through the analytical work-up. Equivalent term(s): Estimated Detection Limit (EDL) from EPA method 8290.

Method Detection Limit (MDL) - determined as specified by EPA Fed. Reg. 40 CFR Part 136 Appendix B (no iteration option). The 99% confidence level MDL is determined based on analysis of a minimum of 7 replicate matrix spikes fortified at 1-10 times the estimated detection limit. MDL is determined as required based on accreditation, contract and workload requirements.

Lower Method Calibration Limit (LMCL) - determined by prorating the concentration of the lowest calibration limit for sample size and extract volume. The following equation is used. ((lowest level cal conc.) x (extract volume))/sample size. Typical extract volume for aqueous and tissue samples is 100 µL for all other matrices typical extract volume is 500 µL.

For the analysis of PAHs SGS AXYS standard is to report sample concentrations using the SDL as the reporting limit.

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Analyte Ions Monitored, Surrogates Used and RRF Determination For PAH

TARGET ANALYTES	Quantification Ion (m/z)	Confirmation Ions (m/z)	Typical Ion Ratio (Conf./Quant.)	SURROGATE	Typical Retention Time (minutes)	RRF DETERMINED FROM
Naphthalene	128	102	0.064	d_8-Naphthalene	6.84	Naphthalene
Acenaphthylene	152	151	0.222	d_8-Acenaphthylene	10.83	Acenaphthylene
Acenaphthene	154	153	1.18	d_8-Acenaphthylene	11.33	Acenaphthene
Fluorene	166	165	1.01	d_{10}-Phenanthrene	12.63	Fluorene
Phenanthrene	178	176	0.202	d_{10}-Phenanthrene	15.04	Phenanthrene
Anthracene	178	176	0.196	d_{10}-Phenanthrene	15.15	Anthracene
Fluoranthene	202	200	0.214	d_{10}-Fluoranthene	18.06	Fluoranthene
Pyrene	202	200	0.219	d_{10}-Fluoranthene	18.60	Pyrene
Benz[a]anthracene[6]	228	226	0.281	d_{12}-Benz[a]anthracene	21.68	Benz[a]anthracene
Chrysene[1]	228	226	0.312	d_{12}-Chrysene	21.79	Chrysene
Benzo[b]fluoranthene	252	253	0.218	d_{12}-Benzo[b]fluoranthene	25.21	Benzo[b]fluoranthene
Benzo[j,k]fluoranthenes	252	253	0.215	d_{12}-Benzo[k]fluoranthene	25.30	Benzo[k]fluoranthene
Benzo[e]pyrene	252	253	0.213	d_{12}-Benzo[a]pyrene	26.36	Benzo[e]pyrene
Benzo[a]pyrene	252	253	0.217	d_{12}-Benzo[a]pyrene	26.58	Benzo[a]pyrene
Perylene	252	253	0.212	d_{12}-Perylene	27.00	Perylene
Dibenzo[ah]anthracene[2]	278	139	0.144	d_{14}-Dibenzo[ah]anthracene	31.86	Dibenz[ah]anthracene
Indeno[1,2,3-cd]pyrene	276	138	0.179	d_{12}-Indeno[1,2,3,cd]pyrene	31.71	Indeno[1,2,3-cd]pyrene
Benzo[ghi]perylene	276	138	0.194	d_{12}-Benzo[ghi]perylene	32.53	Benzo[ghi]perylene
Biphenyl[3]	154	152	0.304	d_{10}- Biphenyl	9.81	Biphenyl
Dibenzothiophene[3]	184	152	0.073	d_8-Dibenzothiophene	14.72	Dibenzothiophene
1-Methylnaphthalene[3]	142	141	0.962	d_{10}-2-Methylnaphthalene	8.81	1-Methylnaphthalene
2-Methylnaphthalene[3]	142	141	0.930	d_{10}-2-Methylnaphthalene	8.55	2-Methylnaphthalene
1-Naphthalenes[3]	142	[4]	[4]	d_{10}-2-Methylnaphthalene	[5]	1- & 2-Methylnaphthalene
2,6-Dimethylnaphthalene[3]	156	141	0.666	d_{12}-2,6 Dimethylnaphthalene	10.17	2,6-Dimethylnaphthalene
1,2-Dimethylnaphthalene	156	141	1.26	d_{12}-2,6 Dimethylnaphthalene	10.90	1,2-Dimethylnaphthalene
C2-Naphthalenes[3]	156	[4]	[4]	d_{12}-2,6 Dimethylnaphthalene	[5]	2,6- & 1,2-Dimethylnaphthalene

[1] Coelutes with Triphenylene
[2] Coelutes with Dibenz[ac]anthracene
[3] These compounds are in addition to the regular suite of analytes, and are analyzed by client request only.
[4] Secondary ion confirmation procedures do not apply
[5] RRT ranges apply to alkylated PAH Totals
[6] Benzo[a]anthracene coelutes with cyclopenta[cd]pyrene, which may contribute response in the second ion to cause a failing ion abundance ratio. Therefore, quantification of benzo[a]anthracene should use response from the first ion only.

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TARGET ANALYTES	Quantification Ion (m/z)	Confirmation Ions (m/z)	Typical Ion Ratio (Conf./Quant.)	SURROGATE	Typical Retention Time (minutes)	RRF DETERMINED FROM
2,3,5-Trimethylnaphthalene[3]	170	155	0.873	d_{12}-2,6 Dimethylnaphthalene	12.35	2,3,5- Trimethylnaphthalene
2,3,6-Trimethylnaphthalene	170	155	0.876	d_{12}-2,6 Dimethylnaphthalene	12.17	2,3,6- Trimethylnaphthalene
C3-Naphthalenes[3]	170			d_{12}-2,6 Dimethylnaphthalene	[5]	2,3,5- & 2,3,6-Trimethylnaphthalene
1,4,6,7-Tetramethylnaphthalene	184	139	0.027	d_{12}-2,6 Dimethylnaphthalene	13.89	1,4,6,7-Tetramethylnaphthalene
C4-Naphthalene		[4]	[4]	d_{12}-2,6 Dimethylnaphthalene	[5]	1,4,6,7-Tetramethylnaphthalene
2-Methylanthracene	192	191	0.531	d_{10}-Phenanthrene	16.45	2-Methylanthracene
3-Methylphenanthrene	192	191	0.608	d_{10}-Phenanthrene	16.27	1- & 2-Methylphenanthrene & 2-Methylanthracene
2-Methylphenanthrene	192	191	0.608	d_{10}-Phenanthrene	16.36	2-Methylphenanthrene
9/4-Methylphenanthrenes	192	191	0.634	d_{10}-Phenanthrene	16.59	1- & 2-Methylphenanthrene & 2-Methylanthracene
1-Methylphenanthrene[3]	192	191	0.634	d_{10}-Phenanthrene	16.64	1-Methylphenanthrene
C1-Phenanthrenes/Anthracenes[3]	192	[4]	[4]	d_{10}-Phenanthrene	[5]	1- & 2-Methylphenanthrene & 2-Methylanthracene
3,6-Dimethylphenanthrene[3]	206	191	0.342	d_{10}-Fluoranthrene	17.46	3,6-Dimethylphenanthrene
2,6-Dimethylphenanthrene	206	191	0.342	d_{10}-Fluoranthrene	17.54	3,6- & 1,7-Dimethyl-phenanthrenes
1,7-Dimethylphenanthrene	206	191	0.332	d_{10}-Fluoranthrene	17.89	1,7-Dimethylphenanthrene
1,8-Dimethylphenanthrene	206	191	0.332	d_{10}-Fluoranthrene	18.13	3,6- & 1,7-Dimethyl-phenanthrenes
C2-Phenanthrenes/Anthracenes[3]	206	[4]	[4]	d_{10}-Fluoranthrene	[5]	3,6- & 1,7-Dimethyl-phenanthrenes
1,2,6-Trimethylphenanthrene	220	205	0.581	d_{10}-Fluoranthrene	19.41	1,2,6-Trimethylphenanthrene
C3-Phenanthrenes/Anthracenes				d_{10}-Fluoranthrene	[5]	1,2,6-Trimethylphenanthrene
Retene[3]	234	219	1.63	d_{10}-Fluoranthene	19.53	Retene
C4-Phenanthrenes/Anthracenes	234	[4]	[4]	d_{10}-Fluoranthrene	[5]	Retene
C1-Biphenyls	168	[4]	[4]	d_{10}- Biphenyl	[5]	Biphenyl
C2-Biphenyls	182	[4]	[4]	d_{10}- Biphenyl	[5]	Biphenyl
C1-Acenaphthenes	168	[4]	[4]	d_8-Acenaphthylene	[5]	Acenaphthene
2-Methylfluorene	180	165	1.23	d_{10}-Phenanthrene	14.06	2-Methylfluorene
C1-Fluorenes	180	[4]	[4]	d_{10}-Phenanthrene	[5]	2-Methylfluorene
1,7-Dimethylfluorene	194	177	0.092	d_{10}-Phenanthrene	15.49	1,7-Dimethylfluorene
C2-Fluorenes	194	[4]	[4]	d_{10}-Phenanthrene	[5]	1,7-Dimethylfluorene

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TARGET ANALYTES	Quantification Ion (m/z)	Confirmation Ions (m/z)	Typical Ion Ratio (Conf./Quant.)	SURROGATE	Typical Retention Time (minutes)	RRF DETERMINED FROM
C3-Fluorenes	208	[4]	[4]	d_{10}-Phenanthrene	[5]	1,7-Dimethylfluorene
2/3-Methyldibenzothiophenes	198	197	0.738	d_8-Dibenzothiophene	16.07	2/3-Methyldibenzothiophenes
C1-Dibenzothiophenes	198	[4]	[4]	d_8-Dibenzothiophene	[5]	2/3-Methyldibenzothiophenes
2,4-Dimethyldibenzothiophene	212	197	0.514	d_8-Dibenzothiophene	17.08	2,4-Dimethyldibenzothiophene
4,6-Dimethyldibenzothiophene	212	197	0.160	d_8-Dibenzothiophene	16.94	4,6-Dimethyldibenzothiophene
C2-Dibenzothiophenes	212	[4]	[4]	d_8-Dibenzothiophene	[5]	2,4-Dimethyldibenzothiophene
C3-Dibenzothiophenes	226	[4]	[4]	d_8-Dibenzothiophene	[5]	2,4-Dimethyldibenzothiophene
C4-Dibenzothiophenes	240	[4]	[4]	d_8-Dibenzothiophene	[5]	2,4-Dimethyldibenzothiophene
3-Methylfluoranthene/Benzo[a]fluorene	216	215	0.880	d_{10}-Fluoranthrene	19.53	3-Methylfluoranthene
C1-Fluoranthenes/Pyrenes	216	[4]	[4]	d_{10}-Fluoranthrene	[5]	3-Methylfluoranthene
C2-Fluoranthenes/Pyrenes	230	[4]	[4]	d_{10}-Fluoranthrene	[5]	3-Methylfluoranthene
C3-Fluoranthenes/Pyrenes	244	[4]	[4]	d_{10}-Fluoranthrene	[5]	3-Methylfluoranthene
C4-Fluoranthenes/Pyrenes	258	[4]	[4]	d_{10}-Fluoranthrene	[5]	3-Methylfluoranthene
5/6-Methylchrysenes	242	[4]	[4]	d_{12}-Chrysene	23.15	6-Methylchrysene
1-Methylchrysene	242	[4]	[4]	d_{12}-Chrysene	23.32	1-Methylchrysene
C1-Benzo[a]anthracenes/Chrysenes	242	[4]	[4]	d_{12}-Chrysene	[5]	1- & 6-Methylchrysenes
5,9-Dimethylchrysene	256	[4]	[4]	d_{12}-Chrysene	24.49	5,9-Dimethylchrysene
C2-Benzo[a]anthracenes/Chrysenes	256	[4]	[4]	d_{12}-Chrysene	[5]	5,9-Dimethylchrysene
C3-Benzo[a]anthracenes/Chrysenes	270	[4]	[4]	d_{12}-Chrysene	[5]	5,9-Dimethylchrysene
C4-Benzo[a]anthracenes/Chrysenes	284	[4]	[4]	d_{12}-Chrysene	[5]	5,9-Dimethylchrysene
7-Methylbenzo[a]pyrene	266	[4]	[4]	d_{12}-Benzo[a]pyrene	29.35	7-Methylbenzo[a]pyrene
C1-Benzofluoranthenes/Benzopyrenes	266	[4]	[4]	d_{12}-Benzo[a]pyrene	[5]	7-Methylbenzo[a]pyrene
C2-Benzofluoranthenes/Benzopyrenes	280	[4]	[4]	d_{12}-Benzo[a]pyrene	[5]	7-Methylbenzo[a]pyrene
LABELLED SURROGATE STANDARDS	**Quantification Ion (m/z)**	**Confirmation Ions (m/z)**		**RECOVERY CALCULATED AGAINST**		
d_8-Naphthalene	136	134	0.095	d_{10}-Acenaphthene	6.80	
d_{10}-2-Methylnaphthalene	152	151	0.195	d_{10}-Acenaphthene	8.47	
d_{10}-Biphenyl	164	[4]	[4]	d_{10}-Acenaphthene	9.75	
d_{12}-2,6-Dimethylnaphthalene	168	150	0.747	d_{10}-Acenaphthene	10.07	
d_8-Acenaphthylene	160	158	0.159	d_{10}-Acenaphthene	10.80	
d_8-Dibenzothiophene	192	160	0.085	d_{10}-Pyrene	14.67	
d_{10}-Phenanthrene	188	184	0.143	d_{10}-Pyrene	14.97	
d_{10}-Fluoranthene	212	208	0.173	d_{10}-Pyrene	18.02	
d_{12}-Benz[a]anthracene	240	236	0.250	d_{10}-Pyrene	21.63	

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TARGET ANALYTES	Quantification Ion (m/z)	Confirmation Ions (m/z)	Typical Ion Ratio (Conf./Quant.)	SURROGATE	Typical Retention Time (minutes)	RRF DETERMINED FROM
d_{12}-Chrysene	240	236	0.278	d_{10}-Pyrene	21.73	
d_{12}-Benzo[b]fluoranthene	264	260	0.216	d_{12}-Benzo[e]pyrene	25.11	
d_{12}-Benzo[k]fluoranthene	264	260	0.208	d_{12}-Benzo[e]pyrene	25.23	
d_{12}-Benzo[a]pyrene	264	260	0.216	d_{12}-Benzo[e]pyrene	26.47	
d_{12}-Perylene	264	260	0.256	d_{12}-Benzo[e]pyrene	26.88	
d_{12}-Indeno[1,2,3,cd]pyrene	288	284	0.192	d_{12}-Benzo[e]pyrene	31.63	
d_{14}-Dibenzo[ah]anthracene	292	288	0.260	d_{12}-Benzo[e]pyrene	31.75	
d_{12}-Benzo[ghi]perylene	288	284	0.205	d_{12}-Benzo[e]pyrene	32.45	
LABELLED RECOVERY STANDARDS	**Quantification Ion (m/z)**	**Confirmation Ions (m/z)**				
d_{10}-Acenaphthene	164	160	0.464		11.24	
d_{10}-Pyrene	212	208	0.176		18.56	
d_{12}-Benzo[e]pyrene	264	260	0.269		26.25	

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5.0 QUALITY ACCEPTANCE CRITERIA

Samples are analyzed in batches consisting of a maximum of twenty samples, one procedural blank and one spiked matrix (OPR) sample. A duplicate is analyzed, provided there is sufficient sample, with batches containing 7-20 samples. Matrix spike/matrix spike duplicate (MS/MSD) pairs may be analyzed on an individual contract basis. The batch is carried through the complete analytical process as a unit. For sample data to be reportable, the batch QC data must meet the established acceptance criteria presented on the analysis reports.

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PAHs - QC Acceptance Criteria

MATRIX	Typical Sample Specific * Detection Limits							Procedural Blank Level				Acceptable OPR
	Solid	Aqueous	Tissue	XAD-2 Column	PUF & SPMD Extract	Filter	Oil	Solid & SPMD Extract	Aqueous	Tissue	All other matrices	
Analyte:	ng/g	ng/L	ng/g	ng	ng	ng	ng/g	ng (tot)	ng (tot)	ng (tot)	ng (tot)	% Recovery
Naphthalene	0.5	1	0.1	5	5	5	50	≤ 10	≤ 10	≤ 50	≤ 10 [1]	70-130
Acenaphthylene	0.5	1	0.1	5	5	5	50	≤ 5	≤ 5	≤ 5	≤ 5	70-140
Acenaphthene [5]	0.5	1	0.1	5	5	5	50	≤ 5	≤ 5	≤ 5	≤ 5	70-130
Fluorene	0.5	1	0.1	5	5	5	50	≤ 5	≤ 5	≤ 5	≤ 5	60-140
Phenanthrene	0.5	1	0.1	5	5	5	50	≤ 10	≤ 10	≤ 10	≤ 10	70-130
Anthracene	0.5	1	0.1	5	5	5	50	≤ 5	≤ 5	≤ 5	≤ 5	70-130
Fluoranthene	0.5	1	0.1	5	5	5	50	≤ 5	≤ 5	≤ 5	≤ 5	70-130
Pyrene	0.5	1	0.1	5	5	5	50	≤ 5	≤ 5	≤ 5	≤ 5	70-130
Benzo[a]anthracene	0.5	1	0.1	5	5	5	50	≤ 1.9	≤ 1	≤ 0.9	≤ 5	70-130
Chrysene	0.5	1	0.1	5	5	5	50	≤ 3.0	≤ 1	≤ 1.3	≤ 5	70-130
Benzo[b]fluoranthene	0.5	1	0.1	5	5	5	50	≤ 4.2	≤ 0.6	≤ 1.6	≤ 5	70-130
Benzo[j+k]fluoranthenes	0.5	1	0.1	5	5	5	50	≤ 2.1	≤ 0.4	≤ 1.2	≤ 5	70-130
Benzo[e]pyrene [5]	0.5	1	0.1	5	5	5	50	≤ 5	≤ 5	≤ 5	≤ 5	70-130
Benzo[a]pyrene	0.5	1	0.1	5	5	5	50	≤ 3.6	≤ 0.9	≤ 0.7	≤ 5	70-130
Perylene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	70-130
Dibenz[a,h]anthracene	1.0	2	0.2	10	10	10	100	≤ 2.8	≤ 0.7	≤ 0.7	≤ 5	70-130
Indeno[1,2,3-cd]pyrene	1.0	2	0.2	10	10	10	100	≤ 2.8	≤ 0.8	≤ 1.5	≤ 5	70-130
Benzo[ghi]perylene	1.0	2	0.2	10	10	10	100	≤ 2.4	≤ 0.9	≤ 0.7	≤ 5	70-130
Biphenyl	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	70-130
Dibenzothiophene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	60-140
1-Methylnaphthalene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5 [1]	70-130
2-Methylnaphthalene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5 [1]	70-130
2,6-Dimethylnaphthalene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10 [1]	70-130
1,2-Dimethylnaphthalene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10 [1]	60-140
2,3,5-Trimethylnaphthalene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10 [1]	50-150

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MATRIX	Typical Sample Specific * Detection Limits							Procedural Blank Level				Acceptable OPR
	Solid	Aqueous	Tissue	XAD-2 Column	PUF & SPMD Extract	Filter	Oil	Solid & SPMD Extract	Aqueous	Tissue	All other matrices	
Analyte:	ng/g	ng/L	ng/g	ng	ng	ng	ng/g	ng (tot)	ng (tot)	ng (tot)	ng (tot)	% Recovery
2,3,6-Trimethylnaphthalene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10 [1]	50-150
1,4,6,7-Tetramethyl-naphthalene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10 [1]	50-200
2-Methylanthracene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
3-Methylphenanthrene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	N.A.
2-Methylphenanthrene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	50-150
9/4-Methylphenanthrenes	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	N.A.
1-Methylphenanthrene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	50-150
3,6-Dimethylphenanthrene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	50-150
2,6-Dimethylphenanthrene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	N.A.
1,7-Dimethyl-phenanthrene [2]	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	50-150
1,8-Dimethylphenanthrene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	N.A.
1,2,6-Trimethyl-phenanthrene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	50-150
Retene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
2-Methylfluorene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
1,7-Dimethylfluorene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
2/3-Methyldibenzo-thiophenes	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	50-150
2,4-Dimethyl-dibenzothiophene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
4,6-Dimethyl-dibenzothiophene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
3-Methylfluoranthene [3]/ Benzo[a]fluorene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
5/6-Methylchrysenes	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
1-Methylchrysene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
5,9-Dimethylchrysene [4]	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150

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MATRIX	Typical Sample Specific * Detection Limits							Procedural Blank Level				Acceptable OPR
	Solid	Aqueous	Tissue	XAD-2 Column	PUF & SPMD Extract	Filter	Oil	Solid & SPMD Extract	Aqueous	Tissue	All other matrices	
Analyte:	ng/g	ng/L	ng/g	ng	ng	ng	ng/g	ng (tot)	ng (tot)	ng (tot)	ng (tot)	% Recovery
7-Methylbenzo[a]pyrene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
C1-Biphenyls	1.0	2	0.2	10	10	10	100	≤ 50	≤ 50	≤ 50	≤ 50	
C2-Biphenyls	1.0	2	0.2	10	10	10	100	≤ 250	≤ 250	≤ 250	≤ 250	
C1-Naphthalenes	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10 [1]	
C2-Naphthalenes	1.0	2	0.2	10	10	10	100	≤ 50	≤ 50	≤ 50	≤ 50 [1]	
C3-Naphthalenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15 [1]	
C4-Naphthalenes	1.0	2	0.2	10	10	10	100	≤ 25	≤ 25	≤ 25	≤ 25 [1]	
C1-Acenaphthenes	0.5	1	0.1	5	5	5	50	≤ 10	≤ 10	≤ 10	≤ 10	
C1-Fluorenes	1.0	2	0.2	10	10	10	100	≤ 25	≤ 25	≤ 25	≤ 25	
C2-Fluorenes	1.0	2	0.2	10	10	10	100	≤ 50	≤ 50	≤ 50	≤ 50	
C3-Fluorenes	1.0	2	0.2	10	10	10	100	≤ 20	≤ 20	≤ 20	≤ 20	
C1-Dibenzothiophenes	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	
C2-Dibenzothiophenes	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	
C3-Dibenzothiophenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
C4-Dibenzothiophenes	1.0	2	0.2	10	10	10	100	≤ 25	≤ 25	≤ 25	≤ 25	
C1-Phenanthrenes/ Anthracenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
C2-Phenanthrenes/ Anthracenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
C3-Phenanthrenes/ Anthracenes	1.0	2	0.2	10	10	10	100	≤ 25	≤ 25	≤ 25	≤ 25	
C4-Phenanthrenes/ Anthracenes	1.0	2	0.2	10	10	10	100	≤ 25	≤ 25	≤ 25	≤ 25	
C1-Fluoranthenes/ Pyrenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
C2-Fluoranthenes/ Pyrenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
C3-Fluoranthenes/ Pyrenes	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	

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MATRIX	Typical Sample Specific * Detection Limits							Procedural Blank Level				Acceptable OPR
	Solid	Aqueous	Tissue	XAD-2 Column	PUF & SPMD Extract	Filter	Oil	Solid & SPMD Extract	Aqueous	Tissue	All other matrices	
Analyte:	ng/g	ng/L	ng/g	ng	ng	ng	ng/g	ng (tot)	ng (tot)	ng (tot)	ng (tot)	% Recovery
C4-Fluoranthenes/ Pyrenes	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	
C1-Benzo[a]anthracenes/ Chrysenes	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	
C2-Benzo[a]anthracenes/ Chrysenes	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	
C3-Benzo[a]anthracenes/ Chrysenes	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	
C4-Benzo[a]anthracenes/ Chrysenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
C1-Benzofluoranthenes/ Benzopyrenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
C2-Benzofluoranthenes/ Benzopyrenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
Typical Sample Size	10 g (dry)	1 L	10 g (wet)	1 col	1 sample	1 filter	0.1 g					
Typical Final Volume (µL)	500	100	100	500	500	500	500					

* Detection limits quoted are those routinely achieved. Detection limits for alkylated PAH Compound Totals are based on detection of a single component.

[1] Procedural blank level limits don't apply for naphthalene and alkylated naphthalene from XAD-2 resin.

[2] Due to limited availability of 1,7-dimethylphenanthrene standard, this compound may be absent from OPR samples.

[3] Due to limited availablilty of 3-methylfluoranthene standard, this compound may be absent from OPR samples. It may be substituted by 2-methylfluoranthene as the exemplar alkylfluoranthene in the OPR.

[4] Due to limited availablilty of 5,9-dimethylchrysene standard, this compound may be absent from OPR samples. It may be substituted by other alternative dimethylchrysene isomers as the exemplar C2/C3/C4-benzo[a]anthracene/chrysene in the OPR; in such cases the recovery specification for 5,9-dimethylchrysene would not apply.

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[5] For solid samples extracted by the optional hexane:acetone 1:1 Soxhlet extraction the OPR method limits are 70 – 170 % recovery for acenaphthene and 70-160 % for benzo[e]pyrene.

NOTE: Reference samples are unavailable for Alkylated PAH Compound Totals, data acceptability evaluation is based on recoveries of the associated individual alkylated PAH compounds

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SURROGATE STANDARD RECOVERIES:	% RECOVERY RANGES ALL MATRICES
d_8-naphthalene	15 – 130
d_8-acenaphthylene	20 – 130
d_{10}-phenanthrene	30 – 130
d_{10}-fluoranthene *	30 – 130
d_{12}-benz[a]anthracene	30 – 130
d_{12}-chrysene	30 – 130
d_{12}-benzo[b]fluoranthene	30 – 130
d_{12}-benzo[k]fluoranthene	30 – 130
d_{12}-benzo[a]pyrene	30 – 130
d_{12}-perylene	30 – 130
d_{14}-dibenz[ah]anthracene *	30 – 130
d_{12}-indeno[1,2,3-cd]pyrene	30 – 130
d_{12}-benzo[ghi]perylene	30 – 130
d_{10}-2-methylnaphthalene	20 – 130
d_{12}-2,6-dimethylnaphthalene	20 – 130
d_{10}-biphenyl	15 – 130
d_8-dibenzothiophene	30 – 130

* Compound not used as surrogate standard for SPMD analysis. Surrogate recovery specifications do not apply for SPMD samples.

Performance Reference Compound (PRC) and Photolysis Standard Recovery Range Specifications for SPMD OPR samples: These ranges can also be used as guidance limits for any Client Day 0 and Field or Trip Blank Samples.

PRC	%RECOVERY RANGES
d_{10}-fluoranthene	70 – 130
d_{14}-dibenz[ah]anthracene	70 – 130
d_{10}-anthracene	70 – 130

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QC Specification Table: Instrumental Analysis, and Analyte Quantification

Parameter	Acceptance Specification
Procedural Blank	Refer to Table "QC Specification Table: Authentic and Surrogate Standard Recoveries, OPR and Samples" above, or 5 times lower than analogous analyte value detected in the samples.
Analysis Duplicate	Duplicates must fall within ±20% of the mean (applicable to concentrations >10 times the DL) These are guidelines – departures based on professional judgement allowed. (Note that ±20% of the mean is equivalent to 40 relative percent difference)
Instrument Sensitivity	S/N 3:1 for 10 pg of acenaphthene, dibenz[a,h]anthracene.
Instrument Resolution	Calibration gas PFTBA (FC43) unit mass resolution at m/e 69/70 and 219/220, Unit mass resolution is demonstrated by the presence of a resolved peak at m/z 70 and m/e 220.
Instrument Linearity	Linearity is demonstrated by a 5-point calibration over the working concentration range with a relative standard deviation of the RRFs ≤20% for targets with a labelled analog present and all labelled compounds, ≤35% for targets with no labelled analog present.
Continuing Cal Ver	Opening Cal Ver: Concentrations of native compounds and labelled surrogates must be within ±25% of expected values for all targets. Closing Cal Ver: Concentrations of native compounds must be within ±25% of expected values. Concentrations of labelled surrogates must be within ±25% of expected values, with any two (2) values allowed to be within ±40%
Bracketing Cal (optional)	RRFs for the opening and closing calibrations over a 12 hour period must agree to within ±20% of the mean (i.e., ≤40 RPD between RRFs and for the opening and closing calibrations, which is equivalent to ≤28.3 % RSD).
GC Resolution	Benzo[b] & [k]fluoranthene valley height must be ≤75% for equal concentrations. Phenanthrene/anthracene valley height must be ≤30% for equal concentrations.
Chromatogram Quality	Maximum peak width must be ≤15 seconds for benzo[ghi]perylene peak at 10% peak height.
Retention Time Window for Target Compounds	RT within ± 3 seconds of the predicted retention time determined from the calibration standard and adjusted relative to the peak retention time reference (i.e. labelled surrogate). A second requirement is that an authentic elute after its labelled analog.
Ion Abundance Ratios	CAL VER: Ion ratios for authentic and labelled dibenz[ah]anthracene, indeno[1,2,3-cd]pyrene and benzo[ghi]perylene must be within ±35% of the mid-point of the I-CAL All other native analytes and labelled surrogates must be within ±20% of the mid-point of the I-CAL Samples: Ion ratios for authentic dibenz[ah]anthracene, indeno[1,2,3-cd]pyrene and benzo[ghi]perylene must be within ±35% of the 12 hour CAL VER (or bracketing) calibration standard. All other native analytes and labelled surrogates must be within ±20% of the 12 hour CAL VER (or bracketing) calibration standard.

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APPENDIX 1 – SUMMARY OF KEY ATTRIBUTES OF METHODS MLA-021, EPA 8270C, 8270D AND 1625B

Analysis by GC/LRMS, Key Attributes of SGS AXYS MLA-021, EPA 8270C/D and EPA 1625B

	MLA-021	EPA 8270C	EPA 8270D	EPA 1625B
MS acquisition mode	SIM[1]	Full Scan or SIM[1]	Full Scan or SIM[1]	Full Scan[1]
Qualitative Identification Criteria	Retention time & ratio of 2 ions	Retention time & ratio of 3 [2] ions	Retention time & ratio of 3 [2] ions	Retention time & ratio of characteristic ions
MS Ion Ratio Criteria	20 %	30 %	30 %	-50 % to +200 %
MS Tuning Type and Check Frequency	PTFBA, daily	DFTTP[1], 12 hrs	DFTTP[1], 12 hrs	DFTTP[1], 8 hrs
Quantification References	Isotopically labeled standards added prior to extraction	Internal standards added prior to instrumental analysis	Internal standards added prior to instrumental analysis	Isotopically labeled standards added prior to extraction
Recovery correction of results	YES	NO	NO	YES
Initial Calibration, # levels	5	5	5	5
Initial Calibration Limit (% RSD)	20 % (35 % if no labeled analog)	15 %	20 %	20 % (35 % if no labeled analog)
Calibration Verification Frequency	12 hrs	12 hrs	12 hrs	8 hrs
Calibration Verification Relative Response Limit (% diff.)	< 25 % of I-CAL	< 20 % of I-CAL	< 20 % of I-CAL	Various; most stringent is -20% to +25% of I-CAL
Calibration Verification IS area (% of I-CAL midpoint)	50-200 %	50-200 %	50-200 %	n.a.
Calibration verification IS RT (diff. from I-CAL midpoint)	n.a.	30 sec.	30 sec.	n.a.
Extraction	DCM (L/L (aqueous) DCM, Soxhlet (solids)	Options specified externally	Options specified externally	DCM, L/L (aqueous), pH>11 or pH other [3]

Notes:

1 SIM (Selected Ion Monitoring) acquisition protocol is permitted by EPA8270 and Federal Register Vol.77 Iss. 97 (May 18, 2012) Part 136

2 Based on availability, use of fewer ions is permitted

3 Modifications are permitted under Federal Register Vol.77 Iss.97 (May 18, 2012) Part 136.6

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APPENDIX 2 – IDENTIFICATION AND QUANTIFICATION OF ALKYLATED PAH COMPOUND TOTALS

RT windows for Alkylated PAH Compound Totals are determined from a retention time reference extract run at the beginning of the instrumental run list; assignment of the sample RT windows is made based on comparison of the peak retention times and peak patterns in samples to that of the retention time reference standard. Table 13 lists estimated 'typical' values.

Analyte Ions Monitored, Surrogate Used and RRF Determination for Alkylated PAH Compound Totals

Alkylated PAH total parameter	Compounds/peaks	Typical peak/ window RT, min	Quant. ion	Conf. ion	Typical ion ratio	RRF reference	RT reference	Typical RT ref., min
C1-Biphenyls	(2 peaks)	11.22 - 11.44	168			Biphenyl	d10-Biphenyl	9.73
C2-Biphenyls	(6 or 7 peaks)	12.42 - 12.96	182			Biphenyl	d10-Biphenyl	9.73
C1-Naphthalenes	1-methylnaphthalene	8.80	142	141	0.965	1-methylnaphthalene	d_{10}-2-Methylnaphthalene	8.46
	2-methylnaphthalene	8.53	142	141	0.930	2-methylnaphthalene		
C2-Naphthalenes	Ethyl/dimethylnaphthalenes	9.96 - 10.75	156	141		Average of 1,2-dimethylnaphthalene and 2,6-dimethylnaphthalene	d12-2,6-dimethyl-naphthalene	10.05
	1,2-dimethylnaphthalene	10.88	156	141	1.320	1,2-dimethylnaphthalene		
C3-Naphthalenes	(≥2 peaks)	11.65 - 12.95	170			Average of 2,3,5-Trimethylnaphthalene and 2,3,6-Trimethylnaphthalene	d12-2,6-dimethyl-naphthalene	10.05
C4-Naphthalenes	(≥2 peaks)	13.05 - 14.65	184			1,4,6,7-Tetramethylnaphthalene	d12-2,6-dimethyl-naphthalene	10.05
C1-Acenaphthenes	(≥2 peaks)	11.60 - 11.70	168			Acenaphthene	d10-Acenaphthene	11.23
C1-Fluorenes	(≥2 peaks)	13.73 - 14.23	180			2-Methylfluorene	d10-Phenanthrene	14.96
C2-Fluorenes	(≥2 peaks)	14.96 - 16.01	194			1,7-Dimethylfluorene	d10-Phenanthrene	14.96
C3-Fluorenes	(≥2 peaks)	16.25 - 17.70	208			1,7-Dimethylfluorene	d10-Phenanthrene	14.96
C1-Dibenzothiophenes	C1-dibenzothiophenes-1	15.84	198			2/3-Methyldibenzothiophenes	d8-Dibenzothiophene	14.67
	2/3-methyldibenzo-thiophenes	16.05	198					
	C1-dibenzothiophenes-2	16.32	198					
C2-Dibenzothiophenes	(≥2 peaks)	16.35 - 17.95	212			2,4-Dimethyldibenzothiophene	d8-Dibenzothiophene	14.67
C3-Dibenzothiophenes	(≥2 peaks)	17.70 - 19.20	226			2,4-Dimethyldibenzothiophene	d8-Dibenzothiophene	14.67
C4-Dibenzothiophenes	(≥2 peaks)	18.80 - 20.50	240			2,4-Dimethyldibenzothiophene	d8-Dibenzothiophene	14.67

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Alkylated PAH total parameter	Compounds/peaks	Typical peak/ window RT, min	Quant. ion	Conf. ion	Typical ion ratio	RRF reference	RT reference	Typical RT ref., min
C1-Phenanthrenes/ Anthracenes	3-methylphenanthrene	16.27	192	191	0.618	Average of 1-Methylphenanthrene, 2-Methylphenanthrene and 2-Methylanthracene	d10-Phenanthrene	14.96
	2-methylphenanthrene	16.33	192	191	0.610	2-methylphenanthrene		
	2-methylanthracene	16.44	192	191	0.531	2-methylanthracene		
	9/4-methylphenanthrenes	16.56	192	191	0.621	Average of 1-Methylphenanthrene, 2-Methylphenanthrene and 2-Methylanthracene		
	1-methylphenanthrene	16.61	192	191	0.629	1-methylphenanthrene		
C2-Phenanthrenes/ Anthracenes	Ethyl/dimethyl-phenanthrenes/anthracenes	17.22 - 17.95	206			Average of 3,6-Dimethylphenanthrene and 1,7-Dimethylphenanthrene	d10-Fluoranthene	17.99
	1,8-dimethylphenanthrene	18.10	206	191	0.365	Average of 3,6-Dimethylphenanthrene and 1,7-Dimethylphenanthrene		
C3-Phenanthrenes/ Anthracenes	(≥2 peaks)	18.40 - 19.40	220			1,2,6-Trimethylphenanthrene	d10-Fluoranthene	17.99
C4-Phenanthrenes/ Anthracenes	(≥2 peaks)	19.45 - 21.55	234			Retene	d10-Fluoranthene	17.99
C1-Fluoranthenes /Pyrenes	C1-Fluoranthenes/Pyrenes-1 (2 peaks)	19.17 - 19.58	216			3-Methylfluoranthene	d10-Fluoranthene	17.99
	C1-Fluoranthenes/Pyrenes-2 (3 or 4 peaks)	19.70 - 20.07	216			3-Methylfluoranthene		
C2-Fluoranthenes/ Pyrenes	(≥2 peaks)	20.50 - 21.50	230			3-Methylfluoranthene	d10-Fluoranthene	17.99
C3-Fluoranthenes/ Pyrenes	(≥2 peaks)	21.80 - 23.15	244			3-Methylfluoranthene	d10-Fluoranthene	17.99
C4-Fluoranthenes/ Pyrenes	(≥2 peaks)	23.20 - 24.50	258			3-Methylfluoranthene	d10-Fluoranthene	17.99
C1-Benzo[a]anthracenes /Chrysenes	(7 or more peaks)	22.60 - 23.38	242			Average of 5/6-methylchrysenes and 1-methylchrysene	d12-Chrysene	21.70
C2-Benzo[a]anthracenes /Chrysenes	(≥2 peaks)	23.70 - 25.00	256			5,9-Dimethylchrysene	d12-Chrysene	21.70
C3-Benzo[a]anthracenes /Chrysenes	(≥2 peaks)	25.20 - 26.20	270			5,9-Dimethylchrysene	d12-Chrysene	21.70
C4-Benzo[a]anthracenes /Chrysenes	(≥2 peaks)	26.20 - 27.90	284			5,9-Dimethylchrysene	d12-Chrysene	21.70
C1-Benzofluoranthenes/ Benzopyrenes	(≥2 peaks)	27.26 - 29.80	266			7-Methylbenzo[a]pyrene	d12-Benzo[a]pyrene	26.40

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Alkylated PAH total parameter	Compounds/peaks	Typical peak/ window RT, min	Quant. ion	Conf. ion	Typical ion ratio	RRF reference	RT reference	Typical RT ref., min
C2-Benzofluoranthenes/ Benzopyrenes	(≥2 peaks)	29.80 - 30.85	280			7-Methylbenzo[a]pyrene	d12-Benzo[a]pyrene	26.40

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APPENDIX 3: ANALYSIS OF 2-CHLORONAPHTHALENE BY GC-HRMS

The analysis of 2-chloronaphthalene in solid and ash samples may be carried out by GC-HRMS analysis of the F2 extract cleanup fraction. Method MLA-021 applies for 2-chloronaphthalene analysis with the following modifications:

Quality Assurance/Quality Control:

QC Acceptance Criteria for 2-Chloronaphthalene

Compound	Procedural Blank Level (ng/sample)	OPR and Sample Recovery Range (%)
2-Chloronaphthalene	< 1	50 – 150
d7-2-Chloronaphthalene	n.a.	30 – 150

Cleanup procedures: Fraction F2 from the silica column cleanup is used for the analysis.

GC-HRMS Analysis: High resolution gas chromatography/high resolution mass spectrometry analysis of the F2 fraction is performed using a capillary gas chromatograph coupled to a high-resolution mass spectrometer. A J&W DB-5 chromatography column (60 m, 0.25 mm i.d., 0.10 µm film thickness)) is coupled directly to the HRMS source. The HRMS is operated at a static (8000) mass resolution (10% valley) in the electron ionization (EI) mode using multiple ion detection (MID) acquiring two characteristic ions for each target analyte and surrogate standard. Sample concentrations are determined by bracketing calibration using the SAR solution.

Analyte Ions Monitored, Surrogates Used and RRF Determination for 2-Chloronaphthalene Analysis

Compound	Quantification and RRT Standard	Typical Retention Time (min.)	Quant. Ion (m/z)	Conf. Ion (m/z)	Typical Ion Ratio (Quant./Conf.)	Ion Ratio Tolerance
2-Chloronaphthalene	d7-2-Chloronaphthalene	11:53	162.0236	164.0207	3.12	20 %
d7-2-Chloronaphthalene	d10-Acenaphthene	11:50	169.0676	171.0646	3.12	20 %
d10-Acenaphthene		13:21	162.1269	164.1410	1.1	20 %
Lock-mass			168.9888			

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APPENDIX 4: QUANTIFICATION OF CLIENT, FIELD (PRC) AND PHOTOLYSIS STANDARDS

This section is applicable for determination of PAHs in PUF (polyurethane foam) and in SPMD (semipermeable membrane device) samples.

Because PUF and SPMD samples require the use of d_{10}-anthracene and d_{10}-fluoranthene as client or field standards, and SPMD samples require the use of d_{14}-dibenz[a,h]anthracene as photolytic standard, the default quantification scheme (the table between sections 4.1 and 5.0) must be modified as per the table below.

Analyte Ions Monitored, Surrogates Used and RRF Determination for PAH using Client, Field, PRC and Photolysis Standards

TARGET ANALYTES	Quantification Ion (m/z)	Confirmation Ions (m/z)	Typical Ion Ratio (Conf./Quant.)	SURROGATE	Typical Retention Time (minutes)	RRF DETERMINED FROM
Naphthalene	128	102	0.064	d_8-Naphthalene	6.84	Naphthalene
Acenaphthylene	152	151	0.222	d_8-Acenaphthylene	10.83	Acenaphthylene
Acenaphthene	154	153	1.18	d_8-Acenaphthylene	11.33	Acenaphthene
Fluorene	166	165	1.01	d_{10}-Phenanthrene	12.63	Fluorene
Phenanthrene	178	176	0.202	d_{10}-Phenanthrene	15.04	Phenanthrene
Anthracene	178	176	0.196	d_{10}-Phenanthrene	15.15	Anthracene
Fluoranthene	202	200	0.214	d_{10}-Phenanthrene	18.06	Fluoranthene
Pyrene	202	200	0.219	d_{10}-Phenanthrene	18.60	Pyrene
Benz[a]anthracene [6]	228	226	0.281	d_{12}-Benz[a]anthracene	21.68	Benz[a]anthracene
Chrysene [1]	228	226	0.312	d_{12}-Chrysene	21.79	Chrysene
Benzo[b]fluoranthene	252	253	0.218	d_{12}-Benzo[b]fluoranthene	25.21	Benzo[b]fluoranthene
Benzo[j,k]fluoranthenes	252	253	0.215	d_{12}-Benzo[k]fluoranthene	25.30	Benzo[k]fluoranthene
Benzo[e]pyrene	252	253	0.213	d_{12}-Benzo[a]pyrene	26.36	Benzo[e]pyrene
Benzo[a]pyrene	252	253	0.217	d_{12}-Benzo[a]pyrene	26.58	Benzo[a]pyrene
Perylene	252	253	0.212	d_{12}-Perylene	27.00	Perylene
Dibenzo[ah]anthracene [2]	278	139	0.144	d_{12}-Indeno[1,2,3,cd]pyrene	31.86	Dibenz[ah]anthracene
Indeno[1,2,3-cd]pyrene	276	138	0.179	d_{12}-Indeno[1,2,3,cd]pyrene	31.71	Indeno[1,2,3-cd]pyrene
Benzo[ghi]perylene	276	138	0.194	d_{12}-Benzo[ghi]perylene	32.53	Benzo[ghi]perylene
Biphenyl [3]	154	152	0.304	d_{10}- Biphenyl	9.81	Biphenyl

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TARGET ANALYTES	Quantification Ion (m/z)	Confirmation Ions (m/z)	Typical Ion Ratio (Conf./Quant.)	SURROGATE	Typical Retention Time (minutes)	RRF DETERMINED FROM
Dibenzothiophene [3]	184	152	0.073	d_8-Dibenzothiophene	14.72	Dibenzothiophene
1-Methylnaphthalene [3]	142	141	0.962	d_{10}-2-Methylnaphthalene	8.81	1-Methylnaphthalene
2-Methylnaphthalene [3]	142	141	0.930	d_{10}-2-Methylnaphthalene	8.55	2-Methylnaphthalene
C1-Naphthalenes [3]	142	[4]	[4]	d_{10}-2-Methylnaphthalene	[5]	1- & 2-Methylnaphthalene
2,6-Dimethylnaphthalene [3]	156	141	0.666	d_{12}-2,6 Dimethylnaphthalene	10.17	2,6-Dimethylnaphthalene
1,2-Dimethylnaphthalene	156	141	1.26	d_{12}-2,6 Dimethylnaphthalene	10.90	1,2-Dimethylnaphthalene
C2-Naphthalenes [3]	156	[4]	[4]	d_{12}-2,6 Dimethylnaphthalene	[5]	2,6- & 1,2-Dimethylnaphthalene
2,3,5-Trimethylnaphthalene [3]	170	155	0.873	d_{12}-2,6 Dimethylnaphthalene	12.35	2,3,5- Trimethylnaphthalene
2,3,6-Trimethylnaphthalene	170	155	0.876	d_{12}-2,6 Dimethylnaphthalene	12.17	2,3,6- Trimethylnaphthalene
C3-Naphthalenes [3]	170			d_{12}-2,6 Dimethylnaphthalene	[5]	2,3,5- & 2,3,6-Trimethylnaphthalene
1,4,6,7-Tetramethylnaphthalene	184	139	0.027	d_{12}-2,6 Dimethylnaphthalene	13.89	1,4,6,7-Tetramethylnaphthalene
C4-Naphthalene	184	[4]	[4]	d_{12}-2,6 Dimethylnaphthalene	[5]	1,4,6,7-Tetramethylnaphthalene
2-Methylanthracene	192	191	0.531	d_{10}-Phenanthrene	16.45	2-Methylanthracene
3-Methylphenanthrene	192	191	0.608	d_{10}-Phenanthrene	16.27	1- & 2-Methylphenanthrene & 2-Methylanthracene
2-Methylphenanthrene	192	191	0.608	d_{10}-Phenanthrene	16.36	2-Methylphenanthrene
9/4-Methylphenanthrenes	192	191	0.634	d_{10}-Phenanthrene	16.59	1- & 2-Methylphenanthrene & 2-Methylanthracene
1-Methylphenanthrene [3]	192	191	0.634	d_{10}-Phenanthrene	16.64	1-Methylphenanthrene
C1-Phenanthrenes/Anthracenes [3]	192	[4]	[4]	d_{10}-Phenanthrene	[5]	1- & 2-Methylphenanthrene & 2-Methylanthracene
3,6-Dimethylphenanthrene [3]	206	191	0.342	d_{10}-Phenanthrene	17.46	3,6-Dimethylphenanthrene
2,6-Dimethylphenanthrene	206	191	0.342	d_{10}-Phenanthrene	17.54	3,6- & 1,7-Dimethyl-phenanthrenes
1,7-Dimethylphenanthrene	206	191	0.332	d_{10}-Phenanthrene	17.89	1,7-Dimethylphenanthrene
1,8-Dimethylphenanthrene	206	191	0.332	d_{10}-Phenanthrene	18.13	3,6- & 1,7-Dimethyl-phenanthrenes
C2-Phenanthrenes/Anthracenes [3]	206	[4]	[4]	d_{10}-Phenanthrene	[5]	3,6- & 1,7-Dimethyl-phenanthrenes
1,2,6-Trimethylphenanthrene	220	205	0.581	d_{10}-Phenanthrene	19.41	1,2,6-Trimethylphenanthrene
C3-Phenanthrenes/Anthracenes	220	[4]	[4]	d_{10}-Phenanthrene	[5]	1,2,6-Trimethylphenanthrene
Retene [3]	234	219	1.63	d_{10}-Phenanthrene	19.53	Retene

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TARGET ANALYTES	Quantification Ion (m/z)	Confirmation Ions (m/z)	Typical Ion Ratio (Conf./Quant.)	SURROGATE	Typical Retention Time (minutes)	RRF DETERMINED FROM
C4-Phenanthrenes/Anthracenes	234	[4]	[4]	d_{10}-Phenanthrene	[5]	Retene
C1-Biphenyls	168	[4]	[4]	d_{10}- Biphenyl	[5]	Biphenyl
C2-Biphenyls	182	[4]	[4]	d_{10}- Biphenyl	[5]	Biphenyl
C1-Acenaphthenes	168	[4]	[4]	d_8-Acenaphthylene	[5]	Acenaphthene
2-Methylfluorene	180	165	1.23	d_{10}-Phenanthrene	14.06	2-Methylfluorene
C1-Fluorenes	180	[4]	[4]	d_{10}-Phenanthrene	[5]	2-Methylfluorene
1,7-Dimethylfluorene	194	177	0.092	d_{10}-Phenanthrene	15.49	1,7-Dimethylfluorene
C2-Fluorenes	194	[4]	[4]	d_{10}-Phenanthrene	[5]	1,7-Dimethylfluorene
C3-Fluorenes	208	[4]	[4]	d_{10}-Phenanthrene	[5]	1,7-Dimethylfluorene
2/3-Methyldibenzothiophenes	198	197	0.738	d_8-Dibenzothiophene	16.07	2/3-Methyldibenzothiophenes
C1-Dibenzothiophenes	198	[4]	[4]	d_8-Dibenzothiophene	[5]	2/3-Methyldibenzothiophenes
2,4-Dimethyldibenzothiophene	212	197	0.514	d_8-Dibenzothiophene	17.08	2,4-Dimethyldibenzothiophene
4,6-Dimethyldibenzothiophene	212	197	0.160	d_8-Dibenzothiophene	16.94	4,6-Dimethyldibenzothiophene
C2-Dibenzothiophenes	212	[4]	[4]	d_8-Dibenzothiophene	[5]	2,4-Dimethyldibenzothiophene
C3-Dibenzothiophenes	226	[4]	[4]	d_8-Dibenzothiophene	[5]	2,4-Dimethyldibenzothiophene
C4-Dibenzothiophenes	240	[4]	[4]	d_8-Dibenzothiophene	[5]	2,4-Dimethyldibenzothiophene
3-Methylfluoranthene/Benzo[a]fluorene	216	215	0.880	d_{10}-Phenanthrene	19.53	3-Methylfluoranthene
C1-Fluoranthenes/Pyrenes	216	[4]	[4]	d_{10}-Phenanthrene	[5]	3-Methylfluoranthene
C2-Fluoranthenes/Pyrenes	230	[4]	[4]	d_{10}-Phenanthrene	[5]	3-Methylfluoranthene
C3-Fluoranthenes/Pyrenes	244	[4]	[4]	d_{10}-Phenanthrene	[5]	3-Methylfluoranthene
C4-Fluoranthenes/Pyrenes	258	[4]	[4]	d_{10}-Phenanthrene	[5]	3-Methylfluoranthene
5/6-Methylchrysenes	242	[4]	[4]	d_{12}-Chrysene	23.15	6-Methylchrysene
1-Methylchrysene	242	[4]	[4]	d_{12}-Chrysene	23.32	1-Methylchrysene
C1-Benzo[a]anthracenes/Chrysenes	242	[4]	[4]	d_{12}-Chrysene	[5]	1- & 6-Methylchrysenes
5,9-Dimethylchrysene	256	[4]	[4]	d_{12}-Chrysene	24.49	5,9-Dimethylchrysene
C2-Benzo[a]anthracenes/Chrysenes	256	[4]	[4]	d_{12}-Chrysene	[5]	5,9-Dimethylchrysene
C3-Benzo[a]anthracenes/Chrysenes	270	[4]	[4]	d_{12}-Chrysene	[5]	5,9-Dimethylchrysene
C4-Benzo[a]anthracenes/Chrysenes	284	[4]	[4]	d_{12}-Chrysene	[5]	5,9-Dimethylchrysene
7-Methylbenzo[a]pyrene	266	[4]	[4]	d_{12}-Benzo[a]pyrene	29.35	7-Methylbenzo[a]pyrene
C1-Benzofluoranthenes/Benzo-pyrenes	266	[4]	[4]	d_{12}-Benzo[a]pyrene	[5]	7-Methylbenzo[a]pyrene

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TARGET ANALYTES	Quantification Ion (m/z)	Confirmation Ions (m/z)	Typical Ion Ratio (Conf./Quant.)	SURROGATE	Typical Retention Time (minutes)	RRF DETERMINED FROM
C2-Benzofluoranthenes/Benzo-pyrenes	280	[4]	[4]	d_{12}-Benzo[a]pyrene	[5]	7-Methylbenzo[a]pyrene
LABELLED CLIENT/FIELD STANDARDS	**Quantification Ion (m/z)**	**Confirmation Ions (m/z)**	**Typical Ion Ratio (Conf./Quant.)**	**RECOVERY CALCULATED AGAINST**	**Typical Retention Time (minutes)**	**RRF DETERMINED FROM**
d_{10}-Anthracene	188	184		d_{10}-Phenanthrene	15.1	
d_{10}-Fluoranthene	212	208	0.173	d_{12}-Benzo[a]anthracene	18.02	
LABELLED PHOTOLYSIS STANDARD	**Quantification Ion (m/z)**	**Confirmation Ions (m/z)**	**Typical Ion Ratio (Conf./Quant.)**	**RECOVERY CALCULATED AGAINST**	**Typical Retention Time (minutes)**	**RRF DETERMINED FROM**
d_{14}-Dibenzo[ah]anthracene	292	288	0.260	d_{12}-Indeno[1,2,3-cd]pyrene	31.75	
LABELLED SURROGATE STANDARDS	**Quantification Ion (m/z)**	**Confirmation Ions (m/z)**	**Typical Ion Ratio (Conf./Quant.)**	**RECOVERY CALCULATED AGAINST**	**Typical Retention Time (minutes)**	
d_{8}-Naphthalene	136	134	0.095	d_{10}-Acenaphthene	6.80	
d_{10}-2-Methylnaphthalene	152	151	0.195	d_{10}-Acenaphthene	8.47	
d_{10}-Biphenyl	164			d_{10}-Acenaphthene	9.75	
d_{12}-2,6-Dimethylnaphthalene	168	150	0.747	d_{10}-Acenaphthene	10.07	
d_{8}-Acenaphthylene	160	158	0.159	d_{10}-Acenaphthene	10.80	
d_{8}-Dibenzothiophene	192	160	0.085	d_{10}-Pyrene	14.67	
d_{10}-Phenanthrene	188	184	0.143	d_{10}-Pyrene	14.97	
d_{12}-Benz[a]anthracene	240	236	0.250	d_{10}-Pyrene	21.63	
d_{12}-Chrysene	240	236	0.278	d_{10}-Pyrene	21.73	
d_{12}-Benzo[b]fluoranthene	264	260	0.216	d_{12}-Benzo[e]pyrene	25.11	
d_{12}-Benzo[k]fluoranthene	264	260	0.208	d_{12}-Benzo[e]pyrene	25.23	
d_{12}-Benzo[a]pyrene	264	260	0.216	d_{12}-Benzo[e]pyrene	26.47	
d_{12}-Perylene	264	260	0.256	d_{12}-Benzo[e]pyrene	26.88	
d_{12}-Indeno[1,2,3,cd]pyrene	288	284	0.192	d_{12}-Benzo[e]pyrene	31.63	
d_{12}-Benzo[ghi]perylene	288	284	0.205	d_{12}-Benzo[e]pyrene	32.45	

[1] Coelutes with triphenylene

[2] Coelutes with dibenz[ac]anthracene

[3] These compounds are in addition to the regular suite of analytes, and are analyzed by client request only.

[4] Secondary ion confirmation procedures do not apply

[5] RRT ranges apply to alkylated PAH Totals

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[6] Benzo[a]anthracene coelutes with cyclopenta[cd]pyrene, which may contribute response in the second ion to cause a failing ion abundance ratio. Therefore quantification of benzo[a]anthracene should use response from the first ion only

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SGS AXYS ANALYTICAL SERVICES LTD

ANALYTICAL METHOD FOR THE DETERMINATION OF 209 PCB CONGENERS BY EPA METHOD 1668A[1], EPA METHOD 1668C[8] OR EPA METHOD CBC01.2[2]

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SCOPE

The analytical method described in this document determines the concentration of 209 PCB congeners in sediment, soil, sludge, tissue (including blood), aqueous samples, milk, solvent extracts, air samples and XAD-2 columns. Samples are spiked with $^{13}C_{12}$-labeled surrogate standards prior to analysis. Samples are extracted, the extracts cleaned up by column chromatography and analyzed by high-resolution gas chromatography with high-resolution mass spectrometric detection (HRGC/HRMS). The method covers both EPA Method 1668A and EPA Method 1668C, which are attached and considered to be part of this document.

This method allows for the determination of twelve PCB congeners designated as "toxic" by the World Health Organization[3].

The analysis of chlorinated pesticides, and/or polybrominated diphenylethers may also be carried out using the extraction procedures described in this document. The standards required, clean-up and instrumental analysis procedures and QA/QC specifications are described in SGS AXYS method documents MLA-007[4], MLA-028[5] and MLA-033[6,7]. Additional details for these optional analyses are summarized in Appendix A.

The procedures documented in this method conform to EPA Methods 1668A and 1668C, but may be modified in order to determine the concentrations of PCBs according to protocols described EPA Method CBC01.2. These modifications are described in Appendix D.

Approved 29-Dec-2020 Shea Hewage, General Manager
Dale Hoover, Senior Technical Director
Rhonda Stoddard, QA Manager

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AXYS ANALYTICAL SERVICES LTD

ANALYTICAL METHOD FOR THE DETERMINATION OF 209 PCB CONGENERS BY EPA METHOD 1668A[1], EPA METHOD 1668C[10] OR EPA METHOD CBC01.2[2]

SCOPE

The analytical method described in this document determines the concentration of 209 PCB congeners in sediment, soil, sludge, tissue (including blood), aqueous samples, milk, solvent extracts, air samples and XAD-2 columns. Samples are spiked with $^{13}C_{12}$-labelled surrogate standards prior to analysis. Samples are extracted, the extracts cleaned up by column chromatography and analyzed by high-resolution gas chromatography with high-resolution mass spectrometric detection (HRGC/HRMS). The method covers both EPA Method 1668A and EPA Method 1668C, which are attached and considered to be part of this document.

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Approved 30-Jul-2015 John Cosgrove, President and Senior Technical Director
Shea Hewage, Director of Operations
Dale Hoover, QA Manager

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REFERENCES:

1. EPA Method 1668, Revision A - *Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS,* with changes and corrections through to August 20, 2003.

2. EPA Method CBC01.2 *Statement of Work (SOW) for Analysis of Chlorinated Biphenyl Congeners (CBCS),* December 2009.

3. Van den Berg et al. 1998. *Toxicity Equivalency Factors (TEF) for PCB's, PCDD's, and PCDF's for Humans and Wildlife. Environmental Health Perspective.* **106** (12): 775-792

4. AXYS Method Document MLA-007. *Analytical Method for the Determination of: Aroclors, Total PCBS, Chlorinated Pesticides, PCB Congeners, Coplanar PCBs, Toxaphene, Chlorobenzenes.*

5. AXYS Method Document MLA-028. *Organochlorine Pesticides by Isotope Dilution HRGC/HRMS.*

6. EPA Method 1614. *Brominated diphenyl ethers in water, soil, sediment, and tissue by HRGC/HRMS*

7. AXYS Method Document MLA-033. *Analytical Method for the Determination of Polybrominated Diphenylethers by EPA Method 1614*.

8. EPA Method 1668, Revision C - *Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS*, April, 2010. (EPA-820-R-10-005)

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CHANGES FROM PREVIOUS REVISIONS or VERSIONS

The table below lists the details of the changes from revision 11 version 05 of this document (Jul 2015).

Page	Change Details
68	Section 6.6: Added note that recovery standards must be respiked after carbon/Celite recolumning.
90	Section 9.2: Added instructions for implementing the EPA minimium level (ML) reporting protocol using LIMS' pkeys.

The table below lists the details of the changes from revision 11 version 04 of this document.

Page	Change Details
66	Section 6.5: Updated the Florisil elution solvent to the current composition (15:85 dichloromethane: hexane).
87-94	Section 9: Added description of MDL. Added instructions for 1668A/C minimum level (ML) reporting limit protocol. Table 8: Added table with EPA MLs and MDLs, and MLA-010 typical SDLs, LMCLs and MLs.

The table below lists the details of the changes from revision 11 version 03 of this document.

Page	Change Details
	Removed all references indicating coextraction with MLA-030. Optional analysis of PCNs has been removed from MLA-010. Removed all references to MLA-026; MLA-026 will be a standalone method.
12, 18, 37, 38, 40, 46, 110	Sections B, 3.2, 5.1.2, 5.2, 5.3 and 5.5.3.4, and Appendix D: Clarified that PCB analysis according to EPA method CBC01.2 requires 3 hours of pre-cleaning of the Soxhlet extraction equipment using toluene solvent.
17-18	Section 3.1: Clarified a few equipment items (chromatography columns, rotary evaporator, water bath)
25-26	Section 3.4: Added missing reagent shelf lives.
27, 87, 92	Sections 3.6.4 and 10, appendix A: Clarified that Florisil currently is used as 2.0% deactivated.
41, 43, 54, 56, 65	Sections 5.3.3, 5.4.3, 5.9, 5.10 and 6.1: Clarified that extracts after Biobead cleanup must be solvent exchanged to hexane before proceeding to other column cleanup.
45	Sections 5.5.2.1 and 5.5.2.2: Clarified extract drying, cleanup standard spiking and evaporation procedures.
46	Sections 5.5.3.2 and 5.5.3.3: Clarified extract drying procedures.
47	Section 5.5.3.5: Changed water bath temperature to 55ºC.
56	Section 5.10: Specified evaporation volume in the last step
64	Section 6: Added reference to MLA-013
66	Section 6.5: Updates to current practice - solvent ratio, column loading/rinsing procedures.
70	Figure2 : Corrected format error in the first decision “diamond”
73	Section 8.1: Document clarification that congener contributions to Aroclor 1254 corresponding to PCB congener 99 includes contribution from coeluting PCB 83

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The table below lists the details of the changes from revision 11 version 02 of this document.

Page	Change Details
18	Section 3.1: Added Ahlstrom 161 filter (for Millipore filtration of stack gas extract in section 5.11)
20, 21, 24, 25, 35, 69	Section 3.3.4, Table 3, Table 4c and section 7.1: Added optional CS-6 calibration level.
28	Sections 3.6.5 and 3.6.6: Corrected SOP references to be SCH-002.
31, 34	Tables 4a and 4b: Removed obsolete CAL/VER warning limits for the cleanup standards.
41, 55, 56	Sections 5.3.2, 5.9 and 5.10: Clarified lipid determination instructions for tissue, milk and blood/serum/ plasma samples.
74	Section 8.1: Updated calculation of Aroclor 1254 to apply for EPA method1668C as well as for 1668A.
75-84	Table 6a: Widened a few RT windows to reflect current practice accommodating to skewed peaks of some coeluting peaks (PCB-95/100/93/102/98, PCB-151/135/154 and PCB-197/200)
87	Table 7: Changed PCB-209 acquisition ions from (m+2)/(m+4) to (m+4)/(m+6) to align with EPA 1668A/C. Added new table “Theoretical Ion Abundance Ratios and QC Limits” (according to 1668A/C)
113	Appendix D: Clarified that aqueous samples according to EPA CBC01.2 are extracted by liquid-liquid extraction in a separatory funnel; the magnetic stirring extraction option is not allowed. Clarified that tissue samples according to EPA CBC01.2 must be Soxhlet extracted for 18-24 hours, rather than 16-20 hours.

The table below lists the details of the changes from revision 11 version 01 of this document.

Page	Change Details
13	Section F: Added “fit for intended use” phrase.
14	Table 1, Changed solids storage temperature to read -20ºC nominal to more closely reflect practice.
17	Section 3.1: Clarified current filter make.
18, 37, 38, 40, 46, 49, 50, 52, 57	Sections 3.2, 5.1.2, 5.2, 5.3.2, 5.5.3.4, 5.6.3, 5.6.4, 5.6.5, 5.7, 5.11: Changed pre-Soxhlet solvent to be dichloromethane.
18-19, 44	Sections 3.3.1 and 5.5.1: Clarified preparation procedures for surrogate standard solution.
23-24	Table 3: Removed “CAL-VER” from the table header (below CS-3)
26	Section 3.5: Changed Florisil deactivation to be by 2.0% water to correspond with current practice.
27	Section 3.6.5: Updated name of cleanup column PB 4g 44%, formerly “Layered Acid/Base Silica Column”. Added section 3.6.6: PB 8g 44% Column.
33	Table 4b: Corrected text error for PCB-170L and PCB-180L; warning limits in sample should be 40-130%.
35, 46	Sections 5 and 5.5.3.3: Added reference to SLA-124 “Liquid-Liquid Extraction Supplemental Techniques”.
37, 39	Sections 5.1.3 and 5.2.3: Changed Biobead cleanup for biosolids from optional to routine.
44	Section 5.5.1: Clarified that aqueous samples are centrifuged, not filtered. Deleted reference to obsolete SLA-086. Added instruction to visually inspect the sample before determining % suspended solids.
45	Section 5.5.2: Clarified aqueous extraction procedures. Deleted reference to obsolete SLA-086.
45-47	Section 5.5.3: Deleted filtration procedures, clarified that centrifugation is the default procedure to separate particulate and aqueous phases. Section 5.5.4: Deleted Biobead cleanup instructions for wastewater, added instead a reference to procedure instructions in section 6.1. Changed wastewater to INFL and EPRO matrices, with EFIN having Biobead as an option.
48	Sections 5.6.2 and 5.6.3: Updated procedure descriptions to current practice.

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52	Section 5.7.2: Added instruction to check for separated water phase in the extract.
55-56	Section 5.10: Clarified that minimum sample weight for blood/serum/plasma is 10 g. Updated lipid determination instruction.
62	Section 5.12: Deleted procedure instructions, instead referring to SLA-123 "Splitting of Sample Extracts".
64	Section 6: Removed all references to FMS procedures.
65	Section 6.1: Clarified that gel permeation cleanup is mandatory for INFL and EPRO extracts, but optional for all other extracts. Added information that the maximum amount of lipid per column is 2 g. Section 6.2: Updated name of cleanup column PB 4g 44%, formerly "Layered Acid/Base Silica Column". Added Section 6.3: Cleanup column PB 8g 44%.
66	Section 6.4: Updated the typical cutpoints for the alumina cleanup column
82, 84	Table 6a: Corrected that PCB-193 is quantified against PCB-180L. Corrected text error (PCB-170L and PCB-180L were entered twice).
1. 87	2. Added section 9 Reporting Criteria and Practices.
3. various	4. Renumbered some sections in accordance with updated method format standard.

The table below lists the details of the changes from revision 10 Version 02 of this document.

Page	**Change Details**
General	Added references to EPA method 1668C where applicable.
2	References: Added EPA method 1668C to the list
9	Clarified how 1668A and 1668C identify congeners 107, 108 and 109
29-30	Table 4a: Pertaining to 1668A only. Added L-PCB-170 and L-PCB-180 specifications.
31-32	Table 4b: Pertaining to 1668C only. Added L-PCB-170 and L-PCB-180 specifications.
36, 37, 39, 41, 44, 45, 47, 48, 63, 64	Section 5.1, 5.2, 5.3, 5.4, 5.5.2.1, 5.5.2.2, 5.5.3.1, 5.5.3.2, 5.5.3.3, 5.6.3, 5.6.4, 6.2, 6.3 and 6.4: Clarified in the section headers which extraction and cleanup options are default and which are optional.
62-63	Section 6: Clarified the two cleanup column sequences that are allowed. Deleted former Figure 2 "Flow chart – Manual Florisil/Silica/Alumina Clean-up Column Sequence".
72-81	Table 6a: Revised PCB 170 and PCB 180 to use 13C-PCB 170 and PCB 180 (respectively) as quantification references.
82	Added Table 6b "Analyte Retention Times, Surrogates Used and RRT Windows for Congener Numbers 107,108, 109 in accordance with 1668A Specification"
83	Added Table 6c "Analyte Retention Times, Surrogates Used and RRT Windows for Optional Reporting of Congener Numbers 107,108, 109 in accordance with 1668C Specification".
84	Section 10: Deleted modification item that powdered rather than granular sodium is used. AXYS currently uses granular.
	Deleted obsolete Appendix E "Optional Reporting of PCB Congeners 107, 108 and 109 in accordance with EPA 1668C specification"

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The table below lists the details of the changes from revision 10 Version 01 of this document.

Page	Change Details
1, 2, 10, 105ff	Updated references to EPA method CBC01 to current CBC01.2
25	Section 3.5 “4.5% carbon/Celite”: Corrected typo about instruction to mix before use.
32	Updated list of worksheets (FWO-)
50-51	Section 5.10: Harmonized blood extraction instructions with MLA-007 (and with current practice)
71	Table 6: Added column with quantification references.
107	Appendix D: Added clarification that hepta-PCBs according to EPA CBC01.2 are quantified against ^{13}C-PCB 188 and ^{13}C-PCB 189 only.
115	Appendix E – added alternate procedure for reporting PCB congener numbers 107, 108, 109 in accordance with EPA 1668C.

The table below lists the details of the changes from revision 9 of this document.

Page	Change Details
35	Section 5.2.2: Deleted requirement to add the toluene in the Dean-Stark receiver to the extract.
58	Section 5.12, point 1: Harmonized instructions for extract transfer with and without sodium sulfate. Deleted rinse instructions for PBDPE.
61	Section 6: Added three manual cleanup options. Clarified instructions for sulphur removal with activated copper. Added Figure 2 – “Flow Chart: Manual Florisil/Silica/Alumina Clean-up Column Sequence”.
62	Section 6.2: Deleted sulphur removal instruction. Clarified that acid/base silica columns may be loaded using extract in any solvent.
62	Section 6.3: Clarified required extract volume (1 mL hexane). Deleted sulphur removal instruction.
63	Section 6.4: Clarified extract loading instructions and composition of rinse solvent.
64	Section 7: Added instructions of how to evaporate the extract after the final column cleanup.

The table below lists the details of the changes from revision 8 of this document.

Page	Change Details
	Introduction, section 2.0, Optional Analyses: Corrected “PCDD/F by EPA Superfund method DLM02.0” to read “PCBs by EPA method CBC01.0”.
	Section 5.5.1: Allow the solution to equilibrate for at least 30 min after addition of surrogate standard solution.
	Sections 5.5.2 and 5.5.3: Added aqueous extraction option using magnetic stirring with dichloromethane.
	Section 5.9, Milk extraction: Changed surrogates to be added directly to the sample prior to the solvent instead of being added after the solvent (similarly as in MLA-007 and MLA-017). After drying with anh. sodium sulphate, added filtration through large fluted filter paper, evaporation and addition of cleanup standards. The following concentration and glass wool filtration steps omitted (similarly as in MLA-013).
	Section 5.13, SPE Disk Extraction: Deleted the SPE (solid phase extraction) procedure from this document.

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The table below lists the details of the changes from revision 7 of this document.

Page	Change Details
	General: Added references to EPA Method CBC01.0 and Appendix D to describe the analysis.
26	Section 4.1 Deleted requirement to spike field standard to OPR sample.
29	Revised the acceptance criterion for sum of PCBs in blank to 300 pg/sample. Included accepting higher concentrations if sample concentrations exceed 10x the blank.
52	Section 5.11 Correction: changed the layer to be discarded to “aqueous (top) layer”
59	Section 5.13.1 Revised procedure for the addition of surrogate standard.
65	Section 7 Revised microvialing procedure to clarify the different final extract volumes required for PCB, PCB/BDE and BDE analyses.
82	Section 10.0 Revised modification to Section 12.4.9 to include the correction of surrogate recoveries for lipid analysis.

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INTRODUCTION

A. ANALYTES OF INTEREST

This analytical method determines the concentrations of all 209 PCB congeners by high resolution GC/MS (HRGC/HRMS). PCBs are reported using the PCB numbering conventions used either in EPA Method 1668A or in EPA Method 1668C; these methods differ in how PCB Congener Numbers 107, 108, 109 are assigned to the chromatographic peaks.

The concentrations of some PCB congeners are reported as the sum of two or more congeners due to coelution of the congeners. The coeluting congeners are listed below.

COELUTING PCB CONGENERS	
PCB 12, 13	PCB 93, 95, 98, 100, 102
PCB 30, 18	PCB 107, 124 (1668A) or PCB 108, 124 (1668C)
PCB 20, 28	PCB 110, 115
PCB 21, 33	PCB 128, 166
PCB 26, 29	PCB 129, 138, 160, 163
PCB 40, 41, 71	PCB 134, 143
PCB 44, 47, 65	PCB 135, 151, 154
PCB 45, 51	PCB 139, 140
PCB 49, 69	PCB 147, 149
PCB 50, 53	PCB 156, 157[1]
PCB 59, 62, 75	PCB 153, 168
PCB 61, 70, 74, 76	PCB 171, 173
PCB 83, 99	PCB 183, 185
PCB 85, 116, 117	PCB 180, 193
PCB 86, 87, 97 108, 119, 125 (in EPA 1668A) or PCB 86, 87, 97 109, 119, 125 (in EPA 1668C)	PCB 197, 200
PCB 88, 91	PCB 198, 199
PCB 90, 101, 113	

The concentrations of all other PCB congeners are reported as the concentration of individual congeners. The occasional formation of an interference during the extraction procedure and the high boiling point of the toluene may limit the quantification of PCBs 1 through 15.

A subset of congeners designated as “toxic” by the World Health Organization, can be reported with the associated toxic equivalents.

[1] A second GC/MS analysis on a DB-1 column (30 m, 0.25 mm I.D., 0.25 µm film thickness) resolves the PCB 156/157 coeluting pair. This second analysis is performed upon request by the client.

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WHO TOXIC PCB CONGENERS	Congener Number
3,4,4',5-Tetrachlorobiphenyl (TeCB)	81
3,3',4,4'-TeCB	77
2,3,3',4,4'-Pentachlorobiphenyl (PeCB)	105
2,3,4,4',5-PeCB	114
2,3',4,4',5-PeCB	118
2',3,4,4',5-PeCB	123
3,3',4,4',5-PeCB	126
2,3,3',4,4',5-Hexachlorobiphenyl (HxCB)	156
2,3,3',4,4',5'-HxCB	157
2,3',4,4',5,5'-HxCB	167
3,3',4,4',5,5'-HxCB	169
2,3,3',4,4',5,5'-HpCB	189

Upon client request, any subset of the 209 PCB congeners may be reported.

A.1 Optional Analyses

The analysis of PCBs by EPA method CBC01.2 is carried out as described in this document, with the modifications described in Appendix D.

The analysis of chlorinated pesticides may be carried out concurrently with this method, using the extraction and cleanup procedures described in this document. The analyst is referred to Appendix A for details of carrying out this analysis.

The analysis of polybrominated diphenylethers is carried out using the extraction and cleanup procedures described in this method, as documented in AXYS Method Document MLA-033, *Analysis Method for the Determination of Polybrominated Diphenylethers by EPA Method 1614.*

B. CONTAMINATION AND INTERFERENCES

Contamination can come from all aspects of the analysis procedure. Care should be taken to ensure that all glassware is cleaned according to the specifications listed in Section 3.2. The Soxhlet apparatus (including thimble) must be pre-extracted with dichloromethane for a minimum of two hours (three hours if brominated analytes are being determined, or three hours with toluene if PCBs are analyzed according to EPA method CBC01.2) to avoid potential contamination from previous samples.

PCB congeners may be destroyed in the presence of acid and caution must be exercised to limit the exposure the sample and extract to acid.

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Records of background levels within reference materials should be maintained in order to correct QC samples run within the same batch.

Further chromatographic cleanup may be carried out to resolve interferences with toxic PCB congeners. These procedures are described in Section 6.6 and Appendix B.

C. SAFETY

Refer to Section 5.0 of EPA Method 1668C for safety precautions when performing the analysis. For general safety procedures refer to SAF-001 *"Safety Manual"*. Refer to standard operating procedures SLA-079 *"Agricultural Hazard Protocols for Soils"* and SLA-082 *"Handling of Human Biohazardous Samples"* for procedures for handling of hazardous samples.

D. POLLUTION PREVENTION AND WASTE MANAGEMENT

AXYS Analytical Services Ltd. complies with all federal, provincial and municipal regulations governing waste management, including land disposal restrictions and sewage discharge regulations. AXYS' waste disposal procedures have been developed to comply with all pollution prevention regulations.

All standards are prepared in volumes consistent with volumes required by the method to minimize the disposal of standards.

Refer to AXYS standard operating procedure SAD-014 *"Sample Disposal"* for procedures for disposing of sample laboratory wastes.

E. DEFINITIONS

Refer to AXYS document QDO-001 "QA/QC Policies and Procedures Manual" for definitions of terms used in this document.

F. METHOD PERFORMANCE

The method performance quality acceptance specifications have been verified at AXYS. Ongoing method performance is monitored by the measurement of percent surrogate recoveries in samples, percent analyte recovery in spiked matrix samples and certified reference samples, background analyte levels in procedural blanks, and relative percent difference between sample duplicates.

This method has been validated to demonstrate that it is fit for the intended use.

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ANALYSIS PROCEDURES

1. SAMPLE PRESERVATION AND STORAGE

All samples must be received and stored under the conditions presented in Table 1.

Table 1. Sample Storage, Sample Receipt and Hold Time Requirements

Matrix	Sample Size (per analysis)	Sample Container[1]	Condition upon Receipt	Storage Conditions[2]	Holding Time[3]	Extract Holding Time[4]	Preservation
Solid (Sediment/Soil/ Sludge)	10 g dry wt basis	Amber Glass	<4°C	-20°C, dark	(1 year)	1 year	none required
Tissue	5-10 g wet	Glass or foil wrapped	<4°C	-20°C, dark	(1 year)	1 year	none required
Blood	5-20 g	Glass	<4°C	-20°C, dark	(1 year)	1 year	none required
Milk	50 - 150 g	Glass	<4°C	-20°C, dark	(1 year)	1 year	none required
Aqueous (water/effluent/ wet sludge)	1 litre	Amber glass	0 – 4°C	4°C, dark	(1 year)	1 year	Add 80 mg of Na_2SO_3 if +ve for residual Cl
XAD-2 Columns	- one column - resin - particulate filter(s) - wound glass filter	-As is -Glass -Foil wrapped -Foil wrapped	0 – 4°C 0 – 4°C <4°C <4°C	4°C, dark 4ºC, dark -20°C, dark -20°C, dark	undefined undefined undefined undefined	undefined undefined undefined undefined	none required none required none required none required
Ambient Air	PUF & Filter(s)	Glass	<4°C	-20°C, dark	undefined	undefined	none required
Sample Trains (Stack Gas)	Several Containers[5]	Glass	0 – 4°C	4°C	(30 days)	undefined	none required
Particulate Filter Papers	1 or more	Glass or foil wrapped	<4°C	-20°C, dark	undefined	undefined	none required
Solvent Extract	1 Sample	Glass	<4°C	-20°C, dark	undefined	undefined	none required

[1] All glass containers should be organically clean; i.e., baked, solvent-rinsed or purchased as certified 'clean'. All containers must be tightly sealed with screw cap lids (PTFE or foil-lined) to prevent loss of volatiles or contamination from volatiles. If samples are received in clear glass containers, they must be protected from the light. Samples for brominated analysis must be in amber glass containers.

[2] Storage temperatures quoted are nominal temperatures. Samples stored at a nominal temperature of 4ºC are permitted a variance of ±2°C and samples stored at -20°C are permitted a variance of ±4°C.

[3] Hold times in brackets are from time of sampling. There is no evidence to indicate that properly stored samples are not stable for longer periods of time. Client requests for specific holding times or other method-specific holding times are adhered to.

[4] Holding times for extracts are from time of extraction.

[5] Refer to Section 5.11 for details of containers and contents.

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2. SAMPLE PRETREATMENT AND PREPARATION

Refer to the following standard operating procedures for details of laboratory techniques for sample preparation.

Document ID	Title
SLA-011	Compositing Samples
SLA-012	Dissection of Samples
SLA-013	Procedures for Homogenization of Solids and Tissues
SLA-014	Thawing Solid and Tissue Samples
SLA-015	Moisture Determination
SLA-043	Removing Sampling Media from Field Sampling Equipment
SLA-084	Preparation of Aqueous Samples for Extraction

2.1 Solids (Sediment/Soil) Samples

Allow a solid sample to thaw prior to homogenization and subsampling procedures following procedures in standard operating procedure SLA-014. Homogenize the entire sample before subsampling for analysis following standard operating procedure SLA-013.

Determine the percent moisture of an accurately weighed subsample (1 - 2 g) according to standard operating procedure SLA-015.

2.2 Tissues

Allow tissue samples to thaw prior to homogenization and subsampling procedures following procedures in standard operating procedure SLA-014. If the tissue is received as a homogenate, stir well with a spatula prior to subsampling. Otherwise, homogenize tissue samples following standard operating procedures. Samples must be extracted within 24 hours of thawing.

If required, determine the moisture content of an accurately weighed subsample (1 g) according to standard operating procedure SLA-015.

2.3 Aqueous Samples

Refer to the standard operating procedures SLA-084, *Preparation of Aqueous Samples for Extraction*, for sample preparation procedures.

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2.4 XAD-2 Columns and Filters

Refer to the following standard operating procedure SLA-043, *Removing Sampling Media from Field Sampling Equipment*, for details of removing the resin from the column. Filters are partially thawed prior to extraction.

2.5 Whole Blood/Blood Serum/Blood Plasma

Allow blood samples to thaw prior to subsampling for analysis following procedures in standard operating procedure SLA-014. Homogenize the sample by shaking well. Samples must be extracted within 24 hours of thawing.

2.6 Milk

Allow a milk sample to thaw prior to homogenization and subsampling following procedures in standard operating procedure SLA-014. Homogenize the sample by shaking well. Samples must be extracted within 24 hours of thawing.

2.7 Ambient Air (PUF and Filter)

If received in the sampling apparatus, use solvent rinsed forceps to withdraw the PUF from the sampler. The associated filter is typically received in a Petri dish or wrapped in foil. Use solvent rinsed forceps to handle the filter.

2.8 Sludge

Homogenize dry sludge according to standard operating procedure SLA-013. Consult with the Lab Supervisor for the treatment of wet sludge samples.

Determine the percent moisture of an accurately weighed subsample (1 - 2 g) of dry sludge according to standard operating procedure SLA-015.

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3. MATERIALS AND REAGENTS

Refer to the following standard operating procedures:

Document ID	Title
SLA-001	Cleaning of Laboratory Items
SLA-002	Glassware and Laboratory Equipment Proofs
SLA-009	Preparation of Standards
SLA-018	Solvent Rinsing of Glassware for Organic Analysis
SLA-019	Solvent Proofs
SLA-022	Use of Drying Ovens and Muffle Furnace
SLA-023	Use of Balances
SLA-036	Cleaning of GC/MS and GC/ECD Microvial Caps
SLA-041	Reagent Preparation
SLA-044	Activation of Copper Foil, Turnings and Powder
SLA-093	Baking of Anhydrous Sodium Sulphate
SQA-003	Standard Solution Validation
SQA-009	Storage and Control of Analytical Standards

3.1 Equipment List

Extraction

Separatory funnels (125 mL, 500 mL, 1000 mL)
Erlenmeyer flasks (250 mL, 500 mL, 1000 mL)
Round bottom flasks (100 mL, 250 mL, 500 mL, 1000 mL))
Beakers
Graduated Cylinders
Class A volumetric flasks, pipettes
Disposable pipettes
Disposable centrifuge tubes (15 mL)
Hamilton Syringes (5 µL, 25 µL, 50 µL, 100 µL, 250 µL, 1000 µL)
Autosampler vials (amber glass, 800 µL)
Chromatography columns (1 cm x 12 cm, 3 cm x 50 cm, 1 cm x 25 cm)
Silanized glass wool
Glass fibre filter paper, Ahlstrom, 161 grade, 1.1 µm, 42.5 mm (or equivalent)
Filter paper – PALL, glass fiber, type A/E, 1.0 µm, 102 mm
PTFE tape
Aluminum Foil
Spatula – aluminum
Disposable spoons
Rotary evaporator with water bath at 30 °C
Magnetic stirring plate, with pre-cleaned PTFE coated magnetic stirring bars

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Millipore Filtration Apparatus
Soxhlet apparatus (pre-cleaned) with heating mantle
Soxhlet/Dean-Stark apparatus (pre-cleaned)
Water bath capable of maintaining up to 55 ºC
Balance – Top loading and analytical (2-, 3-, & 4-place)
Glassware ovens
Drying ovens
Muffle furnace
Nitrogen source with manifold apparatus

Instruments

Hewlett Packard 6890 Series Gas Chromatograph or equivalent;
Micromass/VG Autospec Ultima mass spectrometer or equivalent;
GC Columns: SPB-Octyl (30 m, 0.25 mm i.d., 0.25 µm film thickness)
DB-1 (30 m, 0.25 mm i.d., 0.25 µm film thickness)

3.2 Glassware

All glassware used in the preparation of reagents and in the analytical procedure must be organically clean. Glassware must be washed and baked using standard operating procedures. Baked glassware must be solvent rinsed with toluene and hexane. The Soxhlet apparatus, including thimble, must be or pre-cleaned by Soxhlet extraction with dichloromethane for a minimum of 2 hours for PCB analyses (three hours if brominated analytes are determined, or three hours with toluene if PCBs are analyzed according to EPA method CBC01.2).

If baked glassware is not available, glassware must be washed and water, rinsed with solvent rinsed following the procedures described in standard operating procedure SLA-018.

3.3 Preparation of Standard Solutions

The analysis of PCB congeners by EPA Methods 1668A or 1668C requires the use of the surrogate, cleanup, recovery (internal), authentic spike and calibration solutions described in Section 7.7 - 7.16 of EPA Method 1668A or 1668C (tables 2 and 3 in this method). Stock solutions are prepared from commercially available individual components. Working standards are prepared by diluting stock solutions. The Standard Data Sheets describe the details of the preparation and composition of all stock and working standard solutions used and the validated concentrations of all components.

3.3.1 Surrogate Standard Solution

Prepare the surrogate standard stock solution as described in EPA Method 1668A or 1668C, Section 7.9.1. Modify the preparation of the surrogate spiking solution (Section 7.12, EPA 1668A or 1668C) so that the concentration of the labelled compounds is 100 ng/mL (table 2 of MLA-010). The volume of the aliquot used is adjusted so that the same absolute amount of

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standard, as described in EPA Method 1668A or 1668C is added.

Prior to extraction procedures, each sample and QC sample is spiked with an aliquot of surrogate standard solution, containing a suite of $^{13}C_{12}$-labelled surrogate standards. Typically, an aliquot of 20 µL is added.

For aqueous samples, the surrogate standard is prepared immediately prior to extraction procedures by diluting an aliquot of the stock surrogate standard (1000 ng/mL of each surrogate) in a volume of acetone, to make a solution that is 2 ng/mL of each surrogate. Prepare the surrogate standard in the laboratory space where it will be used. A 1 mL aliquot is added to each aqueous sample. Sufficient solution to spike an entire analysis batch, including QC sample, is prepared.

The amount of surrogate added is dependent upon sample size, final extract volume and concentration of analytes in the sample and may be adjusted accordingly. The Batch List indicates the name of the surrogate standard solution used and the volume added to each sample. The suite of surrogates, the nominal concentration of each surrogate in the standard solution and the amount added to each sample are presented in Table 2.

3.3.2 Cleanup Standard Solution

Prepare the cleanup standard stock solution as described in EPA Method 1668A or 1668C, Section 7.9.2. Modify the preparation of the cleanup standard spiking solution (Section 7.13, EPA Method 1668A or 1668C) so that the concentration of the labelled compounds is 100 ng/mL (table 2 of MLA-010).

Just prior to chromatographic cleanup each extract is spiked with an aliquot of cleanup standard solution, containing $^{13}C_{12}$-labelled standards. Typically an aliquot of 20 µL is added. The Batch List indicates the name of the cleanup standard solution used and the volume added to each sample. The suite of standards used, the nominal concentration of each standard in the standard solution and the amount added to each sample are presented in Table 2.

3.3.3 Recovery (Internal) Standard Solution

Prepare the recovery standard stock solution as described in EPA Method 1668A or 1668C, Section 7.9.3. Prepare the diluted internal (recovery) standard spiking solution as described in EPA Method 1668A or 1668C, Section 7.14 (table 2 of MLA-010).

Prior to instrumental analysis, cleaned up extracts are spiked with $^{13}C_{12}$-labelled recovery (internal) standards prior to instrumental analyses. Typically, an aliquot of 5 µL of recovery standard solution is added. The Batch List indicates the name of the recovery standard solution used and the volume added to each sample. The suite of recovery standards, the nominal concentration of each standard in the solution and the amount added to each sample are presented in Table 2.

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3.3.4 Calibration Solutions

A series of calibration solutions (CS-0.2 through CS-5, CS-6 optional) containing native analytes, labelled surrogate standards and labelled recovery standards are used to establish the linearity of the analytical instrument. The concentration of the native analytes in the solutions varies to encompass the working range of the instrument, while the concentration of the surrogate recovery and cleanup standards remain constant. The composition of the solutions and the nominal concentration of each component are presented in Table 3.

CS-0.2 through CS-5 and optionally CS-6

Prepare six calibration solutions (CS-0.2 through CS-5) as described in EPA Method 1668A or EPA1668C, Section 7.10.1, at the concentrations listed in Table 5 of the EPA method (table 3 of MLA-010). To extend the linear range for PCBs (except for mono- and di-PCBs)_a seventh calibration solution (CS-6) may optionally be prepared. These solutions are used to establish the linearity of the GC/MS.

Calibration Solution for 209 Congeners

Prepare a calibration solution containing all 209 PCB congeners (unlabelled), the surrogate standards and the recovery standards, as described in EPA Method 1668A or 1668C, Section 7.10.2, and where appropriate, the relevant field standards. This single calibration solution is used to determine the relative response factors and retention times of the PCB congeners not in the calibration solutions and is used as the calibration verification (CAL/VER) solution, analyzed at least every twelve hours to verify GC/MS performance and calibration. The nominal concentration of the unlabelled congeners in this solution is: mono-tri-CB: 25 ng/mL; tetra-hp-CB: 50 ng/mL; octa-nona-CB: 75 ng/mL.

3.3.5 Authentic Spike Solution (Precision and Recovery Standard, PAR)

Prepare the authentic spiking solution as described in EPA Method 1668A or 1668C, Section 7.11. This solution is used to prepare the initial precision and recovery (IPR) samples and ongoing performance and recovery (OPR) samples. Typically an aliquot of 1 mL is added to every OPR and IPR sample. The Batch List indicates the name of the PAR standard solution used and the volume added to each sample. The composition of the solution, the nominal concentration of each component and amount added to the sample are presented in Table 2.

3.3.6 Field Surrogate Solutions

If required, a field surrogate standard solution or sampling media spiked with a field surrogate solution is provided to a client. The solution contains $^{13}C_{12}$-labelled PCBs. The Project Manager, in consultation with the client, determines the components in the solution and the appropriate concentration. The most typical field surrogates are $^{13}C_{12}$-PCB 31, $^{13}C_{12}$-PCB 95 and $^{13}C_{12}$-PCB 153. Field surrogate solutions are prepared under the direction of the Standards Chemist, according to instructions from the Project Manager.

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Table 2. Concentration of PCB Standard Solutions - HRGC/HRMS Analysis

Compound Name	Congener No (1)	Conc'n of Stock Solution (Aqueous samples)	Concentration of Standard Solution	Typical Amount Spiked into Sample
Surrogate Std Solution				
$^{13}C_{12}$-2-MoCB	1L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-4-MoCB	3L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-2,2'-DiCB	4L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-4,4'-DiCB	15L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-2,2',6-TriCB	19L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-3,4,4'-TriCB	37L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-2,2',6,6'-TeCB	54L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-3,3',4,4'-TeCB	77L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-3,4,4',5-TeCB	81L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-2,2',4,6,6'-PeCB	104L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-2,3,3',4,4'-PeCB	105L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-2,3,4,4',5-PeCB*	114L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-2,3',4,4',5-PeCB	118L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-2',3,4,4',5-PeCB	123L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-3,3',4,4',5-PeCB	126L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-2,2',4,4',6,6'-HxCB	155L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-2,3,3',4,4',5-HxCB	156L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-2,3,3',4,4',5'-HxCB	157L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-2,3',4,4',5,5'-HxCB	167L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-3,3',4,4',5,5'-HxCB	169L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-2,2',3,3',4,4',5-HpCB	170L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-2,2',3,4,4',5,5'-HpCB	180L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB	188L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-2,3,3',4,4',5,5'-HpCB	189L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB	202L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB	205L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB	206L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-NoCB	208L	1000 ng/mL	100 ng/mL	2000 pg
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-DeCB	209L	1000 ng/mL	100 ng/mL	2000 pg
Cleanup Std Solution				
$^{13}C_{12}$-2,4,4'-TriCB	28L	-	100 ng/mL	2000 pg
$^{13}C_{12}$-2,3,3',5,5'- PeCB	111L	-	100 ng/mL	2000 pg
$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB	178L	-	100 ng/mL	2000 pg
Recovery Std Solution				
$^{13}C_{12}$-2,5-DiCB	9L	-	400 ng/mL	2000 pg
$^{13}C_{12}$-2,2',5,5'-TeCB	52L	-	400 ng/mL	2000 pg
$^{13}C_{12}$-2,2',4',5,5'-PeCB	101L	-	400 ng/mL	2000 pg
$^{13}C_{12}$-2,2',3',4,4',5'-HxCB	138L	-	400 ng/mL	2000 pg
$^{13}C_{12}$-2,2',3,3',4,4',5,5'-OcCB	194L	-	400 ng/mL	2000 pg

(1) Suffix "L" designates labelled compound

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Table 2 Cont'd

Compound Name	Congener No.	Concentration of Standard Solution	Typical Amount Spiked into Sample
Authentic Spike Solution			
2-MoCB	1	1 ng/mL	1000 pg
4-MoCB	3	1 ng/mL	1000 pg
2,2'-DiCB	4	1 ng/mL	1000 pg
4,4'-DiCB	15	1 ng/mL	1000 pg
2,2',6-TriCB	19	1 ng/mL	1000 pg
2,3,5-TriCB	23	1 ng/mL	1000 pg
2',3,5-TriCB	34	1 ng/mL	1000 pg
3,4,4'-TriCB	37	1 ng/mL	1000 pg
2,2',6,6'-TeCB	54	1 ng/mL	1000 pg
3,3',4,4'-TeCB	77	1 ng/mL	1000 pg
3,4,4',5-TeCB	81	1 ng/mL	1000 pg
2,2',4,6,6'-PeCB	104	1 ng/mL	1000 pg
2,3,3',4,4'-PeCB	105	1 ng/mL	1000 pg
2,3,4,4',5-PeCB	114	1 ng/mL	1000 pg
2,3',4,4',5-PeCB	118	1 ng/mL	1000 pg
2',3,4,4',5-PeCB	123	1 ng/mL	1000 pg
3,3',4,4',5-PeCB	126	1 ng/mL	1000 pg
2,2',4,4',6,6'-HxCB	155	1 ng/mL	1000 pg
2,3,3',4,4',5-HxCB	156	1 ng/mL	1000 pg
2,3,3',4,4',5'-HxCB	157	1 ng/mL	1000 pg
2,3',4,4',5,5'-HxCB	167	1 ng/mL	1000 pg
2-3,3',4,4',5,5'-HxCB	169	1 ng/mL	1000 pg
2,2',3,3',4,4',5-HpCB	170	1 ng/mL	1000 pg
2,2',3,4,4',5,5'-HpCB	180	1 ng/mL	1000 pg
2,2',3,4,4',5,6'-HpCB	182	1 ng/mL	1000 pg
2,2',3,4',5,5',6-HpCB	187	1 ng/mL	1000 pg
2,2',3,4',5,6,6'-HpCB	188	1 ng/mL	1000 pg
2,3,3',4,4',5,5'-HpCB	189	1 ng/mL	1000 pg
2,2',3,3',5,5',6,6'-OcCB	202	1 ng/mL	1000 pg
2,3,3',4,4',5,5',6-OcCB	205	1 ng/mL	1000 pg
2,2',3,3',4,4',5,5',6-NoCB	206	1 ng/mL	1000 pg
2,2',3,3',4,5,5',6,6'-NoCB	208	1 ng/mL	1000 pg
2,2',3,3',4,4',5,5',6,6'-DeCB	209	1 ng/mL	1000 pg

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Table 3. Concentration of PCB Calibration Solutions - HRGC/HRMS Analysis

CB CONGENER	Congener No.	Solution concentration (ng/mL)						
		CS-0.2 (Hi sens)[1]	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6 (optional)
Native Compounds								
2-MoCB	1	0.2	1.0	5.0	50	400	2000	n.a.
4-MoCB	3	0.2	1.0	5.0	50	400	2000	n.a.
2,2'-DiCB	4	0.2	1.0	5.0	50	400	2000	n.a.
4,4'-DiCB	15	0.2	1.0	5.0	50	400	2000	n.a.
2,2',6-TriCB	19	0.2	1.0	5.0	50	400	2000	6000
2,3,5-TriCB	23	0.2	1.0	5.0	50	400	2000	6000
2',3,5-TriCB	34	0.2	1.0	5.0	50	400	2000	6000
3,4,4'-TriCB	37	0.2	1.0	5.0	50	400	2000	6000
2,2',6,6'-TeCB	54	0.2	1.0	5.0	50	400	2000	6000
3,3',4,4'-TeCB	77	0.2	1.0	5.0	50	400	2000	6000
3,4,4',5-TeCB	81	0.2	1.0	5.0	50	400	2000	6000
2,2',4,6,6'-PeCB	104	0.2	1.0	5.0	50	400	2000	6000
2,3,3',4,4'-PeCB	105	0.2	1.0	5.0	50	400	2000	6000
2,3,4,4',5-PeCB	114	0.2	1.0	5.0	50	400	2000	6000
2,3',4,4',5-PeCB	118	0.2	1.0	5.0	50	400	2000	6000
2',3,4,4',5-PeCB	123	0.2	1.0	5.0	50	400	2000	6000
3,3',4,4',5-PeCB	126	0.2	1.0	5.0	50	400	2000	6000
2,2',4,4',6,6'-HxCB	155	0.2	1.0	5.0	50	400	2000	6000
2,3,3',4,4',5-HxCB	156	0.2	1.0	5.0	50	400	2000	6000
2,3,3',4,4',5'-HxCB	157	0.2	1.0	5.0	50	400	2000	6000
2,3',4,4',5,5'-HxCB	167	0.2	1.0	5.0	50	400	2000	6000
3,3',4,4',5,5'-HxCB	169	0.2	1.0	5.0	50	400	2000	6000
2,2',3,3'4,4',5-HpCB	170	0.2	1.0	5.0	50	400	2000	6000
2,2',3,4,4',5,5'-HpCB	180	0.2	1.0	5.0	50	400	2000	6000
2,2',3,4,4',5,6'-HpCB	182	0.2	1.0	5.0	50	400	2000	6000
2,2',3,4',5,5',6-HpCB	187	0.2	1.0	5.0	50	400	2000	6000
2,2',3,4',5,6,6'-HpCB	188	0.2	1.0	5.0	50	400	2000	6000
2,3,3',4,4',5,5'-HpCB	189	0.2	1.0	5.0	50	400	2000	6000
2,2',3,3',5,5',6,6'-OcCB	202	0.2	1.0	5.0	50	400	2000	6000
2,3,3',4,4',5,5',6-OcCB	205	0.2	1.0	5.0	50	400	2000	6000
2,2',3,3',4,4',5,5',6-NoCB	206	0.2	1.0	5.0	50	400	2000	6000
2,2',3,3',4,5,5',6,6'-NoCB	208	0.2	1.0	5.0	50	400	2000	6000
2,2',3,3',4,4',5,5',6,6'-DeCB	209	0.2	1.0	5.0	50	400	2000	6000
Surrogate Standards								
$^{13}C_{12}$-2-MoCB	1L[2]	100	100	100	100	100	100	100
$^{13}C_{12}$-4-MoCB	3L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2'-DiCB	4L	100	100	100	100	100	100	100

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CB CONGENER	Congener No.	Solution concentration (ng/mL)						
		CS-0.2 (Hi sens)[1]	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6 (optional)
$^{13}C_{12}$-4,4'-DiCB	15L	100	100	100	100	100	100	100
$^{13}C_{12}$-3,4,4'-TriCB	37L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',6,6'-TeCB	54L	100	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4'-TeCB	77L	100	100	100	100	100	100	100
$^{13}C_{12}$-3,4,4',5-TeCB	81L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',4,6,6'-PeCB	104L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4'-PeCB	105L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,3,4,4',5-PeCB	114L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,3',4,4',5-PeCB	118L	100	100	100	100	100	100	100
$^{13}C_{12}$-2',3,4,4',5-PeCB	123L	100	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4',5-PeCB	126L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',4,4',6,6'-HxCB	155L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5-HxCB	156L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5'-HxCB	157L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,3',4,4',5,5'-HxCB	167L	100	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4',5,5'-HxCB	169L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5-HpCB	170L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,4,4',5,5'-HpCB	180L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB	188L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5,5'-HpCB	189L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB	202L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB	205L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB	206L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-NoCB	208L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-DeCB	209L	100	100	100	100	100	100	100
Cleanup Standards								
$^{13}C_{12}$-2,4,4'-TriCB	28L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',5,5'-PeCB	111L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB	178L	100	100	100	100	100	100	100
Recovery Standards								
$^{13}C_{12}$-2,5-DiCB	9L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',5,5'-TeCB	52L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',4',5,5'-PeCB	101L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3',4,4',5'-HxCB	138L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5,5'-OcCB	194L	100	100	100	100	100	100	100

[1] Additional concentration used for calibration of high sensitivity HRGC/HRMS systems.

[2] Suffix "L" indicates labelled compound.

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3.4 Preparation of Reagents

Activated Copper is prepared as described in the standard operating procedure SLA-044.

Ammonium Sulphate ($(NH_4)_2SO_4$, Saturated) is prepared by dissolving ammonium sulphate (700 g) in ultra pure water (1 L). The solution is extracted by shaking twice with dichloromethane (2 x 100 L) and once with hexane (100 mL). Shelf life 3 months, after expiry re-extract and re-use.

Anhydrous Sodium Sulphate (Na_2SO_4, granular 12-60 mesh, J.T. Baker, or demonstrated equivalent approved for use by Operations Management and QA) is cleaned by baking at a minimum of 300°C as described in SLA-093.

Glass Fibre Filters (Ahlstrom, A/E glass or equivalent) are cleaned by baking overnight at a minimum of 300°C.

Glass Fibre Filter Bed is prepared and cleaned by grinding glass fibre filters in a blender and heating overnight at a minimum of 300°C. The cleaned filter bed is stored in a covered jar.

Hydrochloric Acid (conc., Seastar Chemicals, quartz distilled) is used as received. Indefinite shelf life.

Hydrochloric Acid (1 M) is prepared by adding HCL (100 mL, conc.,) to ultra pure water (1 L). Shelf life 2 years, discard when expired.

Potassium Hydroxide (KOH, 1M) is prepared by dissolving potassium hydroxide pellets (56 g, Fisher, certified) in ethanol (1000 mL). Shelf life 3 months, discard after expiry.

Reagent Sand (Aldrich Chemicals, white quartz, -50 +70 mesh) is proofed by lot prior to use and may be cleaned by Soxhlet extraction with dichloromethane for 16 hours as necessary. The clean sand is stored in a clean glass jar

Ultra Pure Water (Seastar Chemicals (contaminant-free) or equivalent) is used as received.

Silanized Glass Wool is stored in a clean amber jar and is rinsed twice with toluene and twice with hexane prior to use.

Sodium Hydroxide (NaOH, 1 M) is prepared by dissolving sodium hydroxide pellets (AR grade, 40 g) in ultra pure water (1 L). The solution is extracted by shaking with dichloromethane (2 x 100 mL) and once with hexane (100 mL). The solution is stored in an amber jar with PTFE lined lid. Typically 3 L is prepared. Shelf life 3 months, after expiry re-extract and re-use.

Solvents are high purity, distilled in glass solvents, either HPLC grade or pesticide residue grade. Each lot number of solvent must be checked for impurities by performing a solvent proof prior to use. All solvent mixtures used in the analyses are made by mixing the appropriate proportions of solvent on a volume:volume basis.

Soxhlet Thimbles are cleaned according to SLA-018 “Solvent Rinsing of Glassware and Equipment for Organic Analyses”, rinsed well with water and baked overnight at a minimum of 300°C.

Sulphuric Acid (conc., Seastar Chemicals, quartz distilled) is used as received. Indefinite shelf life.

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3.5 Preparation of Chromatography Materials

Refer to the following standard operating procedures:

Q-Pulse ID	Title
SCH-001	Activation/Deactivation Procedures
SCH-002	Column Packing Procedures
SCH-003	Column Cutpoint Procedures
SCH-004	Layered Silica Gel Preparation
SCH-005	Carbon/Celite Column Preparation
SCH-006	Preparation, Use and Maintenance of Biobead Columns

Alumina (Fisher Basic Brockman Activity 1, 60-325 mesh) is baked for a minimum of 8 hours at 450°C and deactivated with ultra pure water (typically 1.0% w/w) as described in SCH-001. The degree of activation may vary depending upon the batch of alumina. Deactivated alumina is stored under nitrogen in a stoppered flask and allowed to equilibrate 24 hours. Cutpoints are determined prior to use (SCH-003).

Biobeads (Bio-Rad, SX-3, 200-400 mesh) are prepared by soaking the beads for 24 hours in 1:1 dichloromethane:hexane prior to column preparation as described in SCH-006.

4.5% Carbon/Celite Mixture is prepared by adding activated carbon (AX-21, Anderson Co., 4.5 g, sieved (1 mm mesh)) to Celite 545 (95.5 g) and shaking until uniform as described in SCH-005. The mixture is stored in a stoppered glass reagent bottle. The mixture must be well mixed by shaking before use. The cutpoints are determined prior to use (SCH-003).

2.0% Florisil (Supelco or US Silica, Pesticide grade, 60-100 mesh) is activated at 450°C for a minimum of 8 hours and deactivated with ultra pure water (typically 2.0% by weight) as described in SCH-001. The degree of activation may vary depending upon the batch of Florisil. Florisil is stored under nitrogen in a stoppered flask and allowed to equilibrate for 24 hours. The cutpoints are determined prior to use (SCH-003).

Silica Gel (SiliCycle G60, 60-200 µm 60 Å, or equivalent) is heated for a minimum of 8 hours at 450°C and stored, under nitrogen, in a stoppered reagent jar as described in SCH-001.

28% NaOH Basic Silica is prepared by adding sodium hydroxide solution (equivalent to 28% of final weight of basic silica) to baked silica and agitating until homogeneous, as described in SCH-004. The silica is stored in a stoppered glass reagent bottle.

22% H_2SO_4 Acidic Silica is prepared by adding sulfuric acid (conc., equivalent to 22% of the final weight of acidified silica) to baked silica and agitating until homogeneous as described in SCH-004 The silica is stored in a stoppered glass reagent bottle.

44% H_2SO_4 Acidic Silica is prepared by adding sulfuric acid (conc., equivalent to 44% of final weight of acidified silica) to baked silica and agitating until homogenous, as described in SCH-004. The silica is stored in a stoppered glass reagent bottle.

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3.6 Preparation of Chromatography Columns

3.6.1 Alumina Column

Basic alumina (6 g, 1% deactivated) is dry packed into hexane in a glass chromatography column (1 cm x 12 cm) as described in SCH-002. The column is capped with a 1 cm bed of anhydrous granular sodium sulphate and eluted with hexane (10 mL).

3.6.2 Biobeads SX-3 Column

A Biobeads (SX-3, 60 g) column is prepared and cutpoints determined as described in SCH-006. The column is cleaned with 20% methanol:dichloromethane and 1:1 dichloromethane:hexane prior to use, as described in SCH-006.

3.6.3 4.5% Carbon/Celite Column

A carbon/Celite column is prepared by packing 4.5% carbon/Celite mixture (0.22 g) into a 9" Pasteur pipette that is fitted with a filter paper disc, as described in SCH-005. The column is pre-eluted with toluene (15 mL) and hexane (15 mL), ensuring all the toluene has been eluted from the column.

3.6.4 Florisil Column

Deactivated Florisil (8 g, 2.0% deactivated) is dry packed in hexane into a glass chromatography column (1 cm x 25 cm with 250 mL reservoir)as described in SCH-002. The column is, capped with a 1 cm layer of anhydrous sodium sulphate (granular) and flushed with hexane (~40 mL).

3.6.5 PB 4g 44% (formerly "Layered Acid/Base Silica Column")

A silica chromatography column (1 cm O.D. x 25 cm long) is prepared by sequentially dry-packing in hexane the following reagents, as described in SCH-002:

neutral silica (0.5 g), silica (28% NaOH, 2.0 g), neutral silica (0.5 g), silica (44% H_2SO_4, 4 g), silica (22% H_2SO_4, 2.0 g) and neutral silica (1.0 g).

3.6.6 PB 8g 44% (formerly "Large Capacity Layered Acid/Base Silica Column")

A silica chromatography column (2 cm O.D. x 30 cm long with glass wool plug in the bottom) is prepared by sequentially dry-packing in hexane the following reagents, as described in SCH-002:

neutral silica (1.0 g), silica (28% NaOH, 4.0 g), neutral silica (1.0 g), silica (44% H_2SO_4, 8.0 g), silica (22% H_2SO_4, 4.0 g) and neutral silica (2.0 g).

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4. QUALITY ASSURANCE/QUALITY CONTROL

Refer to the following standard operating procedure:

Q-Pulse ID	Title
SLA-016	Preparation of QA/QC Samples

4.1 Quality Control Samples

All samples are analyzed in batches. The composition of a batch is detailed on a Batch List, including the method number, quality control samples and standards to use.

- Batch Size - Each batch consists of up to twenty test samples and additional QC samples.

- Blanks - One procedural blank is analyzed with each batch. The procedural blank is prepared by spiking an aliquot of the surrogate standard solution into a clean matrix. If required, an aliquot of field surrogate spiking solution is also added to the blank sample. The clean matrices available for preparing procedural blanks are described in SLA-016. The procedural blank is extracted and analyzed using the same procedures as the test samples in the analysis batch.

  Note: For stationary source air samples, the extraction procedure for the blank (using a filter and XAD-2 resin as the matrix) starts with the acidification of the filter. The surrogate standard solution is spiked into the XAD-2 when it is in the Soxhlet body.

- Initial Precision and Accuracy (IPR) is demonstrated when commencing the method or when significant changes have been made to the method. The IPR is carried out by the analysis of four spiked reference samples (prepared as described below).

- On-going Precision and Recovery (OPR) is demonstrated by the analysis of a spiked reference matrix (SPM) analyzed with each batch. The OPR sample is prepared by spiking an aliquot of the authentic spiking standard solution into an accurately weighed in-house reference matrix (known to contain low background levels of target analytes). The matrix sample is spiked with an aliquot of surrogate standard solution and extracted following procedures in Section 5 of this method. The OPR matrix to be analyzed is assigned to the analyst when the batch is assigned.

  Note: For stationary source air samples, the reference sample (a clean filter and XAD-2 resin) is spiked with an aliquot of authentic spiking standard prior to extraction. The extraction procedure starts with the acidification of the filter. The surrogate standard solution is spiked into the XAD-2 when it is in the Soxhlet body.

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- Reference Samples – Certified reference materials are commercially available and used to validate and periodically check the methods. The type of reference material to be analyzed is assigned to the analyst when the batch is assigned.

- Duplicates - A duplicate sample is analyzed with analysis batches containing 7-20 test samples, or as required by contract, provided sufficient sample is available. For some matrices (XAD-2 columns, filters, air samples) only field duplicates (if available) can be analyzed.

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4.2 QA/QC Criteria

The acceptance ranges for the recovery of target analytes in the CAL/VER solution, the IPR and the OPR samples are presented in Tables 4a and 4b. The applicable AXYS QC acceptance limits meet or exceed the requirements of EPA 1668A or EPA 1668C.

Elements of the QA/QC program at AXYS Analytical Services are documented in the most recent revision of QDO-001 "AXYS Analytical QA/QC Policies and Procedures Manual".

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Table 4a. QC Acceptance Criteria according to EPA method 1668A for Chlorinated Biphenyls in CAL/VER, IPR, OPR, and Samples

Congener	Cong. No. [2]	Test conc. ng/mL	CAL/VER (%)		IPR [1] (%)		OPR [1] (%)		Labelled compound [1] % recovery in samples	
			Warning limits	Acceptance limits	RSD	$\bar{X}$	Warning limits	Acceptance limits	Warning limits	Acceptance limits
2-MoCB	1	50	75-125	70-130	40	60-140	70-130	50-150	-	-
4-MoCB	3	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2'-DiCB	4	50	75-125	70-130	40	60-140	70-130	50-150	-	-
4,4'-DiCB	15	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2'6-TrCB	19	50	75-125	70-130	40	60-140	70-130	50-150	-	-
3,4,4'-TrCB	37	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2'6,6'TeCB	54	50	75-125	70-130	40	60-140	70-130	50-150	-	-
3,3',4,4'-TeCB	77	50	75-125	70-130	40	60-140	70-130	50-150	-	-
3,4,4',5-TeCB	81	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2',4,6,6'-PeCB	104	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3,3',4,4'-PeCB	105	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3,4,4',5-PeCB	114	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3',4,4',5-PeCB	118	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2',3,4,4',5-PeCB	123	50	75-125	70-130	40	60-140	70-130	50-150	-	-
3,3',4,4',5-PeCB	126	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2',4,4',6,6'-HxCB	155	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3,3',4,4',5-HxCB [3]	156	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3,3',4,4',5'-HxCB [3]	157	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3',4,4',5,5'-HxCB	167	50	75-125	70-130	40	60-140	70-130	50-150	-	-
3,3',4,4',5,5'-HxCB	169	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2',3,4',5,6,6'-HpCB	188	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3,3',4,4',5,5'-HpCB	189	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2',3,3',5,5',6,6'-OcCB	202	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3,3',4,4',5,5',6-OcCB	205	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2',3,3',4,4',5,5',6-NoCB	206	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2',3,3,'4,5,5',6,6'-NoCB	208	50	75-125	70-130	40	60-140	70-130	50-150	-	-
DeCB	209	50	75-125	70-130	40	60-140	70-130	50-150	-	-

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Congener	Cong. No. [2]	Test conc. ng/mL	CAL/VER (%)		IPR [1] (%)		OPR [1] (%)		Labelled compound [1] % recovery in samples	
			Warning limits	Acceptance limits	RSD	$\bar{X}$	Warning limits	Acceptance limits	Warning limits	Acceptance limits
Labeled Compounds										
$^{13}C_{12}$-2-MoCB	1L	100	65-135	50-150	50	20-135	15-140	15-140	15-130	15-150
$^{13}C_{12}$-4-MoCB	3L	100	65-135	50-150	50	20-135	15-140	15-140	15-130	15-150
$^{13}C_{12}$-2,2'-DiCB	4L	100	65-135	50-150	50	35-135	30-140	30-140	25-130	25-150
$^{13}C_{12}$-4,4'-DiCB	15L	100	65-135	50-150	50	35-135	30-140	30-140	25-130	25-150
$^{13}C_{12}$-2,2',6-TrCB	19L	100	65-135	50-150	50	35-135	30-140	30-140	30-130	25-150
$^{13}C_{12}$-3,4,4'-TrCB	37L	100	65-135	50-150	50	35-135	30-140	30-140	30-130	25-150
$^{13}C_{12}$-2,2',6,6'-TeCB	54L	100	65-135	50-150	50	35-135	30-140	30-140	30-130	25-150
$^{13}C_{12}$-3,3',4,4'-TCB	77L	100	65-135	50-150	50	35-135	30-140	30-140	30-130	25-150
$^{13}C_{12}$-3,4,4',5-TeCB	81L	100	65-135	50-150	50	35-135	30-140	30-140	30-130	25-150
$^{13}C_{12}$-2,2',4,6,6'-PeCB	104L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,3,3',4,4'-PeCB	105L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,3,4,4',5-PeCB	114L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,3',4,4',5-PeCB	118L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2',3,4,4',5-PeCB	123L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-3,3',4,4',5-PeCB	126L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,2',4,4',6,6'-HxCB	155L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,3,3',4,4',5-HxCB [3]	156L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,3,3',4,4',5'-HxCB [3]	157L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,3',4,4',5,5'-HxCB	167L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-3,3',4,4',5,5'-HxCB	169L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,2',3,3',4,4',5-HpCB	170L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,2',3,4,4',5,5'-HpCB	180L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB	188L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB	189L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB	202L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB	205L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB	206L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-NoCB	208L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-DeCB	209L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
Cleanup Standards										
$^{13}C_{12}$-2,4,4'-TriCB	28L	100		60-130	45	45-120	40-125	40-125	40-130	30-135
$^{13}C_{12}$-2,3,3',5,5'-PeCB	111L	100		60-130	45	45-120	40-125	40-125	40-130	30-135
$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB	178L	100		60-130	45	45-120	40-125	40-125	40-130	30-135

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Table 4b. QC Acceptance Criteria according to EPA method 1668C for Chlorinated Biphenyls in CAL/VER, IPR, OPR, and Samples

Congener	Cong. No.[2]	Test conc. ng/mL	CAL/VER (%)		IPR[1] (%)		OPR[1] (%)		Labelled compound[1] % recovery in samples	
			Warning limits	Acceptance limits	RSD	$\overline{X}$	Warning limits	Acceptance limits	Warning limits	Acceptance limits
2-MoCB	1	50	75-125	75-125	25	70-130	70-130	60-135	-	-
4-MoCB	3	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2'-DiCB	4	50	75-125	75-125	25	70-130	70-130	60-135	-	-
4,4'-DiCB	15	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2'6-TrCB	19	50	75-125	75-125	25	70-130	70-130	60-135	-	-
3,4,4'-TrCB	37	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2'6,6'TeCB	54	50	75-125	75-125	25	70-130	70-130	60-135	-	-
3,3',4,4'-TeCB	77	50	75-125	75-125	25	70-130	70-130	60-135	-	-
3,4,4',5-TeCB	81	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',4,6,6'-PeCB	104	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,3',4,4'-PeCB	105	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,4,4',5-PeCB	114	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3',4,4',5-PeCB	118	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2',3,4,4',5-PeCB	123	50	75-125	75-125	25	70-130	70-130	60-135	-	-
3,3',4,4',5-PeCB	126	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',4,4',6,6'-HxCB	155	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,3',4,4',5-HxCB[3]	156	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,3',4,4',5'-HxCB[3]	157	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3',4,4',5,5'-HxCB	167	50	75-125	75-125	25	70-130	70-130	60-135	-	-
3,3',4,4',5,5'-HxCB	169	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',3,4',5,6,6'-HpCB	188	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,3',4,4',5,5'-HpCB	189	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',3,3',5,5',6,6'-OcCB	202	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,3',4,4',5,5',6-OcCB	205	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',3,3',4,4',5,5',6-NoCB	206	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',3,3,'4,5,5',6,6'-NoCB	208	50	75-125	75-125	25	70-130	70-130	60-135	-	-
DeCB	209	50	75-125	75-125	25	70-130	70-130	60-135	-	-

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Congener	Cong. No.[2]	Test conc. ng/mL	CAL/VER (%)		IPR[1] (%)		OPR[1] (%)		Labelled compound[1] % recovery in samples	
			Warning limits	Acceptance limits	RSD	$\bar{X}$	Warning limits	Acceptance limits	Warning limits	Acceptance limits
Labeled Compounds										
$^{13}C_{12}$-2-MoCB	1L	100	65-135	50-145	70	20-135	15-140	15-145	15-130	5-145
$^{13}C_{12}$-4-MoCB	3L	100	65-135	50-145	70	20-135	15-140	15-145	15-130	5-145
$^{13}C_{12}$-2,2'-DiCB	4L	100	65-135	50-145	70	20-135	30-140	15-145	25-130	5-145
$^{13}C_{12}$-4,4'-DiCB	15L	100	65-135	50-145	70	20-135	30-140	15-145	25-130	5-145
$^{13}C_{12}$-2,2',6-TrCB	19L	100	65-135	50-145	70	20-135	30-140	15-145	30-130	5-145
$^{13}C_{12}$-3,4,4'-TrCB	37L	100	65-135	50-145	70	20-135	30-140	15-145	30-130	5-145
$^{13}C_{12}$-2,2',6,6'-TeCB	54L	100	65-135	50-145	70	20-135	30-140	15-145	30-130	5-145
$^{13}C_{12}$-3,3',4,4'-TCB	77L	100	65-135	50-145	50	45-135	30-140	40-145	30-130	10-145
$^{13}C_{12}$-3,4,4',5-TeCB	81L	100	65-135	50-145	50	45-135	30-140	40-145	30-130	10-145
$^{13}C_{12}$-2,2',4,6,6'-PeCB	104L	100	65-135	50-145	50	45-135	30-140	40-145	40-130	10-145
$^{13}C_{12}$-2,3,3',4,4'-PeCB	105L	100	65-135	50-145	50	45-135	30-140	40-145	40-130	10-145
$^{13}C_{12}$-2,3,4,4',5-PeCB	114L	100	65-135	50-145	50	45-135	30-140	40-145	40-130	10-145
$^{13}C_{12}$-2,3',4,4',5-PeCB	118L	100	65-135	50-145	50	45-135	30-140	40-145	40-130	10-145
$^{13}C_{12}$-2',3,4,4',5-PeCB	123L	100	65-135	50-145	50	45-135	30-140	40-145	40-130	10-145
$^{13}C_{12}$-3,3',4,4',5-PeCB	126L	100	65-135	50-145	50	45-135	30-140	40-145	40-130	10-145
$^{13}C_{12}$-2,2',4,4',6,6'-HxCB	155L	100	65-135	50-145	50	45-135	30-140	40-145	40-130	10-145
$^{13}C_{12}$-2,3,3',4,4',5-HxCB [3]	156L	100	65-135	50-145	50	45-135	30-140	40-145	40-130	10-145
$^{13}C_{12}$-2,3,3',4,4',5'-HxCB [3]	157L	100	65-135	50-145	50	45-135	30-140	40-145	40-130	10-145
$^{13}C_{12}$-2,3',4,4',5,5'-HxCB	167L	100	65-135	50-145	50	45-135	30-140	40-145	40-130	10-145
$^{13}C_{12}$-3,3',4,4',5,5'-HxCB	169L	100	65-135	50-145	50	45-135	30-140	40-145	40-130	10-145
$^{13}C_{12}$-2,2',3,3',4,4',5-HpCB	170L	100	65-135	50-145	50	45-135	30-140	40-145	40-130	10-145
$^{13}C_{12}$-2,2',3,4,4',5,5'-HpCB	180L	100	65-135	50-145	50	45-135	30-140	40-145	40-130	10-145
$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB	188L	100	65-135	50-145	50	45-135	30-140	40-145	40-130	10-145
$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB	189L	100	65-135	50-145	50	45-135	30-140	40-145	40-130	10-145
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB	202L	100	65-135	50-145	50	45-135	30-140	40-145	40-130	10-145
$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB	205L	100	65-135	50-145	50	45-135	30-140	40-145	40-130	10-145
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB	206L	100	65-135	50-145	50	45-135	30-140	40-145	40-130	10-145
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-NoCB	208L	100	65-135	50-145	50	45-135	30-140	40-145	40-130	10-145
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-DeCB	209L	100	65-135	50-145	50	45-135	30-140	40-145	40-130	10-145
Cleanup Standards										
$^{13}C_{12}$-2,4,4'-TriCB	28L	100		65-135	70	20-135	40-125	15-145	40-130	5-145
$^{13}C_{12}$-2,3,3',5,5'-PeCB	111L	100		75-125	50	45-135	40-125	40-145	40-130	10-145
$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB	178L	100		75-125	50	45-135	40-125	40-145	40-130	10-145

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Footnotes to Table 4a and Table 4b:

[1] QC acceptance criteria for IPR, OPR, and samples based on a 20 µL extract final volume
[2] Suffix "L" indicates labelled compound.
[3] PCBs 156 and 157 are tested as the sum of two concentrations

Table 4c. Instrumental Acceptance Criteria

QC Parameter	Specification
Analysis Duplicate	Must agree to within ±20% of the mean (applicable to concentrations >10 times the DL)[1]
Procedural Blank	Analyte concentrations in blank samples for PCB congeners 77, 81, 114, 123, 126 and 169 must be less than 2 pg/congener/sample, and concentrations of PCB congeners 156, 157, 167 and 189 must be less than 10 pg/congener/sample. Concentrations of all other individual PCB congeners or coelutions must be less than 50 pg/congener/sample in blank samples. The sum of all 209 congeners should be less than 300 pg/sample. Higher levels are acceptable where sample concentrations exceed 10 times the blank levels.
Sample Specific Detection Limit	Typical sample specific detection limits, determined from chromatographic noise, are in the range of 0.5 to 2.0 pg.
Initial Calibration	For 6- or 7-point calibration, a relative standard deviation of the RRF's ≤20% for all compounds. Ion ratios for all congeners must be within ±15% of theoretical for CS 0.2. (See Section 7.1.1 for details). Minimum S:N ratio 10:1 for all calibration standards. For CS-0.2, S:N ratio may be as low as 3:1 for di-PCBs and nona-PCBs. If the optional CS-6 calibration point fails, calibration may still be performed using the default CS-0.2 through CS-5 calibration range.
Continuing CAL VER	Refer to Tables 4a and 4b above.
Analyte/Surrogate Ratios	Response must be within the calibrated range of the instrument. Coders may use data from more than one chromatogram to get the responses in the calibrated range.
Ion Ratios	Ion ratios must fall within ±15% of the theoretical values for positive identification of all targets in the calibration standards and samples.
Sensitivity	Minimum S:N ratio 10:1 for all calibration standards. For CS0.2, S:N ratio may be as low as 3:1. for di-PCBs and nona-PCBs.

1. Duplicate criterion is a guideline; final assessment depends upon sample characteristics, overall batch QC and on-going lab performance.

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5. EXTRACTION PROCEDURES

The extraction procedures described in this section are used for the analyses of samples by EPA Method 1668A or 1668C.

An analysis batch is assigned to an analyst. The samples to be analyzed, the batch QC samples, the name and volume of the surrogate, cleanup, recovery and authentic standards required, and any additional information concerning the analysis are documented on a Batch List given to an analyst. Refer to SOP SLA-033 for details of assigning analysis batches.

Each analyst performs the extraction procedure according to the following written extraction procedures and completes an analysis worksheet (refer to table of available worksheets) for each sample during the sample extraction. The analyst is referred to the following Standard Operating Procedures for details of routine laboratory techniques.

Q-Pulse ID	Title
SLA-003	Sample Receipt and Login
SLA-004	Sample Control Procedures
SLA-005	Use and Maintenance of Rotary Evaporation Equipment
SLA-006	Nitrogen Blowdown Concentration Technique
SLA-008	Preparing Extracts for Instrumental Analysis
SLA-017	Spiking Procedures
SLA-020	Gravimetric Lipid Determination by Weight of Extract
SLA-023	Use of Balances
SLA-027	Completing a Worksheet
SLA-028	Independent Gravimetric Determination of Lipid Content
SLA-030	Micro Lipid Determination for Human Blood Analysis
SLA-033	Procedures for Making an Analysis Batch
SLA-045	Removal of Sulphur From Extracts Using Activated Copper
SLA-067	Use of Nitrogen System
SLA-072	Computer Preparation of Labels
SLA-078	Spike Witness Program
SLA-079	Agricultural Hazard Protocols for Soils
SLA-081	Labelling Protocols and Sample Extract Transfer Procedures
SLA-082	Handling of Human Biohazardous Samples
SLA-084	Preparation of Aqueous Samples for Extraction
SLA-085	Subsampling Procedures for Solids and Tissues
SLA-087	Transferring an Ampouled Standard to a Reacti-Vial
SLA-092	Determination of Suspended Solids Content in Aqueous Samples
SLA-095	Use and Maintenance of the Syncore Analyst Evaporator
SLA-124	Liquid-Liquid Extraction Supplemental Techniques

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The following table lists the worksheets used for sample extraction and cleanup.

Number	Name
FWO-305	Sample Labeling Information
FWO-306	Sample Weight and Moisture
FWO-307	Aqueous Samples
FWO-308	Lipid Sheet
FWO-309	Standards Spiking into C
FWO-310	Sample Spiking Sheet
FWO-311	Extract Splitting
FWO-312	Cleanup Information
FWO-314	Notes
FWO-316	Suspended Solids
FWO-317	Extraction – Blood, Milk, Nonylphenols
FWO-318	Extraction – Sample Trains, Resin Acids, Multi-Residue Pesticides
FWO-319	Extraction – Ash, Acid extractable Pesticides
FWO-328	Water Filtration Processing Worksheet
FWO-330	Rotovap Record Sheet
FWO-331	Dean-Stark Extraction Worksheet

5.0.1 Extract Handling Procedures

To minimize the loss of the most volatile PCBs, sample extracts should be handled in the following manner:

a) Extracts or column eluates that are 10 mL or larger must be covered with clean aluminum foil for overnight storage at room temperature.
b) Extracts, at volumes <10 mL must be stored at 4°C (refrigerator or cooler) in centrifuge tubes that are capped and covered with foil.

5.0.2 Extract Evaporation Procedures

A Syncore Analyst evaporator may be used to concentrate extracts, as described in standard operating procedure SLA-095, instead of a rotary evaporator or Kuderna-Danish concentrator.

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5.1 Soxhlet Extraction of Solids (Default Procedure)

The solid Soxhlet extraction in dichloromethane, described below, allows for the determination of all 209 PCB congeners. The extraction procedure is applicable to the matrices and sample size listed below. Alternately, solids may be extracted by a Soxhlet/Dean-Stark procedure (Section 5.2)

Matrix	Sample Size
Sediments/Soil/Sludge	10 g dry weight

The size of the Soxhlet apparatus used for the extraction of sediment, soil and sludge samples is dependent upon the amount of sample used to obtain the equivalent of 10 g dry weight. Use a larger Soxhlet apparatus for sample sizes larger than 25 g wet or with greater than 60% moisture content. Adjust the amount of silica and solvent volumes accordingly.

5.1.1 Sample Preparation

Accurately weigh to a minimum of 3 significant figures, a wet sample (an amount equivalent to 10 g dry) into a beaker and add anhydrous sodium sulphate (75-100 g) to the sample. Mix well by stirring with a clean spatula, cover with clean aluminum foil and allow the mixture to dry to a free flowing powder (30 minutes minimum).

5.1.2 Soxhlet Extraction Procedure

Assemble the Soxhlet apparatus, including the thimble, and thoroughly clean by Soxhlet extraction with dichloromethane for two hours; three hours if brominated analytes are determined, or three hours with toluene if PCBs are determined according to EPA method CBC01.2. Discard the dichloromethane. Rinse the apparatus with dichloromethane.

Place a layer of clean silica or granular anhydrous sodium sulphate into a pre-cleaned Soxhlet thimble (2 g for small thimbles, 5 g for large thimbles). Transfer the dried sample to the Soxhlet thimble. Add an aliquot of surrogate standard solution to the sample and allow to equilibrate for 30 minutes. Place the Soxhlet thimble with sample into the Soxhlet apparatus. Be sure the level of the sample in the thimble is not higher than the siphon arm. Add anti-bumping granules (4-5 granules) and dichloromethane (300 mL for a small Soxhlet apparatus, 600 mL for a large) to the Soxhlet apparatus' round bottom flask. Heat the sample under reflux for 16-20 hours; adjust as necessary to achieve a reflux rate of a minimum of 4 cycles per hour. Cool the solution. Add an aliquot of cleanup standard to the extract. Concentrate the extract to 1 mL by rotary evaporation. The extract is ready for chromatographic cleanup procedures (Section 6).

5.1.3 Biosolids

In the case of biosolid samples, an optional acid wash may be carried out and a gel permeation chromatographic cleanup procedure will routinely be carried out prior to cleanup procedures. Refer to Section 5.2.3 of this method for details.

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5.2 Soxhlet/Dean-Stark Extraction of Solids (Optional Procedure)

The Soxhlet/Dean-Stark extraction in toluene allows for the determination of all PCB congeners except PCB 1 through PCB 15. The occasional formation of an interference during the extraction procedure and the high boiling point of the toluene limit the quantification of these PCB congeners. The Soxhlet/Dean-Stark extraction procedure is applicable to solid samples such as sediments or soils that are >1% solid or particulate filter papers that are received wet.

Matrix	Sample Size
Sediments/Soil/Sludge	10 g dry weight.
Particulate Filter Paper	1 - several

The size of the Soxhlet apparatus used for the extraction of sediment, soil and sludge samples is dependent upon the amount of sample used to obtain the equivalent of 10 g dry weight. Use a larger Soxhlet apparatus for sample sizes larger than 25 g wet or with greater than 60% moisture content. Adjust the amounts of reagent sand, silica and solvent volumes accordingly.

Assemble the Soxhlet/Dean-Stark (SDS) apparatus, including the thimble, and thoroughly clean by Soxhlet extraction with dichloromethane for two hours (three hours if brominated analytes are required, or three hours with toluene if PCBs are determined according to EPA method CBC01.2). Discard the dichloromethane. Rinse the apparatus with dichloromethane.

5.2.1 Sample Preparation

Solids

Accurately weigh to a minimum of 3 significant figures, a solid sample (>1% solids), equivalent to 10 g dry, into a clean beaker. Add an aliquot of surrogate standard solution. Stir well with a clean spatula, cover with clean aluminum foil, and allow to equilibrate for 30 minutes. Add clean reagent sand (50 g) to the sample in the beaker, after equilibration with the surrogate standard solution. Mix well with a clean spatula

Add a layer of silica or granular anhydrous sodium sulphate (2 g for small thimbles, 5 g for large thimbles) to a pre-cleaned Soxhlet thimble. Transfer the sample to the Soxhlet thimble. Rinse the beaker and spatula with the extraction solvent. Add the rinses to the Soxhlet body.

Particulate Filter Paper

Handle the wet particulate filter paper(s) with clean solvent-rinsed forceps. Fold the filter paper(s) to fit into a Soxhlet thimble for extraction. Using clean forceps place the filter(s) into a pre-cleaned Soxhlet thimble that has a layer of neutral silica or granular anhydrous sodium sulphate (2 g for small thimbles, 5 g for large thimbles) in the bottom. Add an aliquot of surrogate standard to the sample and allow to equilibrate for 30 minutes. Refer to the Bach List for spiking and extract splitting instructions.

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5.2.2 Soxhlet/Dean-Stark Extraction Procedure

Place the thimble containing the sample in a clean SDS apparatus. Be sure that the level of sample in the thimble is not higher than the height of the siphon arm. Add anti-bumping granules (4-5 granules) and toluene (300 mL for a small Soxhlet apparatus, 600 mL for a large) to the Soxhlet apparatus' round bottom flask. Fill the Dean-Stark sidearm with toluene. Heat the sample under reflux for 16-24 hours; adjust as necessary to achieve a reflux rate of 4 cycles per hour. Drain the water from the receiver as necessary. When the extraction has finished remove the distilling flask. Drain the water from the receiver and discard any toluene left in the receiver. Allow the solution to cool. Add an aliquot of cleanup standard to the extract. Concentrate the extract to 1 mL by rotary evaporation. The extract is ready for chromatographic cleanup procedures (Section 6).

5.2.3 Acid Wash

In the case of biosolid samples, an optional acid wash may be carried out prior to cleanup procedures (Section 6 of this method). **Do not carry out the acid wash procedure if the analysis of pesticides is required.**

Acid Wash

The following precautions must be observed when carrying out the acid wash procedures.

1. Ensure the extract is dry by drying with anhydrous sodium sulphate prior to washing procedures.
2. Use only concentrated sulphuric acid for the wash.
3. Ensure the contact time with acid is less than 15 minutes per wash.

Transfer the extract, with rinses, to a 125 mL separatory funnel. Add sufficient hexane to the extract to make the volume 50 mL. Add concentrated sulphuric acid (30 mL) to the extract and gently agitate (invert 5 times). Let stand (no more than 15 minutes). If the aqueous layer is strongly coloured drain the acid and repeat the procedure with up to 3 more washes (with H_2SO_4).

Wash the extract by shaking with ultra pure water, followed by potassium hydroxide (KOH, 20%) followed by ultra pure water. Transfer the organic layer to a clean 250 mL Erlenmeyer flask and dry over anhydrous sodium sulphate (5-10 g, 10-15 minutes). Concentrate the extract to 1 mL by rotary evaporation. The extract is ready for chromatographic cleanup procedures (Section 6).

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5.3 Tissue Extraction Procedure – Soxhlet (Default Procedure)

This extraction procedure is applicable to the matrix and sample size listed below.

Matrix	Sample Size
Tissue	1 - 25 g wet weight

5.3.1 Sample Preparation

Accurately weigh, to a minimum of 3 significant figures, a wet homogenized tissue sample into a beaker and add anhydrous sodium sulphate (75 g). Refer to the Batch List for the sample size. Typical sample sizes are: muscle: 10 g, liver: 5 g, fat: 1 g. Stir the mixture well with a spatula and allow the mixture to dry to a free flowing powder (30 minutes minimum).

5.3.2 Soxhlet Extraction

Assemble the Soxhlet apparatus, including a sintered glass Soxhlet thimble, and thoroughly clean by Soxhlet extraction with dichloromethane for two hours (three hours if brominated analytes are required, or three hours with toluene if PCBs are determined according to EPA method CBC01.2). Discard the dichloromethane. Rinse the apparatus with dichloromethane.

Place a layer of clean silica or granular anhydrous sodium sulphate into a pre-cleaned Soxhlet thimble (2 g for small thimbles, 5 g for large thimbles). Quantitatively transfer the dried sample to the pre-Soxhleted thimble. Add an aliquot of the surrogate standard solution to the sample in the thimble and allow to equilibrate for 30 minutes. Place the Soxhlet thimble in the Soxhlet apparatus. Be sure that the level of sample in the thimble is not higher than the height of the siphon arm. Add anti-bumping granules (4-5 granules) and dichloromethane (300 mL for a small Soxhlet apparatus, 600 mL for a large) to the Soxhlet apparatus' round bottom flask. Allow the sample to reflux for 16 to 20 hours; adjust as necessary to achieve a reflux rate of a minimum of 4 cycles per hour. Allow the mixture to cool. Add an aliquot of cleanup surrogate standard.

Refer to the Batch List to determine whether a lipid determination is required. If so, carry out the lipid determination according to SLA-020 "Gravimetric Lipid Determination by Weight of Extract".

If lipid analysis is not performed, concentrate the extract by rotary evaporation to about 1 mL and add hexane (1 mL) to the extract.

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5.3.3 Gel Permeation Cleanup

Load the extract onto a Biobead SX-3 gel permeation column with 1:1 dichloromethane:hexane and elute with 1:1 dichloromethane:hexane at 5 mL/min. Refer to the most recent Biobead cutpoint determination for the elution volume. Typical cutpoints are as follows:

F1	0 - 140 mL
F2	140 - 300 mL

Discard the first fraction. Collect the second fraction and evaporate to a small volume by rotary evaporation. If the lipid content for the sample is greater than 2 g absolute (i.e. 10% of a 20 g sample), repeat the gel permeation cleanup procedure. The extract is ready for chromatographic cleanup procedures (Section 6).

Note: After Biobead cleanup the extract must be solvent exchanged to hexane before proceeding to other column cleanup procedures.

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5.4 Tissue Extraction Procedure - Base Digestion (Optional Procedure)

This extraction procedure is applicable to the matrix and sample size listed below. This method is not suitable if the analysis of pesticides is also required.

Matrix	Sample Size
Tissue	75 - 150 g wet weight.

5.4.1 Sample Preparation

Accurately weigh, to a minimum of 3 significant figures, a homogenized tissue sample into an Erlenmeyer flask (500 mL). Refer to the Batch List for sample size. Add an aliquot of surrogate standard solution to the sample and allow to equilibrate for 30 minutes. Add potassium hydroxide (KOH, 200 mL, 1M in ethanol) to the sample. Swirl, cover the flask, and **STIR SLOWLY** with a magnetic stir bar overnight. The tissue sample must be entirely dissolved. Alternatively, allow the mixture to stand overnight and stir in the morning until the tissue sample is entirely dissolved.

5.4.2 Extraction Procedure

Add ultra pure water, equal in volume to the volume of ethanolic KOH, to the Erlenmeyer flask. Stir well and transfer the ethanolic potassium hydroxide/water mixture into a separatory funnel (1000 mL). Add hexane (150 mL) to the Erlenmeyer, swirl, and then transfer the hexane to the separatory funnel. Shake the separatory funnel vigorously for 2 minutes. Allow the mixture to separate. Drain the bottom aqueous layer into the original 500 mL Erlenmeyer flask and then drain the hexane extract into a clean Erlenmeyer flask (1000 mL). Repeat the extraction of the aqueous layer twice more with hexane (2 x 150 mL). On the final extraction, draw the aqueous layer off, leaving the hexane in the separatory funnel. Add the previous hexane extracts to the separatory funnel.

Rinse the 500 mL Erlenmeyer flask used for the base digest with toluene (20 mL). Use the vortex mixer if necessary to dissolve the residue. Add the toluene to the hexane extract in the separatory funnel. Backwash the hexane extract by shaking with ultra pure water (150 mL) for 30 seconds. Discard the aqueous (lower) layer. Leave the hexane extract in the separatory funnel. Add an aliquot of PCB cleanup standard solution to the hexane extract.

Add concentrated sulphuric acid (H_2SO_4, 100 mL) to the extract and gently shake for two minutes. Let stand (no more than 15 minutes). Discard the lower acid layer. Repeat the sulphuric acid wash (100 mL, conc. H_2SO_4) until the acid layer is colourless. Backwash the extract by shaking with ultra pure water (150 mL) for 30 seconds. Discard the aqueous (lower) layer. Drain the hexane extract into the 1000 mL Erlenmeyer flask and dry over anhydrous sodium sulphate.

Transfer the dried extract to a round bottom flask (500 mL) and concentrate to approximately 1 - 2 mL by rotary evaporation.

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5.4.3 Gel Permeation Cleanup

Load the extract onto a Biobead SX-3 gel permeation column with 1:1 dichloromethane:hexane and elute with 1:1 dichloromethane:hexane at 5 mL/min. Refer to the most recent Biobead cutpoint determination for the elution volume. Typical cutpoints are as follows:

F1	0	- 140 mL
F2	140	- 300 mL

Discard the first fraction. Collect the second fraction and evaporate to a small volume by rotary evaporation. If the lipid content for the sample is greater than 2 g absolute (i.e. 10% of a 20 g sample), repeat the gel permeation cleanup procedure. The extract is ready for chromatographic cleanup procedures (Section 6).

Note: After Biobead cleanup the extract must be solvent exchanged to hexane before proceeding to other column cleanup procedures.

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5.5 Aqueous Sample Extraction

These extraction procedures are suitable for aqueous samples including drinking water, non-potable water, effluents, aqueous sludge and aqueous ash.

Matrix	Sample Size
Aqueous	1 - 4 L

This extraction procedure for aqueous samples depends on the percentage of suspended solid in the sample. A typical sample size is 1 litre.

5.5.1 Sample Preparation

The surrogate standard spiking solution must be prepared prior to sample extraction and in the same laboratory space as where the samples are extracted. Dilute the surrogate stock solution, described in Table 2, in acetone, to make a solution that is 2 ng of each surrogate/mL. Prepare enough solution to spike all the samples in the batch as well as the QC samples. Typically, 50 µL of stock solution is dissolved in 25 mL of acetone in a volumetric flask. Mix the sample well by inverting the flask at least 10 times. A 1 mL aliquot is added to each sample, which is allowed to equilibrate for at least 30 minutes.

Prior to extraction, aqueous samples are homogenized, subsampled and spiked with an aliquot of surrogate standard solution by the analyst. The analyst must refer to SLA-084 “Preparation of Aqueous Samples for Extraction” for complete details of sample homogenization, subsampling, rinsing, surrogate spiking and centrifugation procedures.

The aqueous sample extraction procedure depends on the percentage of suspended solids in the sample. Estimate the percent suspended solids by visual inspection. If in doubt of a reasonably accurate visual solid percent estimation, determine the percent suspended solids on a subsample of the sample according to SLA-092 “Determination of Suspended Solids (SS) in a Sample”.

- Samples with ≤1% suspended solids are neither centrifuged nor filtered prior to extraction (Section 5.5.2).
- Samples with >1% suspended solids are centrifuged to separate the particulate and liquid phases. The particulate and liquid phases are extracted separately (Section 5.5.3).

Two technically equivalent liquid/liquid extraction procedures are described in sections 5.5.2.1/5.5.2.2 and 5.5.3.2/5.5.3.3 for aqueous samples with ≤1% solids and for centrifuged aqueous samples, respectively. The magnetic stirring procedure may offer some advantage in terms of cost efficiency, particularly for larger sample sizes.

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5.5.2 Extraction of Samples with ≤1% Solids

5.5.2.1 Extraction by Magnetic Stirring (Default Procedure)

After homogenization, subsampling and spiking with surrogate standard, as described in SLA-084, add dichloromethane (300 mL) and a pre-cleaned PTFE magnetic stir bar to the sample in an Erlenmeyer flask and extract by stirring the solution (with vortex) for a minimum of 30 minutes. Quantitatively transfer the solution to a separatory funnel and draw off the dichloromethane layer. Discard the aqueous layer. Dry the extract by filtration into a round-bottom flask through a solvent rinsed glass funnel with a glass wool plug and approximately half full of granular anhydrous sodium sulphate. Rinse the filter with dichloromethane to ensure quantitative transfer. Add an aliquot of cleanup standard solution to the extract. Concentrate the extract to 1-2 mL by rotary evaporation. The extract is ready for chromatographic cleanup procedures (Section 6).

Alternate procedure after drying over anhydrous sulphate: Transfer the extract to a Kuderna-Danish flask (500 mL) with dichloromethane. Spike an aliquot of cleanup standard solution. Add hexane (2 mL) and concentrate the extract to 2 mL (water bath 55°C). The extract is ready for chromatographic cleanup procedures (Section 6).

5.5.2.2 Extraction by Shaking in a Separatory Funnel (Optional Procedure)

After homogenization, subsampling and spiking with surrogate standard, as described in SLA-084, extract the sample by adding dichloromethane (100 mL or 10% of the volume of the sample) to the separatory funnel and shaking vigorously for two minutes. Collect the dichloromethane layer in an Erlenmeyer flask. Repeat the extraction twice more. Combine the dichloromethane layers. Dry the extract by filtration into a round-bottom flask through a solvent rinsed glass funnel with a glass wool plug and approximately half full of granular anhydrous sodium sulphate. Rinse the filter with dichloromethane to ensure quantitative transfer. Add an aliquot of cleanup standard solution to the extract. Concentrate the extract to 1-2 mL by rotary evaporation. The extract is ready for chromatographic cleanup procedures (Section 6).

Alternate procedure after drying over anhydrous sulphate: Transfer the extract to a Kuderna-Danish flask (500 mL) with dichloromethane. Spike an aliquot of cleanup standard solution. Add hexane (2 mL) and concentrate the extract to 2 mL (water bath 55°C). The extract is ready for chromatographic cleanup procedures (Section 6).

5.5.3 Extraction of Samples with >1% Solids

The analyst must refer to the following operating procedures SLA-084 for complete details of sample homogenization, subsampling, rinsing, surrogate spiking and centrifugation procedures. The procedures are summarized below:

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5.5.3.1 Default Centrifugation Procedure for Separation of Particulate

Transfer a portion of the sample to a clean 500 mL stainless steel vessel with a clean baked 10 cm filter paper (Pall, type A/E, glass fibre, 1.0 µm) in the bottom of the jar to assist with capturing of the solids. Spin the sample in the centrifuge at 1500 rpm until the solid has settled. Decant the supernatant into a separatory funnel (if proceeding according to Section 5.5.3.3) or an Erlenmeyer flask (if proceeding according to Section 5.5.3.2). Repeat the procedure until the entire sample has been centrifuged, leaving the filter paper in place throughout the process. Once the entire sample has been processed, dry the particulate and filter in the jar by mixing with granular anhydrous sodium sulphate. Extract the solids as described for particulate in Section 5.5.3.4.

Rinse the jar with ultra pure water and dichloromethane. Add the rinses to the separatory funnel/Erlenmeyer flask containing the supernatant. Extract the supernatant as described in Section 5.5.3.2 or 5.5.3.3.

5.5.3.2 Extraction of the Centrifugate by Magnetic Stirring (Default Procedure)

Quantitatively transfer the centrifugate to an Erlenmeyer flask. Add dichloromethane (300 mL) and a pre-cleaned PTFE magnetic stir bar and extract by stirring the solution (with vortex) for a minimum of 30 minutes. Quantitatively transfer the solution to a separatory funnel and draw off the dichloromethane layer. Discard the aqueous layer. Dry the extract by filtration into a round-bottom flask through a solvent rinsed glass funnel with a glass wool plug and approximately half full of granular anhydrous sodium sulphate.

5.5.3.3 Extraction of the Centrifugate by Shaking in a Separatory Funnel (Optional Procedure)

Quantitatively transfer the centrifugate to a separatory funnel. Extract the centrifugate by adding dichloromethane (100 mL or 10% the volume of sample) to the separatory funnel and shaking vigorously for two minutes. Collect the dichloromethane layer in an Erlenmeyer flask. Repeat the extraction twice more. Combine the dichloromethane layers. Dry the extract by filtration into a round-bottom flask through a solvent rinsed glass funnel with a glass wool plug and approximately half full of granular anhydrous sodium sulphate. Rinse the filter with dichloromethane to ensure quantitative transfer.

Should problems be encountered for liquid-liquid extraction of complex samples consult AXYS SOP SLA-124 *Liquid-Liquid Extraction Supplemental Techniques*.

5.5.3.4 Extraction of Particulate

Assemble the Soxhlet apparatus, including the thimble, and thoroughly clean by Soxhlet extraction with dichloromethane for two hours (3 hours if brominated analytes are required, or three hours with toluene if PCBs are determined according to EPA method CBC01.2). Discard the dichloromethane. Rinse the apparatus with dichloromethane.

Place a layer of silica or granular anhydrous sodium sulphate (2 g for small thimbles, 5 g

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for large thimbles) into a pre-cleaned Soxhlet thimble. Transfer the dried particulate with dichloromethane rinses (use extraction solvent) to the thimble. Be sure that the level of sample in the thimble is not higher than the height of the siphon arm. Add the rinses to the thimble in the Soxhlet body. Place the Soxhlet thimble in the Soxhlet apparatus. Add anti-bumping granules (4-5 granules) and dichloromethane (300 mL for a small Soxhlet apparatus, 600 mL for a large) to the Soxhlet apparatus' round bottom flask (use the round bottom flask containing the filtrate extract if option 1 was chosen). Heat the sample under reflux for 16 - 20 hours; adjust as necessary to achieve a reflux rate of a minimum of 4 cycles per hour. Allow the solution to cool.

5.5.3.5 Combining Extracts

Combine the extract from the centrifugate with the extract from the particulate, using dichloromethane rinses to transfer the extract. Add an aliquot of cleanup standard to the combined extract. Transfer the extract to a round-bottom flask (500 mL) with dichloromethane. Concentrate to 1 mL by rotary evaporation. The extract is ready for chromatographic cleanup procedures (Section 6)

Alternately, after the addition of the cleanup standard transfer the extract into a Kuderna-Danish flask. Add hexane (2 mL) and concentrate the extract to 2 mL (water bath 55°C). The extract is ready for chromatographic cleanup procedures (Section 6).

Alternate Procedure for Combining Extracts

If time allows, quantitatively transfer the centrifugate extract with dichloromethane rinses to a round bottom flask. Use this flask for the Soxhlet extraction of the particulate as described in Section 5.5.3.4.

5.5.4 Cleanup of Extracts from Influent and Effluent samples

Extracts from samples with INFL and EPRO as the matrix require a cleanup on a gel permeation column (Section 6.1) prior to the other cleanup procedures described in Section 6. This is an optional step for samples with EFIN as the matrix..

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5.6 XAD-2 Column (Resin and Filter) Extraction

This procedure is applicable to the analysis of an XAD-2 column. The analysis of a XAD-2 column usually consists of two analyses, as the filter(s) and XAD-2 resin are extracted and analyzed separately. Upon client request, the filter and resin extracts may be combined to form a single extract. If the sample is also analyzed for PCDD/F, a Soxhlet/Dean-Stark extraction of the XAD-2 resin must be carried out, as described in Section 5.6.4.

The occasional formation of an interference during the extraction procedure and the high boiling point of the toluene may limit the quantification of PCBs 1 through 15 in the filter/XAD-2 matrix.

5.6.1 Sample Handling Procedures

Refer to document SLA-043 for details of sample handling procedures. If the sample consists of a large amount of XAD-2 resin or multiple filters, it may be necessary to use more than one Soxhlet apparatus to extract the sample. Distribute the XAD-2 resin or filters evenly amongst the required number of Soxhlet apparatuses. Spike the sample in multiple Soxhlet apparatuses with surrogate standard as follows:

1. Dilute the aliquot of surrogate standard in a centrifuge tube with acetone (5 mL). Ensure the solution is homogeneous by using a disposable pipette to withdraw and expel the solution several times.
2. Use a disposable pipette to distribute the surrogate solution evenly amongst the Soxhlet apparatuses.
3. Rinse the centrifuge tube with acetone (5 mL). Mix the rinsate in the centrifuge tube by vortex mixing. Distribute the rinsate evenly amongst the Soxhlet apparatuses. Repeat the rinse step once more with another 5 mL acetone.
4. Once the extraction is complete, combine the extracts.

5.6.2 XAD-2 Resin – Drying Procedure

1. Place a pre-cleaned Soxhlet thimble into a beaker.
2. Quantitatively transfer the XAD-2 resin to the pre-cleaned thimble with ultra pure water and allow to drain.
3. Rinse the resin with ultra pure water and allow to drain. Discard the water.

5.6.3 XAD-2 Resin Soxhlet Extraction Procedure (Default Procedure - **only** if PCDD/F is not determined in the same extract)

1. Assemble a Soxhlet apparatus. Add a small bed of glass wool (weighted down with the pre-cleaned thimble) to the bottom of the Soxhlet body to prevent clogging during the reflux procedure. Thoroughly clean the apparatus by Soxhlet extraction with dichloromethane for two hours (three hours if brominated analytes are required). Discard the dichloromethane. Rinse

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the apparatus with dichloromethane.

2. Transfer the pre-cleaned thimble containing the XAD-2 resin to the Soxhlet body. Be sure that the level of XAD-2 resin is not higher than the height of the siphon arm.
3. Add the appropriate volume of dichloromethane (300 mL for a small Soxhlet apparatus, 600 mL for a large) and anti-bumping granules (4-5 granules) to the round bottom flask of the Soxhlet apparatus.
4. If a single analysis is required, spike an aliquot of the surrogate solution into a centrifuge tube containing acetone (1 mL). Transfer the surrogate standard, along with acetone rinses of the centrifuge tube, onto the XAD-2 resin in the thimble prior to starting the Soxhlet extraction. If multiple analyses are required, spike the surrogates after the extraction and extract splitting procedures (Section 5.12). Refer to the Batch List for spiking instructions.
5. Heat the sample under reflux for 16 -20 hours; adjust as necessary to achieve a reflux rate of a minimum of 4 cycles per hour. Allow the solution to cool. Remove water from the extract (Section 5.6.3.1).

EXTREMELY IMPORTANT – DO NOT CONCENTRATE THE SAMPLE BY ROTARY EVAPORATION WITHOUT A WATER REMOVAL STEP.

5.6.3.1 Removal of Water from XAD-2 Resin Extracts

The water removal step depends on the amount of visible water.

1. If less than 3 mL of water is visible, transfer the entire extract to an Erlenmeyer flask.
2. If greater than 3 mL of water is visible, transfer the extract to a separatory funnel and drain the water from the sample extract. Discard the water. Quantitatively transfer the extract to an Erlenmeyer flask.
3. Dry the extract over anhydrous granular sodium sulphate. Return the extract to the round bottom flask with complete dichloromethane rinses. An aliquot of cleanup surrogate standard may be added at this point or after the extract has been split. Refer to the Batch List for spiking and extract splitting instructions. Concentrate the extract by rotary evaporation to 1 - 2 mL for extract splitting. If using a 1000 mL round bottom flask for the Soxhlet extraction, transfer the extract with dichloromethane rinses to a 500 mL round bottom flask during the evaporation process. The extract is ready to be gravimetrically split according to procedures in Section 5.12 of this method.

5.6.4 Soxhlet/Dean-Stark Extraction of XAD-2 Resin (Default Procedure **when** PCDD/F is determined in the same extract)

This procedure is used only if PCDD/F analysis is required as well as PCB analysis.

1. Assemble a Soxhlet apparatus fitted with a Dean-Stark adapter. Place some clean glass wool into the bottom of the siphon arm. Clean the apparatus by Soxhlet extraction with dichloromethane for two hours (3 hours if brominated analytes are required). Discard the

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dichloromethane. Rinse the apparatus with dichloromethane.

2. Transfer the pre-cleaned thimble containing the XAD-2 resin to the Soxhlet body. Be sure that the level of XAD-2 resin is not higher than the height of the siphon arm.
3. Use a 1000 mL round bottom flask for the solvent.
4. If a single analysis is required, spike the surrogate solution into a centrifuge tube containing acetone (1 mL). Transfer the surrogate solution, along with acetone rinses of the centrifuge tube, onto the resin in the Soxhlet prior to starting the extraction. If multiple analyses are required, refer to the Batch List for spiking details.
5. Add anti-bumping granules (4-5 granules) and toluene (700 mL) to the Soxhlet apparatus' round bottom flask. Fill the sidearm to the top with toluene. Soxhlet extract the resin until the water removal is complete (no water collecting in the sidearm); adjust as necessary to achieve a reflux rate of 4 cycles per hour. When the water extraction has finished, drain the water from the receiver. Continue with the Soxhlet extraction for another 8-12 hours. Allow the solution to cool. The cleanup standard is added at this point or after the extract has been split. Refer to the Batch List for spiking and extract splitting instructions. Concentrate by rotary evaporation to 1-2 mL. The extract is ready to be gravimetrically split according to procedures in Section 5.12.

5.6.5 Filter - Soxhlet/Dean-Stark Extraction

This extraction procedure is applicable to glass fibre filters or filter cartridges (wound glass filters).

1. Assemble a large Soxhlet apparatus, without the thimble, fit with a Dean-Stark adapter and thoroughly clean by Soxhlet extraction with dichloromethane for two hours (three hours if brominated analytes are required). Discard the dichloromethane. Rinse the apparatus with dichloromethane.
2. Place the filters or filter cartridge directly into the pre-cleaned Soxhlet body. Use a large Soxhlet body to hold the filter(s); thimbles are not required. Be sure that the level of filters in the Soxhlet body is not higher than the height of the siphon arm.
3. Use a 1000 mL round bottom flask for the solvent.
4. If a single analysis is required, spike the surrogate solution into a centrifuge tube containing acetone (1 mL). Transfer the surrogate standard along with acetone rinses of the centrifuge tube, onto the filter(s) in the Soxhlet apparatus prior to starting the extraction. If multiple analyses are required, refer to the Batch List for spiking details and extract splitting procedures (Section 5.12).
5. Add anti-bumping granules (4-5 granules) and toluene (700 mL) to the Soxhlet apparatus' round bottom flask. Fill the side arm to the top with toluene. Soxhlet extract the filter(s) or filter cartridge with until the water removal is complete (no water in the sidearm). Adjust as necessary to achieve a reflux rate of a minimum of 4 cycles per hour. When the extraction has finished, drain the water from the receiver. Monitor the amount of water collected, typically 50-150 mL should be present. Contact the Lab Supervisor if the amount of water is anomalous.

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Continue with the Soxhlet extraction for another 8 - 12 hours. Allow the solution to cool. Add the cleanup standard at this point or after the extract has been split. Refer to the Batch List for spiking and extract splitting instructions. Concentrate by rotary evaporation to 1-2 mL. The extract is ready to be gravimetrically split according to procedures in Section 5.12.

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5.7 Air Sampler (PUF/Filter) and Particulate Filter Extraction Procedure

This method is applicable to the analysis of ambient air samples consisting of polyurethane foam plug (PUF) and associated filter(s). The PUF and filter(s) are extracted together as one sample. The method is also applicable to particulate filters received dry.

Assemble the Soxhlet apparatus, including the thimble, and thoroughly clean by Soxhlet extraction with dichloromethane for two hours (three hours if brominated analyses are required). Discard the dichloromethane. Rinse the apparatus with dichloromethane.

5.7.1 Sample Preparation

Particulate Filter Papers

Handle the particulate filter paper(s) with clean solvent-rinsed forceps. Fold the filter paper(s) to fit into a Soxhlet thimble for extraction. Using clean forceps place the filter(s) into a pre-cleaned Soxhlet thimble that has a layer of neutral silica or granular anhydrous sodium sulphate (baked, 2 g for small thimbles, 5 g for large thimbles) in the bottom. Be sure that the level of the filter in the thimble is not higher than the height of the siphon arm.

PUF/Filter

Using clean solvent-rinsed forceps, transfer the PUF and filter(s) to a pre-cleaned Soxhlet thimble that has a layer of neutral silica or granular anhydrous sodium sulphate (baked, 2 g for small thimbles, 5 g for large thimbles) in the bottom. Be sure that the level of the filter in the thimble is not higher than the height of the siphon arm.

5.7.2 Soxhlet Extraction Procedure

Place the Soxhlet thimble containing the PUF and filter(s) in a pre-cleaned Soxhlet apparatus. If a single analysis is required, spike the surrogate standard solution onto the sample prior to starting the extraction and allow to equilibrate for 30 minutes. If multiple analyses are required, refer to the Batch List for surrogate spiking instructions. Add anti-bumping granules (4-5 granules) and dichloromethane (300 mL for a small Soxhlet apparatus, 600 mL for a large) to the Soxhlet apparatus' round bottom flask. Heat the sample under reflux for 16-20 hours; adjust as necessary to achieve a reflux rate of a minimum of 4 cycles per hour. If extract is also being analyzed for PCDD/PCDF compounds, use 80:20 toluene:acetone as solvent. Allow the solution to cool. Ensure that the extract doesn't have any water present. Add and aliquot of cleanup standard to the extract at this point or after the extract has been split. Refer to the Batch list for spiking instructions. Concentrate the extract to 1 - 2 mL by rotary evaporation. The extract is ready to be gravimetrically split according to procedures in Section 5.12.

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5.8 Solvent Extracts

This procedure is suitable for samples submitted as solvent extracts, where only extract splitting and chromatographic cleanup are required prior to instrumental analysis.

5.8.1 Sample Preparation

Determine the weight of the extract as received, as follows:

- Weigh the extract, to a minimum of 3 decimal places, in the original container (usually jar or ampoule).
- Transfer the extract to a centrifuge tube (if received in an ampoule) or to an Erlenmeyer flask with solvent rinses using the same solvent as in the sample.
- Reweigh the original container once it has dried.
- Record all weights on the worksheet.
- Calculate the weight of the extract.

Determine from the Batch List if the sample requires a single analysis or multiple analyses. Check the Batch List for detailed splitting and surrogate spiking instructions.

Single PCB Analysis

If only PCB congener analysis is required, add aliquots of surrogate standard solution and cleanup standard solution to the extract and allow to equilibrate for 30 minutes. Dry the extract according to procedures in Section 5.8.2.

Multiple Analyses

If multiple analyses are required, the surrogate and cleanup standards may be spiked before or after extract drying and splitting procedures. Refer to the Batch List for detailed splitting and surrogate spiking instructions. Dry the extract according to procedures in Section 5.8.2.

5.8.2 Extract Drying Procedures

Inspect the extract for the presence of water.

1. If less than 3 mL of water is visible, transfer the entire extract to an Erlenmeyer flask with solvent rinses (the same solvent as the sample).
2. If greater than 3 mL of water is visible, transfer the extract to a separatory funnel with solvent rinses (the same solvent as the sample) and drain the water from the sample extract. Discard the water. Quantitatively transfer the solvent layer to an Erlenmeyer flask.
3. Dry the extract over anhydrous granular sodium sulphate. Quantitatively transfer the extract to a round bottom flask with solvent rinses. Concentrate the extract by rotary evaporation to 1 - 2 mL for extract splitting. Refer to the Batch List for extract splitting instructions. The extract is ready to be gravimetrically split according to procedures in Section 5.12.

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5.9 Milk Sample Extraction

This extraction procedure is suitable for both human and cow's milk.

1. Accurately weigh to a minimum of 3 significant figures, a subsample of milk (50 g). Add an aliquot of the surrogate standard solution that has been dissolved in ~1 mL acetone and allow to equilibrate for at least 30 minutes.

2. Add the sample to 200 mL of 2:1 acetone:hexane in a 500 mL separatory funnel. Shake the mixture for 2 min and allow the layers to separate.

3. Transfer the aqueous layer to an Erlenmeyer flask and the hexane layer to a separate Erlenmeyer. Return the aqueous layer to the separatory funnel and repeat the extraction with hexane (200 mL). Combine the hexane extracts. Discard the aqueous layer.

4. Return the hexane extracts to the separatory funnel and wash by shaking with ultra pure water (2 x 50 mL). Drain the hexane extract into an Erlenmeyer flask and dry over anhydrous granular sodium sulphate.

5. Using a glass funnel lined with a large fluted filter paper, quantitatively transfer the dried extract to a pre-weighed round bottom flask and concentrate to ~5 mL by rotary evaporation. Add aliquots of the PCB cleanup standards to the extract.

6. **Lipid Determination** - Refer to the Batch List to determine whether a lipid determination is required. If so, carry out the lipid determination according to SLA-020 "Gravimetric Lipid Determination by Weight of Extract".

7. If no lipid analysis is required, concentrate the extract to ~1 mL. Add 1 mL dichloromethane.

8. Load the extract onto a clean Biobead SX-3 column and elute with 1:1 dichloromethane:hexane. Refer to the most recent Biobead cutpoint determination for the elution volume. Discard the first fraction. Collect the second fraction. Concentrate the extract to 1 mL by rotary evaporation followed by evaporation under a gentle stream of nitrogen. The extract is ready for chromatographic cleanup procedure (Section 6).

Note: After Biobead cleanup the extract must be solvent exchanged to hexane before proceeding to other column cleanup procedures.

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5.10 Blood/Serum/Plasma Sample Extraction

This extraction procedure is applicable to the matrix and sample size listed below.

Matrix	Sample Size
Whole Blood/Serum/Plasma	10 - 20 g

Refer to the standard operating procedure SLA-082, *Handling of Human Biohazards*, for details of safety precautions when handling blood samples. Use caution when using bleach to decontaminate equipment used in the analysis of human biohazards and **avoid contact between acid and bleach.** Note that ammonium sulphate, used in the extraction below, is an acidic solution.

1. Accurately weigh a blood/serum/plasma sample (at least 10 g) to a minimum of 3 significant figures into a 250 mL round-bottom flask. Add an aliquot of surrogate standard, cover the flask with clean aluminum foil and allow to equilibrate for 30 minutes.
2. Add ethanol, hexane and saturated ammonium sulphate to the sample (in the proportions described below) and shake for 30 min on the shaker table. Refer to the Batch List for the volume of reagents to use.

   NOTE: The proportion of reagents used is critical to complete extraction of analytes. The minimum sample size required is 10 g and the volumes of reagents are scaled to sample size in a ratio of sample:ethanol:saturated ammonium sulphate:hexane of 1:1:1:3. An alternate procedure for sample sizes less than 10 g, using 10 mL ethanol, 50 mL hexane and 10 mL saturated ammonium may be applied on a custom basis to meet client or historical project requirements - this option requires written pre-approval of the Project Manager including confirmation that the client has been made aware of potential solvent system effects on gravimetric determination of lipids in blood matrices.
3. Decant the hexane layer into a 500 mL separatory funnel.
4. Add additional hexane (100 mL) to the aqueous layer in the round bottom flask and repeat the extraction step. Add the hexane layer to the hexane in the separatory funnel. Discard the aqueous phase.
5. Wash the hexane extracts by shaking with ultra pure water (2 x 50 mL) to remove residual ethanol. Discard the aqueous layer.
6. Transfer the hexane extract to an Erlenmeyer flask and dry over anhydrous sodium sulphate for a minimum of 30 minutes.
7. Add an aliquot of cleanup standard to the extract.
8. Refer to the Batch List to determine whether a lipid determination is required. If so, carry out the lipid determination according to SLA-020 “Gravimetric Lipid Determination by Weight of Extract”.
9. Quantitatively transfer the extract with hexane rinses to a clean round bottom flask. Concentrate the extract by rotary evaporation to about 1 mL. Add dichloromethane (1 mL) to

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the extract.

10. Load the extract onto a Biobeads SX-3 column and elute with 1:1 dichloromethane:hexane. Refer to the most recent Biobead cutpoint determination for the elution volume. Discard the first fraction. Collect the second fraction.
11. Concentrate the extract to a small volume by rotary evaporation to about 1 mL. Transfer to a centrifuge tube with hexane rinses. Concentrate to 1 mL under a stream of nitrogen. The extract is ready for the column cleanup procedure (Section 6).

Note: After Biobead cleanup the extract must be solvent exchanged to hexane before proceeding to other column cleanup procedures.

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5.11 Stationary Source Air Samples (Stack Gas)

5.11.1 Sample Preparation

Stack gas samples are usually submitted as sample trains in several containers labelled A to F or 1 to 6. It is essential that the analyst confirm that the labels on the sample container match those on the sample log-in information prior to commencing extraction procedures.

Prior to proceeding with the sample processing, the analyst must refer to the LIMS and/or Project Notes for information regarding the solvents present in the various containers. A flow chart of the sample processing procedure is presented in Figure 1.

QC Samples

Refer to Section 4.1 for details of preparing the procedural blank and reference (OPR) sample.

Glassware

Assemble the Soxhlet apparatus, including the thimble, and thoroughly clean by Soxhlet extraction with dichloromethane for two hours. Discard the dichloromethane. Rinse the apparatus with dichloromethane.

Front Half of Train

a) Container A (1)

The solvent rinses, that may include dichloromethane, toluene, and/or methanol, from the probe liner, nozzle, cyclone and front half of the filter housing are in container A. **If the rinses contain additional or different solvents, or two or more phases, consult the Supervisor for instructions.**

1. Pour the contents of container A through a Millipore filtration apparatus containing a baked filter and if needed, a glass fibre filter/filter bed. Rinse the container with dichloromethane and filter the rinses.

   CAUTION: To avoid loss of volatile compounds when filtering, DO NOT draw air through the filter. DO NOT use acetone rinses to dry the filter.

2. The filter/filter bed is ready for the acidification/sonication procedure (Section 5.11.1b). (Residual water in the filter is acceptable).

3. If methanol is not present in the filtrate, treat the filtrate as described in Step 5.

4. If methanol is present in the filtrate, transfer the filtrate to a separatory funnel with dichloromethane rinses. Make the volume of dichloromethane in the separatory funnel to 100 mL. Add ultra pure water (about equal in volume to the volume of the filtrate) to the

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separatory funnel. Shake the mixture gently for 10 inversions. Allow the layers to separate. If there is no phase separation, consult the Supervisor for assistance. Corrective action may include the addition of additional water or dichloromethane and another gentle shake of the mixture for 10 inversions. Draw off the organic (bottom) layer into an Erlenmeyer flask. Discard the aqueous (top) layer. Proceed with the dichloromethane extract as described below.

Note: Depending upon the mix of solvent in the containers, the aqueous and organic layers may be inverted from that described above. Ensure that the organic layer is the layer retained for processing.

5. Dry the filtrate (or filtrate extract) over granular anhydrous sodium sulphate (Na_2SO_4) for a minimum of 30 minutes. Transfer the filtrate extract to a round bottom flask (500 mL) with dichloromethane rinses; add toluene (1 mL) as a "keeper" and concentrate to 1 mL by rotary evaporation. Use this round bottom flask for the Soxhlet extraction of the XAD-2 resin (container C) and filters (Section 5.11.2).

b) Container B (2)

A particulate filter is in container B.

1. Transfer the filter to a clean, tared, piece of foil. Weigh the filter and record the weight to a minimum of 3 significant figures on the work sheet. Transfer the filter/filter bed from the processing of container A (Section 5.11.1a) and the particulate filter and all visible particulate from container B to a clean beaker. Add enough hydrochloric acid (1M, HCL) to the beaker to cover the filters and sonicate for 30 minutes. Filter the mixture through a Millipore filtration apparatus and rinse with ultra pure water until the rinsate is at the pH of ultra pure water (typically 6, check with pH paper).

   CAUTION: To avoid loss of volatile compounds when filtering, DO NOT draw air through the filter. DO NOT use acetone rinses to dry the filter.

2. Transfer the wet filters and particulate to the Soxhlet thimble containing the XAD-2 resin from container C (Section 5.11.1c).

3. Combine the filtrate with the contents of container E (impinger water washes) in a separatory funnel (Section 5.11.1e).

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Back Half of Train

c) Container C (3)

XAD-2 resin is in container C.

1. Put a layer of neutral silica or granular anhydrous sodium sulphate (2 g for small thimbles, 5 g for large thimbles) into a pre-cleaned Soxhlet thimble. Place the Soxhlet thimble into a large glass funnel that is set in a beaker (400 mL). Handle the thimble with a clean piece of foil. Remove the glass wool from the top of XAD-2 trap and place into the Soxhlet thimble. Rinse the XAD-2 resin from the container into the Soxhlet thimble with ultra pure water. Place the glass wool from the other end of the trap on top of the resin. Allow the water to drain from the thimble.

2. Transfer the filters and particulate from containers A and B (Section 5.11.1b) to the top of the Soxhlet thimble containing the XAD-2 resin. Be sure that the level of the XAD-2 and filters in the thimble is not higher than the height of the siphon arm.

3. Add an aliquot of the surrogate standard to the Soxhlet thimble. If multiple analyses of the extract are required, refer to the Batch List for the name(s) and volume(s) of surrogate standard solutions to add.

4. Discard the water rinses of the XAD-2 resin.

d) Container D (4)

Solvent rinses, which may contain dichloromethane, toluene, and/or methanol, of the condenser, coil and back half of filter housing, are in container D. **If the rinses contain additional or different solvents, consult the Supervisor for instructions.**

1. If methanol is in the Container D rinses, combine the rinses with the filtrate from Container A, and backwash as described n Step 4 in Section 5.11.1a

2. If methanol is not present, dry the contents of container D with granular anhydrous sodium sulphate for a minimum of 30 minutes. Transfer the extract with dichloromethane to the 500 mL round bottom flask used for the Soxhlet extraction of the XAD-2 and filters (Section 5.11.2). Concentrate to 1 mL by rotary evaporation. (Add 1 mL toluene keeper to the flask if it has not been previously added.)

e) Container E (5)

Impinger contents and water rinses, or perhaps only water are in container E. **If the rinses contain ethylene glycol or solvents other than dichloromethane, toluene or methanol, consult the Supervisor for instructions.**

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1. Transfer the contents of Container E to a separatory funnel. Add the aqueous filtrate from the acidification step (Section 511.1b) to the separatory funnel. Rinse the containers with dichloromethane and add the rinses to the separatory funnel. Extract by shaking with dichloromethane (3 x 100 mL). Combine the extracts, dry with granular anhydrous sodium sulphate for a minimum of 30 minutes. Proceed as described below:

   i) Once the Soxhlet extraction of the XAD-2 resin and filters is complete, concentrate the 80:20 toluene:acetone extract to 1 mL by rotary evaporation. Quantitatively transfer the extract from Container E with dichloromethane rinses to the round bottom flask containing the extract of the XAD-2 and filters. Concentrate the extract to 1 mL by rotary evaporation.

   OR

   ii) If time permits prior to the Soxhlet extraction, quantitatively transfer the dichloromethane extract of Container E with dichloromethane rinses to the round bottom flask to be used for the Soxhlet extraction of the XAD-2 resin and filters. Concentrate the extract to 1 mL by rotary evaporation. Proceed with the Soxhlet extraction (Section 5.11.2)

f) Container F (6) (Final Rinses)

Solvent rinses, that may include dichloromethane, toluene, and/or methanol, of the condenser coil, back half of filter holder and impingers are in container F. The client does not always submit this container. **If the rinses contain additional or different solvents, consult the Supervisor for instructions.**

1. If methanol is in the Container F rinses, combine the rinses with the filtrate from Container A, and backwash as described in Step 4 of Section 5.11.1a.

2. If methanol is not present, dry the contents of Container F with granular anhydrous sodium sulphate for a minimum of 30 minutes. Transfer the extract, using dichloromethane, to the 500 mL round bottom flask used for the Soxhlet extraction of the XAD-2 and filters (Section 5.11.2) and concentrate by rotary evaporation to 1 mL. (Add 1 mL toluene keeper to the flask if it has not been previously added.)

5.11.2 Soxhlet Extraction

1. Add 80:20 toluene:acetone (300 mL) and anti-bumping granules (4-5 granules) to a pre-cleaned 500 mL round bottom flask and extract the XAD-2/filters under reflux for 16-24 hours; adjust as necessary to achieve a reflux rate of 4 cycles per hour. Allow the extract to cool.

2. Remove water from the extract as follows:
   - If less than 3 mL of water is visible, quantitatively transfer the entire extract to an Erlenmeyer flask with toluene rinses.

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- If greater than 3 mL of water is visible, transfer the extract to a separatory funnel and drain the water from the sample extract. Discard the water. Quantitatively transfer the entire extract to an Erlenmeyer flask with toluene rinses.
- Dry the extract with anhydrous granular sodium sulphate for a minimum of 30 minutes. Return the extract to the round bottom flask with complete toluene rinses. An aliquot of cleanup surrogate standard is added at this point or after the extract has been split. Refer to the Batch List for extract splitting instructions. Concentrate by rotary evaporation to 1 mL.

3. If not added prior to the Soxhlet extraction, add the dichloromethane extract from Container E (Section 5.11.1e) to the concentrated XAD-2/Filter extract in the Soxhlet extraction round bottom flask. Concentrate by rotary evaporation to 1 mL.

4. Elute the extract (and rinses of the round bottom flask) through a glass wool column to remove residual sodium sulphate prior to gravimetrically splitting the extract as specified on the Batch List, as described in Section 5.12.

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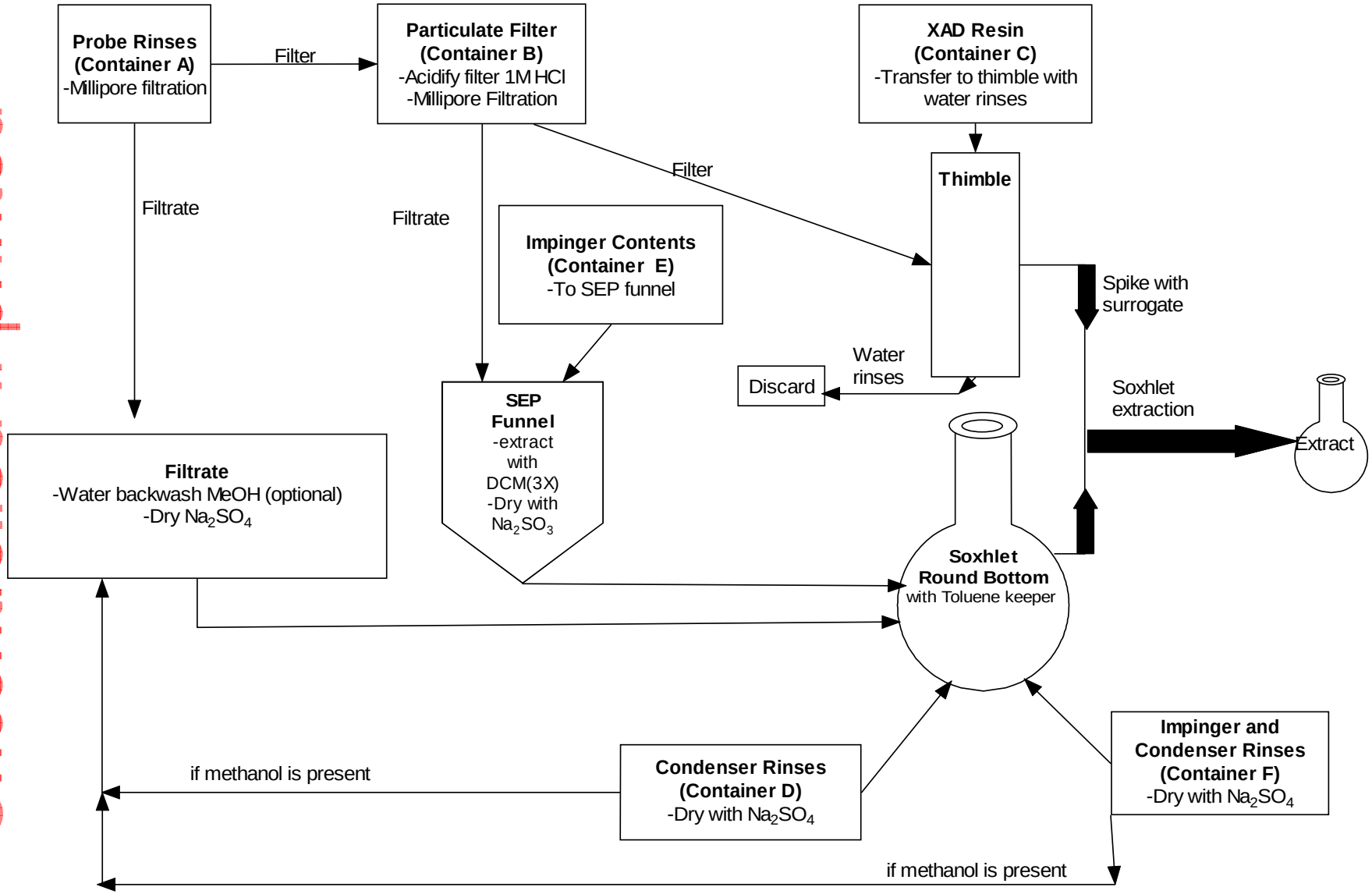
Figure 1: Processing of Sample Trains



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5.12 Extract Splitting Procedures

For detailed instructions refer to SLA-123 "*Splitting of Sample Extracts*".

Gravimetrically split the extract into two portions, one for the PCB analysis and one as backup. If multiple analyses are required, the extract may be split into many portions. Refer to the Batch List for extract splitting instructions. Record all weights on the worksheets.

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6. CLEANUP PROCEDURES

The default chromatographic cleanup procedures used for the analysis of 209 PCB congeners are described in Sections 6.2, 6.4 and 6.5. Optional cleanup columns described in Sections 6.1, 6.3 and 6.6 may be used depending upon the nature of the extract and the quality of the instrumental data. The Batch List indicates if optional columns are to be included as part of the initial extract cleanup procedures.

The nature of the extract or additional analysis requirements may necessitate using the columns in a different sequence. In this event, the analyst must ensure that the extracts are in a solvent suitable for the column.

For manual cleanup two options for sequencing the Silica/Alumina/Florisil columns are permitted. The default cleanup column sequence is A/B silica, Alumina, Florisil. The alternate sequence is A/B silica, Florisil, Alumina, and this option may be used as necessary to conform to sample turnaround times. Effectiveness of extract cleanup is not affected by the use of the alternate sequence.

At least two tests for sulphur with activated copper must be performed (refer to SLA-045 “Removal of Sulphur from Extracts using Activated Copper”). One of these tests must be performed after the Florisil column. The test for sulphur with activated copper is ineffective in the presence of toluene. If cleanup for pesticide analysis is required refer to Appendix A for details.

In the case where polybrominated diphenylethers are also to be determined along with the PCBs, the cleanup may be carried out using modified manual cleanup procedures. The analyst is referred to the following documents for details of manual cleanup procedures:

MLA-013	Coextraction for 1613B PCDD/F, 1668A PCBs, BDE, Pest and Toxaphene
MLA-033	Analytical Method for the Determination of Brominated Diphenylethers (PBDE) by EPA Method 1614

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6.1 Gel Permeation Cleanup Column (Default Procedure for Biosolid, INFL and EPRO matrices, Optional Procedure Otherwise. Note that for "tissue" type matrices this step is required and is described in the extraction sections)

Gel Permeation cleanup is mandatory for Biosolid, INFL and EPRO sample extracts (where it should be performed prior to the other cleanup procedures) and optional for other sample extracts, e.g. from sediment, soil, XAD-2 column, ambient air, solvent extract, EFIN and other aqueous samples. The Biobead column may be used prior to the chromatographic sequences described in Section 6.2 and 6.4. The cleanup is carried out as described below.

If necessary, carry out a solvent exchange on the extract to ensure that the extract is in 1:1 dichloromethane:hexane prior to cleanup on the Biobead column. Minor amounts of other solvents (i.e. up to 1 mL of toluene) can be tolerated.

Load the extract onto a Biobead SX-3 gel permeation column (prepared as described in Section 3.6.2) with 1:1 dichloromethane:hexane and elute with 1:1 dichloromethane:hexane at 5 mL/min. Refer to the most recent Biobead cutpoint determination for the elution volumes. Typical cutpoints are as follows:

F1	0 - 140 mL
F2	140 - 300 mL

Collect the second fraction and evaporate to a small volume by rotary evaporation. If any further column cleanup is required perform a solvent change to hexane by evaporating the extract to 300 µL under a gentle stream of nitrogen.

If the lipid content for the sample is greater than 2 g absolute (i.e. 10% of a 20 g sample), repeat the gel permeation cleanup procedure.

6.2 PB 4g 44% Multi-layered Acid/Base Silica Column (Default Procedure)

Extract may be loaded on to acid/base silica columns in any solvent. This column may be loaded from a round bottom flask.

Transfer the extract and hexane rinses (3 x 1-2 mL from the elution volume) to a layered acid/base silica column, prepared as described in Section 3.6.5 and elute with hexane (100 mL) at 5 mL/min. Collect the eluate in a round bottom flask and concentrate to a volume of 1 mL by rotary evaporation.

6.3 PB 8g 44% Large Capacity Multi-layered Acid/Base Silica Column (Optional Procedure)

Extract may be loaded on to acid/base silica columns in any solvent. This column may be loaded from a round bottom flask.

Transfer the extract and hexane rinses (3 x 1-2 mL from the elution volume) to a layered acid/base silica column, prepared as described in Section 3.6.6 and elute with hexane (100 mL) at 5 mL/min. Collect the eluate in a round bottom flask and concentrate to a volume of 1 mL by rotary evaporation.

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6.4 Alumina Column (Default Procedure)

The extract must be in 1 mL of hexane prior to loading onto the alumina column. Alumina columns may be loaded from a round bottom flask as long as the volume is 1 mL and the solvent is hexane. If other solvents are present in the extract a solvent exchange must be carried out as described below.

Solvent Exchange

Transfer the extract to a centrifuge tube with hexane and evaporate to 300 µL (50 µL if toluene is present) under a stream of nitrogen. Add hexane to 1 mL.

Load the extract with hexane rinses (3 x 1 mL, from the elution volume) onto an alumina column (6 g, 1% deactivated) prepared as described in Section 3.6.1. Refer to the most recent alumina cutpoint determination for the volume of solvent to use. Typical cutpoints are as follows:

F1	hexane	10 mL
F2	1:1 dichloromethane:hexane	45 mL

Elute the column with hexane (F1) and discard the eluate. Elute the column with 1:1 dichloromethane:hexane (F2) and retain the eluate. Concentrate to 1 mL by rotary evaporation and transfer to a centrifuge tube. Concentrate under a stream of nitrogen.

6.5 Florisil Column (Default Procedure)

Florisil columns can be loaded from a round-bottom flask. The extract must be 1 mL in hexane. If other solvents are present a solvent exchange must be carried out as described in section 6.4.

Quantitatively transfer the extract to a Florisil column (8 g, 2.0% deactivated) prepared as described in Section 3.6.4. Rinse the container (3 x 1 mL) using a portion of the 15:85 dichloromethane:hexane E1 elution solvent volume, and add the rinses to the column. Drain the solvent to the top of the column bed, collecting the eluate in a round bottom flask. Apply an additional 1 mL rinse from the E1 solvent to the column, drain to the top of the column bed and elute the column with the remaining volume of E1 solvent collecting all of the eluates in the round bottom flask. Use the elution volume determined from the Florisil cutpoint determination of the particular batch of Florisil. A typical cutpoint is as follows:

E1	15:85 dichloromethane:hexane	45 mL

6.6 4.5% Carbon/Celite Column (Optional Procedure for Toxic PCB Congeners)

This column may be used as part of the initial extract cleanup where it is used just prior to the alumina column (Section 6.4). Alternately, it is may be used only after the successful HRGC/HRMS analysis of all 209 PCB congeners has been carried out.

Prepare a 4.5% carbon/Celite column as described in Section 3.6.3, (0.22 g, 4.5% carbon AX 21 on Celite, pre-eluted with 15 mL toluene, and 15 mL hexane).

Note - Watch carefully for the packing slipping in the column. Be generous with the glass wool plugs when packing columns.

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Ensure the extract is in hexane (1 mL). Load the extract with hexane rinses (2 x 1 mL, taken from a 15 mL elution volume) onto the column. (A total of 3 mL of hexane should have been applied to the column). Elute the column with remaining hexane and store the eluate (E1) in a centrifuge tube that is capped and covered with clean foil.

Invert the column and elute with toluene. Refer to the most recent cutpoint determination for the elution volume. The typical elution volume is 50 - 60 mL. Collect this fraction in a 100 mL round-bottom flask. This fraction is E2. Concentrate the E2 fraction by rotary evaporation to a small volume. Transfer the extract to a clean centrifuge tube with toluene rinses and concentrate to 500 µL by evaporation under a gentle stream of nitrogen.

Note: Extracts recolumned on carbon/Celite must be respiked with recovery standards before instrumental analysis.

6.7 Preparation for GC/MS Analysis

After the final column concentrate the eluate to 1 mL by rotary evaporation.

If both copper tests have been done, add 1 mL of toluene to the extract in the round bottom flask before reducing on a Rotovap.

If both copper tests have not been done, add 1 mL of toluene to the centrifuge tube before blow down. Transfer the extract to a centrifuge tube with hexane rinses (3 x 1 mL) and concentrate to 300 µL in a stream of nitrogen, using a flow rate that produces an obvious dip in the surface of the solvent. The presence of toluene during this final blow down step is essential to reduce losses of the more volatile PCBs.

Add nonane (15 µL) to a clean autosampler vial (if the extract is also being analyzed for PBDE add 15 µL of toluene rather than 15 µL of nonane). Mark the vial at the 15 µL volume. Transfer the extract to the autosampler vial and concentrate to 150 – 200 µL under a gentle stream of nitrogen. Rinse the centrifuge tube with 350 µL of hexane and add the rinse to the autosampler vial. Concentrate the extract to a 150 – 200 µL using a gentle stream of nitrogen. Make sure the volume of extract does not go below 100 µL. Rinse the centrifuge tube with 350 µL of hexane and add the rinse to the autosampler vial. Concentrate as described below:

6.7.1 PCB or PCB/BDE Analysis

Concentrate the extract to 15 µL. Add an aliquot of recovery standard (5 µL) to the autosampler vial. Cap the autosampler vial. Store the vials in the freezer until just prior to GC/MS analysis.

6.7.2 BDE Analysis

Concentrate the extract to 45 µL (15 µL for blood extracts). Add an aliquot of recovery standard (5 µL) to the autosampler vial. Cap the autosampler vial. Store the vials in the freezer until just prior to GC/MS analysis.

Refer to the following Standard Operating Procedures:

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SLA-008	Preparing Extracts for Instrumental Analysis
SLA-072	Computer Preparation of Labels

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7. INSTRUMENTAL HRGC/HRMS ANALYSIS

Analysis by high resolution GC/MS is carried out using a Micromass Ultima high resolution mass spectrometer equipped with an HP 6890 gas chromatograph, a CTC autosampler, and an Alpha workstation running VG software. Chromatographic separation is achieved using an SPB-Octyl column (30 m, 0.25 mm I.D., 0.25 µm film thickness).

Perform the GC/MS analyses as described in Section 14 of EPA Method 1668A or 1668C. Instrumental analysis details are described in the most recent revision of AXYS instrumental method MIN-010, *EPA Method 1668A/C - Instrument Parameters and Specifications*.

Use the calibration standard level CS-0.2 as the sensitivity standard. Analyse the solution every time an initial calibration procedure is carried out.

Table 8 of EPA Method 1668A or 1668C presents the ions monitored, the ion abundance ratios, and quality control limits for all chlorination groups. The QC limits for the theoretical ratios must be satisfied for all analyses, including calibration solutions, QC samples, and test samples.

7.1 Initial Calibration

Refer to Section 10.0 of EPA Method 1668A or 1668C for details of calibration procedures. Perform the initial calibration as described in Section 10.4, using the calibration solutions described on Table 3 of this method.

The initial calibration solutions establish retention times and response factors for congeners present in the calibration solutions. After analysis of the multi-level standards (CS-1 through CS-5, or CS-0.2 through CS-5 for high sensitivity option; optionally including CS-6), analyze the calibration solution for 209 PCB congeners to establish the relative response factors and retention times for those PCB congeners not present in the multi-level standards solutions. The 209 congener standard need not be analyzed if only the analysis of toxic congeners is requested.

Optional Initial Calibration Procedure: A series of six calibration solutions, where the lowest concentration solution is one-half the concentration of the CS-1 solution, may be analyzed and unsmoothed data acquired and used to determine the linearity of the GC/MS. This option is used upon client request.

7.1.1 CS-0.2 Initial Calibration Criteria

An option is provided for evaluation of the CS-0.2 initial calibration results using duplicate injections performed sequentially. The CS-0.2 standard may be run twice at the beginning of the initial calibration series; the first run is quantified as a calibration run and included in the calculation of mean RRFs and the second run is quantified in concentration as a calibration verification run. Acceptability of ion ratios for the CS-0.2 standard may be based on

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evaluation of both the first and the second injections (see Figure 2). The 12-hour calibration time window always starts with the first injection of the CS-0.2 standard.

First Injection of CS-0.2 - Ion Ratio Acceptance Criteria

If the peak ion ratios for more than four congeners differ by more than 15% from theoretical or the peak ion ratio for any congener differs from theoretical by more than 35%, the instrument must be adjusted or serviced and the initial calibration sequence re-started with injection of CS-0.2.

If all ion peak ratios are within 15% of theoretical then the CS-0.2 peak ion ratio criteria have been met and a second injection of the CS-0.2 standard is not required.

If all ion peak ratios are not within 15% of theoretical for the first injection of the CS-0.2 standard but the ion peak ratios for a maximum of four congeners differ from theoretical by no more than 35% and the ion ratios for all remaining congeners are within 15% of theoretical, then further evaluation of CS 0-2 peak ion ratios may be made based on a second injection of CS-0.2.

Second Injection CS-0.2 - Ion Ratio Acceptance Criteria

If the ion peak ratios for a maximum of four congeners differ from theoretical by no more than 35%, ion ratios for all remaining congeners fall within 15% of theoretical and the ion ratio for any congener differing from theoretical by more than 15% in the first injection CS-0.2 falls within 15% of theoretical for the second injection CS-0.2 then the CS-0.2 peak ion ratio criteria have been met.

If the above conditions are not all met the instrument must be adjusted or serviced and the initial calibration sequence re-started with the first injection of CS-0.2.

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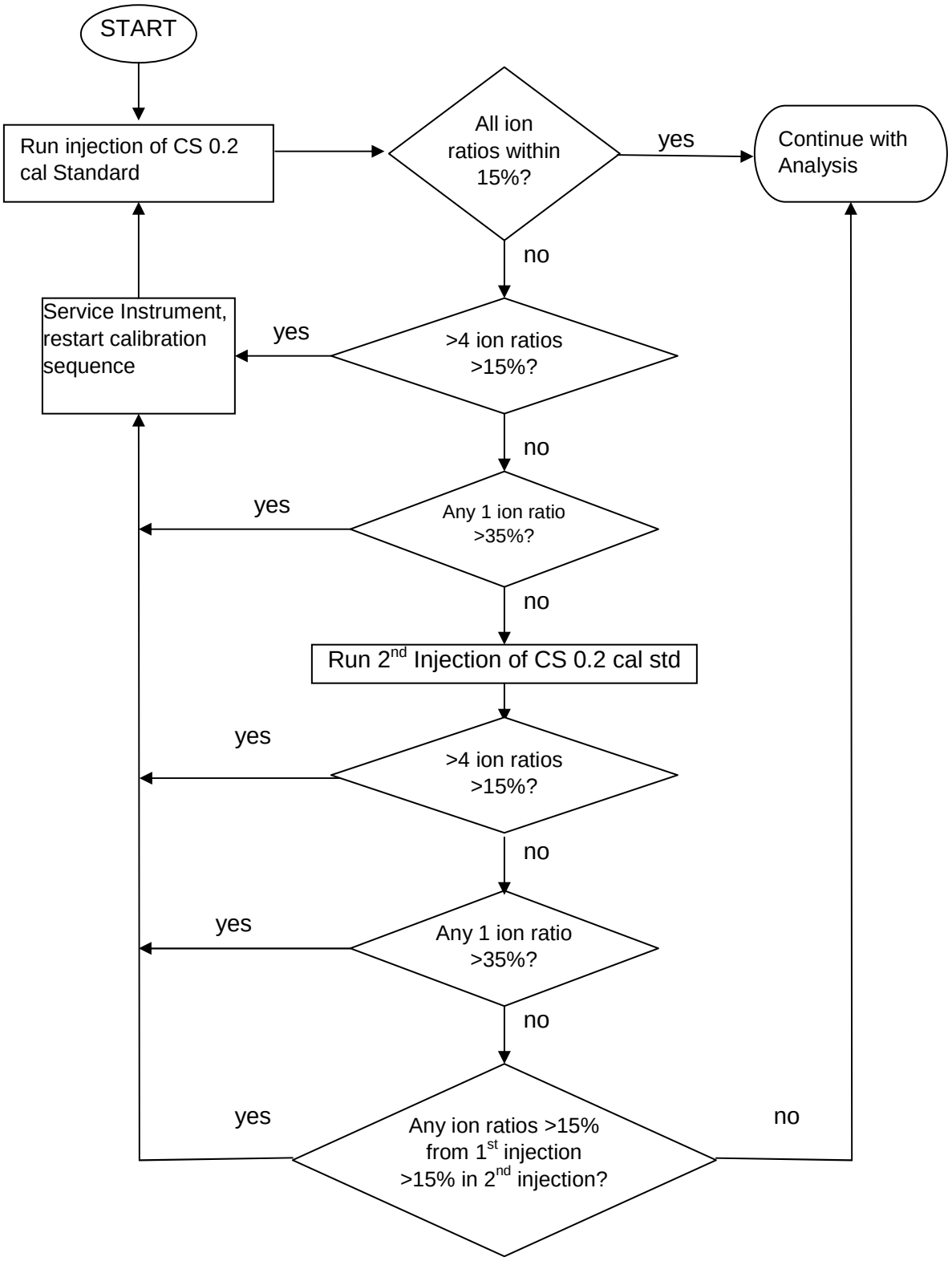
Figure 2. Procedure for Ion Ratio Criteria for CS-0.2 Initial Calibration Standard



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7.2 Calibration Verification

Every 12 hours the GC/MS system performance and calibration must be verified by the analysis of the 209 PCB congener solution. The QC criteria for the CAL/VER solution, as presented in Table 4a (EPA 1668A) or Table 4b (EPA 1668C), must be met prior to the analysis of samples. Details of the calibration verification procedures are described in the EPA Method 1668A or 1668C, Section 15.0.

7.3 Analysis of Samples

Refer to Section 14.0 of EPA Method 1668A or 1668C for details of instrumental analysis. Table 5 of this document summarizes the calibration and sample injection sequence.

Refer to Section 15.0 of EPA Method 1668A or 1668C for details of system and laboratory performance.

Extracts may be diluted with solvent and re-injected to bring responses within linear range. Extracts may not be diluted beyond the point at which reliable isotope quantification can be performed. Routine extract dilutions should not exceed 10 times (final extract volume of 200 µL). For any extracts analyzed at a greater dilution the chromatograms from both before and after the dilution must be inspected to ensure that detector 'phantom peak' phenomenon will not, and has not, impacted the reliability of results; supervisory approval of the dilution results must be documented. Appendix C gives details on performing the check for phantom peak phenomenon. Extracts with concentrations exceeding the range for reliable isotope dilution quantification must be reanalyzed using smaller sample sizes. For very high level samples and where allowable by contract it may be permissible to respike a diluted portion of the extract with an additional aliquot of surrogate standard, reinject and correct final concentrations for dilution/respike factors and surrogate recoveries observed initially.

7.4 Resolution of Coeluting Congeners

Resolution of the PCB 156/157 co-elution is achieved by high resolution GC/MS using a Micromass Ultima high resolution mass spectrometer coupled to an HP5890 or HP6890 gas chromatograph equipped with a CTC autosampler and an Alpha workstation running VG software. Chromatographic separation is achieved using a DB-1 chromatography column (30 m, 0.25 mm id, 0.25 µm film thickness).

7.5 Interferences

Analysis of the extract on either a DB-1 or DB-5 column is useful to resolve potential interferences with PCB 169 (from PCB 190). If the data show that such interference is present, carry out a GC/MS analysis on DB-1 or DB-5 column.

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Table 5. EPA Method 1668A and 1668C Instrumental Injection Sequence

TEST	Maximum Allowable Time *	Cycle
PFK Tune/Mass Res #1		Start of first cycle
CS3 run 1		
CS1, CS2, CS4, CS5		
209 Congener CS		
Instr blank		
QC Samples, Samples		
PFK Tune/Mass Res #2	PFK Tune/Mass Res #1 +12.0 hr	
CS3 run 2	CS3 run1 + 12.0 hr	Completion of first cycle
209 Congener CS		
Instr blank		
QC Samples, Samples		
PFK Tune/Mass Res #3	PFK Tune/Mass Res #2 +12.0 hr	
CS3 run 3	CS3 run2 + 12.0 hr	Completion of second cycle
Instr blank		
QC Samples, Samples		
…continue cycles or stop as below		
CS3 run 3		
PFK Tune/Mass Res #4	PFK Tune/Mass Res #3 +12.0 hr	Stop

*Time at injection

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8. QUALITATIVE AND QUANTITATIVE DETERMINATION

Refer to Section 16.0 of EPA Method 1668A or 1668C for details of qualitative determination and to Section 17.0 of EPA Method 1668A or 1668C for details of quantitative determination incorporating any modifications noted in Section 10 of this document and the modification below.

Peaks are assigned as target compounds on the basis of correct ion ratio within a specified retention time window. The retention time windows, around the observed retention time in the calibration standard, adjusted for any peak shifting in samples, are shown in Tables 6a, 6b and 6c. For Toxic/LOC/Window Defining/Labelled congeners retention time (RT) acceptance limits are determined from the mean RTs of the multi-point calibration runs; for other compounds the RT windows are determined from the single point 209 calibration standard.

8.1 Calculation of Aroclor Equivalent Concentrations from SPB-Octyl GC Column Data

Aroclor equivalent concentrations may be calculated by converting the summed concentrations of a suite of characteristic PCB congeners to concentrations using empirical factors determined from the analysis of Aroclor mixtures.

Aroclor 1016[1] = The sum of PCBs 8, 18/30, 31, 28/20 concentrations multiplied by 2.7;

Aroclor 1221 = The sum of PCBs 1, 3, 8 concentrations multiplied by 1.4;

Aroclor 1232 = The sum of PCBs 1, 3, 18/30 concentrations multiplied by 3.4;

Aroclor 1242[1] = The sum of PCBs 8, 18/30, 31, 28/20 concentrations multiplied by 3.0;

Aroclor 1248 = The sum of PCBs 44/47/65, 49/69, 66 concentrations multiplied by 6.1;

Aroclor 1254 = The sum of PCBs 86/87/97/**108**/119/125, 83/99 concentrations multiplied by 8.0 (for EPA 1668A); OR
The sum of PCBs 86/87/97/**109**/119/125, 83/99 concentrations multiplied by 8.0 (for EPA 1668C);

Aroclor 1260 = the sum of PCBs 183/185, 180/193, 170 concentrations multiplied by 5.0.

Environmental samples with no clearly identified Aroclor signature are quantified as 1242/1254/1260 mixtures. Results may be reported as Aroclor 1248 instead of Aroclor 1242 and 1254 where the congener pattern clearly indicates this formulation. Other Aroclor formulations may be reported by calibration against the specific Aroclor solutions.

[1] Aroclors 1016 and 1242 may be reported as combined 1016/1242 using the 1242 factor if allowed by contract

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Table 6a. Analyte Retention Times, Surrogates Used and RRT Windows for 209 PCB's by HRGC/MS

COMPOUND	Congener Number	CAS NO.	QUANTIFICATION REFERENCE	CO-ELUTIONS	COMPOUND RT	RT Reference	Labelled RT	RRT	RT Window (sec)	RRT Lower Limit	RRT Upper Limit
2 - MoCB	1	2051-60-7	1L		11:34:00	1L	11:34:00	1.000	-1,3	0.999	1.004
3 - MoCB	2	2051-61-8	1L/3L		13:38:00	3L	13:48:00	0.988	6	0.984	0.992
4 - MoCB	3	2051-62-9	3L		13:49:00	3L	13:48:00	1.001	-1,3	0.999	1.004
22' - DiCB	4	13029-08-8	4L		14:04:00	4L	14:03:00	1.001	-1,3	0.999	1.004
26 - DiCB	10	33146-45-1	4L/15L		14:14:00	4L	14:03:00	1.013	6	1.009	1.017
25 - DiCB	9	34883-39-1	4L/15L		16:06:00	4L	14:03:00	1.146	6	1.142	1.149
24 - DiCB	7	33284-50-3	4L/15L		16:16:00	4L	14:03:00	1.158	6	1.154	1.161
23' - DiCB	6	25569-80-6	4L/15L		16:31:00	4L	14:03:00	1.176	6	1.172	1.179
23 - DiCB	5	16605-91-7	4L/15L		16:49:00	4L	14:03:00	1.197	6	1.193	1.200
24' - DiCB	8	34883-43-7	4L/15L		16:58:00	4L	14:03:00	1.208	6	1.204	1.211
35 - DiCB	14	34883-41-5	4L/15L		18:39:00	15L	20:08:00	0.926	6	0.924	0.929
33' - DiCB	11	2050-67-1	4L/15L		19:32:00	15L	20:08:00	0.970	6	0.968	0.973
34' - DiCB	13	2974-90-5	4L/15L	12 + 13							
34 - DiCB	12	2974-92-7	4L/15L	12 + 13	19:51:00	15L	20:08:00	0.986	6	0.983	0.988
44' - DiCB	15	2050-68-2	15L		20:09:00	15L	20:08:00	1.001	-1,3	0.999	1.002
22'6 - TriCB	19	38444-73-4	19L		17:15:00	19L	17:13:00	1.002	-1,3	0.999	1.003
246 - TriCB	30	35693-92-6	19L/37L	18 + 30							
22'5 - TriCB	18	37680-65-2	19L/37L	18 + 30	19:09:00	19L	17:13:00	1.112	6	1.109	1.115
22'4 - TriCB	17	37680-66-3	19L/37L		19:36:00	19L	17:13:00	1.138	6	1.136	1.141
23'6 - TriCB	27	38444-76-7	19L/37L		19:50:00	19L	17:13:00	1.152	6	1.149	1.155
236 - TriCB	24	55702-45-9	19L/37L		19:58:00	19L	17:13:00	1.160	6	1.157	1.163
22'3 - TriCB	16	38444-78-9	19L/37L		20:05:00	19L	17:13:00	1.167	6	1.164	1.169
24'6 - TriCB	32	38444-77-8	19L/37L		20:37:00	19L	17:13:00	1.197	6	1.195	1.200
2'35 - TriCB	34	37680-68-5	19L/37L		21:56:00	19L	17:13:00	1.274	6	1.271	1.277
235 - TriCB	23	55720-44-0	19L/37L		22:06:00	19L	17:13:00	1.284	6	1.281	1.287
245 - TriCB	29	15862-07-4	19L/37L	26 + 29							
23'5 - TriCB	26	38444-81-4	19L/37L	26 + 29	22:26:00	19L	17:13:00	1.303	10	1.298	1.308
23'4 - TriCB	25	55712-37-3	19L/37L		22:40:00	37L	27:27:00	0.826	6	0.824	0.828
24'5 - TriCB	31	16606-02-3	19L/37L		22:59:00	37L	27:27:00	0.837	6	0.835	0.839

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COMPOUND	Congener Number	CAS NO.	QUANTIFICATION REFERENCE	CO-ELUTIONS	COMPOUND RT	RT Reference	Labelled RT	RRT	RT Window (sec)	RRT Lower Limit	RRT Upper Limit
244' - TriCB	28	7012-37-5	19L/37L	20 + 28							
233' - TriCB	20	38444-84-7	19L/37L	20 + 28	23:18:00	37L	27:27:00	0.849	10	0.846	0.852
234 - TriCB	21	55702-46-0	19L/37L	21 + 33	23:30:00	37L	27:27:00	0.856	10	0.853	0.859
2'34 - TriCB	33	38444-86-9	19L/37L	21 + 33							
234' - TriCB	22	38444-85-8	19L/37L		23:57:00	37L	27:27:00	0.872	6	0.871	0.874
33'5 - TriCB	36	38444-87-0	19L/37L		25:35:00	37L	27:27:00	0.932	6	0.930	0.934
34'5 - TriCB	39	38444-88-1	19L/37L		25:58:00	37L	27:27:00	0.946	6	0.944	0.948
345 - TriCB	38	53555-66-1	19L/37L		26:33:00	37L	27:27:00	0.967	6	0.965	0.969
33'4 - TriCB	35	37680-69-6	19L/37L		27:03:00	37L	27:27:00	0.985	6	0.984	0.987
344' - TriCB	37	38444-90-5	37L		27:28:00	37L	27:27:00	1.001	-1,3	0.999	1.002
22'66' - TeCB	54	15968-05-5	54L		20:25:00	54L	20:25:00	1.000	-1,3	0.999	1.002
22'46 - TeCB	50	62796-65-0	54L/81L/77L	50 + 53	22:41:00	54L	20:25:00	1.111	10	1.107	1.115
22'56' - TeCB	53	41464-41-9	54L/81L/77L	50 + 53							
22'36 - TeCB	45	70362-45-7	54L/81L/77L	45 + 51	23:24:00	54L	20:25:00	1.146	10	1.142	1.150
22'46' - TeCB	51	68194-04-7	54L/81L/77L	45 + 51							
22'36' - TeCB	46	41464-47-5	54L/81L/77L		23:41:00	54L	20:25:00	1.160	6	1.158	1.162
22'55' - TeCB	52	35693-99-3	54L/81L/77L		25:11:00	54L	20:25:00	1.233	6	1.231	1.236
23'5'6 - TeCB	73	74338-23-1	54L/81L/77L		25:20:00	54L	20:25:00	1.241	6	1.238	1.243
22'35 - TeCB	43	70362-46-8	54L/81L/77L		25:26:00	54L	20:25:00	1.246	6	1.243	1.248
23'46 - TeCB	69	60233-24-1	54L/81L/77L	49 + 69							
22'45' - TeCB	49	41464-40-8	54L/81L/77L	49 + 69	25:40:00	54L	20:25:00	1.257	10	1.253	1.261
22'45 - TeCB	48	70362-47-9	54L/81L/77L		25:59:00	54L	20:25:00	1.273	6	1.270	1.275
2356 - TeCB	65	33284-54-7	54L/81L/77L	44 + 47 + 65							
22'44' - TeCB	47	2437-79-8	54L/81L/77L	44 + 47 + 65							
22'35' - TeCB	44	41464-39-5	54L/81L/77L	44 + 47 + 65	26:14:00	54L	20:25:00	1.285	10	1.281	1.289
2346 - TeCB	62	54230-22-7	54L/81L/77L	59 + 62 + 75							
244'6 - TeCB	75	32598-12-2	54L/81L/77L	59 + 62 + 75							
233'6 - TeCB	59	74472-33-6	54L/81L/77L	59 + 62 + 75	26:34:00	54L	20:25:00	1.301	10	1.297	1.305
22'34' - TeCB	42	36559-22-5	54L/81L/77L		26:45:00	54L	20:25:00	1.310	6	1.308	1.313
22'34 - TeCB	41	52663-59-9	54L/81L/77L	40 + 41 + 71							
23'4'6 - TeCB	71	41464-46-4	54L/81L/77L	40 + 41 + 71							
22'33' - TeCB	40	38444-93-8	54L/81L/77L	40 + 41 + 71	27:14:00	54L	20:25:00	1.334	10	1.330	1.338

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COMPOUND	Congener Number	CAS NO.	QUANTIFICATION REFERENCE	CO-ELUTIONS	COMPOUND RT	RT Reference	Labelled RT	RRT	RT Window (sec)	RRT Lower Limit	RRT Upper Limit
234'6 - TeCB	64	52663-58-8	54L/81L/77L		27:31:00	54L	20:25:00	1.348	6	1.345	1.350
23'55' - TeCB	72	41464-42-0	54L/81L/77L		28:24:00	81L	34:31:00	0.823	6	0.821	0.824
23'45' - TeCB	68	73575-52-7	54L/81L/77L		28:42:00	81L	34:31:00	0.831	6	0.830	0.833
233'5 - TeCB	57	70424-67-8	54L/81L/77L		29:09:00	81L	34:31:00	0.845	6	0.843	0.846
233'5' - TeCB	58	41464-49-7	54L/81L/77L		29:23:00	81L	34:31:00	0.851	6	0.850	0.853
23'45 - TeCB	67	73575-53-8	54L/81L/77L		29:34:00	81L	34:31:00	0.857	6	0.855	0.858
234'5 - TeCB	63	74472-34-7	54L/81L/77L		29:50:00	81L	34:31:00	0.864	6	0.863	0.866
2345 - TeCB	61	33284-53-6	54L/81L/77L	61 + 70 + 74 + 76	30:11:00	81L	34:31:00	0.874	12	0.872	0.877
23'4'5 - TeCB	70	32598-11-1	54L/81L/77L	61 + 70 + 74 + 76							
2'345 - TeCB	76	70362-48-0	54L/81L/77L	61 + 70 + 74 + 76							
244'5 - TeCB	74	32690-93-0	54L/81L/77L	61 + 70 + 74 + 76							
23'44' - TeCB	66	32598-10-0	54L/81L/77L		30:32:00	81L	34:31:00	0.885	6	0.883	0.886
233'4 - TeCB	55	74338-24-2	54L/81L/77L		30:41:00	81L	34:31:00	0.889	6	0.887	0.890
233'4' - TeCB	56	41464-43-1	54L/81L/77L		31:13:00	81L	34:31:00	0.904	6	0.903	0.906
2344' - TeCB	60	33025-41-1	54L/81L/77L		31:27:00	81L	34:31:00	0.911	6	0.910	0.913
33'55' - TeCB	80	33284-52-5	54L/81L/77L		31:54:00	81L	34:31:00	0.924	6	0.923	0.926
33'45' - TeCB	79	41464-48-6	54L/81L/77L		33:29:00	81L	34:31:00	0.970	6	0.969	0.972
33'45 - TeCB	78	70362-49-1	54L/81L/77L		34:05:00	81L	34:31:00	0.987	6	0.986	0.989
344'5 - TeCB	81	70362-50-4	81L		34:32:00	81L	34:31:00	1.000	-1,3	1.000	1.001
33'44' - TeCB	77	32598-13-3	77L		35:08:00	77L	35:07:00	1.000	-1,3	1.000	1.001
22'466' - PeCB	104	56558-16-8	104L		26:10:00	104L	26:08:00	1.001	-1,3	0.999	1.002
22'366' - PeCB	96	73575-54-9	104L/123L/114L/ 118L/105L		26:32:00	104L	26:08:00	1.015	10	1.012	1.018
22'45'6 - PeCB	103	60145-21-3	104L/123L/114L/ 118L/105L		28:34:00	104L	26:08:00	1.093	6	1.091	1.095
22'356' - PeCB	94	73575-55-0	104L/123L/114L/ 118L/105L		28:48:00	104L	26:08:00	1.102	6	1.100	1.104
22'35'6 - PeCB	95	38379-99-6	104L/123L/114L/ 118L/105L	93 + 95 + 98 + 100 + 102							
22'44'6 - PeCB	100	39485-83-1	104L/123L/114L/ 118L/105L	93 + 95 + 98 + 100 + 102							
22'356 - PeCB	93	73575-56-1	104L/123L/114L/ 118L/105L	93 + 95 + 98 + 100 + 102	29:31:00	104L	26:08:00	1.129	34 *	1.123	1.136
22'456' - PeCB	102	68194-06-9	104L/123L/114L/ 118L/105L	93 + 95 + 98 + 100 + 102							

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COMPOUND	Congener Number	CAS NO.	QUANTIFICATION REFERENCE	CO-ELUTIONS	COMPOUND RT	RT Reference	Labelled RT	RRT	RT Window (sec)	RRT Lower Limit	RRT Upper Limit
22'3'46 - PeCB	98	60233-25-2	104L/123L/114L/ 118L/105L	93 + 95 + 98 + 100 + 102							
22'346 - PeCB	88	55215-17-3	104L/123L/114L/ 118L/105L	88 + 91	30:07:00	104L	26:08:00	1.152	12	1.149	1.156
22'34'6 - PeCB	91	68194-05-8	104L/123L/114L/ 118L/105L	88 + 91							
22'33'6 - PeCB	84	52663-60-2	104L/123L/114L/ 118L/105L		30:23:00	104L	26:08:00	1.163	6	1.161	1.165
22'346' - PeCB	89	73575-57-2	104L/123L/114L/ 118L/105L		30:53:00	104L	26:08:00	1.182	6	1.180	1.184
23'45'6 - PeCB	121	56558-18-0	104L/123L/114L/ 118L/105L		31:20:00	104L	26:08:00	1.199	6	1.197	1.201
22'355' - PeCB	92	52663-61-3	104L/123L/114L/ 118L/105L		31:44:00	123L	37:12:00	0.853	6	0.852	0.854
233'5'6 - PeCB	113	68194-10-5	104L/123L/114L/ 118L/105L	90 + 101 + 113							
22'34'5 - PeCB	90	68194-07-0	104L/123L/114L/ 118L/105L	90 + 101 + 113	32:19:00	123L	37:12:00	0.869	10	0.866	0.871
22'455' - PeCB	101	37680-73-2	104L/123L/114L/ 118L/105L	90 + 101 + 113							
22'33'5 - PeCB	83	60145-20-2	104L/123L/114L/ 118L/105L	83 + 99	32:53:00	123L	37:12:00	0.884	12	0.881	0.887
22'44'5 - PeCB	99	38380-01-7	104L/123L/114L/ 118L/105L	83 + 99							
233'56 - PeCB	112	74472-36-9	104L/123L/114L/ 118L/105L		33:04:00	123L	37:12:00	0.889	6	0.888	0.890
23'44'6 - PeCB											
233'45' - PeCB											
22'345 - PeCB	See Tables 6b and 6c for information specific to EPA 1668A or 1668C										
22'3'45 - PeCB											
2'3456' - PeCB											
22'345' - PeCB											
234'56 - PeCB	117	68194-11-6	104L/123L/114L/ 118L/105L	85 + 116 + 117							
23456 - PeCB	116	18259-05-7	104L/123L/114L/ 118L/105L	85 + 116 + 117							
22'344' - PeCB	85	65510-45-4	104L/123L/114L/ 118L/105L	85 + 116 + 117	34:12:00	123L	37:12:00	0.919	12	0.917	0.922

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COMPOUND	Congener Number	CAS NO.	QUANTIFICATION REFERENCE	CO-ELUTIONS	COMPOUND RT	RT Reference	Labelled RT	RRT	RT Window (sec)	RRT Lower Limit	RRT Upper Limit
233'4'6 - PeCB	110	38380-03-9	104L/123L/114L/ 118L/105L	110 + 115	34:27:00	123L	37:12:00	0.926	10	0.924	0.928
2344'6 - PeCB	115	74472-38-1	104L/123L/114L/ 118L/105L	110 + 115							
22'33'4 - PeCB	82	52663-62-4	104L/123L/114L/ 118L/105L		34:43:00	123L	37:12:00	0.933	6	0.932	0.935
233'55' - PeCB	111	39635-32-0	104L/123L/114L/ 118L/105L		35:10:00	123L	37:12:00	0.945	6	0.944	0.947
23'455' - PeCB	120	68194-12-7	104L/123L/114L/ 118L/105L		35:38:00	123L	37:12:00	0.958	6	0.957	0.959
233'4'5 - PeCB											
2'3455' - PeCB			See Tables 6b and 6c for information specific to EPA 1668A or 1668C								
233'46 - PeCB											
2'344'5 - PeCB	123	65510-44-3	123L		37:13:00	123L	37:12:00	1.000	-1,3	1.000	1.001
233'45 - PeCB	106	70424-69-0	104L/123L/114L/ 118L/105L		37:21:00	123L	37:12:00	1.004	6	1.003	1.005
23'44'5 - PeCB	118	31508-00-6	118L		37:34:00	118L	37:32:00	1.001	-1,3	1.000	1.002
2'33'45 - PeCB	122	76842-07-4	104L/123L/114L/ 118L/105L		37:55:00	118L	37:32:00	1.010	6	1.009	1.012
2344'5 - PeCB	114	74472-37-0	114L		38:07:00	114L	38:06:00	1.000	-1,3	1.000	1.001
233'44' - PeCB	105	32598-14-4	105L		38:48:00	105L	38:46:00	1.001	-1,3	0.999	1.001
33'455' - PeCB	127	39635-33-1	104L/123L/114L/ 118L/105L		40:21:00	105L	38:46:00	1.041	6	1.040	1.042
33'44'5 - PeCB	126	57465-28-8	126L		42:02:00	126L	42:01:00	1.000	-1,3	1.000	1.001
22'44'66' - HxCB	155	33979-03-2	155L		32:06:00	155L	32:05:00	1.001	-1,3	0.999	1.002
22'3566' - HxCB	152	68194-09-2	155L/156L/157L/ 167L/169L		32:17:00	155L	32:05:00	1.006	6	1.005	1.008
22'34'66' - HxCB	150	68194-08-1	155L/156L/157L/ 167L/169L		32:28:00	155L	32:05:00	1.012	6	1.010	1.014
22'33'66' - HxCB	136	38411-22-2	155L/156L/157L/ 167L/169L		32:51:00	155L	32:05:00	1.024	6	1.022	1.025
22'3466' - HxCB	145	74472-40-5	155L/156L/157L/ 167L/169L		33:10:00	155L	32:05:00	1.034	6	1.032	1.035
22'34'56' - HxCB	148	74472-41-6	155L/156L/157L/ 167L/169L		34:45:00	155L	32:05:00	1.083	6	1.082	1.085
22'355'6 - HxCB	151	52663-63-5	155L/156L/157L/ 167L/169L	135 + 151 + 154							

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COMPOUND	Congener Number	CAS NO.	QUANTIFICATION REFERENCE	CO-ELUTIONS	COMPOUND RT	RT Reference	Labelled RT	RRT	RT Window (sec)	RRT Lower Limit	RRT Upper Limit
22'33'56' - HxCB	135	52744-13-5	155L/156L/157L/ 167L/169L	135 + 151 + 154	35:29:00	155L	32:05:00	1.106	22 *	1.103	1.109
22'44'5'6 - HxCB	154	60145-22-4	155L/156L/157L/ 167L/169L	135 + 151 + 154							
22'345'6 - HxCB	144	68194-14-9	155L/156L/157L/ 167L/169L		35:57:00	155L	32:05:00	1.121	6	1.119	1.122
22'34'56 - HxCB	147	68194-13-8	155L/156L/157L/ 167L/169L	147 + 149	36:20:00	155L	32:05:00	1.132	10	1.130	1.135
22'34'5'6 - HxCB	149	38380-04-0	155L/156L/157L/ 167L/169L	147 + 149							
22'33'56 - HxCB	134	52704-70-8	155L/156L/157L/ 167L/169L	134 + 143	36:36:00	155L	32:05:00	1.141	10	1.138	1.143
22'3456' - HxCB	143	68194-15-0	155L/156L/157L/ 167L/169L	134 + 143							
22'344'6 - HxCB	139	56030-56-9	155L/156L/157L/ 167L/169L	139 + 140	36:58:00	155L	32:05:00	1.152	10	1.150	1.155
22'344'6' - HxCB	140	59291-64-4	155L/156L/157L/ 167L/169L	139 + 140							
22'33'46 - HxCB	131	61798-70-7	155L/156L/157L/ 167L/169L		37:11:00	155L	32:05:00	1.159	6	1.157	1.161
22'3456 - HxCB	142	41411-61-4	155L/156L/157L/ 167L/169L		37:20:00	155L	32:05:00	1.164	6	1.162	1.165
22'33'46' - HxCB	132	38380-05-1	155L/156L/157L/ 167L/169L		37:39:00	155L	32:05:00	1.174	10	1.171	1.176
22'33'55' - HxCB	133	35694-04-3	155L/156L/157L/ 167L/169L		38:11:00	155L	32:05:00	1.190	6	1.189	1.192
233'55'6 - HxCB	165	74472-46-1	155L/156L/157L/ 167L/169L		38:37:00	167L	43:57:00	0.879	6	0.878	0.880
22'34'55' - HxCB	146	51908-16-8	155L/156L/157L/ 167L/169L		38:52:00	167L	43:57:00	0.884	6	0.883	0.885
233'45'6 - HxCB	161	74472-43-8	155L/156L/157L/ 167L/169L		39:01:00	167L	43:57:00	0.888	6	0.887	0.889
22'44'55' - HxCB	153	35065-27-1	155L/156L/157L/ 167L/169L	153 + 168	39:32:00	167L	43:57:00	0.900	10	0.898	0.901
23'44'5'6 - HxCB	168	59291-65-5	155L/156L/157L/ 167L/169L	153 + 168							
22'3455' - HxCB	141	52712-04-6	155L/156L/157L/ 167L/169L		39:43:00	167L	43:57:00	0.904	6	0.903	0.905
22'33'45' - HxCB	130	52663-66-8	155L/156L/157L/ 167L/169L		40:08:00	167L	43:57:00	0.913	6	0.912	0.914

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COMPOUND	Congener Number	CAS NO.	QUANTIFICATION REFERENCE	CO-ELUTIONS	COMPOUND RT	RT Reference	Labelled RT	RRT	RT Window (sec)	RRT Lower Limit	RRT Upper Limit
22'344'5 - HxCB	137	35694-06-5	155L/156L/157L/ 167L/169L		40:22:00	167L	43:57:00	0.918	6	0.917	0.920
233'4'5'6 - HxCB	164	74472-45-0	155L/156L/157L/ 167L/169L		40:29:00	167L	43:57:00	0.921	6	0.920	0.922
22'344'5' - HxCB	138	35065-28-2	155L/156L/157L/ 167L/169L	129 + 138 + 160 + 163							
233'4'56 - HxCB	163	74472-44-9	155L/156L/157L/ 167L/169L	129 + 138 + 160 + 163							
22'33'45 - HxCB	129	55215-18-4	155L/156L/157L/ 167L/169L	129 + 138 + 160 + 163	40:53:00	167L	43:57:00	0.930	14	0.928	0.933
233'456 - HxCB	160	41411-62-5	155L/156L/157L/ 167L/169L	129 + 138 + 160 + 163							
233'44'6 - HxCB	158	74472-42-7	155L/156L/157L/ 167L/169L		41:13:00	167L	43:57:00	0.938	6	0.937	0.939
2344'56 - HxCB	166	41411-63-6	155L/156L/157L/ 167L/169L	128 + 166							
22'33'44' - HxCB	128	38380-07-3	155L/156L/157L/ 167L/169L	128 + 166	42:08:00	167L	43:57:00	0.959	10	0.957	0.961
233'455' - HxCB	159	39635-35-3	155L/156L/157L/ 167L/169L		43:10:00	167L	43:57:00	0.982	6	0.981	0.983
233'4'55' - HxCB	162	39635-34-2	155L/156L/157L/ 167L/169L		43:28:00	167L	43:57:00	0.989	6	0.988	0.990
23'44'55' - HxCB	167	52663-72-6	167L		43:59:00	167L	43:57:00	1.001	-1,3	1.000	1.001
233'44'5 - HxCB	156	38380-08-4	156L/157L		45:11:00	156L/157L	45:10:00	1.000	6	0.999	1.001
233'44'5' - HxCB	157	69782-90-7	156L/157L	156 + 157							
33'44'55' - HxCB	169	32774-16-6	169L		48:36:00	169L	48:34:00	1.001	-1,3	1.000	1.001
22'34'566' - HpCB	188	74487-85-7	188L		38:06:00	188L	38:04:00	1.001	-1,3	1.000	1.001
22'33'566' - HpCB	179	52663-64-6	188L/189L		38:26:00	188L	38:04:00	1.010	6	1.008	1.011
22'344'66' - HpCB	184	74472-48-3	188L/189L		39:00:00	188L	38:04:00	1.025	6	1.023	1.026
22'33'466' - HpCB	176	52663-65-7	188L/189L		39:22:00	188L	38:04:00	1.034	6	1.033	1.035
22'34566' - HpCB	186	74472-49-4	188L/189L		39:50:00	188L	38:04:00	1.046	6	1.045	1.048
22'33'55'6 - HpCB	178	52663-67-9	188L/189L		41:17:00	188L	38:04:00	1.085	6	1.083	1.086
22'33'45'6 - HpCB	175	40186-70-7	188L/189L		41:57:00	188L	38:04:00	1.102	6	1.101	1.103
22'34'55'6 - HpCB	187	52663-68-0	188L/189L		42:15:00	188L	38:04:00	1.110	6	1.109	1.111
22'344'56' - HpCB	182	60145-23-5	188L/189L		42:27:00	188L	38:04:00	1.115	6	1.114	1.116

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22'344'5'6 - HpCB	183	52663-69-1	188L/189L	183 + 185	42:56:00	188L	38:04:00	1.128	6	1.127	1.129
22'3455'6 - HpCB	185	52712-05-7	188L/189L	183 + 185							
22'33'456' - HpCB	174	38411-25-5	188L/189L		43:07:00	188L	38:04:00	1.133	6	1.131	1.134
22'33'4'56 - HpCB	177	52663-70-4	188L/189L		43:35:00	188L	38:04:00	1.145	6	1.144	1.146
22'344'56 - HpCB	181	74472-47-2	188L/189L		44:00:00	188L	38:04:00	1.156	6	1.155	1.157
22'33'44'6 - HpCB	171	52663-71-5	188L/189L	171 + 173	44:13:00	188L	38:04:00	1.162	10	1.159	1.164
22'33'456 - HpCB	173	68194-16-1	188L/189L	171 + 173							
22'33'455' - HpCB	172	52663-74-8	188L/189L		45:57:00	189L	51:13:00	0.897	6	0.896	0.898
233'455'6 - HpCB	192	74472-51-8	188L/189L		46:15:00	189L	51:13:00	0.903	6	0.902	0.904
233'4'55'6 - HpCB	193	69782-91-8	180L	180 + 193							
22'344'55' - HpCB	180	35065-29-3	180L	180 + 193	46:36:00	180L	46:35:00	1.000	6	0.999	1.001
233'44'5'6 - HpCB	191	74472-50-7	188L/189L		47:00:00	189L	51:13:00	0.918	6	0.917	0.919
22'33'44'5 - HpCB	170	35065-30-6	170L		47:57:00	170L	47:56:00	1.000	6	0.999	1.001
233'44'56 - HpCB	190	41411-64-7	188L/189L		48:31:00	189L	51:13:00	0.947	6	0.946	0.948
233'44'55' - HpCB	189	39635-31-9	189L		51:14:00	189L	51:13:00	1.000	-1,3	1.000	1.001
22'33'55'66' - OcCB	202	2136-99-4	202L		43:43:00	202L	43:41:00	1.001	-1,3	1.000	1.001
22'33'45'66' - OcCB	201	40186-71-8	202L/205L		44:41:00	202L	43:41:00	1.023	10	1.021	1.025
22'344'566' - OcCB	204	74472-52-9	202L/205L		45:23:00	202L	43:41:00	1.039	6	1.038	1.040
22'33'44'66' - OcCB	197	33091-17-7	202L/205L	197 + 200	45:41:00	202L	43:41:00	1.046	14 *	1.045	1.047
22'33'4566' - OcCB	200	52663-73-7	202L/205L	197 + 200							
22'33'455'6 - OcCB	198	68194-17-2	202L/205L	198 + 199	48:40:00	202L	43:41:00	1.114	10	1.112	1.116
22'33'455'6' - OcCB	199	52663-75-9	202L/205L	198 + 199							
22'33'44'56' - OcCB	196	42740-50-1	202L/205L		49:23:00	205L	53:54:00	0.916	6	0.915	0.917
22'344'55'6 - OcCB	203	52663-76-0	202L/205L		49:35:00	205L	53:54:00	0.920	6	0.919	0.921
22'33'44'56 - OcCB	195	52663-78-2	202L/205L		50:59:00	205L	53:54:00	0.946	6	0.945	0.947
22'33'44'55' - OcCB	194	35694-08-7	202L/205L		53:26:00	205L	53:54:00	0.991	6	0.990	0.992
233'44'55'6 - OcCB	205	74472-53-0	205L		53:56:00	205L	53:54:00	1.001	-1,3	1.000	1.001
22'33'455'66' - NoCB	208	52663-77-1	208L		50:43:00	208L	50:42:00	1.000	-1,3	1.000	1.001
22'33'44'566' - NoCB	207	52663-79-3	208L/206L		51:42:00	208L	50:42:00	1.020	6	1.019	1.021
22'33'44'55'6 - NoCB	206	40186-72-9	206L		55:45:00	206L	55:44:00	1.000	-1,3	1.000	1.001

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COMPOUND	Congener Number	CAS NO.	QUANTIFICATION REFERENCE	CO-ELUTIONS	COMPOUND RT	RT Reference	Labelled RT	RRT	RT Window (sec)	RRT Lower Limit	RRT Upper Limit
22'33'44'55'66' - DeCB	209	2051-24-3	209L		57:26:00	209L	57:25:00	1.000	-1,3	1.000	1.001
LABELLED COMPOUND											
13C12-2 - MoCB	**1L**		9L		11:34:00	9L	16:05:00	0.719	30	0.704	0.735
13C12-4 - MoCB	**3L**		9L		13:48:00	9L	16:05:00	0.858	30	0.842	0.874
13C12-22' - DiCB	**4L**		9L		14:03:00	9L	16:05:00	0.874	30	0.858	0.889
13C12-44' - DiCB	**15L**		9L		20:08:00	9L	16:05:00	1.252	30	1.236	1.267
13C12-22'6 - TriCB	**19L**		9L		17:13:00	9L	16:05:00	1.070	30	1.055	1.086
13C12-344' - TriCB	**37L**		52L		27:27:00	52L	25:09:00	1.091	30	1.082	1.101
13C12-22'66' - TeCB	**54L**		52L		20:25:00	52L	25:09:00	0.812	20	0.805	0.818
13C12-33'44' - TeCB	**77L**		52L		35:07:00	52L	25:09:00	1.396	20	1.390	1.403
13C12-344'5 - TeCB	**81L**		52L		34:31:00	52L	25:09:00	1.372	20	1.366	1.379
13C12-22'466' - PeCB	**104L**		101L		26:08:00	101L	32:19:00	0.809	20	0.804	0.814
13C12-233'44' - PeCB	**105L**		101L		38:46:00	101L	32:19:00	1.200	20	1.194	1.205
13C12-2344'5 - PeCB	**114L**		101L		38:06:00	101L	32:19:00	1.179	20	1.174	1.184
13C12-23'44'5 - PeCB	**118L**		101L		37:32:00	101L	32:19:00	1.161	20	1.156	1.167
13C12-2'344'5 - PeCB	**123L**		101L		37:12:00	101L	32:19:00	1.151	20	1.146	1.156
13C12-33'44'5 - PeCB	**126L**		101L		42:01:00	101L	32:19:00	1.300	20	1.295	1.305
13C12-22'44'66' - HxCB	**155L**		138L		32:05:00	138L	40:48:00	0.786	20	0.782	0.790
13C12-233'44'5 - HxCB and	**156L**		138L		45:10:00	138L	40:48:00	1.107	20	1.103	1.111
13C12-233'44'5' - HxCB	**157L**		138L								
13C12-23'44'55' - HxCB	**167L**		138L		43:57:00	138L	40:48:00	1.077	20	1.073	1.081
13C12-33'44'55' - HxCB	**169L**		138L		48:34:00	138L	40:48:00	1.190	20	1.186	1.194
13C12-22'33'44'5 - HpCB	**170L**		194L		47:57:00	194L	53:25:00	0.898	20	0.891	0.904
13C12-22'344'55' - HpCB	**180L**		194L		46:36:00	194L	53:25:00	0.872	20	0.866	0.879
13C12-22'34'566' - HpCB	**188L**		194L		38:04:00	194L	53:25:00	0.713	20	0.710	0.716
13C12-233'44'55' - HpCB	**189L**		194L		51:13:00	194L	53:25:00	0.959	20	0.956	0.962
13C12-22'33'55'66' - OcCB	**202L**		194L		43:41:00	194L	53:25:00	0.818	20	0.815	0.821
13C12-233'44'55'6 - OcCB	**205L**		194L		53:54:00	194L	53:25:00	1.009	30	1.004	1.014
13C12-22'33'44'55'6 - NoCB	**206L**		194L		55:44:00	194L	53:25:00	1.043	30	1.039	1.048
13C12-22'33'455'66' - NoCB	**208L**		194L		50:42:00	194L	53:25:00	0.949	20	0.946	0.952
13C12-22'33'44'55'66' - DeCB	**209L**		194L		57:26:00	194L	53:25:00	1.075	30	1.071	1.080
LABELLED CLEAN-UP STANDARD											
13C12-244' - TriCB	**28L**		52L		23:16:00	52L	25:09:00	0.925	20	0.918	0.932
13C12-233'55' - PeCB	**111L**		101L		35:09:00	101L	32:19:00	1.088	20	1.083	1.093

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COMPOUND	Congener Number	CAS NO.	QUANTIFICATION REFERENCE	CO-ELUTIONS	COMPOUND RT	RT Reference	Labelled RT	RRT	RT Window (sec)	RRT Lower Limit	RRT Upper Limit
13C12-22'33'55'6 - HpCB	**178L**		138L		41:16:00	138L	40:48:00	1.011	20	1.007	1.016
LABELLED INJECTION INTERNAL STANDARD											
13C12-25 - DiCB	**9L**		138L		16:05:00	138L	40:48:00	0.394	25	0.389	0.399
13C12-22'55' - TeCB	**52L**		138L		25:09:00	138L	40:48:00	0.616	25	0.611	0.622
13C12-22'455' - PeCB	**101L**		138L		32:19:00	138L	40:48:00	0.792	25	0.787	0.797
13C12-22'344'5' - HxCB	**138L**		138L		40:48:00	138L	40:48:00	1.000	100	0.980	1.020
13C12-22'33'44'55' - OcCB	**194L**		138L		53:25:00	138L	40:48:00	1.309	25	1.304	1.314

(1) Suffix "L" indicates labelled compound.

(2) C = co-eluting congener

* RT window wider than prescribed by 1668A/C to accommodate for skewed peaks of coeluting peaks (PCB-95/100/93/102/98, PCB-151/135/154 and PCB-197/200)

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Table 6b. Analyte Retention Times, Surrogates Used and RRT Windows for Congener Numbers 107, 108 and 109 in accordance with 1668A Specification

COMPOUND	Congener Number	CAS NO.	QUANTIFICATION REFERENCE	CO-ELUTIONS	COMPOUND RT	RT Reference	Labelled RT	RRT	RT Window (sec)	RRT Lower Limit	RRT Upper Limit
23'44'6 - PeCB	119	56558-17-9	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 108 + 119 + 125							
233'45' - PeCB	108	70362-41-3	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 108 + 119 + 125							
22'345 - PeCB	86	55312-69-1	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 108 + 119 + 125	33:29:00	123L	37:12:00	0.900	16	0.897	0.904
22'3'45 - PeCB	97	41464-51-1	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 108 + 119 + 125							
2'3456' - PeCB	125	74472-39-2	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 108 + 119 + 125							
22'345' - PeCB	87	38380-02-8	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 108 + 119 + 125							
233'4'5 - PeCB	107	70424-68-9	104L/123L/114L/ 118L/105L	107 + 124	36:50:00	123L	37:12:00	0.990	10	0.988	0.992
2'3455' - PeCB	124	70424-70-3	104L/123L/114L/ 118L/105L	107 + 124							
233'46 - PeCB	109	74472-35-8	104L/123L/114L/ 118L/105L		37:05:00	123L	37:12:00	0.997	6	0.996	0.998

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Table 6c. Analyte Retention Times, Surrogates Used and RRT Windows for Optional Reporting of Congener Numbers 107, 108 and 109 in accordance with 1668C Specification

COMPOUND	Congener Number	CAS NO.	QUANTIFICATION REFERENCE	CO-ELUTIONS	COMPOUND RT	RT Reference	Labelled RT	RRT	RT Window (sec)	RRT Lower Limit	RRT Upper Limit
23'44'6 - PeCB	119	56558-17-9	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 109 + 119 + 125							
2,3,3',4,6 - PeCB	109	74472-35-8	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 109 + 119 + 125							
2,2',3,4,5 - PeCB	86	55312-69-1	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 109 + 119 + 125	33:29:00	123L	37:12:00	0.900	16	0.897	0.904
2,2',3',4,5 - PeCB	97	41464-51-1	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 109 + 119 + 125							
2',3,4,5,6' - PeCB	125	74472-39-2	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 109 + 119 + 125							
2,2',3,4,5' - PeCB	87	38380-02-8	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 109 + 119 + 125							
2,3,3',4,5' - PeCB	108	70362-41-3	104L/123L/114L/ 118L/105L	108 + 124	36:50:00	123L	37:12:00	0.990	10	0.988	0.992
2',3,4,5,5' - PeCB	124	70424-70-3	104L/123L/114L/ 118L/105L	108 + 124							
2,3,3',4',5 - PeCB	107	70424-68-9	104L/123L/114L/ 118L/105L		37:05:00	123L	37:12:00	0.997	6	0.996	0.998

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Table 7. Theoretical Ion Abundance Ratios and QC Limits

Chlorine Atoms	m/z's Forming Ratio	Theoretical Ratio	Lower QC Limit	Upper QC Limit
1	M/(M+2)	3.13	2.66	3.60
2	M/(M+2)	1.56	1.33	1.79
3	M/(M+2)	1.04	0.88	1.20
4	M/(M+2)	0.77	0.65	0.89
5	(M+2)/(M+4)	1.55	1.32	1.78
6	(M+2)/(M+4)	1.24	1.05	1.43
7	(M+2)/(M+4)	1.05	0.89	1.21
8	(M+2)/(M+4)	0.89	0.76	1.02
9	(M+2)/(M+4)	0.77	0.65	0.89
10	(M+4)/(M+6)	1.16	0.99	1.33

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9. REPORTING CRITERIA AND PRACTICES

Concentrations and detection limits for target compounds are reported. The isotope dilution/internal standard method of quantification, used to determine concentrations of target analytes, corrects the concentrations based on the percent recovery of the surrogate. Typical reporting units are pg/g, pg/L, or pg/sample. Concentrations for solids are reported on a dry weight basis. Concentrations in tissues (including blood/serum/plasma and milk) are reported on a wet weight basis and/or on a lipid weight basis when requested. Concentrations in aqueous samples are reported on a volume basis. Concentrations in XAD-2 resin, filters and stack gas samples are reported on a per sample basis or a per volume basis. Concentrations in particulate filters are reported on a per sample basis. Results may be expressed in other units if specified by contract. Unless otherwise specified, concentrations are reported to three significant figures.

Percent moisture is reported for all sediments, soils, and ash samples and for tissues when percent moisture is requested. Percent moisture is not reported for pulps and sludge, although it is determined for the calculation of a dry weight.

Percent lipid is reported if determined on a sample.

Various reporting limit options are offered to meet particular client and data quality objectives and should be defined specific to contract by the client and agreed to by AXYS in advance of instrumental analysis:

9.1 Sample Specific Detection Limit (SDL) Reporting Limit

SDLs are determined from the analysis data by converting the minimum detectable area to a concentration following the same quantification procedures used to convert target peak responses to concentrations. The estimated minimum detectable area is determined as 2.5 times the height of the noise in the m/z channel of interest, converted to an area using the area:height ratio of the corresponding labelled surrogate peak. The SDL accounts for any effect of matrix on the detection system, for recovery achieved through the analytical work-up and for the exact sample size used.

SDLs do not account for background contribution, and sample results must be compared to the associated QC blank results for significance of detection.

The lower reporting limit for blood/serum/plasma samples is the SDL, for other matrices the lower reporting limit is the greater of SDL or 0.5 pg/sample.

9.2 EPA1668A and EPA1668C Specified Reporting Limit

An alternate reporting limit approach may be applied on written request of the customer: sample results are reported to minimum levels (“MLs”) according to section 17.6.1.4 of EPA methods 1668A/C. Depending on client request, the MLs used may be the MLs listed in 1688A/C (see Table 8) or laboratory MLs. Laboratory MLs are determined as the greater of the low calibration equivalent or of the (mean + 2 standard deviations) values of laboratory blank concentrations. Values for laboratory blanks are updated annually for aqueous, solid and tissue sample matrices, using a minimum of 10 consecutively processed blanks representative of

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typical lab performance.

MLs do not account for sample specific matrix effect or sample specific recovery achieved through the analytical work-up. ML reporting limits do account for background detectable in laboratory blanks. ML reporting limit is a recognized convention for compliance monitoring based on Total PCB values determined from summation of detected congeners.

The blank statistics are retained on file. QC blank results are reported to the SDL.

MLs can be implemented in the data edit process by updating the QDL column in the Client Specific PKey (CS-PKey). Before activating the CS-PKey, PM's will send a request to the LIMS group to update the QDL field with the MLs for a specific matrix (W/S/T); the LIMS group will employ "update statements" in the applicable BLK-00# file to update the CS-PKey (LJ#) directly. Project Notes will require data evaluation for all samples (but not the blank) to be the greater of the SDL and QDL.

9.3 Laboratory Method detection limit (MDL) Reporting Limit

Laboratory MDLs are determined as specified by EPA Fed. Reg. 40 CFR Part 136 Appendix B (no iteration option). The 99% confidence level MDL is statistically determined based on analysis of a minimum of 7 replicate matrix spikes fortified at 1-5 times the estimated detection limit. MDLs are an estimate of detection limit determined at a point in time and do not compensate for sample specific matrix effect, sample specific recovery achieved through the analytical work-up; an implicit assumption of the MDL procedure followed is that blank results are centred around zero, and consequently, MDLs may not adequately account for background contribution as evidenced in QC blanks.

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Table 8. Reporting Limit Options - EPA MLs and MDLs, AXYS MLs

PCB Congener	EPA1668C MDL pg (tot)	EPA1668C ML pg (tot)	AXYS Aqueous ML [4] pg (tot)	AXYS Solid ML [4] pg (tot)	AXYS Tissue ML [4] pg (tot)
PCB-1	10	20	4.8	4.3	8.7
PCB-2	7	20	18.0	13.3	4.0
PCB-3	11	50	19.1	6.1	21.8
PCB-4	13	50	5.4	6.4	7.0
PCB-5	8	20	4.1	4.8	4.0
PCB-6	7	20	4.0	4.7	4.0
PCB-7	8	20	26.3	4.7	4.0
PCB-8	15	50	4.5	4.0	4.0
PCB-9	7	20	4.0	4.2	4.0
PCB-10	13	50	4.0	4.1	4.0
PCB-11	34	100	43.4	12.4	24.3
PCB-12/13	19	50	4.1	4.5	4.0
PCB-14	8	20	4.0	4.3	4.0
PCB-15	16	50	5.2	4.4	4.0
PCB-16	9	20	4.0	4.0	4.0
PCB-17	9	20	4.0	4.0	4.0
PCB-30/18	16	50	6.1	4.0	4.0
PCB-19	8	20	4.0	4.0	4.0
PCB-28/20	22	50	10.4	7.4	7.9
PCB-21/33	21	50	4.5	4.0	4.0
PCB-22	9	20	4.0	4.0	4.0
PCB-23	7	20	4.0	4.0	4.0
PCB-24	10	20	4.0	4.0	4.0
PCB-25	8	20	4.0	4.0	4.0
PCB-26/29	12	50	4.0	4.0	4.0
PCB-27	8	20	4.0	4.0	4.0
PCB-31	18	50	7.4	4.0	4.8
PCB-32	8	20	4.0	4.0	4.0
PCB-34	7	20	4.0	4.0	4.0

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PCB Congener	EPA1668C MDL pg (tot)	EPA1668C ML pg (tot)	AXYS Aqueous ML [4] pg (tot)	AXYS Solid ML [4] pg (tot)	AXYS Tissue ML [4] pg (tot)
PCB-35	9	20	4.0	4.0	4.0
PCB-36	8	20	4.0	4.0	4.0
PCB-37	10	20	4.0	4.0	4.0
PCB-38	7	20	4.0	4.0	4.0
PCB-39	8	20	4.0	4.0	4.0
PCB-41/40/71	42	100	4.6	4.0	4.0
PCB-42	16	50	4.0	4.0	4.0
PCB-43	14	50	4.0	4.0	4.0
PCB-44/47/65	40	100	10.0	6.3	6.9
PCB-45/51	22	50	4.0	4.0	4.0
PCB-46	10	20	4.0	4.0	4.0
PCB-48	14	50	4.0	4.0	4.0
PCB-69/49	26	100	4.6	4.0	4.4
PCB-50/53	25	100	4.0	4.0	4.0
PCB-52	15	50	9.3	5.9	8.5
PCB-54	14	50	4.0	4.0	4.0
PCB-55	12	50	4.0	4.0	4.0
PCB-56	15	50	4.0	4.0	4.0
PCB-57	11	50	4.0	4.0	4.0
PCB-58	14	50	4.0	4.0	4.0
PCB-59/62/75	37	100	4.0	4.0	4.0
PCB-60	14	50	4.0	4.0	4.0
PCB-61/70/74/76	59	200	11.0	5.9	8.8
PCB-63	12	50	4.0	4.0	4.0
PCB-64	13	50	4.0	4.0	4.0
PCB-66	17	50	5.1	4.0	4.9
PCB-67	12	50	4.0	4.0	4.0
PCB-68	14	50	4.0	4.0	4.0
PCB-72	13	50	4.0	4.0	4.0
PCB-73	14	50	4.0	4.0	4.0
PCB-77	14	50	4.0	4.0	4.0

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PCB Congener	EPA1668C MDL pg (tot)	EPA1668C ML pg (tot)	AXYS Aqueous ML [4] pg (tot)	AXYS Solid ML [4] pg (tot)	AXYS Tissue ML [4] pg (tot)
PCB-78	16	50	4.0	4.0	4.0
PCB-79	13	50	4.0	4.0	4.0
PCB-80	11	50	4.0	4.0	4.0
PCB-81	18	50	4.0	4.0	4.0
PCB-82	15	50	4.0	4.0	4.0
PCB-83/99	29	100	4.0	4.0	7.8
PCB-84	11	20	4.0	4.0	4.0
PCB-117/116/85	38	100	4.0	4.0	4.0
PCB-109/119/86/97/125/87	74	200	6.0	4.0	5.1
PCB-88/91	22	50	4.0	4.0	4.0
PCB-89	13	50	4.0	4.0	4.0
PCB-113/90/101	47	200	5.9	4.0	9.0
PCB-92	13	50	4.0	4.0	4.0
PCB-95/100/93/102/98	77	200	4.4	4.0	5.7
PCB-94	13	50	4.0	4.0	4.0
PCB-96	15	50	4.0	4.0	4.0
PCB-103	11	50	4.0	4.0	4.0
PCB-104	14	50	4.0	4.0	4.0
PCB-105	17	50	4.0	4.0	4.0
PCB-106	17	50	4.0	4.0	4.0
PCB-108/124	29	100	4.0	4.0	4.0
PCB-107	17	50	4.0	4.0	4.0
PCB-110/115	39	100	6.7	4.6	6.0
PCB-111	14	50	4.0	4.0	4.0
PCB-112	14	50	4.0	4.0	4.0
PCB-114	15	50	4.0	4.0	4.0
PCB-118	30	100	7.9	4.1	7.0
PCB-120	13	50	4.0	4.0	4.0
PCB-121	12	50	4.0	4.0	4.0
PCB-122	12	50	4.0	4.0	4.0
PCB-123	17	50	4.0	4.0	4.0

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PCB Congener	EPA1668C MDL pg (tot)	EPA1668C ML pg (tot)	AXYS Aqueous ML [4] pg (tot)	AXYS Solid ML [4] pg (tot)	AXYS Tissue ML [4] pg (tot)
PCB-126	16	50	4.0	4.0	4.0
PCB-127	14	50	4.0	4.0	4.0
PCB-128/166	29	100	4.0	4.0	4.0
PCB-138/163/129/160	63	200	5.2	5.8	9.0
PCB-130	13	50	4.0	4.0	4.0
PCB-131	17	50	4.0	4.0	4.0
PCB-132	16	50	4.0	4.0	4.0
PCB-133	12	50	4.0	4.0	4.0
PCB-134/143	33	100	4.0	4.0	4.0
PCB-151/135/154	46	100	4.0	4.0	4.0
PCB-136	16	50	4.0	4.0	4.0
PCB-137	15	50	4.0	4.0	4.0
PCB-139/140	29	100	4.0	4.0	4.0
PCB-141	17	50	4.0	4.0	4.0
PCB-142	17	50	4.0	4.0	4.0
PCB-144	15	50	4.0	4.0	4.0
PCB-145	16	50	4.0	4.0	4.0
PCB-146	14	50	4.0	4.0	4.0
PCB-147/149	35	100	4.0	4.0	5.0
PCB-148	14	50	4.0	4.0	4.0
PCB-150	15	50	4.0	4.0	4.0
PCB-152	14	50	4.0	4.0	4.0
PCB-153/168	30	100	4.9	4.5	9.1
PCB-155	14	50	4.0	4.0	4.0
PCB-156/157	23	100	4.0	4.0	4.0
PCB-158	16	50	4.0	4.0	4.0
PCB-159	14	50	4.0	4.0	4.0
PCB-161	13	50	4.0	4.0	4.0
PCB-162	13	50	4.0	4.0	4.0
PCB-164	15	50	4.0	4.0	4.0
PCB-165	13	50	4.0	4.0	4.0

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PCB Congener	EPA1668C MDL pg (tot)	EPA1668C ML pg (tot)	AXYS Aqueous ML [4] pg (tot)	AXYS Solid ML [4] pg (tot)	AXYS Tissue ML [4] pg (tot)
PCB-167	13	50	4.0	4.0	4.0
PCB-169	15	50	4.0	4.0	4.0
PCB-170	12	50	4.0	4.0	4.0
PCB-171/173	30	100	4.0	4.0	4.0
PCB-172	13	50	4.0	4.0	4.0
PCB-174	15	50	4.0	4.0	4.0
PCB-175	14	50	4.0	4.0	4.0
PCB-176	12	50	4.0	4.0	4.0
PCB-177	11	50	4.0	4.0	4.0
PCB-178	14	50	4.0	4.0	4.0
PCB-179	14	50	4.0	4.0	4.0
PCB-180/193	30	100	4.2	4.0	4.0
PCB-181	13	50	4.0	4.0	4.0
PCB-182	13	50	4.0	4.0	4.0
PCB-183/185	28	100	4.0	4.0	4.0
PCB-184	14	50	4.0	4.0	4.0
PCB-186	15	50	4.0	4.0	4.0
PCB-187	17	50	4.0	4.0	4.0
PCB-188	15	50	4.0	4.0	4.0
PCB-189	13	50	4.0	4.0	4.0
PCB-190	14	50	4.0	4.0	4.0
PCB-191	13	50	4.0	4.0	4.0
PCB-192	13	50	4.0	4.0	4.0
PCB-194	18	50	4.0	4.0	4.0
PCB-195	22	50	4.0	4.0	4.0
PCB-196	20	50	4.0	4.0	4.0
PCB-197/200	43	100	4.0	4.0	4.0
PCB-198/199	37	100	4.0	4.0	4.0
PCB-201	20	50	4.0	4.0	4.0
PCB-202	24	100	4.0	4.0	4.0
PCB-203	18	50	4.0	4.0	4.0

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PCB Congener	EPA1668C MDL pg (tot)	EPA1668C ML pg (tot)	AXYS Aqueous ML [4] pg (tot)	AXYS Solid ML [4] pg (tot)	AXYS Tissue ML [4] pg (tot)
PCB-204	21	50	4.0	4.0	4.0
PCB-205	15	50	4.0	4.0	4.0
PCB-206	16	50	4.0	4.0	4.0
PCB-207	19	50	4.0	4.0	4.0
PCB-208	16	50	4.0	4.0	4.8
PCB-209	16	50	4.0	4.0	4.3

[1] CL5-PCB-108/119/86/97/125/87 (EPA 1668A congener naming)

[2] CL5-PCB-107/124 (EPA 1668A congener naming)

[3] CL5-PCB-109 (EPA 1668A congener naming)

[4] AXYS MLs determined according to EPA 1668C, section 17.6.1.4, as the greater of the low calibration equivalent or of the (mean + 2 standard deviations) values of laboratory blank concentrations.

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10. SUMMARY OF MODIFICATIONS TO EPA METHODS 1668A and 1668C

The following sections of EPA Methods 1668A and 1668C have been modified as described below.

Section 4.2.1, 4.2.2: The protocol for washing reusable glassware includes a detergent wash, water rinse and baking at a minimum of 300°C for 8 hours. Immediately prior to use, glassware is solvent rinsed with toluene and hexane.

Section 4.7: The first cleanup column for tissue extracts is a gravity gel permeation column (SX-3 Biobeads). An anthropogenic isolation column 7.5.3 is not used.

Section 6.5.1: Glass wool is cleaned by rinsing twice with toluene and twice with hexane.

Section 7.12, 7.13, 9.0, 11.0: The concentration of the labelled toxics/LOC and the cleanup standard spiking solutions is 100 ng/mL and the sample spiking volume is 20 µL. The resulting final concentrations in the extracts are as specified in the method.

Section 7.14: Concentration of the labelled injection internal standard spiking solution (recovery standard) is modified so that a volume of 5 µL is added. The resulting amount of standard added to the final extract is the same as specified in the method. The solution is spiked into a 15 µL extract volume for a final extract volume of 20 µL.

Section 7.2.1: Sodium sulphate is baked at a minimum of 300°C for 8 hrs.

Section 7.5.1: Silica is activated by baking at 450°C in a muffle oven for at least 8 hrs.

Section 7.5.4.1.1: Florisil is baked at 450°C in a muffle over for at least 8 hrs, then deactivated with water to 2.0% deactivation.

Section 10.3.3, 15.3.3: A S:N ratio of 3:1 for di-PCBs and nona-PCBs in CS0.2 calibration solution is acceptable.

Section 11.5.6: Unless requested by the client, the aqueous portion after filtration of aqueous samples with >1% solids is not discarded but is extracted.

Section 11.5, 11.5.2, 11.5.5, 12.3 Solid samples are dried by mixing with anhydrous sodium sulphate. The dried solid is extracted using a Soxhlet extraction apparatus. The surrogate spike is incorporated after the drying step. Equilibration time for the surrogate is 30 minutes. The extracting solvent for solids is dichloromethane.

Section 12.4, 11.8: The surrogate spike is incorporated into the sample after the drying step to eliminate the possibility of disproportional loss of volatile labelled and target compounds.

Section 12.4.2: The precleaning of the Soxhlet apparatus is carried out using toluene instead of

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dichloromethane, for 2 hours.

Section 12.4.9: Lipid analysis is carried out by sub-sampling two 2 g portions of the extract from a total 30 g extract weight. The cleanup standard is spiked into the extract after Soxhlet extraction and before any lipid analysis or rotary evaporation is done. The percent surrogate recoveries are corrected for the amount of extract used for lipid analysis.

Section 12.6.1.1: Rotary evaporation is done at 30°C. Daily cleaning of the rotary evaporators include dismantling and rinsing/soaking with solvent. Proofs are run periodically but are not archived daily.

Section 12.7.4: Before Florisil or alumina cleanup procedures, a solvent exchange is done by reducing under nitrogen to 300 µL and bulking up to 1mL in hexane. If toluene is present the extract is reduced to 50 µL under nitrogen and bulked up to 1 mL.

Section 12.7.7: Toluene (1 mL) is added to the eluate from the final column prior to rotary evaporation and nitrogen blow down concentration steps.

Section 13.1.1: GPC chromatography, by a gravity column, is routinely used only for tissue extracts. The GPC cleanup is optional for all other matrices.

Section 13.3.1: Routine layered silica column is as follows: 0.5 g neutral silica, 2 g 28% basic silica, 0.5 g neutral silica, 4 g 44% acidic silica, 4 g 22% acidic silica, 1 g neutral silica.

Section 13.3.4: The sample is loaded onto the column followed by 2-3 rinses of a least 1 mL, and eluted with 100 mL of hexane.

Section 14.2: The volume of labelled injection internal standard (recovery standard) added to the extract is 5 µL, for a final extract volume of 20 µL. Hexane rather than nonane is used as the solvent to bring extract back to volume for re-analysis or to dilute extracts.

Section 15.3: The calibration solution containing all 209 PCB congeners is used as the CAL/VER solution.

*Section 17.*5: Extracts are diluted with hexane. The concentration of the labelled injection internal (recovery) standard is not re-adjusted to 100 pg/µL when dilutions are performed.

Section 17.0

$Conc_i$ - the concentrations of target analytes, and the labelled compound concentrations and recoveries, are calculated using the equations below. These procedures are equivalent to those described in the method but are more direct.

$$Conc_i = \frac{A_i}{A_{si}} \times \frac{M_{si}}{RRF_{i,si}} \times \frac{1}{M_x}$$

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where A_i = summed areas of the primary and secondary m/z's for the analyte peak of interest (compound *i*)

A_{si} = summed areas of the primary and secondary m/z's for the labelled surrogate peak used to quantify *i*)

M_x = mass of sample taken for analysis

M_{si} = mass of labelled surrogate (compound *si*) added to sample as calculated by the concentration of standard spiked (pg/mL) multiplied by the volume spiked (mL)

$RRF_{i,si}$ = mean relative response factor of *i* to *si* from the five-point calibration range and defined individually as:

$$\frac{A_i}{A_{si}} \times \frac{M_{si}}{M_i}$$

Calculation of Surrogate Standard Concentrations and Percent Recoveries:

Concentrations of surrogate standards are calculated using the following equation:

$$Conc_{si} = \frac{A_{si}}{A_{rs}} \times \frac{M_{rs}}{RRF_{si,rs}}$$

and, the percent recoveries of the surrogate standards are calculated using the following equation:

$$\%\text{Recovery} = \frac{A_{si}}{A_{rs}} \times \frac{M_{rs}}{RRF_{si,rs}} \times \frac{1}{M_{si}} \times 100$$

where A_{rs} and A_{si} are the summed peak areas (from the primary and secondary m/z channels) of recovery standard and labelled surrogate added to the sample;
M_{rs} and M_{si} are the masses of recovery standard and labelled surrogate added to the sample, and;
$RRF_{si,rs}$ is the mean relative response factor of the labelled surrogate to the recovery standard as determined by the five-point calibration range and defined individually as:

$$\frac{A_{si}}{A_{rs}} \times \frac{M_{rs}}{M_{si}}$$

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APPENDIX A

OPTIONAL ANALYSES

Chlorinated Pesticides

Polybrominated Diphenylethers

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A OPTIONAL ANALYSES

Chlorinated Pesticides

The analysis of chlorinated pesticides may be carried out using the extraction procedures described in Section 5. The analyst is cautioned to avoid the use of concentrated H_2SO_4 during the cleanup procedures as pesticides are destroyed by strong acid.

The analyst is referred to AXYS Method Document MLA-007, *Analytical Method for the Determination of: Aroclors, Total PCBS, Chlorinated Pesticides, PCB Congeners, Coplanar PCBs, Toxaphene, Chlorobenzenes* and AXYS Method Document MLA-028, *Organochlorine Pesticides by Isotope Dilution HRGC/HRMS* for details regarding standard solutions, instrumental analysis, analyte identification and quantification procedures and QA/QC criteria for the analysis of chlorinated pesticides by LRGC/MS and HRGC/MS respectively.

Sample matrices are spiked with the appropriate surrogate for the pesticide analysis, as listed on the Batch List. The choice and quantity of surrogate and recovery standard is dependent upon the instrumental analysis (LRMS or HRMS). The final extract is cleaned up and concentrated using a modified Florisil column procedure as described below.

Florisil Cleanup Procedure

There may be no residual toluene in the extract prior cleanup on Florisil. If toluene is present, carry out a solvent exchange by transferring the extract to a clean centrifuge tube with hexane and concentrating to 50 µL under a gentle stream of nitrogen. Make to 1 mL with hexane.

Quantitatively transfer the extract and hexane rinses (3 x 1 mL from the elution volume) to a Florisil column (8 g, 2.0% deactivated) prepared as described in Section 3.6.4. Elute the column with 15:85 dichloromethane:hexane (E1) followed by dichloromethane, using the volumes of solvent determined from the most recent Florisil cutpoint determination. Typical cutpoints are as follows:

E1	15:85 dichloromethane:hexane	45 mL
E2	Dichloromethane	50 mL.

Concentrate the eluate to 1 mL by rotary evaporation Add activated copper to all E1 extracts to remove sulphur. Transfer the extract to a centrifuge tube with hexane and concentrate to 1 mL in a stream of nitrogen.

The cleaned up extract may be treated in one of two ways:

a) The extract is analyzed for pesticides by either LRGC/MS or HRGC/MS according to procedure described in either MLA-007 or MLA-028 respectively. Once the data has been accepted, the extract is subjected to further cleanup on layered acid/base silica (Section 6.2) followed by alumina (Section 6.4), prior to analysis by HRGC/MS for PCB congeners (Sections 7 through 9).

b) The extract is split into two portions, the proportions as determined by the Project QC Chemist

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and documented on the Batch List. One fraction is analyzed by either LRGC/MS or HRGC/MS according to procedure described in either MLA-007 or MLA-028 respectively. The other fraction is subjected to further cleanup on layered acid/base silica (Section 6.2) followed by alumina (Section 6.4), prior to analysis by HRGC/MS for PCB congeners (Sections 7 through 9).

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Brominated Diphenylethers (PBDE)

The analysis of brominated diphenylethers may be carried out using the extraction procedure as described in Section 5. Sample matrices are spiked with the appropriate surrogate, as listed on the Batch List. The analyst is referred to the most recent version of AXYS Method Document MLA-033, *Analysis Method for the Determination of Polybrominated Diphenylethers by EPA Method 1614* for details regarding special sample handling procedures, standard solutions, chromatographic cleanup, instrumental analysis, analyte identification and quantification procedures and QA/QC criteria.

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APPENDIX B

FRACTIONATION OF PCB CONGENERS

BY

CARBON COLUMNING

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B.1 Fractionation of Toxic PCB Congeners

Fractionation on a carbon column to remove poly-ortho-substituted PCB's as described in Section 6.6 is an option to improve the data quality for certain of the "toxic" group of PCB's. When a carbon column cleanup is required, it must be done after initial analysis on the SPB-Octyl column where the results for all 209 congeners are required.

The decision to perform carbon column cleanup is based on the degree of resolution achieved for PCB congeners 81, 123, 126 and 169 from interfering peaks arising from closely eluting poly-ortho-substituted congeners or fragment peaks from these – notably for PCB-126, due to its high toxicity. The following information is presented as a guideline on when a column carbon cleanup would be beneficial to congener resolution.

The concentrations of PCB-126 from a well-defined shouldering peak (Figure B-1) are compared before and after carbon columning in Table B-1. Table B-2 shows a comparison of results from a less-defined peak, shown in Figure B-2. Shouldering peaks with less definition than Figure B-2 should be confirmed by carbon column and reinjection on a DB-1 GC column.

Carbon column clean-up may be required to adequately resolve all toxic PCB congeners from interferences.

a) Depending upon the particular congener profile carbon column cleanup may be required due to the following chromatographic issues:

PCB 81 shoulders on a fragment peak from PCB 110/115 on the SPB-Octyl column and shoulders on a fragment peak from PCB 86 on the DB-1 column.

PCB 123 shoulders on the PCB 109 peak on both the SPB-Octyl and the DB-1 columns.

PCB 126 shoulders on a fragment peak from PCB 128/166 on the SPB-Octyl column and shoulders or may co-elute with a fragment peak from PCB 129 on the DB-1 column. Quantification of PCB 126 from the DB-1 column without prior carbon column cleanup may lead to false positive detection.

PCB 157 co-elutes with a fragment peak from PCB 202 on the DB-1 column.

PCB 169 co-elutes with a fragment peak from PCB 190/198 on the SPB-Octyl column and shoulders on a fragment peak from PCB 170 on the DB-1 column. Quantification of PCB 169 on the SPB-Octyl column without prior carbon column cleanup may lead to false positive detection. In extreme cases analysis on an SPB-Octyl following carbon column cleanup may still result in false positive detection due to co-elution with a fragment peak from any residual PCB 190/198 not removed by the carbon column.

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b) The following PCB congeners are adequately resolved on the SPB-Octyl column without the need for carbon column cleanup

PCB 77 is resolved adequately on the SPB-Octyl and the DB-1 columns

PCB 105 is resolved adequately on the SPB-Octyl column. It co-elutes with a fragment peak of PCB 132 and shoulders on a fragment peak of PCB 153 on the DB-1 column.

PCB 114 is resolved adequately on the SPB-Octyl column and the DB-1 columns

PCB 118 is resolved adequately on the SPB-Octyl column and co-elutes with PCB 106 on the DB-1 column

PCB 167 is resolved adequately on the SPB-Octyl and the DB-1 columns

PCB 189 is resolved adequately on the SPB-Octyl and the DB-1 columns.

c) The following PCB congener is adequately resolved on the DB-1 column without the need for carbon column cleanup

PCB 156 co-elutes with PCB 157 on the SPB-Octyl column and is resolved adequately on the DB-1 column

Table B-1. Comparison of PCB-126 Results Pre- and Post-Carbon Columning for a Well-defined Peak (see Figure B-1)

Compound	Pre-Carbon Octyl PB21_080 S:22	Pre-Carbon DB1 PB23_102 S:23	Post-Carbon Octyl PB2c_016 S:26
PCB-81	5.4		4.8
PCB-77	6.4		7.4
PCB-123	119		114
PCB-118	7011		7032
PCB-114	126		113
PCB-105	1903		1736
PCB-126	**24**		**29**
PCB-169	ND (8.0)	ND (1.9)	9.8

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Table B-2. Comparison of PCB-126 Results Pre- and Post-Carbon Columning for a Less-defined Peak (see Figure B-2)

Compound	Pre-Carbon Octyl PB21_080 S:27	Pre-Carbon DB1 PB23_102 S:25	POST-Carbon Octyl PB2c_017b S:18
81	2.5		2.5
77	32		36
123	85		74
118	4748		4541
114	87.1		77
105	1272		1128
126	**9.4**		**11**
169	ND (4.9)	ND (.9)	2.9

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Figure B-1: Typical Chromatogram of a Well-defined Shoulder of PCB-126 on SPB-Octyl Column

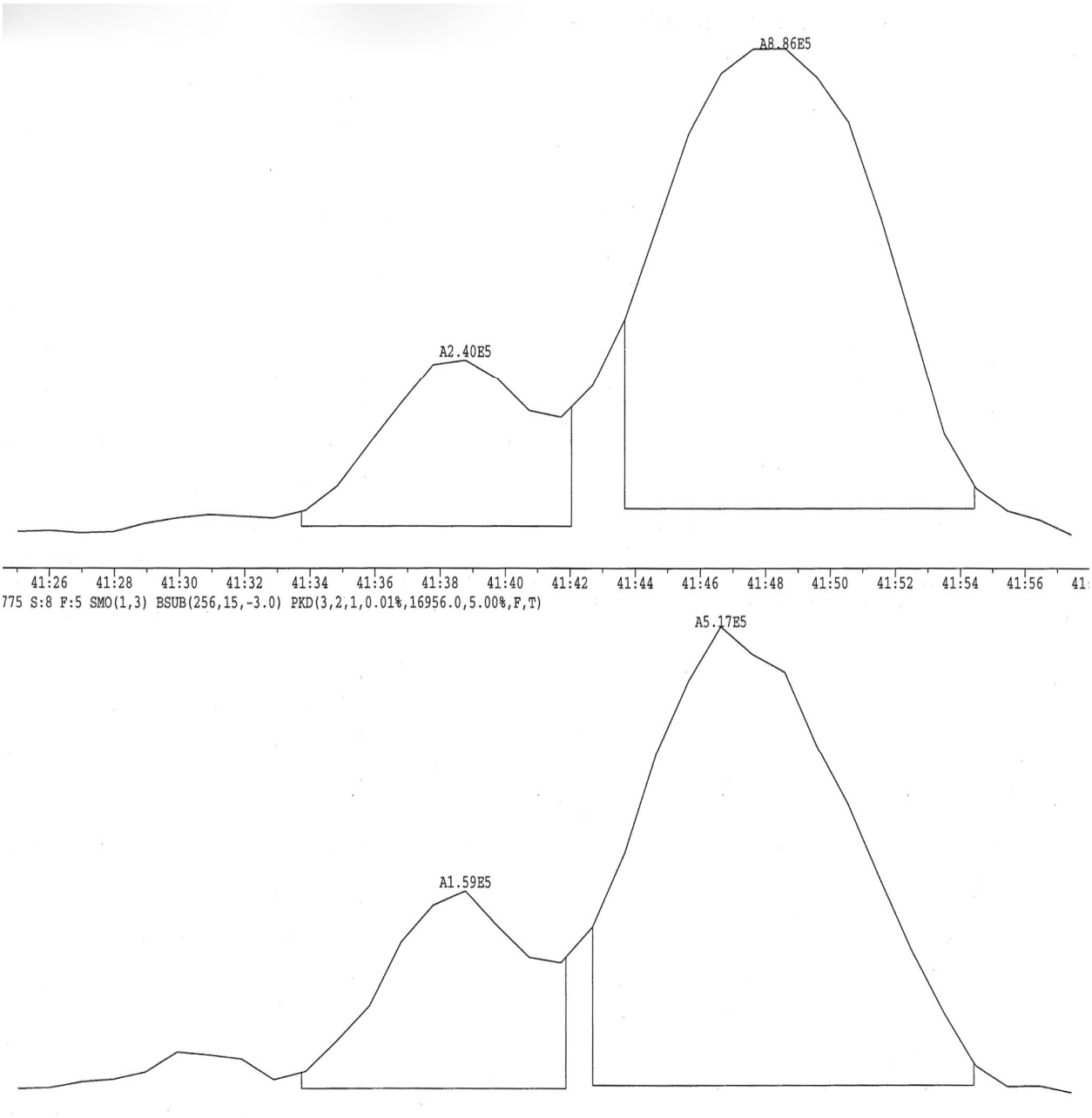

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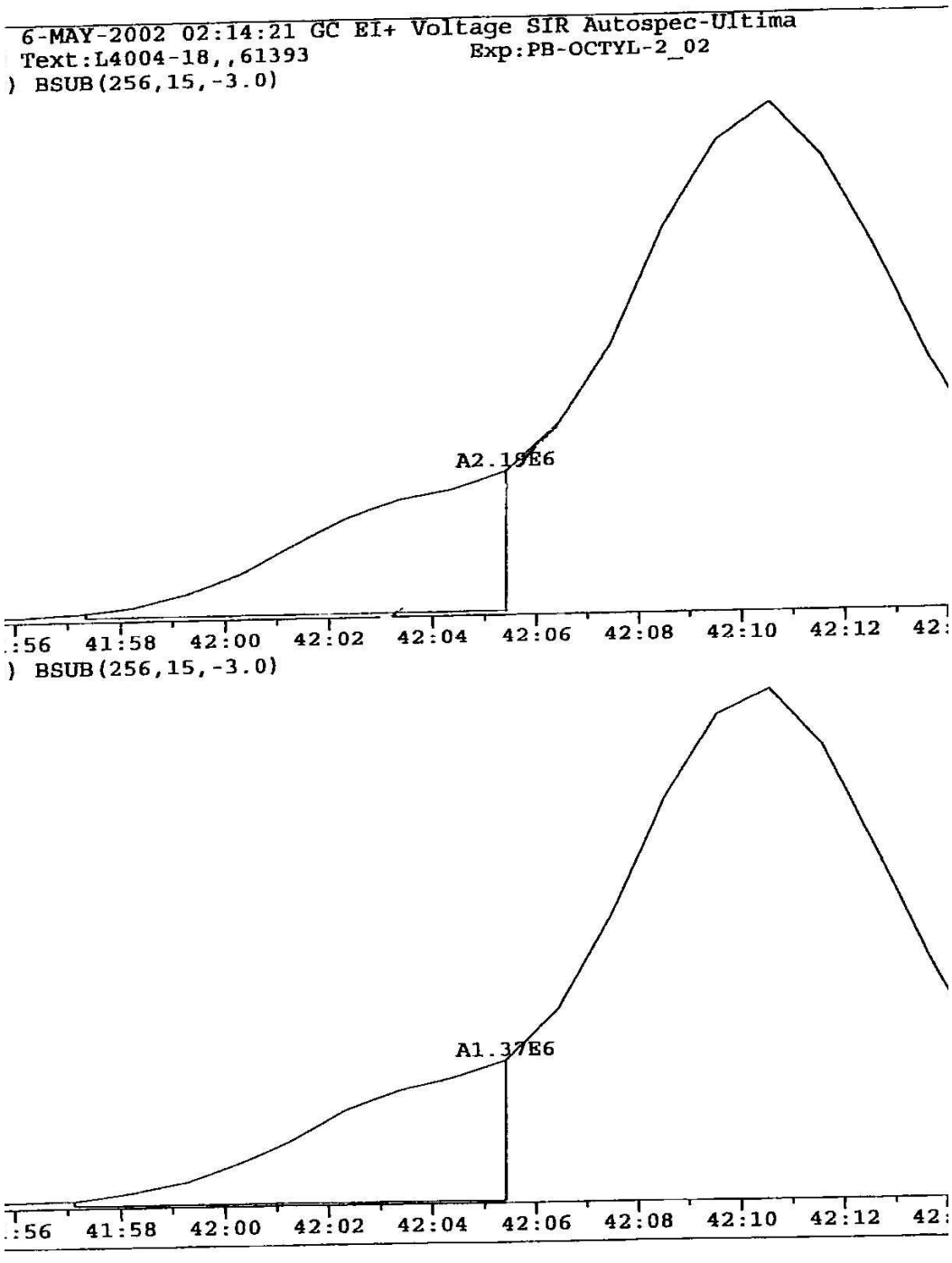
Figure B-2: Typical Chromatogram of a Less-defined Shoulder of PCB-126 on SPB-Octyl Column



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APPENDIX C

DETECTOR 'PHANTOM PEAK' PHENOMENON

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C.1 'Phantom Peak' Phenomenon

The term detector 'phantom peak' phenomenon refers to the appearance of peak response across a wide mass range resulting from high concentration impact on the MS detector, a phenomenon recognized to occur by the instrument manufacturer. While these responses are of very low relative intensity (typically less than 1/1000 of the 'true' response in the mass range of interest) at very high native to surrogate concentration ratios surrogate response may be enhanced significantly. To assess the effect of 'phantom peak' occurrence first determine the response ratio of phantom vs. native congener response from a high concentration native congener for which a labelled surrogate is not present in the extract. Then, for each surrogate compound determine the potential response enhancement by multiplying the response of the associated native congener by the native:phantom ratio. Where the resulting value is greater than 1/10 of the observed surrogate response isotope dilution analysis should not be performed and re-analysis of a smaller sample (or surrogate re-spiking of the extract were allowable) are required.

Example:

The area response for PCB 153 is 2.5E9. An inspection of the chromatogram reveals a response at the mass corresponding to ^{13}C-PCB 153 of 1.2E6 yet the extract has not been spiked with ^{13}C-PCB 153. This native PCB 153: phantom response ratio is 1.2E6/2.3E9=0.0004.

The PCB 118 response is 9.8E8. The estimated phantom ^{13}C-PCB 118 response is therefore 9.8E8 x 0.0004=3.9E5. If this response is less than 1/10 of the observed ^{13}C-PCB 118 response then isotope dilution quantification of PCB 118 can proceed from these data, otherwise, re-analysis of a smaller sample is required.

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APPENDIX D

ANALYSIS OF PCBs BY EPA METHOD CBC01.2

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D. ANALYSIS OF PCB BY EPA METHOD CBC01.2

The analysis of polychlorinated biphenyls by EPA Method CBC01.2 is carried out using the procedures described in this document and according to the following additional protocols. An electronic copy of CBC01.2 is filed on the AXYS network at G:\information\Published Methods\EPA Methods\.

1. Solid samples are extracted using the Soxhlet/Dean-Stark extraction procedure, described in Section 5.2 of this document. This requirement may be waived by contract to permit Soxhlet extraction in dichloromethane (Section 5.1 of this document).
2. The Soxhlet apparatus including thimble must be pre-cleaned for at least **3 hours** by Soxhlet extraction with toluene.
3. Tissue samples are extracted by Soxhlet extraction for 18-24 hours, rather than 16-20 hours.
4. Aqueous samples are extracted by liquid-liquid extraction using separatory funnels, rather that by magnetic stirring extraction.
5. Carbon column isolation of PCBs 77, 126, and 169 is required if detected. This requirement may be waived by contract.
6. Hepta-PCBs are quantified against ^{13}C-PCB 188 and ^{13}C-PCB 189 only.
7. Quantification reports include the signal:noise (S:N) ratio for all analytes and standards.
8. Data reports, sample naming conventions and data flagging protocols are in accordance with the reporting requirements specified in CBC01.2, Exhibit B, Reporting and Deliverables Requirements.

Modifications:

The following modifications have been made to EPA Method CBC01.2.

1. Additional recovery (internal standard) is not added to a diluted extract.
2. The compounds typically present in the Window Defining Mixture (WDM) and in the Combined 209-Congener Standard solution are in a single solution that is analyzed as the CAL/VER solution. Analysis of the CAL/VER solution at least once every 12 hours satisfies the analysis frequency requirement for the WDM.
3. Modifications made to EPA Method 1668C, described in Section 10 of this document, are applicable.

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SUMMARY OF SGS AXYS METHOD MLA-010 REV. 12 VER. 06:

ANALYTICAL METHOD FOR THE DETERMINATION OF 209 PCB CONGENERS BY EPA METHOD 1668A, EPA METHOD 1668C OR EPA METHOD CBC01.2

This analytical method determines the concentrations of all 209 PCB congeners in sediment, soil, sludge, tissue (including blood), aqueous samples, milk, solvent extracts, air samples and XAD-2 columns by high resolution gas chromatography and mass spectrometry (HRGC/HRMS). For EPA Method 1668A applications PCBs are reported using the congener numbering convention used in Method 1668A, for EPA Method 1668C and EPA Method CBC01.2 applications PCBs are reported using the congener numbering convention in EPA Method 1668C.

The concentrations of some PCB congeners are reported as the sum of two or more congeners due to coelution of the congeners. The coeluting congeners are listed below.

COELUTING PCB CONGENERS	
PCB 12, 13	PCB 93, 95, 98, 100, 102
PCB 30, 18	PCB 107, 124 (1668A) or PCB 108, 124 (1668C)
PCB 20, 28	PCB 110, 115
PCB 21, 33	PCB 128, 166
PCB 26, 29	PCB 129, 138, 160, 163
PCB 40, 41, 71	PCB 134, 143
PCB 44, 47, 65	PCB 135, 151, 154
PCB 45, 51	PCB 139, 140
PCB 49, 69	PCB 147, 149
PCB 50, 53	PCB 156, 157[1]
PCB 59, 62, 75	PCB 153, 168
PCB 61, 70, 74, 76	PCB 171, 173
PCB 83, 99	PCB 183,185
PCB 85, 116, 117	PCB 180, 193
PCB 86, 87, 97 108, 119, 125 (1668A) or PCB 86, 87, 97 109, 119, 125 (1668C)	PCB 197, 200
PCB 88, 91	PCB 198, 199
PCB 90, 101, 113	

[1] A second GC/MS analysis on a DB-1 column (30 m, 0.25 mm I.D., 0.25 µm film thickness) resolves the PCB 156/157 coeluting pair. This second analysis is performed upon request by the client.

The concentrations of all other PCB congeners are reported as the concentration of individual congeners.

A subset of congeners designated as “toxic” by the World Health Organization, can be reported with the associated toxic equivalents.

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WHO TOXIC PCB CONGENERS	CONGENER NUMBER
3,4,4',5-Tetrachlorobiphenyl (TeCB)	81
3,3',4,4'-TeCB	77
2,3,3',4,4'-Pentachlorobiphenyl (PeCB)	105
2,3,4,4',5-PeCB	114
2,3',4,4',5-PeCB	118
2',3,4,4',5-PeCB	123
3,3',4,4',5-PeCB	126
2,3,3',4,4',5-Hexachlorobiphenyl (HxCB)	156
2,3,3',4,4',5'-HxCB	157
2,3',4,4',5,5'-HxCB	167
3,3',4,4',5,5'-HxCB	169
2,3,3',4,4',5,5'-HpCB	189

Upon client request, any subset of the 209 PCB congeners may be reported.

1.0 EXTRACTION AND CLEANUP PROCEDURES

All samples are spiked with ^{13}C-labelled surrogate standards prior to extraction and extracted as per the table below. Optional extraction procedures are shown within parentheses.

Matrix	Extraction
Aqueous	≤1% suspended solids: Liquid-liquid extraction with dichloromethane. >1% suspended solids: The sample is centrifuged prior to extraction and the particulate fraction is separately extracted by Soxhlet extraction with dichloromethane. The filtrate is liquid-liquid extracted with dichloromethane. The two extracts are then combined.
Solid (sediment, soil, sludge, particles on filter paper)	Soxhlet extraction with dichloromethane (Optional: Dean-Stark Soxhlet extraction with toluene, or Soxhlet extraction with 1:1 hexane:acetone)
Tissue	Soxhlet extraction with dichloromethane (Optional: Base digestion and liquid-liquid extraction with hexane)
Whole blood/serum/plasma	Liquid-liquid extraction with ethanol:hexane:saturated ammonium sulphate.
Milk	Liquid-liquid extraction with acetone and hexane.

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XAD-2 column and filter	XAD-2 adsorbent is dried and Soxhlet extracted with dichloromethane (optional Dean-Stark Soxhlet extraction with toluene). The filter is extracted by Dean-Stark Soxhlet extraction using toluene.
Ambient air (PUF and filter)	The PUF and filter(s) are Soxhlet extracted together using dichloromethane

Extracts are routinely cleaned up using the following chromatography columns:

Multi-layered Acid/Base Silica
Alumina
Florisil

Extracts may additionally be cleaned up using some or all of the following procedures as necessary:

Column chromatography on Biobeads
Column chromatography on 4.5% carbon/Celite

2.0 INSTRUMENTATION

Instrumental analysis is performed on a SPB-Octyl capillary gas chromatographic column coupled to a high-resolution mass spectrometer (HRMS). Selected PFK ions are used as a reference for mass lock. Two masses from the molecular ion cluster are used to monitor each of the target analytes and ^{13}C-labelled surrogate standards.

Upon client request resolution of the PCB 156/157 co-elution is achieved using a DB-1 chromatography column.

Analysis of the extract on either a DB-1 or DB-5 column is useful to resolve potential interferences with PCB 169 (from PCB 190).

3.0 CALIBRATION

Initial calibration is performed using a 5 or 6 point calibration series of solutions that encompass the working concentration range. Initial calibration solutions contain the suite of labeled surrogate and recovery standards and natives listed in the table below. Calibration procedures use the mean RRFs determined from the initial calibration to calculate analyte concentrations. Calibration is verified at least once every 12 hours by analysis of a mid-level calibration solution. The mid level calibration solution contains 209 PCB congeners and is used to establish the relative response factors and retention times for those PCB congeners not present in the multi-level standards solutions.

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Nominal Concentration of PCB Calibration Solutions

PCB CONGENER	Congener No.	Solution concentration (ng/mL)						
		B CS-0.5 (Hi sens)[1]	C CS-1	D CS-2	E CS-3	F CS-4	G CS-5	H CS-6 (optional)
Native Compounds								
2-MoCB	1	0.5	1.0	5.0	50	400	2000	n.a.
4-MoCB	3	0.5	1.0	5.0	50	400	2000	n.a.
2,2'-DiCB	4	0.5	1.0	5.0	50	400	2000	n.a.
4,4'-DiCB	15	0.5	1.0	5.0	50	400	2000	n.a.
2,2',6-TriCB	19	0.5	1.0	5.0	50	400	2000	6000
2,3,5-TriCB	23	0.5	1.0	5.0	50	400	2000	6000
2',3,5-TriCB	34	0.5	1.0	5.0	50	400	2000	6000
3,4,4'-TriCB	37	0.5	1.0	5.0	50	400	2000	6000
2,2',6,6'-TeCB	54	0.5	1.0	5.0	50	400	2000	6000
3,3',4,4'-TeCB	77	0.5	1.0	5.0	50	400	2000	6000
3,4,4',5-TeCB	81	0.5	1.0	5.0	50	400	2000	6000
2,2',4,6,6'-PeCB	104	0.5	1.0	5.0	50	400	2000	6000
2,3,3',4,4'-PeCB	105	0.5	1.0	5.0	50	400	2000	6000
2,3,4,4',5-PeCB	114	0.5	1.0	5.0	50	400	2000	6000
2,3',4,4',5-PeCB	118	0.5	1.0	5.0	50	400	2000	6000
2',3,4,4',5-PeCB	123	0.5	1.0	5.0	50	400	2000	6000
3,3',4,4',5-PeCB	126	0.5	1.0	5.0	50	400	2000	6000
2,2',4,4',6,6'-HxCB	155	0.5	1.0	5.0	50	400	2000	6000
2,3,3',4,4',5-HxCB	156	0.5	1.0	5.0	50	400	2000	6000
2,3,3',4,4',5'-HxCB	157	0.5	1.0	5.0	50	400	2000	6000
2,3',4,4',5,5'-HxCB	167	0.5	1.0	5.0	50	400	2000	6000
3,3',4,4',5,5'-HxCB	169	0.5	1.0	5.0	50	400	2000	6000
2,2',3,3'4,4',5-HpCB	170	0.5	1.0	5.0	50	400	2000	6000
2,2',3,4,4',5,5'-HpCB	180	0.5	1.0	5.0	50	400	2000	6000

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PCB CONGENER	Congener No.	Solution concentration (ng/mL)						
		B CS-0.5 (Hi sens)[1]	C CS-1	D CS-2	E CS-3	F CS-4	G CS-5	H CS-6 (optional)
2,2',3,4,4',5,6'-HpCB	182	0.5	1.0	5.0	50	400	2000	6000
2,2',3,4',5,5',6-HpCB	187	0.5	1.0	5.0	50	400	2000	6000
2,2',3,4',5,6,6'-HpCB	188	0.5	1.0	5.0	50	400	2000	6000
2,3,3',4,4',5,5'-HpCB	189	0.5	1.0	5.0	50	400	2000	6000
2,2',3,3',5,5',6,6'-OcCB	202	0.5	1.0	5.0	50	400	2000	6000
2,3,3',4,4',5,5',6-OcCB	205	0.5	1.0	5.0	50	400	2000	6000
2,2',3,3',4,4',5,5',6-NoCB	206	0.5	1.0	5.0	50	400	2000	6000
2,2',3,3',4,5,5',6,6'-NoCB	208	0.5	1.0	5.0	50	400	2000	6000
2,2',3,3',4,4',5,5',6,6'-DeCB	209	0.5	1.0	5.0	50	400	2000	6000
Surrogate Standards								
$^{13}C_{12}$-2-MoCB	1L[2]	100	100	100	100	100	100	100
$^{13}C_{12}$-4-MoCB	3L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2'-DiCB	4L	100	100	100	100	100	100	100
$^{13}C_{12}$-4,4'-DiCB	15L	100	100	100	100	100	100	100
$^{13}C_{12}$-3,4,4'-TriCB	37L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',6,6'-TeCB	54L	100	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4'-TeCB	77L	100	100	100	100	100	100	100
$^{13}C_{12}$-3,4,4',5-TeCB	81L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',4,6,6'-PeCB	104L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4'-PeCB	105L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,3,4,4',5-PeCB	114L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,3',4,4',5-PeCB	118L	100	100	100	100	100	100	100
$^{13}C_{12}$-2',3,4,4',5-PeCB	123L	100	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4',5-PeCB	126L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',4,4',6,6'-HxCB	155L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5-HxCB	156L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5'-HxCB	157L	100	100	100	100	100	100	100

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PCB CONGENER	Congener No.	Solution concentration (ng/mL)						
		B CS-0.5 (Hi sens)[1]	C CS-1	D CS-2	E CS-3	F CS-4	G CS-5	H CS-6 (optional)
$^{13}C_{12}$-2,3',4,4',5,5'-HxCB	167L	100	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4',5,5'-HxCB	169L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5-HpCB	170L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,4,4',5,5'-HpCB	180L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB	188L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5,5'-HpCB	189L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB	202L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB	205L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB	206L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-NoCB	208L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-DeCB	209L	100	100	100	100	100	100	100
Cleanup Standards								
$^{13}C_{12}$-2,4,4'-TriCB	28L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',5,5'-PeCB	111L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB	178L	100	100	100	100	100	100	100
Recovery Standards								
$^{13}C_{12}$-2,5-DiCB	9L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',5,5'-TeCB	52L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',4',5,5'-PeCB	101L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3',4,4',5'-HxCB	138L	100	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5,5'-OcCB	194L	100	100	100	100	100	100	100

[1] Additional concentration used for calibration of high sensitivity HRGC/HRMS systems.
[2] Suffix "L" indicates labeled compound.

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4.0 QUANTIFICATION PROCEDURES

Analyte Identification

A chromatographic peak is identified as a target compound if the following criteria are met for the quantification and confirmation ions (where confirmation ions are available):

- Peak responses must be at least 2.5 times the background noise level.
- All peaks are acquired in the appropriate acquisition windows, when compared to the window standard.
- Peak centroids for the quantification and confirmation ions must coincide within two seconds.
- The relative ion abundance ratios must be within 15% of the expected ratio.

Calculations

Refer to Section 17.0 of EPA Method 1668A or 1668C for details of quantitative determination.

Aroclor equivalent concentrations may be calculated by converting the summed concentrations of a suite of characteristic PCB congeners to concentrations using empirical factors determined from the analysis of Aroclor mixtures.

Aroclor 1016[1] =	The sum of PCBs 8, 18/30, 31, 28/20 concentrations multiplied by 2.7;
Aroclor 1221 =	The sum of PCBs 1, 3, 8 concentrations multiplied by 1.4;
Aroclor 1232 =	The sum of PCBs 1, 3, 18/30 concentrations multiplied by 3.4;
Aroclor 1242[1] =	The sum of PCBs 8, 18/30, 31, 28/20 concentrations multiplied by 3.0;
Aroclor 1248 =	The sum of PCBs 44/47/65, 49/69, 66 concentrations multiplied by 6.1;
Aroclor 1254 =	The sum of PCBs 86/87/97/**108**/119/125, 83/99 concentrations multiplied by 8.0 (for EPA 1668A); OR The sum of PCBs 86/87/97/**109**/119/125, 83/99 concentrations multiplied by 8.0 (for EPA 1668C);
Aroclor 1260 =	The sum of PCBs 183/185, 180/193, 170 concentrations multiplied by 5.0;
Aroclor 1262 =	The sum of PCBs 183/185, 180/193, 170 concentrations multiplied by 5.3;
Aroclor 1268 =	The sum of PCBs 198/199, 206, 208, 209 concentrations multiplied by 1.3;

Environmental samples with no clearly identified Aroclor signature are quantified as 1242/1254/1260 mixtures. Results may be reported as Aroclor 1248 instead of Aroclor 1242 and 1254 where the congener pattern clearly indicates this formulation. Other Aroclor formulations may be reported by calibration against the specific Aroclor solutions.

[1] Aroclors 1016 and 1242 may be reported as combined 1016/1242 using the 1242 factor

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Analyte Retention Times, Surrogates Used and RRT Windows for 209 PCB's by HRGC/MS

COMPOUND	Congener Number	CAS NO.	QUANTIFICATION REFERENCE	CO-ELUTIONS	COMPOUND RT	RT Reference	Labeled RT	RRT	RT Window (sec)	RRT Lower Limit	RRT Upper Limit
2 - MoCB	1	2051-60-7	1L		11:34:00	1L	11:34:00	1.000	-1,3	0.999	1.004
3 - MoCB	2	2051-61-8	1L/3L		13:38:00	3L	13:48:00	0.988	6	0.984	0.992
4 - MoCB	3	2051-62-9	3L		13:49:00	3L	13:48:00	1.001	-1,3	0.999	1.004
22' - DiCB	4	13029-08-8	4L		14:04:00	4L	14:03:00	1.001	-1,3	0.999	1.004
26 - DiCB	10	33146-45-1	4L/15L		14:14:00	4L	14:03:00	1.013	6	1.009	1.017
25 - DiCB	9	34883-39-1	4L/15L		16:06:00	4L	14:03:00	1.146	6	1.142	1.149
24 - DiCB	7	33284-50-3	4L/15L		16:16:00	4L	14:03:00	1.158	6	1.154	1.161
23' - DiCB	6	25569-80-6	4L/15L		16:31:00	4L	14:03:00	1.176	6	1.172	1.179
23 - DiCB	5	16605-91-7	4L/15L		16:49:00	4L	14:03:00	1.197	6	1.193	1.200
24' - DiCB	8	34883-43-7	4L/15L		16:58:00	4L	14:03:00	1.208	6	1.204	1.211
35 - DiCB	14	34883-41-5	4L/15L		18:39:00	15L	20:08:00	0.926	6	0.924	0.929
33' - DiCB	11	2050-67-1	4L/15L		19:32:00	15L	20:08:00	0.970	6	0.968	0.973
34' - DiCB	13	2974-90-5	4L/15L	12 + 13							
34 - DiCB	12	2974-92-7	4L/15L	12 + 13	19:51:00	15L	20:08:00	0.986	6	0.983	0.988
44' - DiCB	15	2050-68-2	15L		20:09:00	15L	20:08:00	1.001	-1,3	0.999	1.002
22'6 - TriCB	19	38444-73-4	19L		17:15:00	19L	17:13:00	1.002	-1,3	0.999	1.003
246 - TriCB	30	35693-92-6	19L/37L	18 + 30							
22'5 - TriCB	18	37680-65-2	19L/37L	18 + 30	19:09:00	19L	17:13:00	1.112	6	1.109	1.115
22'4 - TriCB	17	37680-66-3	19L/37L		19:36:00	19L	17:13:00	1.138	6	1.136	1.141
23'6 - TriCB	27	38444-76-7	19L/37L		19:50:00	19L	17:13:00	1.152	6	1.149	1.155
236 - TriCB	24	55702-45-9	19L/37L		19:58:00	19L	17:13:00	1.160	6	1.157	1.163
22'3 - TriCB	16	38444-78-9	19L/37L		20:05:00	19L	17:13:00	1.167	6	1.164	1.169
24'6 - TriCB	32	38444-77-8	19L/37L		20:37:00	19L	17:13:00	1.197	6	1.195	1.200
2'35 - TriCB	34	37680-68-5	19L/37L		21:56:00	19L	17:13:00	1.274	6	1.271	1.277
235 - TriCB	23	55720-44-0	19L/37L		22:06:00	19L	17:13:00	1.284	6	1.281	1.287
245 - TriCB	29	15862-07-4	19L/37L	26 + 29							
23'5 - TriCB	26	38444-81-4	19L/37L	26 + 29	22:26:00	19L	17:13:00	1.303	10	1.298	1.308
23'4 - TriCB	25	55712-37-3	19L/37L		22:40:00	37L	27:27:00	0.826	6	0.824	0.828
24'5 - TriCB	31	16606-02-3	19L/37L		22:59:00	37L	27:27:00	0.837	6	0.835	0.839

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COMPOUND	Congener Number	CAS NO.	QUANTIFICATION REFERENCE	CO-ELUTIONS	COMPOUND RT	RT Reference	Labeled RT	RRT	RT Window (sec)	RRT Lower Limit	RRT Upper Limit
244' - TriCB	28	7012-37-5	19L/37L	20 + 28							
233' - TriCB	20	38444-84-7	19L/37L	20 + 28	23:18:00	37L	27:27:00	0.849	10	0.846	0.852
234 - TriCB	21	55702-46-0	19L/37L	21 + 33	23:30:00	37L	27:27:00	0.856	10	0.853	0.859
2'34 - TriCB	33	38444-86-9	19L/37L	21 + 33							
234' - TriCB	22	38444-85-8	19L/37L		23:57:00	37L	27:27:00	0.872	6	0.871	0.874
33'5 - TriCB	36	38444-87-0	19L/37L		25:35:00	37L	27:27:00	0.932	6	0.930	0.934
34'5 - TriCB	39	38444-88-1	19L/37L		25:58:00	37L	27:27:00	0.946	6	0.944	0.948
345 - TriCB	38	53555-66-1	19L/37L		26:33:00	37L	27:27:00	0.967	6	0.965	0.969
33'4 - TriCB	35	37680-69-6	19L/37L		27:03:00	37L	27:27:00	0.985	6	0.984	0.987
344' - TriCB	37	38444-90-5	37L		27:28:00	37L	27:27:00	1.001	-1,3	0.999	1.002
22'66' - TeCB	54	15968-05-5	54L		20:25:00	54L	20:25:00	1.000	-1,3	0.999	1.002
22'46 - TeCB	50	62796-65-0	54L/81L/77L	50 + 53	22:41:00	54L	20:25:00	1.111	10	1.107	1.115
22'56' - TeCB	53	41464-41-9	54L/81L/77L	50 + 53							
22'36 - TeCB	45	70362-45-7	54L/81L/77L	45 + 51	23:24:00	54L	20:25:00	1.146	10	1.142	1.150
22'46' - TeCB	51	68194-04-7	54L/81L/77L	45 + 51							
22'36' - TeCB	46	41464-47-5	54L/81L/77L		23:41:00	54L	20:25:00	1.160	6	1.158	1.162
22'55' - TeCB	52	35693-99-3	54L/81L/77L		25:11:00	54L	20:25:00	1.233	6	1.231	1.236
23'5'6 - TeCB	73	74338-23-1	54L/81L/77L		25:20:00	54L	20:25:00	1.241	6	1.238	1.243
22'35 - TeCB	43	70362-46-8	54L/81L/77L		25:26:00	54L	20:25:00	1.246	6	1.243	1.248
23'46 - TeCB	69	60233-24-1	54L/81L/77L	49 + 69							
22'45' - TeCB	49	41464-40-8	54L/81L/77L	49 + 69	25:40:00	54L	20:25:00	1.257	10	1.253	1.261
22'45 - TeCB	48	70362-47-9	54L/81L/77L		25:59:00	54L	20:25:00	1.273	6	1.270	1.275
2356 - TeCB	65	33284-54-7	54L/81L/77L	44 + 47 + 65							
22'44' - TeCB	47	2437-79-8	54L/81L/77L	44 + 47 + 65							
22'35' - TeCB	44	41464-39-5	54L/81L/77L	44 + 47 + 65	26:14:00	54L	20:25:00	1.285	10	1.281	1.289
2346 - TeCB	62	54230-22-7	54L/81L/77L	59 + 62 + 75							
244'6 - TeCB	75	32598-12-2	54L/81L/77L	59 + 62 + 75							
233'6 - TeCB	59	74472-33-6	54L/81L/77L	59 + 62 + 75	26:34:00	54L	20:25:00	1.301	10	1.297	1.305
22'34' - TeCB	42	36559-22-5	54L/81L/77L		26:45:00	54L	20:25:00	1.310	6	1.308	1.313
22'34 - TeCB	41	52663-59-9	54L/81L/77L	40 + 41 + 71							
23'4'6 - TeCB	71	41464-46-4	54L/81L/77L	40 + 41 + 71							
22'33' - TeCB	40	38444-93-8	54L/81L/77L	40 + 41 + 71	27:14:00	54L	20:25:00	1.334	10	1.330	1.338

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COMPOUND	Congener Number	CAS NO.	QUANTIFICATION REFERENCE	CO-ELUTIONS	COMPOUND RT	RT Reference	Labeled RT	RRT	RT Window (sec)	RRT Lower Limit	RRT Upper Limit
234'6 - TeCB	64	52663-58-8	54L/81L/77L		27:31:00	54L	20:25:00	1.348	6	1.345	1.350
23'55' - TeCB	72	41464-42-0	54L/81L/77L		28:24:00	81L	34:31:00	0.823	6	0.821	0.824
23'45' - TeCB	68	73575-52-7	54L/81L/77L		28:42:00	81L	34:31:00	0.831	6	0.830	0.833
233'5 - TeCB	57	70424-67-8	54L/81L/77L		29:09:00	81L	34:31:00	0.845	6	0.843	0.846
233'5' - TeCB	58	41464-49-7	54L/81L/77L		29:23:00	81L	34:31:00	0.851	6	0.850	0.853
23'45 - TeCB	67	73575-53-8	54L/81L/77L		29:34:00	81L	34:31:00	0.857	6	0.855	0.858
234'5 - TeCB	63	74472-34-7	54L/81L/77L		29:50:00	81L	34:31:00	0.864	6	0.863	0.866
2345 - TeCB	61	33284-53-6	54L/81L/77L	61 + 70 + 74 + 76	30:11:00	81L	34:31:00	0.874	12	0.872	0.877
23'4'5 - TeCB	70	32598-11-1	54L/81L/77L	61 + 70 + 74 + 76							
2'345 - TeCB	76	70362-48-0	54L/81L/77L	61 + 70 + 74 + 76							
244'5 - TeCB	74	32690-93-0	54L/81L/77L	61 + 70 + 74 + 76							
23'44' - TeCB	66	32598-10-0	54L/81L/77L		30:32:00	81L	34:31:00	0.885	6	0.883	0.886
233'4 - TeCB	55	74338-24-2	54L/81L/77L		30:41:00	81L	34:31:00	0.889	6	0.887	0.890
233'4' - TeCB	56	41464-43-1	54L/81L/77L		31:13:00	81L	34:31:00	0.904	6	0.903	0.906
2344' - TeCB	60	33025-41-1	54L/81L/77L		31:27:00	81L	34:31:00	0.911	6	0.910	0.913
33'55' - TeCB	80	33284-52-5	54L/81L/77L		31:54:00	81L	34:31:00	0.924	6	0.923	0.926
33'45' - TeCB	79	41464-48-6	54L/81L/77L		33:29:00	81L	34:31:00	0.970	6	0.969	0.972
33'45 - TeCB	78	70362-49-1	54L/81L/77L		34:05:00	81L	34:31:00	0.987	6	0.986	0.989
344'5 - TeCB	81	70362-50-4	81L		34:32:00	81L	34:31:00	1.000	-1,3	1.000	1.001
33'44' - TeCB	77	32598-13-3	77L		35:08:00	77L	35:07:00	1.000	-1,3	1.000	1.001
22'466' - PeCB	104	56558-16-8	104L		26:10:00	104L	26:08:00	1.001	-1,3	0.999	1.002
22'366' - PeCB	96	73575-54-9	104L/123L/114L/ 118L/105L		26:32:00	104L	26:08:00	1.015	10	1.012	1.018
22'45'6 - PeCB	103	60145-21-3	104L/123L/114L/ 118L/105L		28:34:00	104L	26:08:00	1.093	6	1.091	1.095
22'356' - PeCB	94	73575-55-0	104L/123L/114L/ 118L/105L		28:48:00	104L	26:08:00	1.102	6	1.100	1.104
22'35'6 - PeCB	95	38379-99-6	104L/123L/114L/ 118L/105L	93 + 95 + 98 + 100 + 102							
22'44'6 - PeCB	100	39485-83-1	104L/123L/114L/ 118L/105L	93 + 95 + 98 + 100 + 102							
22'356 - PeCB	93	73575-56-1	104L/123L/114L/ 118L/105L	93 + 95 + 98 + 100 + 102	29:31:00	104L	26:08:00	1.129	34 *	1.123	1.136
22'456' - PeCB	102	68194-06-9	104L/123L/114L/ 118L/105L	93 + 95 + 98 + 100 + 102							

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COMPOUND	Congener Number	CAS NO.	QUANTIFICATION REFERENCE	CO-ELUTIONS	COMPOUND RT	RT Reference	Labeled RT	RRT	RT Window (sec)	RRT Lower Limit	RRT Upper Limit
22'3'46 - PeCB	98	60233-25-2	104L/123L/114L/ 118L/105L	93 + 95 + 98 + 100 + 102							
22'346 - PeCB	88	55215-17-3	104L/123L/114L/ 118L/105L	88 + 91	30:07:00	104L	26:08:00	1.152	12	1.149	1.156
22'34'6 - PeCB	91	68194-05-8	104L/123L/114L/ 118L/105L	88 + 91							
22'33'6 - PeCB	84	52663-60-2	104L/123L/114L/ 118L/105L		30:23:00	104L	26:08:00	1.163	6	1.161	1.165
22'346' - PeCB	89	73575-57-2	104L/123L/114L/ 118L/105L		30:53:00	104L	26:08:00	1.182	6	1.180	1.184
23'45'6 - PeCB	121	56558-18-0	104L/123L/114L/ 118L/105L		31:20:00	104L	26:08:00	1.199	6	1.197	1.201
22'355' - PeCB	92	52663-61-3	104L/123L/114L/ 118L/105L		31:44:00	123L	37:12:00	0.853	6	0.852	0.854
233'5'6 - PeCB	113	68194-10-5	104L/123L/114L/ 118L/105L	90 + 101 + 113							
22'34'5 - PeCB	90	68194-07-0	104L/123L/114L/ 118L/105L	90 + 101 + 113	32:19:00	123L	37:12:00	0.869	10	0.866	0.871
22'455' - PeCB	101	37680-73-2	104L/123L/114L/ 118L/105L	90 + 101 + 113							
22'33'5 - PeCB	83	60145-20-2	104L/123L/114L/ 118L/105L	83 + 99	32:53:00	123L	37:12:00	0.884	12	0.881	0.887
22'44'5 - PeCB	99	38380-01-7	104L/123L/114L/ 118L/105L	83 + 99							
233'56 - PeCB	112	74472-36-9	104L/123L/114L/ 118L/105L		33:04:00	123L	37:12:00	0.889	6	0.888	0.890
23'44'6 - PeCB											
233'45' - PeCB											
22'345 - PeCB	See Tables 6b and 6c for information specific to EPA 1668A or 1668C										
22'3'45 - PeCB											
2'3456' - PeCB											
22'345' - PeCB											
234'56 - PeCB	117	68194-11-6	104L/123L/114L/ 118L/105L	85 + 116 + 117							
23456 - PeCB	116	18259-05-7	104L/123L/114L/ 118L/105L	85 + 116 + 117							
22'344' - PeCB	85	65510-45-4	104L/123L/114L/ 118L/105L	85 + 116 + 117	34:12:00	123L	37:12:00	0.919	12	0.917	0.922

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COMPOUND	Congener Number	CAS NO.	QUANTIFICATION REFERENCE	CO-ELUTIONS	COMPOUND RT	RT Reference	Labeled RT	RRT	RT Window (sec)	RRT Lower Limit	RRT Upper Limit
233'4'6 - PeCB	110	38380-03-9	104L/123L/114L/ 118L/105L	110 + 115	34:27:00	123L	37:12:00	0.926	10	0.924	0.928
2344'6 - PeCB	115	74472-38-1	104L/123L/114L/ 118L/105L	110 + 115							
22'33'4 - PeCB	82	52663-62-4	104L/123L/114L/ 118L/105L		34:43:00	123L	37:12:00	0.933	6	0.932	0.935
233'55' - PeCB	111	39635-32-0	104L/123L/114L/ 118L/105L		35:10:00	123L	37:12:00	0.945	6	0.944	0.947
23'455' - PeCB	120	68194-12-7	104L/123L/114L/ 118L/105L		35:38:00	123L	37:12:00	0.958	6	0.957	0.959
233'4'5 - PeCB											
2'3455' - PeCB	See Tables 6b and 6c for information specific to EPA 1668A or 1668C										
233'46 - PeCB											
2'344'5 - PeCB	123	65510-44-3	123L		37:13:00	123L	37:12:00	1.000	-1,3	1.000	1.001
233'45 - PeCB	106	70424-69-0	104L/123L/114L/ 118L/105L		37:21:00	123L	37:12:00	1.004	6	1.003	1.005
23'44'5 - PeCB	118	31508-00-6	118L		37:34:00	118L	37:32:00	1.001	-1,3	1.000	1.002
2'33'45 - PeCB	122	76842-07-4	104L/123L/114L/ 118L/105L		37:55:00	118L	37:32:00	1.010	6	1.009	1.012
2344'5 - PeCB	114	74472-37-0	114L		38:07:00	114L	38:06:00	1.000	-1,3	1.000	1.001
233'44' - PeCB	105	32598-14-4	105L		38:48:00	105L	38:46:00	1.001	-1,3	0.999	1.001
33'455' - PeCB	127	39635-33-1	104L/123L/114L/ 118L/105L		40:21:00	105L	38:46:00	1.041	6	1.040	1.042
33'44'5 - PeCB	126	57465-28-8	126L		42:02:00	126L	42:01:00	1.000	-1,3	1.000	1.001
22'44'66' - HxCB	155	33979-03-2	155L		32:06:00	155L	32:05:00	1.001	-1,3	0.999	1.002
22'3566' - HxCB	152	68194-09-2	155L/156L/157L/ 167L/169L		32:17:00	155L	32:05:00	1.006	6	1.005	1.008
22'34'66' - HxCB	150	68194-08-1	155L/156L/157L/ 167L/169L		32:28:00	155L	32:05:00	1.012	6	1.010	1.014
22'33'66' - HxCB	136	38411-22-2	155L/156L/157L/ 167L/169L		32:51:00	155L	32:05:00	1.024	6	1.022	1.025
22'3466' - HxCB	145	74472-40-5	155L/156L/157L/ 167L/169L		33:10:00	155L	32:05:00	1.034	6	1.032	1.035
22'34'56' - HxCB	148	74472-41-6	155L/156L/157L/ 167L/169L		34:45:00	155L	32:05:00	1.083	6	1.082	1.085
22'355'6 - HxCB	151	52663-63-5	155L/156L/157L/ 167L/169L	135 + 151 + 154							
22'33'56' - HxCB	135	52744-13-5	155L/156L/157L/	135 + 151 + 154	35:29:00	155L	32:05:00	1.106	22 *	1.103	1.109

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COMPOUND	Congener Number	CAS NO.	QUANTIFICATION REFERENCE	CO-ELUTIONS	COMPOUND RT	RT Reference	Labeled RT	RRT	RT Window (sec)	RRT Lower Limit	RRT Upper Limit
22'44'5'6 - HxCB	154	60145-22-4	167L/169L 155L/156L/157L/ 167L/169L	135 + 151 + 154							
22'345'6 - HxCB	144	68194-14-9	155L/156L/157L/ 167L/169L		35:57:00	155L	32:05:00	1.121	6	1.119	1.122
22'34'56 - HxCB	147	68194-13-8	155L/156L/157L/ 167L/169L	147 + 149	36:20:00	155L	32:05:00	1.132	10	1.130	1.135
22'34'5'6 - HxCB	149	38380-04-0	155L/156L/157L/ 167L/169L	147 + 149							
22'33'56 - HxCB	134	52704-70-8	155L/156L/157L/ 167L/169L	134 + 143	36:36:00	155L	32:05:00	1.141	10	1.138	1.143
22'3456' - HxCB	143	68194-15-0	155L/156L/157L/ 167L/169L	134 + 143							
22'344'6 - HxCB	139	56030-56-9	155L/156L/157L/ 167L/169L	139 + 140	36:58:00	155L	32:05:00	1.152	10	1.150	1.155
22'344'6' - HxCB	140	59291-64-4	155L/156L/157L/ 167L/169L	139 + 140							
22'33'46 - HxCB	131	61798-70-7	155L/156L/157L/ 167L/169L		37:11:00	155L	32:05:00	1.159	6	1.157	1.161
22'3456 - HxCB	142	41411-61-4	155L/156L/157L/ 167L/169L		37:20:00	155L	32:05:00	1.164	6	1.162	1.165
22'33'46' - HxCB	132	38380-05-1	155L/156L/157L/ 167L/169L		37:39:00	155L	32:05:00	1.174	10	1.171	1.176
22'33'55' - HxCB	133	35694-04-3	155L/156L/157L/ 167L/169L		38:11:00	155L	32:05:00	1.190	6	1.189	1.192
233'55'6 - HxCB	165	74472-46-1	155L/156L/157L/ 167L/169L		38:37:00	167L	43:57:00	0.879	6	0.878	0.880
22'34'55' - HxCB	146	51908-16-8	155L/156L/157L/ 167L/169L		38:52:00	167L	43:57:00	0.884	6	0.883	0.885
233'45'6 - HxCB	161	74472-43-8	155L/156L/157L/ 167L/169L		39:01:00	167L	43:57:00	0.888	6	0.887	0.889
22'44'55' - HxCB	153	35065-27-1	155L/156L/157L/ 167L/169L	153 + 168	39:32:00	167L	43:57:00	0.900	10	0.898	0.901
23'44'5'6 - HxCB	168	59291-65-5	155L/156L/157L/ 167L/169L	153 + 168							
22'3455' - HxCB	141	52712-04-6	155L/156L/157L/ 167L/169L		39:43:00	167L	43:57:00	0.904	6	0.903	0.905
22'33'45' - HxCB	130	52663-66-8	155L/156L/157L/ 167L/169L		40:08:00	167L	43:57:00	0.913	6	0.912	0.914

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COMPOUND	Congener Number	CAS NO.	QUANTIFICATION REFERENCE	CO-ELUTIONS	COMPOUND RT	RT Reference	Labeled RT	RRT	RT Window (sec)	RRT Lower Limit	RRT Upper Limit
22'344'5 - HxCB	137	35694-06-5	155L/156L/157L/ 167L/169L		40:22:00	167L	43:57:00	0.918	6	0.917	0.920
233'4'5'6 - HxCB	164	74472-45-0	155L/156L/157L/ 167L/169L		40:29:00	167L	43:57:00	0.921	6	0.920	0.922
22'344'5' - HxCB	138	35065-28-2	155L/156L/157L/ 167L/169L	129 + 138 + 160 + 163							
233'4'56 - HxCB	163	74472-44-9	155L/156L/157L/ 167L/169L	129 + 138 + 160 + 163							
22'33'45 - HxCB	129	55215-18-4	155L/156L/157L/ 167L/169L	129 + 138 + 160 + 163	40:53:00	167L	43:57:00	0.930	14	0.928	0.933
233'456 - HxCB	160	41411-62-5	155L/156L/157L/ 167L/169L	129 + 138 + 160 + 163							
233'44'6 - HxCB	158	74472-42-7	155L/156L/157L/ 167L/169L		41:13:00	167L	43:57:00	0.938	6	0.937	0.939
2344'56 - HxCB	166	41411-63-6	155L/156L/157L/ 167L/169L	128 + 166							
22'33'44' - HxCB	128	38380-07-3	155L/156L/157L/ 167L/169L	128 + 166	42:08:00	167L	43:57:00	0.959	10	0.957	0.961
233'455' - HxCB	159	39635-35-3	155L/156L/157L/ 167L/169L		43:10:00	167L	43:57:00	0.982	6	0.981	0.983
233'4'55' - HxCB	162	39635-34-2	155L/156L/157L/ 167L/169L		43:28:00	167L	43:57:00	0.989	6	0.988	0.990
23'44'55' - HxCB	167	52663-72-6	167L		43:59:00	167L	43:57:00	1.001	-1,3	1.000	1.001
233'44'5 - HxCB	156	38380-08-4	156L/157L		45:11:00	156L/157L	45:10:00	1.000	6	0.999	1.001
233'44'5' - HxCB	157	69782-90-7	156L/157L	156 + 157							
33'44'55' - HxCB	169	32774-16-6	169L		48:36:00	169L	48:34:00	1.001	-1,3	1.000	1.001
22'34'566' - HpCB	188	74487-85-7	188L		38:06:00	188L	38:04:00	1.001	-1,3	1.000	1.001
22'33'566' - HpCB	179	52663-64-6	188L/189L		38:26:00	188L	38:04:00	1.010	6	1.008	1.011
22'344'66' - HpCB	184	74472-48-3	188L/189L		39:00:00	188L	38:04:00	1.025	6	1.023	1.026
22'33'466' - HpCB	176	52663-65-7	188L/189L		39:22:00	188L	38:04:00	1.034	6	1.033	1.035
22'34566' - HpCB	186	74472-49-4	188L/189L		39:50:00	188L	38:04:00	1.046	6	1.045	1.048
22'33'55'6 - HpCB	178	52663-67-9	188L/189L		41:17:00	188L	38:04:00	1.085	6	1.083	1.086
22'33'45'6 - HpCB	175	40186-70-7	188L/189L		41:57:00	188L	38:04:00	1.102	6	1.101	1.103
22'34'55'6 - HpCB	187	52663-68-0	188L/189L		42:15:00	188L	38:04:00	1.110	6	1.109	1.111
22'344'56' - HpCB	182	60145-23-5	188L/189L		42:27:00	188L	38:04:00	1.115	6	1.114	1.116
22'344'5'6 - HpCB	183	52663-69-1	188L/189L	183 + 185	42:56:00	188L	38:04:00	1.128	6	1.127	1.129

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COMPOUND	Congener Number	CAS NO.	QUANTIFICATION REFERENCE	CO-ELUTIONS	COMPOUND RT	RT Reference	Labeled RT	RRT	RT Window (sec)	RRT Lower Limit	RRT Upper Limit
22'3455'6 - HpCB	185	52712-05-7	188L/189L	183 + 185							
22'33'456' - HpCB	174	38411-25-5	188L/189L		43:07:00	188L	38:04:00	1.133	6	1.131	1.134
22'33'4'56 - HpCB	177	52663-70-4	188L/189L		43:35:00	188L	38:04:00	1.145	6	1.144	1.146
22'344'56 - HpCB	181	74472-47-2	188L/189L		44:00:00	188L	38:04:00	1.156	6	1.155	1.157
22'33'44'6 - HpCB	171	52663-71-5	188L/189L	171 + 173	44:13:00	188L	38:04:00	1.162	10	1.159	1.164
22'33'456 - HpCB	173	68194-16-1	188L/189L	171 + 173							
22'33'455' - HpCB	172	52663-74-8	188L/189L		45:57:00	189L	51:13:00	0.897	6	0.896	0.898
233'455'6 - HpCB	192	74472-51-8	188L/189L		46:15:00	189L	51:13:00	0.903	6	0.902	0.904
233'4'55'6 - HpCB	193	69782-91-8	180L	180 + 193							
22'344'55' - HpCB	180	35065-29-3	180L	180 + 193	46:36:00	180L	46:35:00	1.000	6	0.999	1.001
233'44'5'6 - HpCB	191	74472-50-7	188L/189L		47:00:00	189L	51:13:00	0.918	6	0.917	0.919
22'33'44'5 - HpCB	170	35065-30-6	170L		47:57:00	170L	47:56:00	1.000	6	0.999	1.001
233'44'56 - HpCB	190	41411-64-7	188L/189L		48:31:00	189L	51:13:00	0.947	6	0.946	0.948
233'44'55' - HpCB	189	39635-31-9	189L		51:14:00	189L	51:13:00	1.000	-1,3	1.000	1.001
22'33'55'66' - OcCB	202	2136-99-4	202L		43:43:00	202L	43:41:00	1.001	-1,3	1.000	1.001
22'33'45'66' - OcCB	201	40186-71-8	202L/205L		44:41:00	202L	43:41:00	1.023	10	1.021	1.025
22'344'566' - OcCB	204	74472-52-9	202L/205L		45:23:00	202L	43:41:00	1.039	6	1.038	1.040
22'33'44'66' - OcCB	197	33091-17-7	202L/205L	197 + 200	45:41:00	202L	43:41:00	1.046	14 *	1.045	1.047
22'33'4566' - OcCB	200	52663-73-7	202L/205L	197 + 200							
22'33'455'6 - OcCB	198	68194-17-2	202L/205L	198 + 199	48:40:00	202L	43:41:00	1.114	10	1.112	1.116
22'33'455'6' - OcCB	199	52663-75-9	202L/205L	198 + 199							
22'33'44'56' - OcCB	196	42740-50-1	202L/205L		49:23:00	205L	53:54:00	0.916	6	0.915	0.917
22'344'55'6 - OcCB	203	52663-76-0	202L/205L		49:35:00	205L	53:54:00	0.920	6	0.919	0.921
22'33'44'56 - OcCB	195	52663-78-2	202L/205L		50:59:00	205L	53:54:00	0.946	6	0.945	0.947
22'33'44'55' - OcCB	194	35694-08-7	202L/205L		53:26:00	205L	53:54:00	0.991	6	0.990	0.992
233'44'55'6 - OcCB	205	74472-53-0	205L		53:56:00	205L	53:54:00	1.001	-1,3	1.000	1.001
22'33'455'66' - NoCB	208	52663-77-1	208L		50:43:00	208L	50:42:00	1.000	-1,3	1.000	1.001
22'33'44'566' - NoCB	207	52663-79-3	208L/206L		51:42:00	208L	50:42:00	1.020	6	1.019	1.021
22'33'44'55'6 - NoCB	206	40186-72-9	206L		55:45:00	206L	55:44:00	1.000	-1,3	1.000	1.001
22'33'44'55'66' - DeCB	209	2051-24-3	209L		57:26:00	209L	57:25:00	1.000	-1,3	1.000	1.001
LABELED COMPOUND											

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COMPOUND	Congener Number	CAS NO.	QUANTIFICATION REFERENCE	CO-ELUTIONS	COMPOUND RT	RT Reference	Labeled RT	RRT	RT Window (sec)	RRT Lower Limit	RRT Upper Limit
13C12-2 - MoCB	**1L**		9L		11:34:00	9L	16:05:00	0.719	30	0.704	0.735
13C12-4 - MoCB	**3L**		9L		13:48:00	9L	16:05:00	0.858	30	0.842	0.874
13C12-22' - DiCB	**4L**		9L		14:03:00	9L	16:05:00	0.874	30	0.858	0.889
13C12-44' - DiCB	**15L**		9L		20:08:00	9L	16:05:00	1.252	30	1.236	1.267
13C12-22'6 - TriCB	**19L**		9L		17:13:00	9L	16:05:00	1.070	30	1.055	1.086
13C12-344' - TriCB	**37L**		52L		27:27:00	52L	25:09:00	1.091	30	1.082	1.101
13C12-22'66' - TeCB	**54L**		52L		20:25:00	52L	25:09:00	0.812	20	0.805	0.818
13C12-33'44' - TeCB	**77L**		52L		35:07:00	52L	25:09:00	1.396	20	1.390	1.403
13C12-344'5 - TeCB	**81L**		52L		34:31:00	52L	25:09:00	1.372	20	1.366	1.379
13C12-22'466' - PeCB	**104L**		101L		26:08:00	101L	32:19:00	0.809	20	0.804	0.814
13C12-233'44' - PeCB	**105L**		101L		38:46:00	101L	32:19:00	1.200	20	1.194	1.205
13C12-2344'5 - PeCB	**114L**		101L		38:06:00	101L	32:19:00	1.179	20	1.174	1.184
13C12-23'44'5 - PeCB	**118L**		101L		37:32:00	101L	32:19:00	1.161	20	1.156	1.167
13C12-2'344'5 - PeCB	**123L**		101L		37:12:00	101L	32:19:00	1.151	20	1.146	1.156
13C12-33'44'5 - PeCB	**126L**		101L		42:01:00	101L	32:19:00	1.300	20	1.295	1.305
13C12-22'44'66' - HxCB	**155L**		138L		32:05:00	138L	40:48:00	0.786	20	0.782	0.790
13C12-233'44'5 - HxCB and	**156L**		138L		45:10:00	138L	40:48:00	1.107	20	1.103	1.111
13C12-233'44'5' - HxCB	**157L**		138L								
13C12-23'44'55' - HxCB	**167L**		138L		43:57:00	138L	40:48:00	1.077	20	1.073	1.081
13C12-33'44'55' - HxCB	**169L**		138L		48:34:00	138L	40:48:00	1.190	20	1.186	1.194
13C12-22'33'44'5 - HpCB	**170L**		194L		47:57:00	194L	53:25:00	0.898	20	0.891	0.904
13C12-22'344'55' - HpCB	**180L**		194L		46:36:00	194L	53:25:00	0.872	20	0.866	0.879
13C12-22'34'566' - HpCB	**188L**		194L		38:04:00	194L	53:25:00	0.713	20	0.710	0.716
13C12-233'44'55' - HpCB	**189L**		194L		51:13:00	194L	53:25:00	0.959	20	0.956	0.962
13C12-22'33'55'66' - OcCB	**202L**		194L		43:41:00	194L	53:25:00	0.818	20	0.815	0.821
13C12-233'44'55'6 - OcCB	**205L**		194L		53:54:00	194L	53:25:00	1.009	30	1.004	1.014
13C12-22'33'44'55'6 - NoCB	**206L**		194L		55:44:00	194L	53:25:00	1.043	30	1.039	1.048
13C12-22'33'455'66' - NoCB	**208L**		194L		50:42:00	194L	53:25:00	0.949	20	0.946	0.952
13C12-22'33'44'55'66' - DeCB	**209L**		194L		57:26:00	194L	53:25:00	1.075	30	1.071	1.080
LABELED CLEAN-UP STANDARD											
13C12-244' - TriCB	**28L**		52L		23:16:00	52L	25:09:00	0.925	20	0.918	0.932
13C12-233'55' - PeCB	**111L**		101L		35:09:00	101L	32:19:00	1.088	20	1.083	1.093
13C12-22'33'55'6 - HpCB	**178L**		138L		41:16:00	138L	40:48:00	1.011	20	1.007	1.016

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COMPOUND	Congener Number	CAS NO.	QUANTIFICATION REFERENCE	CO-ELUTIONS	COMPOUND RT	RT Reference	Labeled RT	RRT	RT Window (sec)	RRT Lower Limit	RRT Upper Limit
LABELED INJECTION INTERNAL STANDARD											
13C12-25 - DiCB	**9L**		138L		16:05:00	138L	40:48:00	0.394	25	0.389	0.399
13C12-22'55' - TeCB	**52L**		138L		25:09:00	138L	40:48:00	0.616	25	0.611	0.622
13C12-22'455' - PeCB	**101L**		138L		32:19:00	138L	40:48:00	0.792	25	0.787	0.797
13C12-22'344'5' - HxCB	**138L**		138L		40:48:00	138L	40:48:00	1.000	100	0.980	1.020
13C12-22'33'44'55' - OcCB	**194L**		138L		53:25:00	138L	40:48:00	1.309	25	1.304	1.314

(1) Suffix "L" indicates labeled compound.

(2) C = co-eluting congener

* RT window wider than prescribed by 1668A/C to accommodate for skewed peaks of coeluting peaks (PCB-95/100/93/102/98, PCB-151/135/154 and PCB-197/200)

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Theoretical Ion Abundance Ratios and QC Limits

Chlorine Atoms	m/z's Forming Ratio	Theoretical Ratio	Lower QC Limit	Upper QC Limit
1	M/(M+2)	3.13	2.66	3.60
2	M/(M+2)	1.56	1.33	1.79
3	M/(M+2)	1.04	0.88	1.20
4	M/(M+2)	0.77	0.65	0.89
5	(M+2)/(M+4)	1.55	1.32	1.78
6	(M+2)/(M+4)	1.24	1.05	1.43
7	(M+2)/(M+4)	1.05	0.89	1.21
8	(M+2)/(M+4)	0.89	0.76	1.02
9	(M+2)/(M+4)	0.77	0.65	0.89
10	(M+4)/(M+6)	1.16	0.99	1.33

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The peak assignment of PCB Congener numbers 107, 108, 109 differs between EPA Method 1668A and EPA 1668C/CBC01.2. Congeners named PCB 108, 107 and 109 in Method 1668A are named PCB 109, 108 and 107, respectively, for EPA 1668C and EPA CBC01.2 applications.

Analyte Retention Times, Surrogates Used and RRT Windows for Congener Numbers 107, 108 and 109 in accordance with 1668A Specification

COMPOUND	Congener Number	CAS NO.	QUANTIFICATION REFERENCE	CO-ELUTIONS	COMPOUND RT	RT Reference	Labeled RT	RRT	RT Window (sec)	RRT Lower Limit	RRT Upper Limit
23'44'6 - PeCB	119	56558-17-9	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 108 + 119 + 125							
233'45' - PeCB	108	70362-41-3	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 108 + 119 + 125							
22'345 - PeCB	86	55312-69-1	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 108 + 119 + 125	33:29:00	123L	37:12:00	0.900	16	0.897	0.904
22'3'45 - PeCB	97	41464-51-1	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 108 + 119 + 125							
2'3456' - PeCB	125	74472-39-2	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 108 + 119 + 125							
22'345' - PeCB	87	38380-02-8	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 108 + 119 + 125							
233'4'5 - PeCB	107	70424-68-9	104L/123L/114L/ 118L/105L	107 + 124	36:50:00	123L	37:12:00	0.990	10	0.988	0.992
2'3455' - PeCB	124	70424-70-3	104L/123L/114L/ 118L/105L	107 + 124							
233'46 - PeCB	109	74472-35-8	104L/123L/114L/ 118L/105L		37:05:00	123L	37:12:00	0.997	6	0.996	0.998

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Analyte Retention Times, Surrogates Used and RRT Windows for Optional Reporting of Congener Numbers 107, 108 and 109 in accordance with 1668C Specification

COMPOUND	Congener Number	CAS NO.	QUANTIFICATION REFERENCE	CO-ELUTIONS	COMPOUND RT	RT Reference	Labeled RT	RRT	RT Window (sec)	RRT Lower Limit	RRT Upper Limit
23'44'6 - PeCB	119	56558-17-9	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 109 + 119 + 125							
2,3,3',4,6 - PeCB	109	74472-35-8	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 109 + 119 + 125							
2,2',3,4,5 - PeCB	86	55312-69-1	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 109 + 119 + 125	33:29:00	123L	37:12:00	0.900	16	0.897	0.904
2,2',3',4,5 - PeCB	97	41464-51-1	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 109 + 119 + 125							
2',3,4,5,6' - PeCB	125	74472-39-2	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 109 + 119 + 125							
2,2',3,4,5' - PeCB	87	38380-02-8	104L/123L/114L/ 118L/105L	86 + 87 + 97 + 109 + 119 + 125							
2,3,3',4,5' - PeCB	108	70362-41-3	104L/123L/114L/ 118L/105L	108 + 124	36:50:00	123L	37:12:00	0.990	10	0.988	0.992
2',3,4,5,5' - PeCB	124	70424-70-3	104L/123L/114L/ 118L/105L	108 + 124							
2,3,3',4',5 - PeCB	107	70424-68-9	104L/123L/114L/ 118L/105L		37:05:00	123L	37:12:00	0.997	6	0.996	0.998

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4.1 Reporting Limits

Concentrations and detection limits for target compounds are reported. The isotope dilution/internal standard method of quantification, used to determine concentrations of target analytes, corrects the concentrations based on the percent recovery of the surrogate. Typical reporting units are pg/g, pg/L, or pg/sample. Concentrations for solids are reported on a dry weight basis. Concentrations in tissues (including blood/serum/plasma and milk) are reported on a wet weight basis and/or on a lipid weight basis when requested. Concentrations in aqueous samples are reported on a volume basis. Concentrations in XAD-2 resin and filter samples are reported on a per sample basis or a per volume basis. Concentrations in particulate filters are reported on a per sample basis. Results may be expressed in other units if specified by contract. Unless otherwise specified, concentrations are reported to three significant figures.

Percent moisture is reported for all sediments, soils, and ash samples and for tissues when percent moisture is requested. Percent moisture is not reported for pulps and sludge, although it is determined for the calculation of a dry weight.

Percent lipid is reported if determined on a sample.

All sample results below the LOQ (limit of quantification) will be flagged in reports.

Various reporting limit options are offered to meet particular client and data quality objectives and should be defined specific to contract by the client and agreed to by SGS AXYS in advance of instrumental analysis:

Sample Specific Detection Limit (SDL) Reporting Limit (Default Option)

SDLs are determined from the analysis data by converting the minimum detectable area to a concentration following the same quantification procedures used to convert target peak responses to concentrations. The estimated minimum detectable area is determined as 2.5 times the height of the noise in the m/z channel of interest, converted to an area using the area:height ratio of the corresponding labelled surrogate peak. The SDL accounts for any effect of matrix on the detection system, for recovery achieved through the analytical work-up and for the exact sample size used.

SDLs do not account for background contribution, and sample results must be compared to the associated QC blank results for significance of detection.

The lower reporting limit for blood/serum/plasma samples is the SDL, for other matrices the lower reporting limit is the greater of SDL or 0.5 pg/sample.

EPA1668A and EPA1668C Specified Reporting Limit

Sample result reporting to minimum levels (“MLs”) according to section 17.6.1.4 of EPA methods 1668A/C is an alternate reporting limit approach which may be applied on written request of the customer.

MLs do not account for sample specific matrix effect or sample specific recovery achieved through the analytical work-up. ML reporting limits do account for background detectable in laboratory blanks. ML reporting limit is a recognized convention for compliance monitoring based on Total PCB values determined from summation of detected congeners.

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Laboratory Method detection limit (MDL) Reporting Limit

Laboratory MDLs are determined as specified by EPA Fed. Reg. 40 CFR Part 136 Appendix B, revision 2. The 99% confidence level MDL is statistically determined based on analysis of replicate matrix spikes and historical blank levels. MDLs are an estimate of detection limit determined at a point in time and do not compensate for sample specific matrix effect, sample specific recovery achieved through the analytical work-up.

5.0 QUALITY ACCEPTANCE CRITERIA

Samples are analyzed in batches consisting of a maximum of twenty samples, one procedural blank and one spiked matrix (OPR) sample. A duplicate is analyzed, provided there is sufficient sample, with batches containing 7-20 samples. Matrix spike/matrix spike duplicate (MS/MSD) pairs may be analyzed on an individual contract basis. The batch is carried through the complete analytical process as a unit. For sample data to be reportable, the batch QC data must meet the established acceptance criteria presented on the analysis reports.

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QC Acceptance Criteria according to EPA method 1668A for Chlorinated Biphenyls in CAL/VER, IPR, OPR, and Samples

Congener	Cong. No. [2]	Test conc. ng/mL	CAL/VER (%)		IPR [1] (%)		OPR [1] (%)		Labelled compound [1] % recovery in samples	
			Warning limits	Acceptance limits	RSD	$\bar{X}$	Warning limits	Acceptance limits	Warning limits	Acceptance limits
2-MoCB	1	50	75-125	70-130	40	60-140	70-130	50-150	-	-
4-MoCB	3	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2'-DiCB	4	50	75-125	70-130	40	60-140	70-130	50-150	-	-
4,4'-DiCB	15	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2'6-TrCB	19	50	75-125	70-130	40	60-140	70-130	50-150	-	-
3,4,4'-TrCB	37	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2'6,6'TeCB	54	50	75-125	70-130	40	60-140	70-130	50-150	-	-
3,3',4,4'-TeCB	77	50	75-125	70-130	40	60-140	70-130	50-150	-	-
3,4,4',5-TeCB	81	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2',4,6,6'-PeCB	104	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3,3',4,4'-PeCB	105	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3,4,4',5-PeCB	114	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3',4,4',5-PeCB	118	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2',3,4,4',5-PeCB	123	50	75-125	70-130	40	60-140	70-130	50-150	-	-
3,3',4,4',5-PeCB	126	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2',4,4',6,6'-HxCB	155	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3,3',4,4',5-HxCB [3]	156	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3,3',4,4',5'-HxCB [3]	157	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3',4,4',5,5'-HxCB	167	50	75-125	70-130	40	60-140	70-130	50-150	-	-
3,3',4,4',5,5'-HxCB	169	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2',3,4',5,6,6'-HpCB	188	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3,3',4,4',5,5'-HpCB	189	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2',3,3',5,5',6,6'-OcCB	202	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3,3',4,4',5,5',6-OcCB	205	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2',3,3',4,4',5,5',6-NoCB	206	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2',3,3,'4,5,5',6,6'-NoCB	208	50	75-125	70-130	40	60-140	70-130	50-150	-	-
DeCB	209	50	75-125	70-130	40	60-140	70-130	50-150	-	-
Labeled Compounds										
$^{13}C_{12}$-2-MoCB	1L	100	65-135	50-150	50	20-135	15-140	15-140	15-130	15-150
$^{13}C_{12}$-4-MoCB	3L	100	65-135	50-150	50	20-135	15-140	15-140	15-130	15-150
$^{13}C_{12}$-2,2'-DiCB	4L	100	65-135	50-150	50	35-135	30-140	30-140	25-130	25-150
$^{13}C_{12}$-4,4'-DiCB	15L	100	65-135	50-150	50	35-135	30-140	30-140	25-130	25-150
$^{13}C_{12}$-2,2',6-TrCB	19L	100	65-135	50-150	50	35-135	30-140	30-140	30-130	25-150
$^{13}C_{12}$-3,4,4'-TrCB	37L	100	65-135	50-150	50	35-135	30-140	30-140	30-130	25-150
$^{13}C_{12}$-2,2',6,6'-TeCB	54L	100	65-135	50-150	50	35-135	30-140	30-140	30-130	25-150

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Congener	Cong. No. [2]	Test conc. ng/mL	CAL/VER (%)		IPR [1] (%)		OPR [1] (%)		Labelled compound [1] % recovery in samples	
			Warning limits	Acceptance limits	RSD	$\bar{X}$	Warning limits	Acceptance limits	Warning limits	Acceptance limits
$^{13}C_{12}$-3,3',4,4'-TCB	77L	100	65-135	50-150	50	35-135	30-140	30-140	30-130	25-150
$^{13}C_{12}$-3,4,4',5-TeCB	81L	100	65-135	50-150	50	35-135	30-140	30-140	30-130	25-150
$^{13}C_{12}$-2,2',4,6,6'-PeCB	104L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,3,3',4,4'-PeCB	105L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,3,4,4',5-PeCB	114L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,3',4,4',5-PeCB	118L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2',3,4,4',5-PeCB	123L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-3,3',4,4',5-PeCB	126L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,2',4,4',6,6'-HxCB	155L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,3,3',4,4',5-HxCB [3]	156L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,3,3',4,4',5'-HxCB [3]	157L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,3',4,4',5,5'-HxCB	167L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-3,3',4,4',5,5'-HxCB	169L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,2',3,3',4,4',5-HpCB	170L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,2',3,4,4',5,5'-HpCB	180L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB	188L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,3,3',4,4',5,5'-HpCB	189L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB	202L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB	205L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB	206L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-NoCB	208L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-DeCB	209L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
Cleanup Standards										
$^{13}C_{12}$-2,4,4'-TriCB	28L	100		60-130	45	45-120	40-125	40-125	40-130	30-135
$^{13}C_{12}$-2,3,3',5,5'-PeCB	111L	100		60-130	45	45-120	40-125	40-125	40-130	30-135
$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB	178L	100		60-130	45	45-120	40-125	40-125	40-130	30-135

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QC Acceptance Criteria according to EPA method 1668C for Chlorinated Biphenyls in CAL/VER, IPR, OPR, and Samples

Congener	Cong. No.[2]	Test conc. ng/mL	CAL/VER (%)		IPR [1] (%)		OPR [1] (%)		Labelled compound [1] % recovery in samples	
			Warning limits	Acceptance limits	RSD	$\bar{X}$	Warning limits	Acceptance limits	Warning limits	Acceptance limits
2-MoCB	1	50	75-125	75-125	25	70-130	70-130	60-135	-	-
4-MoCB	3	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2'-DiCB	4	50	75-125	75-125	25	70-130	70-130	60-135	-	-
4,4'-DiCB	15	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2'6-TrCB	19	50	75-125	75-125	25	70-130	70-130	60-135	-	-
3,4,4'-TrCB	37	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2'6,6'TeCB	54	50	75-125	75-125	25	70-130	70-130	60-135	-	-
3,3',4,4'-TeCB	77	50	75-125	75-125	25	70-130	70-130	60-135	-	-
3,4,4',5-TeCB	81	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',4,6,6'-PeCB	104	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,3',4,4'-PeCB	105	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,4,4',5-PeCB	114	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3',4,4',5-PeCB	118	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2',3,4,4',5-PeCB	123	50	75-125	75-125	25	70-130	70-130	60-135	-	-
3,3',4,4',5-PeCB	126	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',4,4',6,6'-HxCB	155	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,3',4,4',5-HxCB [3]	156	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,3',4,4',5'-HxCB [3]	157	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3',4,4',5,5'-HxCB	167	50	75-125	75-125	25	70-130	70-130	60-135	-	-
3,3',4,4',5,5'-HxCB	169	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',3,4',5,6,6'-HpCB	188	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,3',4,4',5,5'-HpCB	189	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',3,3',5,5',6,6'-OcCB	202	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,3',4,4',5,5',6-OcCB	205	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',3,3',4,4',5,5',6-NoCB	206	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',3,3,'4,5,5',6,6'-NoCB	208	50	75-125	75-125	25	70-130	70-130	60-135	-	-
DeCB	209	50	75-125	75-125	25	70-130	70-130	60-135	-	-
Labeled Compounds										
$^{13}C_{12}$-2-MoCB	1L	100	65-135	50-145	70	20-135	15-145	15-145	15-130	5-145
$^{13}C_{12}$-4-MoCB	3L	100	65-135	50-145	70	20-135	15-145	15-145	15-130	5-145
$^{13}C_{12}$-2,2'-DiCB	4L	100	65-135	50-145	70	20-135	15-145	15-145	25-130	5-145
$^{13}C_{12}$-4,4'-DiCB	15L	100	65-135	50-145	70	20-135	15-145	15-145	25-130	5-145
$^{13}C_{12}$-2,2',6-TrCB	19L	100	65-135	50-145	70	20-135	15-145	15-145	30-130	5-145
$^{13}C_{12}$-3,4,4'-TrCB	37L	100	65-135	50-145	70	20-135	15-145	15-145	30-130	5-145
$^{13}C_{12}$-2,2',6,6'-TeCB	54L	100	65-135	50-145	70	20-135	15-145	15-145	30-130	5-145

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Congener	Cong. No.[2]	Test conc. ng/mL	CAL/VER (%)		IPR[1] (%)		OPR[1] (%)		Labelled compound[1] % recovery in samples	
			Warning limits	Acceptance limits	RSD	$\bar{X}$	Warning limits	Acceptance limits	Warning limits	Acceptance limits
$^{13}C_{12}$-3,3',4,4'-TCB	77L	100	65-135	50-145	50	45-135	40-145	40-145	30-130	10-145
$^{13}C_{12}$-3,4,4',5-TeCB	81L	100	65-135	50-145	50	45-135	40-145	40-145	30-130	10-145
$^{13}C_{12}$-2,2',4,6,6'-PeCB	104L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
$^{13}C_{12}$-2,3,3',4,4'-PeCB	105L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
$^{13}C_{12}$-2,3,4,4',5-PeCB	114L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
$^{13}C_{12}$-2,3',4,4',5-PeCB	118L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
$^{13}C_{12}$-2',3,4,4',5-PeCB	123L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
$^{13}C_{12}$-3,3',4,4',5-PeCB	126L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
$^{13}C_{12}$-2,2',4,4',6,6'-HxCB	155L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
$^{13}C_{12}$-2,3,3',4,4',5-HxCB [3]	156L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
$^{13}C_{12}$-2,3,3',4,4',5'-HxCB [3]	157L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
$^{13}C_{12}$-2,3',4,4',5,5'-HxCB	167L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
$^{13}C_{12}$-3,3',4,4',5,5'-HxCB	169L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
$^{13}C_{12}$-2,2',3,3',4,4',5-HpCB	170L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
$^{13}C_{12}$-2,2',3,4,4',5,5'-HpCB	180L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB	188L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
$^{13}C_{12}$-2,3,3',4,4',5,5'-HpCB	189L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB	202L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB	205L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB	206L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-NoCB	208L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-DeCB	209L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
Cleanup Standards										
$^{13}C_{12}$-2,4,4'-TriCB	28L	100		65-135	70	20-135	15-145	15-145	40-130	5-145
$^{13}C_{12}$-2,3,3',5,5'-PeCB	111L	100		75-125	50	45-135	40-145	40-145	40-130	10-145
$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB	178L	100		75-125	50	45-135	40-145	40-145	40-130	10-145

Footnotes to the tables above:

1. QC acceptance criteria for IPR, OPR, and samples based on a 20 µL extract final volume
2. Suffix "L" indicates labelled compound.
3. PCBs 156 and 157 are tested as the sum of two concentrations

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General QC Acceptance Criteria

QC Parameter	Specification
Analysis Duplicate	±20% of the mean (applicable to concentrations >10 times the DL)[1]
Procedural Blank	The following blank acceptance guidelines apply: **PCB Congeners** Congeners 77, 81, 114, 123, 126, 169: 2 pg/sample Congeners 156, 157, 167 and 189: 10 pg/sample Congener PCB 11: 150 pg/sample All other congeners: 50 pg/sample **Total PCB (sum of 209 congeners) *** Aqueous and serum: 95% confidence level 350 pg/sample; 99% confidence level 450 pg/sample Solids, tissue and all other matrices: 95% confidence level 800 pg/sample; 99% confidence level 1100 pg/sample * Total PCB blank specification may be project dependent and must be agreed with clients prior to work commencement. Where marginal exceedance against acceptance specification is allowable based on lab judgement of data fitness for purpose, the 95% confidence level specification is appropriate. Higher levels are acceptable where sample concentrations exceed 10 times the blank levels.
Sample Specific Detection Limit	Typical sample specific detection limits, determined from chromatographic noise, are in the range of 0.5 to 2.0 pg.
Initial Calibration	For 6- or 7-point calibration, a relative standard deviation of the RRF's ≤20% for all compounds. Ion ratios for all congeners must be within ±15% of theoretical for CS-0.5. Minimum S:N ratio 10:1 for all calibration standards. For CS-0.5, S:N ratio may be as low as 3:1 for di-PCBs and nona-PCBs.
Continuing CAL VER	Refer to Tables above.
Analyte/Surrogate Ratios	Response must be within the calibrated range of the instrument. Coders may use data from more than one chromatogram to get the responses in the calibrated range.
Ion Ratios	Ion ratios must fall within ±15% of the theoretical values for positive identification of all targets in the calibration standards and samples.
Sensitivity	Minimum S:N ratio 10:1 for all calibration standards. For CS-0.5, S:N ratio may be as low as 3:1 for di-PCBs and nona-PCBs.

[1] Duplicate criterion is a guideline; final assessment depends upon sample characteristics, overall batch QC and on-going lab performance.

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Summary of Modifications to EPA Methods 1668A and 1668C

The following sections of EPA Methods 1668A and 1668C have been modified as described below.

Section 4.2.1, 4.2.2: The protocol for washing reusable glassware includes a detergent wash, water rinse and baking at a minimum of 300 °C for 8 hours. Immediately prior to use, glassware is solvent rinsed with toluene and hexane.

Section 4.7: The first cleanup column for tissue extracts is a gravity gel permeation column (SX-3 Biobeads). An anthropogenic isolation column 7.5.3 is not used.

Section 6.5.1: Glass wool is cleaned by rinsing twice with toluene and twice with hexane.

Section 7.12, 7.13, 9.0, 11.0: The concentration of the labeled toxics/LOC and the cleanup standard spiking solutions is 100 ng/mL and the sample spiking volume is 20 µL. The resulting final concentrations in the extracts are as specified in the method.

Section 7.14: Concentration of the labeled injection internal standard spiking solution (recovery standard) is modified so that a volume of 5 µL is added. The resulting amount of standard added to the final extract is the same as specified in the method. The solution is spiked into a 15 µL extract volume for a final extract volume of 20 µL.

Section 7.2.1: Sodium sulfate is baked at a minimum of 300 °C for 8 hrs.

Section 7.5.1: Silica is activated by baking at 450 °C in a muffle oven for at least 8 hrs.

Section 7.5.4.1.1: Florisil is baked at 450 °C in a muffle over for at least 8 hrs, then deactivated with water to 2.0% deactivation.

Section 10.3.3, 15.3.3: A S:N ratio of 3:1 for di-PCBs and nona-PCBs in CS0.5 calibration solution is acceptable.

Section 11.3, 11.5.7, 11.7: To avoid carry-over contamination particles larger than 1 mm in solid samples are by default not subject to grinding prior to extraction. Instead solid samples are homogenized prior to sub-sampling by breaking up clumps with a disposable spoon and mixing/stirring. If required by the client rocks larger than 0.4 cm are removed. Surrogate standard is added after homogenization and sub-sampling.

Section 11.5.6: Unless requested by the client, the aqueous portion after filtration of aqueous samples with >1% solids is not discarded but is extracted.

Section 11.5, 11.5.2, 11.5.5, 12.3: Solid samples are dried by mixing with anhydrous sodium sulfate. The dried solid is extracted using a Soxhlet extraction apparatus. The surrogate spike is incorporated after the drying step. Equilibration time for the surrogate is 30 minutes. The extracting solvent for solids is dichloromethane.

Section 12.4, 11.8: The surrogate spike is incorporated into the sample after the drying step to eliminate the possibility of disproportional loss of volatile labeled and target compounds.

Section 12.4.2: The precleaning of the Soxhlet apparatus is carried out using toluene instead of dichloromethane, for 2 hours.

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Section 12.4.9: Lipid analysis is carried out by sub-sampling two 2 g portions of the extract from a total 30 g extract weight. The cleanup standard is spiked into the extract after Soxhlet extraction and before any lipid analysis or rotary evaporation is done. The percent surrogate recoveries are corrected for the amount of extract used for lipid analysis.

Section 12.6.1.1: Rotary evaporation is done at 30 °C. Daily cleaning of the rotary evaporators include dismantling and rinsing/soaking with solvent. Proofs are run periodically but are not archived daily.

Section 12.7.4: Before Florisil or alumina cleanup procedures, a solvent exchange is done by reducing under nitrogen to 300 µL and bulking up to 1mL in hexane. If toluene is present the extract is reduced to 50 µL under nitrogen and bulked up to 1 mL.

Section 12.7.7: Toluene (1 mL) is added to the eluate from the final column prior to rotary evaporation and nitrogen blow down concentration steps.

Section 13.1.1: GPC chromatography, by a gravity column, is routinely used only for tissue extracts. The GPC cleanup is optional for all other matrices.

Section 13.3.1: Routine layered silica column is as follows: 0.5 g neutral silica, 2 g 28% basic silica, 0.5 g neutral silica, 4 g 44% acidic silica, 4 g 22% acidic silica, 1 g neutral silica.

Section 13.3.4: The sample is loaded onto the column followed by 2-3 rinses of a least 1 mL, and eluted with 100 mL of hexane.

Section 14.2: The volume of labeled injection internal standard (recovery standard) added to the extract is 5 µL, for a final extract volume of 20 µL. Hexane rather than nonane is used as the solvent to bring extract back to volume for re-analysis or to dilute extracts.

Section 15.3: The calibration solution containing all 209 PCB congeners is used as the CAL/VER solution.

*Section 17.*5: Extracts are diluted with hexane. The concentration of the labeled injection internal (recovery) standard is not re-adjusted to 100 pg/µL when dilutions are performed.

Section 17.0

$Conc_i$ - the concentrations of target analytes, and the labeled compound concentrations and recoveries, are calculated using the equations below. These procedures are equivalent to those described in the method but are more direct.

$$Conc_i = \frac{A_i}{A_{si}} \times \frac{M_{si}}{RRF_{i,si}} \times \frac{1}{M_x}$$

where A_i = summed areas of the primary and secondary m/z's for the analyte peak of interest (compound *i*)

A_{si} = summed areas of the primary and secondary m/z's for the labeled surrogate peak used to quantify *i*)

M_x = mass of sample taken for analysis

M_{si} = mass of labeled surrogate (compound *si*) added to sample as calculated by the concentration of standard spiked (pg/mL) multiplied by the volume spiked (mL)

$RRF_{i,si}$ = mean relative response factor of *i* to *si* from the five-point calibration range and defined individually as:

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$$\frac{A_i}{A_{si}} \times \frac{M_{si}}{M_i}$$

Calculation of Surrogate Standard Concentrations and Percent Recoveries:

Concentrations of surrogate standards are calculated using the following equation:

$$Conc_{si} = \frac{A_{si}}{A_{rs}} \times \frac{M_{rs}}{RRF_{si,rs}}$$

and, the percent recoveries of the surrogate standards are calculated using the following equation:

$$\%\text{Recovery} = \frac{A_{si}}{A_{rs}} \times \frac{M_{rs}}{RRF_{si,rs}} \times \frac{1}{M_{si}} \times 100$$

where A_{rs} and A_{si} are the summed peak areas (from the primary and secondary m/z channels) of recovery standard and labeled surrogate added to the sample;
M_{rs} and M_{si} are the masses of recovery standard and labeled surrogate added to the sample, and;
$RRF_{si,rs}$ is the mean relative response factor of the labeled surrogate to the recovery standard as determined by the five-point calibration range and defined individually as:

$$\cdot \frac{A_{si}}{A_{rs}} \times \frac{M_{rs}}{M_{si}}$$

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TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010D

Prepared By: George Brewer Date: 09/19/16

Approved By:

Department Manager: George Brewer Date: 09/19/16

Operations Manager: Michael P. Hanson Date: 9/19/16

QA Officer: Lorie Dimond Date: 09.19.16

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Updated Tables 4 and 5. Updated references	LAD	04/19	04/15
02	Sect. 5.5 - Added rinse solution which is different from the blank solution. Sect. 7.8 - Added linear range criteria of 90+10%R. & Removed sect 8.5 concerning the non-linear behavior of the alkali/alkaline earth metals - This is not applicable w/ our current calibration model.	LAD	06/20	06/20

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010D

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010D**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-638-02**, titled **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010D.**

Recipient: _____ Date: _____

I acknowledge receipt of copy ___ of document **SOP CA-638-02**, titled **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010D.**

Recipient: _____ Date: _____

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010D**

1.0 SCOPE AND APPLICATION

Inductively coupled plasma atomic-emission spectroscopy (ICP-AES) determines trace elements, including metals, in solution. The purpose of this SOP is to describe the procedures used by Katahdin Analytical Services, LLC personnel to analyze aqueous and solid samples for trace metals by USEPA Method 6010D (Test Methods for Evaluating Solid Waste, Physical/ Chemical Methods, USEPA SW846).

Sample types that may be analyzed using these methods include drinking waters, ground waters, aqueous samples, TCLP, SPLP and EP Toxicity extracts, industrial and organic wastes, soils, sludges, sediments, biological tissue and other solid wastes. The following elements may be analyzed under this SOP: Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Se, Si, Ag, Na, Sn, Sr, Tl, Ti, V, and Zn.

All samples, except filtered ground water samples, analyzed under USEPA Method 6010D require digestion prior to analysis. USEPA Methods 3005, 3010, and 3050 describe appropriate digestion procedures for samples to be analyzed by ICP-AES under EPA Method 6010D. Refer to current revisions of Katahdin SOPs CA-604 and CA-605, current revisions, for sample digestion procedures.

1.1 Definitions

Analytical Spike - An aliquot of a sample to which a known amount of analyte has been added before analysis and after digestion, if digestion is required.

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy.

ICP-AES - Inductively Coupled Plasma Atomic Emission Spectroscopy.

ICS - Interference Check Sample - Two standards (ICSA and ICSAB) used to verify the effectiveness of interelement correction and background correction. Solution ICSA contains only interferents (Al, Ca, Fe, and Mg) at high concentrations (200 to 500 mg/L); solution ICSAB contains interferents at the same concentrations as well as analytes at low (20 mg/L or less) concentrations.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 99% confidence.

LLOQ – Lower Limit of Quantitation – The lowest point of quantitation. In this laboratory, the LLOQ is considered to be equivalent to the PQL.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

LRS - Linear Range Standard - A high-concentration standard used to determine the upper reporting limit of the ICP calibration.

PB - Preparation Blank - Reagent water that has been brought through the sample preparation process.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

Hardness – The sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in mg/L.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in ICP analysis by EPA Method 6010D. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in ICP analysis by Method 6010D to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in

the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

Samples, sample digestates, standards, and other reagents used in ICP analysis may contain high concentrations of acids and toxic metals. Safety glasses should be worn when changing or adjusting argon tanks.

1.4 Pollution Prevention and Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes from ICP analysis should be disposed of in a manner appropriate to the hazards they present. Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual I and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010D**

2.0 SUMMARY OF METHOD

This method describes multi-elemental determinations by ICP-AES using simultaneous optical systems and radial and axial viewing of the plasma. The basis of the method is the measurement of atomic emission from sample atoms entrained in an argon plasma by optical spectroscopy. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where thermal excitation of entrained atoms and ions occurs. Characteristic atomic-line and ionic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating and the intensities of the emitted lines are monitored by a solid state charge injection device (CID) camera system. Photocurrents from the CID camera system are measured by a computer system. Element concentrations of unknown samples are quantitated by comparison of sample emission intensities to emission intensities of standards of known concentration. A background correction technique is used to compensate for variable background contribution to the determination of trace elements. Background is measured adjacent to the analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, has been determined by the complexity of the spectrum adjacent to the analytical line. The position used must be relatively free of spectral interference and must reflect the same change in background intensity as occurs at the analyte wavelength. Physical interferences are corrected through the use of an internal standard (yttrium) that is automatically added to all samples and standards prior to nebulization. The possibility of additional interferences (noted in section 3) must be recognized and appropriate corrections applied.

3.0 INTERFERENCES

Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as spectral interferences, physical interferences, and chemical interferences.

Spectral interferences can be categorized as 1) overlap of a spectral line from another element; 2) unresolved overlap of molecular band spectra; 3) background contribution from continuous or recombination phenomena; and 4) background from stray light from the line emission of high concentration elements. The first of these effects is compensated by utilizing the computer correction of raw data, requiring the monitoring and measurement of the interfering element (interelement correction). The second effect is controlled by choosing analytical wavelengths that are free from overlapping molecular emission spectra. The third and fourth effects are usually compensated by a background correction adjacent to the analyte line. Uncorrected spectral interferences may be detected through examination of serial dilution and matrix spike data.

Physical interferences are generally considered to be effects associated with sample nebulization and transport processes. Such properties as changes in viscosity and surface tension can cause significant inaccuracies, especially in samples that may contain high

dissolved solids and/or acid concentrations. Matrix matching of standards and samples and the use of a peristaltic pump may lessen these interferences. If these types of interferences are operative, they must be reduced by dilution of the sample and/or utilization of standard addition techniques. Another problem that can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aerosol flow rate causing instrumental drift. Regular cleaning of nebulizer tips and dilution of samples with high dissolved solids contents are used to control this problem. Physical interferences are also corrected by this laboratory through the use of an internal standard. Uncorrected physical interferences may be detected through examination of serial dilution and matrix spike data. Instrument drift caused by the salting up of nebulizer tips may also be detected by looking for oriented drift in calibration verification standards analyzed regularly throughout the run.

Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not pronounced with the ICP technique; however, if observed they can be minimized by careful selection of operating conditions (i.e., incident power, observation position, etc.), by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element. Uncorrected chemical interferences may be detected through examination of serial dilution data.

4.0 APPARATUS AND MATERIALS

4.1 Computer-controlled inductively coupled plasma atomic emission spectrometer (plasma viewed radially or axially) equipped for internal standardization, and capable of performing automatic background correction and interelement correction. For more information refer to the current revision of Katahdin SOP CA-632, “Operation and Maintenance of the Thermo ICAP 6500 ICP Spectrophotometer”.

4.2 Computer-controlled autosampler.

4.3 Argon gas supply – high purity.

4.4 Volumetric glassware of suitable precision and accuracy.

4.5 Automatic pipets of suitable precision and accuracy. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

Refer to the appropriate instrument-specific SOP for additional required equipment.

5.0 REAGENTS AND STANDARDS

5.1 Hydrochloric acid, concentrated (HCl) – spectroscopic grade.

5.2 Nitric acid, concentrated (HNO_3) – spectroscopic grade.

5.3 Reagent water, trace metals free.

5.4 Calibration blank – reagent water containing HCl (5% v/v) and HNO_3 (5% v/v). Calibration blank solution is prepared in large volumes (up to 20 liters) and stored in a carboy. Calibration blank solution is used in establishing the analytical curve, and in all initial and continuing calibration blank determinations. Intermediate and working standards are prepared by diluting stock standards and intermediate standards with calibration blank solution so that all standards and blanks are acid matrix-matched to sample digestates.

5.5 Rinse Solution – Calibration Blank plus two drops of Triton X. This solution is used to flush the system between standards and samples.

5.6 Single element and multielement stock standard solutions – purchased standards prepared from high purity salts or metals and supplied by the vendors with certificates of purity and analysis. Refer to Tables 3 and 4 for a listing of stock standards required, and to Table 7 for element concentrations in stock standards.

5.7 Intermediate standard solutions – laboratory-prepared multielement standards that are used in the subsequent preparation of working standards. Refer to Table 4 for a listing of intermediate standards required and for preparation instructions. Refer to Table 6 for element concentrations in intermediate standards.

5.8 Working standard solutions – laboratory-prepared multielement standards that are used to calibrate the instrument and to perform all necessary QC checks. Refer to Table 3 for a listing of working standards and for preparation instructions. Refer to Table 5 for element concentrations in working standards.

5.9 5 mg/L yttrium internal standard solution – add 0.5 mL 10000 mg/L yttrium stock standard to a 1000 mL volumetric flask half filled with calibration blank solution. Bring to volume with calibration blank solution.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for trace metals by ICP should be collected and preserved as described in the following table.

Matrix	Container[1]	Volume / Weight	Preservation / Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	6 months
Aqueous (dissolved)	P, G	250 mL	Filter, HNO_3 to pH < 2	6 months
Solid	P, G	10 g	Cool, 4°C	6 months
Tissue	P, G	25 g	Cool, 4°C	6 months

[1] P = polyethylene or, G = glass

7.0 PROCEDURES

7.1 Begin by following the startup and calibration instructions provided in the current revision of Katahdin SOP CA-632, "Operation and Maintenance of the Thermo ICAP 6500 ICP Spectrophotometer"

7.2 Analysis must proceed in the sequence described in Table 8 to ensure that all necessary quality control samples are analyzed at the appropriate frequencies. A minimum of two replicate integrations is required for all standards and samples. Analysis always begins with the analysis of a calibration blank solution (S0) followed by analysis of a multi-element calibration standard (S1 in Table 5) to calibrate the instrument. The system is flushed with calibration blank for two minutes between each sample and standard, and each sample and standard is aspirated for one minute prior to the beginning of emission measurements.

7.3 Analysis continues with analysis of the initial calibration verification standard (ICV) and the initial calibration blank (ICB) to verify the accuracy of the calibration. Refer to Section 8 and Tables 1 through 3 for additional information.

7.4 A continuing calibration verification standard (CCV) and a continuing calibration blank (CCB) must be analyzed at the beginning of the run, after every ten samples, and at the end of the run to verify the continued accuracy of the calibration. Refer to Section 8 and Tables 3 and 5 for additional information.

7.5 Interference check standard solutions (ICSA and ICSAB) must be analyzed at the beginning of the sample run to verify the accuracy of the IEC factors. Refer to Section 8 and Tables 3 and 5 for additional information.

7.6 A practical quantitation limit standard (PQL) must be analyzed at the beginning of each run to determine the accuracy of the calibration at the reporting limit. Refer to Section 8 and Tables 3 and 5 for additional information.

7.7 All sample analytical results for a particular element that are bracketed (preceded or followed) by failing results in a QC sample (ICV, ICB, CCV, CCB, ICSA, or ICSAB) for that element must not be reported. The sample must be reanalyzed for the element in question.

7.8 All samples that exceed the linear dynamic range criteria of 90-110%R must be diluted and reanalyzed. This includes samples with interfering elements that exceed the calibration ranges, because accurate quantitation of interfering elements is necessary for reliable interelement correction. For example, if a sample has been submitted to the laboratory for lead analysis, and the measured aluminum

concentration of that sample exceeds the calibration range for aluminum, it must be diluted sufficiently to bring aluminum within the linear dynamic range and the lead result must be reported from that dilution analysis.

7.9 If dilutions of digested samples are performed, the measured element concentrations must be multiplied by the dilution factor prior to reporting. This is accomplished automatically by entering the dilution factor in the autosampler table prior to initiation of analysis.

7.10 All analyses are performed using yttrium as an internal standard to compensate for enhancement or depression of the analytical signal due to matrix effects. Yttrium solution is pumped at a constant rate through one channel of the peristaltic pump. Samples and standards are pumped through a second channel of the pump. The tubing carrying the internal standard is connected to the tubing carrying samples and standards downstream from the pump and mixing of the two streams is accomplished in a mixing coil downstream from the connection, prior to nebulization. For each sample or standard, the computer that controls the spectrometer divides the detected emission signal for each element by the detected yttrium emission signal prior to quantitation, thus normalizing all emission signals to that of yttrium. The yttrium recovery must be within ± 20% of the counts of the initial calibration blank. If the recovery is outside of this, the sample must be diluted and reanalyzed.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 6010D requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases, data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases, “qualified” data with narration may be advisable after consultation with the client.

In some cases, the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions

may be different than those specified in this SOP. In these cases, the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed. Table 1 lists the QC Checks, minimum frequencies, acceptance criteria, corrective actions, flagging criteria and additional comments for work analyzed in accordance with Method 6010D.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined at least once using new equipment and after major instrument maintenance. This determination requires ten replicate analyses of reagent water blank. The IDL is calculated as the mean blank concentration plus three times the standard deviation of the measured blank concentrations (use zero for the mean if the mean is negative). For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 The lower limit of quantitation (LLOQ) is the lowest point of quantitation.The LLOQ is initially verified by analysis of 7 replicate samples spiked at the LLOQ and taken through all sample preparation and analysis steps. The mean recovery of these standards should be $\pm$ 35% of the true value and the RSD should be $\leq$ 20%.

8.3 Ongoing LLOQ verification must be performed at least quarterly to validate quantitation capability at low analyte concentrations. The LLOQ sample is equivalent to the PQL standard but is carried through the entire sample preparation and analysis process. Element recoveries for the LLQC sample must fall within 70% to 130% of the expected concentrations to confirm the previously established PQLs.

8.4 The upper limit of the linear dynamic range (LDR) must be established for each wavelength utilized. It must be determined from a linear calibration prepared in the normal manner using the established analytical operating procedure for the instrument. The LDR should be determined by analyzing succeedingly higher standard concentrations of the analyte until the observed analyte concentration differs by no more than 10% from the stated concentration of the standard. Determined LDRs must be documented and kept on file. The LDR which may be used for the analyses of samples should be judged by the analyst from the resulting data. Determined sample analyte concentrations that are greater than the determined upper LDR limit must be diluted and reanalyzed. The LDR standards must be analyzed daily after each calibration anywhere during the analytical run. If the LDR standard is not analyzed, or if the recovery in the LDR standard is not within 10% of the true value, the highest standard in the calibration becomes the linear range.

8.5 Spectral interference checks (SIC) - Two types of SIC checks are used. Individual element SIC checks are performed when the instrument is initially setup, and periodically (at least once every 6 months) thereafter. The mixed element SIC solution is used daily to check that the instrument is free from interference from elements typically observed in high concentration and to check that and interference corrections applied are still valid.

8.5.1 Single element interference checks:

At a minimum, single element SIC checks must be performed for the following elements:

- Aluminum 500mg/L
- Boron 50mg/L
- Barium, 50mg/L
- Calcium 500mg/L
- Copper 50mg/L
- Iron 200mg/L
- Magnesium 500mg/L
- Manganese 50mg/L
- Molybdenum 20mg/L
- Sodium 1000mg/L
- Nickel 20mg/L
- Selenium 20mg/L
- Silicon 200mg/L
- Tin 20mg/L
- Vanadium 20mg/L
- Zinc 20mg/L

8.5.2 The absolute value of the concentration observed for any unspiked analyte in the single element SIC checks must be less than two times the analytes' LLOQ. The concentration of the SIC checks are suggested, but become the highest concentration allowed in a sample analysis, and cannot be higher than the highest established linear range. Samples with concentrations of elements higher than the SIC check must be diluted until the concentration is less than the SIC check solution. Note that reanalysis of a diluted sample is required even if the high concentration element is not required to be reported for the specific sample, since the function of the SIC check is to evaluate spectral interferences on other elements.

The single element SIC checks are performed when the instrument is setup and periodically (at least once every 6 months) thereafter.

ANALYTICAL RUN QC SAMPLES

8.6 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared by combining compatible elements from a standard source different than that of the calibration standard and at concentrations within the linear working range of the instrument. The results of the ICV must fall within 90% to 110% of the expected values. If the ICV fails, result for the failing elements may not be reported from the run unless the ICV recovery is greater than 110% and the sample result is less than the PQL.

8.7 Continuing Calibration Verification (CCV) solutions are analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standards used for calibration at concentrations near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected values. If a CCV fails, results for the failing elements may not be reported from the run unless the CCV recovery is greater than 110% and the sample result is less than the PQL. Also, for failing elements, all samples analyzed after the last passing CCV must be reanalyzed.

8.8 Calibration blank solution is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICB must be less than ½ the PQL, and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed, with the following exception. If the result for a CCB or ICB is greater than the PQL, sample results that are less than the PQL or greater than or equal to ten times the measured CCB concentration may be reported. Also, for failing elements, all samples analyzed after the last passing CCB must be reanalyzed, with the exception noted above.

8.9 Interference check solutions ICSA and ICSAB (refer to Section 1.1) are analyzed at the beginning of each run to verify interelement correction factors and background correction. ICSA contains interferent elements (Al, Ca, Fe, and Mg) only, at concentrations of 200 mg/L to 500 mg/L. Results for interfering elements in the ICSA must fall within 80% to 120% of the expected values. Results for unspiked elements in ICSA must fall within ± PQL. ICSAB contains interferent elements at concentrations of 200 mg/L to 500 mg/L, and analytes at concentrations of 20 mg/L or less. Results for all elements (interferents and analytes) in ICSAB must fall within 80% to 120% of the expected values. If the ICSA or ICSAB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICSA or ICSAB has been analyzed.

8.10 A Practical Quantitation Limit (PQL) Check Standard or low level readback is analyzed at the beginning (after the ICV and ICB samples) of each run. Element concentrations in this solution are at the laboratories practical quantitation limit. Element recoveries for the PQL check Standard must fall between 80% - 120% of the expected values. If the PQL Check Standard fails, the results for the failing elements may not be reported from the run, unless the PQL Check Standard recovery is greater than 120% and the samples results are less than the PQL.

PREPARATION BATCH QC SAMPLES

8.11 Each digestion batch of twenty or fewer samples will contain a preparation blank and a laboratory control sample. Each batch will also contain one or more of the following QC samples: laboratory control sample duplicate, sample duplicate, matrix spike sample or matrix spike sample duplicate.

8.12 A preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than ½ the PQL except for common contaminants which must be less than the PQL. If the preparation blank fails, it should be reanalyzed once. If still unacceptable, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL, associated sample results that are less than the PQL or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.13 A laboratory control sample (LCS), consisting of a spiked clean matrix (reagent water or Teflon boiling chips) or a solid reference material carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless vendor-supplied limits (for solid reference materials) or laboratory-generated statistical limits are available. If a laboratory control sample fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested with the following exception. If the LCS fails high, sample results less than the PQL may be reported.

SAMPLE MATRIX QC SAMPLES

8.14 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, the associated sample result must be flagged on the report of analysis. If DoD QSM acceptance criteria are being used, recoveries must be the same as stated for laboratory control samples.

The relative percent difference between sample duplicate, matrix spiked duplicate or LCS duplicate, is calculated as follows:

$$RPD\ (\%) = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = sample result
D_2= duplicate sample result

A control limit of 20% RPD is applied to duplicate analysis if the original sample result is greater than 50X the IDL. If the matrix spike duplicate analysis fails, the associated sample result must be flagged on the report of analysis.

8.15 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 25 x PQL), the measured concentration of a serial dilution (1:5 dilution) of the sample should agree within 80% to 120% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$Difference\ (\%) = \frac{|L-S|}{S} * 100\%$$

where: L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The associated sample result should be flagged on the report of analysis or the sample should be reanalyzed at dilution to eliminate the interference.

8.16 Post-digestion Spike (PDS) additions must be performed for DoD QSM samples if the serial dilution is not within acceptance criteria or if the analyte concentrations in all samples are less than 50x the LOD. The spike addition should produce a concentration that is between 10 and 100x the PQL. The recovery of the PDS must be within 75-125%. If the PDS fails, all samples must be run by method of standard additions or appropriately flagged.

8.17 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The

MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Practical Quantitation Limit (PQL) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. A PQL is analyte and matrix specific and may be laboratory dependent.

The Practical Quantitation Limit (PQL) is the minimum level, concentration, or quantity of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The PQL shall be set at the lowest point in the calibration curve for all analyses utilizing an initial multipoint calibration. PQLs must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters.

MDLs are filed with the Inorganic Department Manager and then with the QAO.

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 6010D for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB, IV, and V July 2014, Method 6010D.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM, Current Version.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, current revision.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010D**

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TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010D**

TABLE 1

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010D	Initial Calibration, minimum 1 point plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient (r) ≥ 0.995	Recalibrate
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	1) Do not use results for failing elements unless the ICV > 110% and the sample < the PQL. 2) Investigate and correct
	Initial Calibration Blank (ICB)	Immediately after the ICV.	Absolute value of ICB < ½ PQL.	1) Do not use results if ≥ PQL and 10x< CCB level. 2) Investigate and correct problem.
	Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of run.	Recovery within ± 10% of true value.	1) Do not use results for failing elements unless the CCV > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Continuing Calibration Blank (CCB)	After every 10 samples and at end of the run.	Absolute value of CCB < PQL.	1) Do not use results if ≥ PQL and < 10x CCB level. 2) Investigate and correct problem.
	Practical Quantitation Level Check Standard (PQL) or low level readback	At beginning of run after ICV and ICB.	Recovery within ± 20% of true value.	1) Do not use results for failing elements unless the recovery > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Interference Check Solution A (ICSA)	At beginning of run after ICV and ICB	For Al, Ca, Fe, and Mg, recovery within ± 20% of true value. For analytes not spiked, ± PQL.	1) Do not use results for failing elements. 2) Investigate and correct problem.
	Interference Check Solution AB (ICSAB)	At beginning of run after ICV and ICB	Recovery of each analyte within ± 20% of true value.	1) Do not use results for failing elements. 2) Investigate and correct problem.
	Preparation Blank (PBW/PBS)	One per digestion batch of 20 or fewer samples.	Less than ½ PQL.	1) Reanalyze once to confirm 2) Investigate source of contamination. 3) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and <10x the blank concentration.
	Laboratory Control Sample (LCSW/LCSS)	One per digestion batch of 20 or fewer samples.	Recovery within ± 20% of true value, unless vendor-supplied or statistical limits have been established.	1) Reanalyze once to confirm 2) Investigate source of problem. 3) Redigest and reanalyze all associated samples if sample concentration ≥ PQL
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample < 4x spike added.	1) Perform serial dilution test and/or post-digestion spike to test for matrix interferences 2) Flag results.
	Matrix Spike Duplicate Sample (P) or sample duplicate	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample < 4x spike added. RPD ≤20% for duplicate spikes and sample duplicates.	1) Flag results.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010D**

TABLE 1

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010D (cont.)	Serial Dilution (L)	One per digestion batch.	If original sample result is at least 25 x PQL, 5-fold dilution must agree within ± 20% of the original result. Flag result or dilute and reanalyze sample to eliminate interference	Perform post digestion spike addition (PDS)
	Post-Digestion Spike Sample (A)	When dilution test fails or analyte concentration in all samples <25 x PQL	Recovery within ± 25%.	Run associated samples by method of standard addition or flag results.
	Internal Standard	Every sample	± 20% (compared to the initial calibration blank)	Dilute sample and reanalyze.
	Instrument Detection Limit (IDL) Study	At least once using new equipement or after major instrument maintenance	IDL < PQL	1) Repeat IDL study. 2) Raise PQL.
	LLOQ (PQL) initial determination	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
	Lower Limit of Quantitation Check (LLQC) Sample	Digest and analyze quarterly or as needed to confirm PQLs	70% - 130% of true value	Re-evaluate PQLs
	Linear Dynamic Range Study	Every six months	Run succeedingly higher stds until recovery not within ± 10%. Use highest passing concentration as upper limit of linear range.	Only accept data to highest passing concentration until next linear range study.
	Linear Dynamic Range verification	During each analytical run	Recovery within 10% of expected value	Do not report results greater than concentration of highest calibration standard

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010D**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-638-02	Method 6010D, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		
QC - Calibration Blanks		

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010D**

TABLE 3

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Calibration Standard (STD1 or S1)	ICP- intermediate Standard	Lab Prepared (see Table 6)	10.0
	QCS 26	High Purity Standards	1.0
Initial Calibration Verification (ICV)	Calibration Standard 3	Claritas PPT	0.96
	1000 mg/L Si	Inorganic Ventures	0.98
	1000 mg/L Al	High Purity Standards	0.96
	IV-28	Inorganic Ventures	0.4
	1000 mg/L Sn, Au	Inorganic Ventures	0.04
Interference Check Sample A (ICSA)	CLPP-ICS-A	Inorganic Ventures	10.0
Interference Check Sample AB (ICSAB)	CLPP-ICS-A	Inorganic Ventures	10.0
	CLPP-ICS-B4	Inorganic Ventures	1.0
	ICSAB-INT	Lab Prepared (see Table 6)	5.0
Continuing Calibration Verification (CCV)	ICP intermediate standard	Lab Prepared (see Table 6)	5.0
	QCS 26	High Purity Standards	0.5
Practical Quantitation Limit Sample (PQL)	PQL-INT	Lab Prepared (see Table 6)	1.0

TABLE 4

PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
PQL-INT	1000 mg/L Li, Sn, Au	High Purity Standards	1.0 each
	10000 mg/L K, Na	High Purity Standards o	1.0 each
	1000 mg/l B	High Purity Standards	0.50
	1000 mg/l Zn	High Purity Standards	0.20
	1000 mg/L Cu	High Purity Standards	0.25
	10000 mg/L Si	High Purity Standards	0.20
	1000 mg/L Ti, Tl	High Purity Standards	0.15 each
	1000 mg/L Se, Mo, Co, Ni, Ag, Sr, V, Cr	High Purity Standards	0.1 each
	10000 mg/L Al	High Purity Standards	0.3
	1000 mg/L As,Sb	High Purity Standards	0.08 each
	1000 mg/L Ba, Be, Cd, Mn, Pb	High Purity Standards	0.05 each
	10000 mg/L Fe, Ca, Mg	High Purity Standards	0.1 each
ICSAB-INT	10000 mg/L K,Na	High Purity Standards or Inorganic Ventures	4.0 each
	10000 mg/L B, Li, Mo,Sr,Sn,Ti, Au	High Purity Standards	1.0 each
	10000 mg/L Si	High Purity Standards	0.40
ICP-INT STD (Intermediate)	10000 mg/L Si	High Purity Standards	2.5
	10000 mg/L Ca, Mg, Fe, Al, Na	High Purity Standards	2.4
	10000 mg/L K	High Purity Standards	1.5
	1000 mg/L Au, Li, Sn. Sr	High Purity Standards	1.0

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010D**

TABLE 5

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L							
	STD1	ICV	PQL	ICSA	ICSAB	CCV	AL_IEC	FE_IEC
Aluminum	25	10	0.3	500	500	12.5	500	
Antimony	1	0.4	0.008		0.6	0.5		
Arsenic	1	0.4	0.008		0.1	0.5		
Barium	1	0.4	0.005		0.5	0.5		
Beryllium	1	0.4	0.005		0.5	0.5		
Boron	1	0.4	0.05		0.5	0.5		
Cadmium	1	0.4	0.005		1.0	0.5		
Calcium	25	10	0.10	500	500	12.5		
Chromium	1	0.4	0.01		0.5	0.5		
Cobalt	1	0.4	0.01		0.5	0.5		
Copper	1	0.4	0.025		0.5	0.5		
Gold	1	0.4	0.1		0.5	0.5		
Iron	25	10	0.1	200	200	12.5		200
Lead	1	0.4	0.005		0.05	0.5		
Lithium	1	0.4	0.1		0.5	0.5		
Magnesium	25	10	0.10	500	500	12.5		
Manganese	1	0.4	0.005		0.5	0.5		
Molybdenum	1	0.4	0.01		0.5	0.5		
Nickel	1	0.4	0.01		1	0.5		
Potassium	25	13.6	1		20	12.5		
Selenium	1	0.4	0.01		0.05	0.5		
Silicon	25.5	10	0.2		2	12.75		
Silver	1	0.4	0.01		0.2	0.5		
Sodium	25	10	1		20	12.5		
Strontium	1	0.4	0.01		0.5	0.5		
Thallium	1	0.4	0.015		0.1	0.5		
Tin	1	0.4	0.1		0.5	0.5		
Titanium	1	0.4	0.015		0.5	0.5		
Vanadium	1	0.4	0.01		0.5	0.5		
Zinc	1	0.4	0.02		1.0	0.5		

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010D**

TABLE 6

ELEMENT CONCENTRATIONS IN INTERMEDIATE STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L		
	ICP Intermed STD	PQL-INT	ICSAB-INT
Aluminum	240	30	
Antimony		0.8	
Arsenic		0.8	
Barium		0.5	
Beryllium		0.5	
Boron		5	10
Cadmium		0.5	
Calcium	240	10	
Chromium		1.0	
Cobalt		1.0	
Copper		2.5	
Iron	240	10	
Lead		0.5	
Lithium	10	10	10
Magnesium	240	10	
Manganese		0.5	
Molybdenum		1.0	10
Nickel		1.0	
Potassium	150	100	400
Selenium		1.0	
Silicon	250	20	40
Silver		1.0	
Sodium	240	100	400
Strontium	10	1.0	10
Thallium		1.5	
Tin	10	10	10
Titanium		1.5	10
Vanadium		1.0	
Zinc		2.0	
Gold	10	10	10

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010D**

TABLE 7

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L				
	IV-28	QCS-26	CLPP-ICS-A	CLPP-ICS-B4	CL-CAL-3
Aluminum	100	100	5000		
Antimony	100	100		60	
Arsenic	100	100		10	
Barium	100	100		50	
Beryllium	100	100		50	
Boron	100	100			
Cadmium	100	100		100	
Calcium	100	100	5000		1000
Chromium	100	100		50	
Cobalt	100	100		50	
Copper	100	100		50	
Iron	100	100	2000		1000
Lead	100	100		5	
Lithium	100				
Magnesium	100	100	5000		1000
Manganese	100	100		50	
Molybdenum	100	100			
Nickel	100	100		100	
Potassium	1000	1000			1000
Selenium	100	100		5	
Silicon	50	50			
Silver	100	100		20	
Sodium	100	100			1000
Strontium	100				
Thallium	100	100		10	
Tin					
Titanium	100	100			
Vanadium	100	100		50	
Zinc	100	100		100	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010D**

TABLE 8

REQUIRED ANALYTICAL SEQUENCE

Sequence Number	Standard/Sample	Purpose
1	Blank (Calibration Blank)	Initial calibration
2	S1 (Calibration Standard)	Initial calibration
3	ICV (Initial Calibration Verification)	Check calibration accuracy
4	ICB (Initial Calibration Blank)	Check calibration accuracy
5	PQL (Practical Quantitation Level Sample)	Check calibration accuracy near PQL, repeat before final CCV, CCB
6	ICSA (Interference Check Solution A)	Verify accuracy of IEC factors, repeat before final CCV, CCB
7	ICSAB (Interference Check Solution AB)	Verify accuracy of IEC factors, repeat before final CCV, CCB
8	CCV (Continuing Calibration Verification)	Check calibration stability
9	CCB (Continuing Calibration Blank)	Check calibration stability
10-19	Analyze up to 10 samples	
20	CCV (Continuing Calibration Verification)	Check calibration stability
25	CCB (Continuing Calibration Blank)	Check calibration stability
…	Continue analyzing sequences of up to 10 samples, followed by a CCV and a CCB	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010D**

ATTACHMENT 1

HARDNESS BY CALCULATION

As referenced in "Standard Methods for the Examination if Water and Wastewater," Methods 2340 A & B, Hardness Introduction and Hardness by Calculation, American Public Health Association, 18th Edition, Revised 1992, total hardness is the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in milligrams per liter.

Once the calcium and magnesium concentrations have been determined by EPA methods 6010D, 6020, 200.7 or 200.8, the total hardness of an aqueous sample may be calculated as follows:

$$\text{Total Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L) + 4.118\ (Mg, mg/L)$$

The calcium hardness of an aqueous sample may also be calculated as follows:

$$\text{Calcium Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L)$$

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010D**

ATTACHMENT 2

ANALYSIS OF PALLADIUM BY SW846 6010D

Palladium may be analyzed by EPA Method SW846 6010DC following the method outlined in this SOP. However, due to significant spectral interferences caused by addition of palladium to the calibration and check standards used in this method, palladium is added to aliquots of the regular standards as needed for analysis. Two stock standards (1000 mg/L) are currently kept for palladium analysis. One is purchased from High Purity Standards and is used for calibration, PQL, ICSAB, and CCV. The other is purchased from Inorganic Ventures and is used as the independent check standard (ICV). Analysts should add palladium stock to the regular standards according to the table below:

Name of Working Standard	Volume of Standard Aliquot (mL)	Volume of Palladium Stock Added (mL)	Concentration of Palladium (mg/L)	Source of Palladium Stock
Calibration Std.	50	0.05	1.0	High Purity
ICV	50	0.02	0.4	Inorganic Ventures
PQL	50	0.005	0.1	High Purity
ICSAB	50	0.025	0.5	High Purity
CCV	50	0.025	0.5	High Purity

Prior to starting the run, a palladium-only standard should be analyzed along with the iron and aluminum standards to evaluate interelement correction factors as outlined in Katahdin SOP CA-632, Section 7.1.

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: CA-627
Revision History
Cover Page
Page 1

TITLE: TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020

Prepared By: George Brewer Date: 03/01

Approved By:

Group Supervisor: George Brewer Date: 04/02/01

Operations Manager: Jol C Butler Date: 3/29/01

QA Officer: Deborah J. Nadeau Date: 03.27.01

General Manager: Duncan F. Hughes Date: 04/03/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Changed acid solution conc. Changed Run ID Naming convention added data reduction and reporting procedures Updated Standards tables (4-8) Updated Table 10 in include ISIS configuration	LAD	02-16-05	02-16-05
02	Sect. 4.2 - changed tedlar bag size Sect. 5 - changed acid conc.'s Sect. 7 - major changes to reflect current practises including reporting data in the metals database. Sect. 8 - major changes updating acceptance criteria. Updated tables 1, 3 thru 8, 10 & 11	LAD	04/06	04/06
03	Updated Tables 4.5 and 6 with current standards. Updated Table 1 with serial dilution, Post Digestion Matrix spike, MSA, ICS-A, ICS-AB and IDL minimum frequency or criteria. Updated Sect. 8 regarding client specific requirements.	LAD	07/07	07/07
04	Section 7.18-changed instrument identifier to reflect new instrument; section 8-changed acceptance criteria and ICSAB analyte list; Table 1. updated acceptance criteria and corrective action for QC. Table 3-added all analytes to list-removed "for information only" list.	LAD	04/08	04/08
05	Updates to reflect changes from 6020 to 6020A. Added Hardness by calculation attachment. Added LLOC requirement and criteria to Sect 8 and Table 1. Added criteria to analyze PQL Std. at beginning and END of run.	LAD	02/09	02/09

TITLE: TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Sect. 8 and QC Tables - Added DoD QSM references and criteria. Section 10 - Added references. Tables 4 → 7 - Added information pertaining to CCV conc. change	LAD	08/09	08/09
07	Added Table 2 with DoD QSM ver. 4.1 QC requirements. Updated Section 4.1, Table 10 and Table 11 with new autosampler information.	LAD	04/10	04/10
08	Sect. 1.1 - Added definitions. Sect. 5.4.1, 4.2, 5.2 7.9, 7.10, 7.11, 7.16 and 9 8.7 - minor changes to reflect current practice. Sect. 9 - added MDL, LOD and LOQ information. Sect. 10 - Added, ~~edited references~~. Updated Tables edited references LAD 042512 1, 5, 6, 7, 8 and 9	LAD	04/12	04/12
09	Sect. 7 - Added reference to autosampler software, added printing calibration and removed printing of run summary	LAD	08/13	08/13
10	Sect. 7 - Updated for changes made in the metals database for importing and handling data. Sect. 10 - updated and added references. Added Table 3 - DoD QSM 5.0 QC Requirements	LAD	06/14	06/14
11	Sect. 7, Table 1, 2, 3, 6, 8 & 11 - Updated to reflect change from 5 pt. to 2 pt. calibration. Table 7, 8 & 9 - Updated to reflect change in Aluminum PQL	LAD	04/16	04/16
12	Change title of Section 5.0. Update method references for NELAC + DoD. Minor additions/corrections to Sections 3.0, 4.2, 7.3.5 and table 5.	LAD	09/17	09/17
13	Table 1 - Added ms/msD, corrected section references - Table 2 - Removed, DoD QSM 4.2 QC Requirements. Renumbered subsequent sections - Table 7 - Added Thorium - Updated Table references throughout SOP	LAD	01/19	01/19
14	Sect.s 4, 7, 8 and 10 updated for the new Agilent 7800 ICPMS System and the Mass Hunter Software.	LAD	06/20	06/20

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-627-14**, titled **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**
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Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-627-14**, titled **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

Recipient: _____ Date: _____

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

1.0 SCOPE AND APPLICATION

Inductively coupled plasma-mass spectrometry (ICP-MS) is applicable to the determination of sub-ppb (ug/L) concentrations of a large number of elements in water samples and in waste extracts or digests. When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are required.

ICP-MS has been applied to the determination of over 60 elements in various matrices. Analytes for which EPA has demonstrated the acceptability Method 6020 in a multi-laboratory study on solid wastes are listed as “analytes” in Table 4. Instrument detection limits, sensitivities, and linear ranges will vary with the matrices, and operating conditions.
If Method 6020 is used to determine any analyte not listed in Table 4, it is the responsibility of the analyst to demonstrate the accuracy and precision of the method in the waste to be analyzed. The analyst is always required to monitor potential sources of interferences and take appropriate action to ensure data of known quality.

An appropriate internal standard is required for each analyte determined by ICP-MS. Recommended internal standards are ^{6}Li, ^{45}Sc, ^{89}Y, ^{103}Rh, ^{115}In, ^{159}Tb, ^{165}Ho, and ^{209}Bi. The lithium internal standard should have an enriched abundance of ^{6}Li, so that interference from lithium native to the sample is minimized. Other elements may need to be used as internal standards when samples contain significant amounts of the recommended internal standards.

4.1. Definitions:

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy.

ICP-MS - Inductively Coupled Plasma Mass Spectrometry.

ICS - Interference Check Samples - Two standards (ICS-A and ICS-AB) used to verify the effectiveness of interference correction equations. Solution ICS-A contains only interferents (Al, Ca, Fe, Mg, Na, K, P, S, Mo, Ti, C, Cl) at high

concentrations; solution ICS-AB contains interferents at the same concentrations as well as analytes at low (20 ug/L) concentrations.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence.

Internal Standard - Pure analytes added to a sample, extract, or standard solution in known amounts and used to measure the relative responses of other method analytes that are components of the same sample or solution. Internal standards must be analytes that are not native to the sample.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

LDR - Linear Dynamic Range - The concentration range over which the instrument response to an analyte is linear.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

LOQ – Limit of Quantitation.- The minimum concentration of a target analyte that produces a quantitative result within specified limits of precision and bias.

PB - Preparation Blank - Reagent water that has been brought through the sample preparation process.

Post-Digestion Spike - An aliquot of a sample to which a known amount of analyte has been added before analysis and after digestion, if digestion is required.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in ICP-MS analysis by USEPA Method 6020 who are knowledgeable in the recognition and in the correction of spectral, chemical, and physical interferences in ICP-MS. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in ICP-MS analysis by USEPA Method 6020 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented, and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

Samples, sample digestates, standards, and other reagents used in ICP analysis may contain high concentrations of acids and toxic metals. Spilled samples and reagents should be cleaned up from instrument and laboratory surfaces immediately.

Liquid argon represents a potential cryogenic and suffocation hazard and safe handling procedures should be employed at all times when handling liquid argon

tanks and fittings. Safety glasses and cryogenic-resistant gloves should be worn when changing or adjusting argon tanks.

The Agilent 7800 ICP-MS spectrometer is safety-interlocked to prevent user exposure to harmful electrical voltages, radio frequency emissions, ultraviolet radiation, high temperatures, and other hazards. At no time should the operator attempt to disable these interlocks or operate the instrument if any safety interlock is suspected to be disabled

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention and waste minimization techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in ICP-MS spectrometry may contain high concentrations of acids and toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested samples and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled “Waste Stream A”) that is located in the Metals Instrument lab. Further information regarding waste classification and disposal may be obtained by consulting Katahdin Analytical Environmental Health and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, samples that require total (“acid-leachable”) values must be digested using appropriate sample preparation methods (such as USEPA Methods 3005 - 3051).

2.2 USEPA Method 6020 describes the multi-elemental determination of analytes by ICP-MS. The method measures ions produced by a radio-frequency inductively coupled argon 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 a vacuum 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.

3.0 INTERFERENCES

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). The Agilent 7800 MassHunter data system is 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. 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 which could affect ICP-MS determinations have been identified. Examples include $ArCl^+$ ions on the As signal and MoO^+ ions on the cadmium isotopes. While the approach used to correct for molecular isobaric interferences is demonstrated below using the natural isotopic abundances from the literature, the most precise coefficients for an instrument must be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1 percent) counting statistics. Because the ^{35}Cl natural abundance of 75.77 percent is 3.13 times the ^{37}Cl abundance of 24.23 percent, the chloride correction for arsenic can be calculated (approximately) as follows (where the $^{38}Ar^{37}Cl^+$ contribution at m/z 75 is a negligible 0.06 percent of the $^{40}Ar\ ^{35}Cl^+$ signal):

Corrected ^{75}As signal (using natural isotopic abundances for coefficient approximations) = (m/z 75 signal) - (2.95) (m/z 77 signal) + (2.548) (m/z 82 signal) – (2.571) (m/z 83 signal), where the final term adjusts for any selenium contribution at 77 m/z.

NOTE: Arsenic values can be biased high by this type of equation when the net signal at m/z 82 is caused by ions other than $^{82}Se^+$, (e.g., $^{81}BrH^+$ from bromine wastes or ^{82}Kr from krypton contamination in the Ar).

Similarly:
Corrected ^{114}Cd signal (using natural isotopic abundances for coefficient approximations) = (m/z 114 signal) - (0.027) (m/z 118 signal) - (1.84)(m/z 108 signal),

where last 2 terms adjust for any tin or MoO^+ contributions at m/z 114.

NOTE: Cadmium values will be biased low by this type of equation when $^{92}ZrO^+$ ions contribute at m/z 108. Also, use of m/z 111 for Cd is even subject to direct ($^{92}ZrOH^+$) ions and indirect ($^{90}ZrO^+$) additive interferences when Zr is present.

NOTE: As for the arsenic equation above, the coefficients in the Cd equation are only illustrative. The most appropriate coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<l percent) counting precision.

The interference correction equations that are used by this laboratory in performing USEPA Method 6020 are listed in Table 4. The accuracy of these types of equations is based upon the constancy of the observed isotopic ratios for the interfering species. Corrections that presume a constant fraction of a molecular ion relative to the “parent” ion have not been

found to be reliable, e.g., oxide levels can vary. If a correction for an oxide ion is based upon the ratio of parent-to-oxide ion intensities, the correction must be adjusted for the degree of oxide formation by the use of an appropriate oxide internal standard previously demonstrated to form a similar level of oxide as the interferent. This type of correction has been reported for oxide-ion corrections using ThO^+/Th^+ for the determination of rare earth elements. The use of aerosol desolvation and/or mixed plasmas have been shown to greatly reduce molecular interferences (the Agilent 7800 ICP-MS spectrometer employs spray chamber cooling to effect aerosol desolvation). These techniques can be used provided that method detection limit, accuracy, and precision requirements for analysis of the samples can be met.

3.1 Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000 mg/L) are recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes. The internal standard used should differ from the analyte of interest by no more than 50 amu. See Table 14 for a list of internal standards used. When the intensity level of an internal standard is less than 70 percent or greater than 120 percent of the intensity of the first standard used during calibration, the sample must be reanalyzed after a fivefold (1+4) or greater dilution has been performed.

3.2 Memory interferences can occur when there are large concentration differences between samples or standards that are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affect the extent of the memory interferences that are observed. The rinse period between samples must be long enough to eliminate significant memory interference.

4.0 APPARATUS AND MATERIALS

4.1 Agilent 7800 ICP-MS system, consisting of the Agilent 7800 ICP-mass spectrometer and its controlling computer data station. The spectrometer is capable of providing resolution better than or equal to unit resolution at 10% peak height. The Agilent 7800 mass range of 2-260 amu exceeds the method requirement of 2- 240 amu. The Agilent 7800 MassHunter software allows automatic corrections for isobaric interferences and correction for internal standard responses as required by the method. All critical argon flows including nebulizer argon are under mass flow controller control and a peristaltic pump is used for sample introduction. Peripheral equipment includes a Elemental Scientific SC-4 PX Fast Autosampler and Sample

Introduction system, and Bullzip PDF printer set to print to file ICPMS_CP.pdf located in folder PDF_PRINTS on the desktop.

4.2 Peristaltic pump tubing – 3-stop ESI PVC flared black-black (0.76 mm ID) and orange-green-orange (0.38 mm ID). 2-stop ESI PVC flared red-red (1.14 mm ID).

4.3 15 ml 17x100 mm polypropylene or polystyrene disposable test tubes for samples and 50 ml polypropylene centrifuge tubes for standards.

4.4 Automatic adjustable volume pipetters of suitable precision and accuracy. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.5 Trace metal grade pipette tips.

4.6 Volumetric glassware or plasticware of suitable precision and accuracy.

4.7 Talc free vinyl gloves.

4.8 Argon gas supply (high purity grade gas or liquid, 99.99%).

4.9 For the determination of trace levels of elements, contamination and loss are of prime consideration. Potential contamination sources include improperly cleaned laboratory apparatus and general contamination within the laboratory environment from dust etc. A clean laboratory work area designed for trace element sample handling must be used. Standards, samples, and blanks should be exposed to the laboratory environment as little as possible. The use of preparation blanks and spikes should be used to verify the absence of sources of contamination and loss. If necessary, polypropylene sample tubes should be rinsed and stored in dilute acid prior to use.

NOTE: Chromic acid must not be used for cleaning glassware for trace metals analysis.

5.0 REAGENTS AND STANDARDS

5.1 Acids used in the preparation of standards and for sample processing must be of high purity. Redistilled acids are recommended because of the high sensitivity of ICP-MS. Mallincrodt/Baker “Instra-Analyzed” trace-metals grade acids are appropriate. It is important to match the acid concentration in standards and samples. Concentrations of antimony and silver between 50-500 ug/L require 1% (v/v) HCl for stability; for concentrations above 500 ug/L additional HCl will be needed. For this reason, it is recommended that antimony and silver concentrations in samples and standards be maintained below 500 ppb wherever possible. Acids are received in poly-coated glass bottles, and are stored under the hood in the

Metals sample preparation laboratory at room temperature until use. All acids are considered to have a shelf life of three years from date of receipt unless otherwise indicated by the vendor. Refer to the current revision of Katahdin SOP CA-105, Reagent and Solvent Handling, for further details.

5.2 Laboratory reagent grade water, trace metals free, equivalent to ASTM Type 1 (ASTM D 1193), >18 Megohm/centimeter resistivity.

5.3 Single element and multielement stock standard solutions – purchased standards prepared from high purity salts or metals and supplied by the vendors with certificates of purity and analysis. Refer to Tables 6 and 7 for a listing of stock standards required, and to Table 9 for element concentrations in stock standards. Purchased stock standards are received in polyethylene containers and are stored in their original containers at room temperature in the Metals Standards Preparation Laboratory. All purchased stock standards are given an expiration date as indicated by the manufacturer. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.4 Intermediate standard solutions – laboratory-prepared multielement standards that are used in the subsequent preparation of working standards. Refer to Table 8 for a listing of intermediate standards required and for preparation instructions. Refer to Table 7 for element concentrations in intermediate standards. Intermediate standards are stored at room temperature in acid-washed polyethylene containers in the Metals Standards Preparation Laboratory. Intermediate standards are assigned an expiration date of three months from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.5 Working standard solutions – laboratory-prepared multielement standards that are used to calibrate the instrument, to provide internal standardization through on-line addition, and to perform all necessary QC checks. Refer to Table 5 for a listing of working standards and for preparation instructions. Refer to Table 7 for element concentrations in working standards. Working standards are stored at room temperature in acid-washed polyethylene containers in the Metals Standards Preparation Laboratory. All working standards except the ICSA and ICSAB solutions are assigned an expiration date of three months from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. The ICSA and ICSAB solutions are assigned an expiration date of one week from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.6 Blanks: Three types of blanks are required for the analysis. The calibration blank is used in establishing the calibration curve. The preparation blank is used to monitor

for possible contamination resulting from the sample preparation procedure. The rinse blank is used to flush the system between all samples and standards.

5.6.1 The calibration blank consists of the same concentrations of the same acid(s) used to prepare the final dilution of the analyte calibration solutions (currently 1% HNO_3 and 0.5% HCl, v/v, in laboratory reagent grade water). Use of HCl for antimony and silver is cited in Section 5.1.

5.6.2 The preparation blank must be carried through the complete preparation procedure and contain the same volumes of reagents as the associated digested sample solutions.

5.6.3 The rinse blank consists of 4% HNO_3 and 0.5% HCl,v/v, in reagent water. Prepare a sufficient quantity to flush the system between standards and samples.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for trace metals by ICP-MS should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	6 months
Aqueous (dissolved)	P, G	250 mL	Filter, HNO_3 to pH < 2	6 months
Solid	P, G	10 g	Cool, 4°C	6 months

[1] P = polyethylene or, G = glass

7.0 PROCEDURES

7.1 Instrument control and data acquisition are completely automated through the use of the Agilent MassHunter software.

7.2 Initialize the autosampler by opening “ESI SC” located on the Desktop and click “Initialize.” Wait for the autosampler to initialize and the COM port to be opened, indicated by the text color changing to GREEN.

7.3 The main MassHunter software is accessed by double-clicking the “ICP-MS Instrument Control” icon on the Desktop.

7.4 Turn on the argon supply at the tank and set the pressure to >700 kPa.

7.5 Turn on the water chiller/recirculator.

7.6 Verify that the exhaust hood is in operation.

7.7 Ensure that the internal standard solution bottle is adequately full. Consumption is approximately 2.5 mL/hour.

7.8 Verify that the rinse station reservoir has an adequate supply of reagent water.

7.9 Verify that the drain reservoir has adequate room to accept the day's drain waste. Empty the reservoir as necessary into an appropriate waste container (Waste Stream A) located in the Hazardous Waste Storage Area.

7.10 Inspect the peristaltic pump tubes for signs of flattening and wear, and replace them as necessary. Clamp the peristaltic pump tubes into the peristaltic pump.

7.11 Move the carrier and internal standard probe to a blank solution.

7.12 Open the MassHunter software by double-clicking the "ICP-MS Top" icon. Initiate the plasma by selecting Plasma>>Plasma On>>Okay and allow the instrument to aspirate calibration blank solution for at least 10 minutes. During this warm-up, select Start Signal Monitor to start the instrument scanning the mass range. Verify that the flow of sample and internal standard solutions through the uptake lines and into the nebulizer is free from pulsations by introducing an air bubble into each line and observing its progress. Adjust the pump clamp tension on each line to obtain a constant, pulse-free flow.

7.13 Move the carrier line to the Agilent Tune Solution (ATS). Verify there is ATS in the ATS autosampler tube. Increase the nebulizer pump speed to quickly pump solution through the lines. Verify there are no air bubbles in the lines by allowing a large bubble to pass through both the carrier and internal standard lines followed by the previously mentioned solutions.

7.14 After the warm-up, check the responses of masses 82 and 83 to insure minimal krypton intereference with selenium. Mass 83 response should be < 2000 counts per second. Then aspirate the Instrument Tune Solution (10 ppb Li, Y, Ce, Tl) and check the responses and RSDs at masses 7, 89, and 205.

7.15 Open a new batch by selecting Batch>>Open Batch from Template>> 1PT_Cal_ORS. Rename the batch to the meet standard format for ICP-MS runs. The protocol for naming data files is as follows: the 1st character is a letter that identifies the instrument ("L" for the Agilent 78000 ICP-MS), the 2nd character is a letter that identifies the year of the run ("G" for 2013, "H" for 2014, etc.), the 3rd character is a letter that identifies the month of the run ("A" for January, "B" for

February, etc.), the 4th and 5th characters are numbers that identify the date of the run ("01" for the first day of the month, etc.), and the 6th character is a letter that sequentially identifies the run ("A" for the first run of the day on that instrument, "B" for the second run, etc.). For example, the run identified as "LME01A" is the first run of the day that was performed on May, 1, 2019, using the Agilent 7800 ICP-MS.

7.16 Generate a tune report by selecting Acq Method>>Tune>>Perform Auto Tune>> All Tune Modes. Evaluate the tune report against the tune specifications listed in Table 12. If the tune passes all specifications, proceed to step 7.14.

7.17 If the tune report indicates unacceptable instrument performance for any parameter, initiate an autotune by selecting Tune>>Autotune>>Run. The MassHunter software will attempt to tune the instrument to meet the tune specifications, and will generate a new tune report after autotuning. Evaluate the new tune report against the tune specifications listed in Table 13.

7.18 Repeat step 7.18 until all tune specifications have been met. File the final tune report.

7.19 Edit the sequence template to create an analytical sequence table listing all of the samples to be analyzed. Fill in the sample table with sample type, sample IDs, vial numbers, dilution factors.

7.20 Load the autosampler racks according to the analytical sequence.

7.21 Select Add to QUEUE. The instrument will analyze all samples in the order listed in the table. Analysis must proceed in the sequence described in Table 10 to ensure that all necessary quality control samples are analyzed at the appropriate frequencies. A minimum of three replicate scans is required for all standards and samples. Analysis always begins with the analysis of a calibration blank followed by at least one multielement calibration standard to calibrate the instrument. The system is flushed with rinse blank between each sample and standard, and each sample and standard is aspirated for at least one minute prior to the beginning of mass scanning.

7.22 Analysis continues with analysis of the initial calibration verification standard (ICV) and the initial calibration blank (ICB) to verify the accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.23 A practical quantitation limit standard (PQL) is analyzed at the beginning of the run to verify calibration accuracy at the reporting limit. Refer to Section 8 and Table 1 for additional information.

7.24 A continuing calibration verification standard (CCV) and a continuing calibration blank (CCB) must be analyzed at the beginning of the run, after every ten samples,

and at the end of the run to verify the continued accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.25 Interference check standard solutions ICS-A and ICS-AB must be analyzed at the beginning of each run and every 12 hours to verify the adequacy of interference corrections. Refer to Section 8 and Table 1 for additional information.

7.26 All sample analytical results for a particular element that are bracketed (preceded or followed) by failing results in a calibration verification sample (ICV, ICB, CCV, or CCB) for that element must not be reported, except as noted in Sections 8.5, 8.6, and 8.9. The sample must be reanalyzed for the element in question.

7.27 All samples that exceed the linear dynamic range must be diluted and reanalyzed.

7.28 If dilutions of digested samples are performed, the measured element concentrations must be multiplied by the dilution factor prior to reporting. This is accomplished automatically by entering the dilution factor in the sample log table prior to initiation of analysis.

7.29 If an element has more than one monitored isotope, examination of the concentration calculated for each isotope, or the isotope ratios, will provide useful information for the analyst in detecting a possible spectral interference. Consideration should therefore be given to both primary and secondary isotopes in the evaluation of the element concentration. In some cases, secondary isotopes may be less sensitive or more prone to interferences than the primary recommended isotopes, therefore differences between the results do not necessarily indicate a problem with data calculated for the primary isotopes. In the case of Pb, quantitation is based on the sum of isotopes 206, 207 and 208 to compensate for any variation in naturally occurring isotope ratios. This is accomplished through the use of the interference correction equation for lead.

7.30 Calculations, aqueous samples: Final element concentrations for aqueous samples are reported in units of micrograms per liter (ug/L). The reported concentrations are calculated from measured digestate concentrations as follows:

$$\text{Concentration (ug/L)} = \frac{MC \times DF \times FV}{IV}$$

where: MC = Measured digestate concentration (ug/L)
DF = Instrument dilution factor
FV = Final digestate volume (L)
IV = Digested sample volume (L)

7.31 Calculations, solid samples: Final element concentrations for solid samples are reported in units of milligrams per kilogram (mg/kg) on a dry weight basis. The

reported concentrations are calculated from measured digestate concentrations as follows:

$$\text{Concentration (mg/kg dry weight)} = \frac{MC \times DF \times FV \times 100}{W \times S}$$

where: MC = Measured digestate concentration (ug/L)
DF = Instrument dilution factor
FV = Final digestate volume (L)
W = Weight of digested wet sample (g)
S = Percent solids

DATA REDUCTION AND REPORTING

7.32 Follow these steps to create the data import file: From the Online (or Offline) ICP-MS Data Analysis window, open the specific batch to be imported to the database.

7.33 Once opened, use the number column (furthest to the left) to highlight all samples to be imported. Click and drag down, or alternatively click, shift, scroll and click. Always include two rinses after the closing PQL in the import file to ensure proper database functionality.

7.34 Once all samples are highlighted, click the down-facing arrow next to the “Report” icon at the top of the ICP-MS Data Analysis window, click “LIMS – Export Selected Samples,” and wait for a text file to open and notification to appear. Close the text file and notification. The text file that appears is the import file to be imported into the database.

To import data into the Metals Database:

7.35 Open the Metals Database “**KIMS_Metals**” from the Desktop.

7.36 Click “**Data Import Menu**,” then **enter analyst initials** and in this order click: “**Agilent 7800**,” and “**Import and Validate Run**.”

7.37 In the window that appears, in this order, click: “**Desktop**”, “**metals on Katahdin File Server (Server_a)**,” ”**L-ICPMSDATA**.” From here, open the text file created in the process above (i.e. “LNF09A.txt”).

7.38 Allow all queries to run and wait for batch QC results to appear. Uncheck the boxes next to all failing batch QC results. Click away from the last unchecked box to finalize the selection. Click “**Accept Selected Batch QC**.”

7.39 Minimize the main menu form. In the window **“KIMS_Metals : Database**,” Click the header: “**Queries**,” and find the query “**transfer requested data from archive to**

instr data." To the right, click "**Design**." The fourth column in the window that opens should have the header called "**File**." Find that column, and under it in the row that calls for "**Criteria**," type the file name you just imported (i.e. "LNF09A"). Close and save this window. Note: This window can be used to filter by many other criteria as well.

7.40 Review and accept or reject the data in the table "**Archive Data**" based on the QC limits in the Table 1 and Table 2 of the following sections.

7.41 Restore the main menu.

7.42 Click "**Additional Data Settings**"

7.43 Click "**Report Added Compounds**"

7.44 Accept the data that comes through. If this process takes more than five minutes, click only once and DO NOT HOLD **Ctrl + Break**. Once the main menu reappears, open the "**Instrument Data**" table and delete its contents.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 6020 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases, data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases, "qualified" data with narration may be advisable after consultation with the client.

In some cases, the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases, the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument. This determination requires seven replicate analyses of a reagent blank, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed by each method on each instrument. This determination requires at least seven replicate digestions and analyses of reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the seven replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 A Lower Limit of Quantitation Check (LLQC) sample must be prepared and analyzed annually or on an as-needed basis to confirm the laboratory's Practical Quantitation Limits (PQLs). The LLQC sample is equivalent to the PQL standard (Section 8.9) but is carried through the entire sample preparation and analysis process. Element recoveries for the LLQC sample must fall within 70% to 130% of the expected concentrations to confirm the previously established PQLs.

ANALYTICAL RUN QC SAMPLES

8.4 Initial instrument calibration: The instrument is calibrated by running a calibration blank and at least one multielement calibration standard. For each element, calibration is performed fitting a single order equation to the calibration data, as follows:

$$Y=aX + [Blank]$$

where: Y = Concentration (ug/L)
X = Measured signal intensity (counts per second)
a = Slope of the calibration line
[Blank] = Measured signal intensity of the calibration blank

8.5 The intensities of all internal standards must be monitored for every analysis. When the intensity of any internal standard fails to fall between 30 and 120 percent of the intensity of that internal standard in the initial calibration standard, the following procedure is followed. The sample must be diluted and reanalyzed. This procedure must be repeated until the internal standard intensities fall within the prescribed window. The intensity levels of the internal standards for the calibration blanks (ICB and CCBs) and calibration verification standards (ICV and CCVs) must agree within

± 20 percent of the intensity level of the internal standard of the original calibration solution. If they do not agree, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and reanalyze the affected samples.

8.6 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared by combining compatible elements from standard sources different than those of the calibration standards and at concentrations within the linear working range of the instrument. The results of the ICV must fall within 90% to 110% of the expected values. If the ICV fails, result for the failing elements may not be reported from the run, unless the ICV recovery is greater than 110% and the sample result is less than the PQL.

8.7 Continuing Calibration Verification (CCV) solutions are analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standards used for calibration at concentrations near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected values. If a CCV fails, results for the failing elements in samples bracketed by the failing CCV may not be reported, unless the CCV recovery is greater that 110% and the sample result is less that the PQL. For DoD analyses, results may not be reported without a valid CCV or report flagged results if reanalysis is not possible.

8.8 A Practical Quantitation Limit (PQL) Check Standard or low level continuing calibration verification (LLCCV) is analyzed at the beginning of each run (after the ICV and ICB samples) and at the end of each run. Element concentrations in this solution are one-fifth the laboratory's practical quantitation limit (assuming a 5-fold dilution of all digestates during analysis). Element recoveries for the PQL Check Standard must fall within 70% to 130% of the expected values (unless the samples analyzed are for the Department of Defense (80% to 120% recovery limits) or other client-specific limits are imposed). If the PQL Check Standard fails, results for the failing elements may not be reported from the run, unless the PQL Check Standard recovery is greater than the high limit and the sample result is less than the PQL.

8.9 A calibration blank solution is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the applicable reporting limit (or PQL) for each element. The reporting limit should be determined on a project specific basis, taking into account the data quality objectives for the project. This information must be communicated through a project QAPP and through the Katahdin project manager. When no project specific reporting limit is specified, the laboratory PQL shall be used. Some project specific limits may require reporting down to the MDL or IDL and taking corrective action based on these levels. Results that fall between the PQL and the IDL or MDL must always be flagged as "estimated" with a "J".

8.10 If an ICB or a CCB fails the acceptance criteria of less than the reporting limit, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed, with the following exception. If the result for an ICB or CCB is greater than the PQL (or reporting limit), sample results that are less than the PQL (or reporting limit) or that are greater than or equal to ten times the measured ICB or CCB concentration may be reported. Also, for failing elements, all samples analyzed after the last passing CCB must be reanalyzed, with the exception noted above.

8.11 To obtain analyte data of known quality, it is necessary to measure more than the analytes of interest in order to apply corrections or to determine whether interference corrections are necessary. If the concentrations of interference sources (such as C, Cl, Mo, Zr, W) are such that, at the correction factor, the analyte is less than the limit of quantitation and the concentration of interferents are insignificant, then the data may go uncorrected. Note that monitoring the interference sources does not necessarily require monitoring the interferent itself, but that a molecular species may be monitored to indicate the presence of the interferent. When correction equations are used, all QC criteria must also be met. Extensive QC for interference corrections are required at all times. The monitored masses must include those elements whose hydrogen, oxygen, hydroxyl, chlorine, nitrogen, carbon and sulfur molecular ions could impact the analytes of interest. Interference check solutions ICS-A and ICS-AB are analyzed at the beginning of each run and at least every 12 hours during the run to verify the effectiveness of interference corrections. Solution ICS-A contains high concentrations of interferents (Al, Ca, Fe, Mg, Na, P, K, S, C, Cl, Mo, and Ti) only. These should recover between 80% and 120% of the true value. The measured concentrations of other elements in this solution should be very low, indicating that interfering mass correction equations are adequate. Solution ICS-AB contains interferents at the same high concentrations, and all other analytes at 20 ug/L. Measured recoveries for all analytes should be within 80% to 120% of the true values.

PREPARATION BATCH QC SAMPLES

8.12 Each digestion batch of twenty or fewer samples will contain a preparation blank and a laboratory control sample. Each batch will also contain one or more of the following QC samples: laboratory control sample duplicate, sample duplicate, matrix spiked sample, or matrix spiked sample duplicate.

8.12.1 A preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) (or project specific reporting limit, if applicable) for each element. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL or

reporting limit, associated sample results that are less than the PQL or reporting limit or that are greater than or equal to ten times the measured preparation blank concentration may be reported.

8.12.2 A laboratory control sample (LCSW, LCSO, or LCSS), consisting of spiked reagent water or a solid reference material carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless vendor-supplied limits (for solid reference materials) or laboratory-generated statistical limits are available. If a laboratory control sample fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the recovery of a laboratory control sample is greater than 120%, associated sample results that are less than the PQL or reporting limit may be reported.

SAMPLE MATRIX QC SAMPLES

8.13 The relative percent difference (RPD) between matrix duplicate, matrix spike duplicate, or laboratory control duplicate sample results is calculated as follows:

$$RPD\ (\%) = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = First sample or LCS result
D_2= Second (duplicate) sample or LCS result

A control limit of 20% RPD is applied to duplicate analysis, if the result is greater than 100 times the instrument detection limit. If the matrix duplicate analysis fails, the associated sample result must be flagged on the report of analysis.

8.14 The recovery for each element in a spiked sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If a recovery fails, the associated sample result must be flagged on the report of analysis. The spike recovery should be calculated as follows:

$$Recovery\ (\%) = \frac{S-U}{SA} *100\%$$

where: S = Measured concentration of spiked aliquot
U = Measured concentration of unspiked aliquot
SA = Amount of spike added

8.15 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL), the measured concentration of a five-fold dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$\text{Difference (\%)} = \frac{|L-S|}{S} * 100\%$$

where: L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The action taken is dependent upon project requirements. The associated sample result may be flagged on the report of analysis, the sample may be reanalyzed at dilution to eliminate the interference, or a post-digestion spike may be performed (see section 8.16).

8.16 An analyte spike that is added to an aliquot of a prepared sample, or its dilution, should be recovered within 80% to 120% of the known value if the result for the unspiked aliquot is less than four times the amount of spike added. If the post-digestion spike is not recovered within these limits, the sample should be diluted and reanalyzed to compensate for the matrix interference or the method standard additions may be employed.

8.17 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revisions of USEPA Method 6020 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 6020A.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM, Current Version 5.1.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision

Agilent 7800/7900 ICP-MS Hardware Maintenance Manual, Agilent Technologies, Inc., 2018.

Agilent 7800 ICP-MS MassHunter Workstation Intelligent Sequence Software, Agilent Technologies, Inc., 2018.

Agilent 7800 ICP-MS Familiarization Guide, Agilent Technologies, Inc., 2018.

Agilent 7800 ICP-MS(7800/7900) MassHunter Workstation User Guide, Agilent Technologies, Inc., 2018.

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TABLE 1

QC REQUIREMENTS

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration, minimum 1 point plus a calibration blank.	Daily prior to sample analysis.	If more than 1 caibration std is used, correlation coefficient (r) $\geq$ 0.998	Recalibrate
Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within $\pm$ 10% of true value.	Do not use results for failing elements, unless ICV >110% and sample result < PQL/reporting limit.
Initial Calibration Blank (ICB)	Immediately after the ICV.	Absolute value of ICB < PQL or project specific reporting limit.	Do not use results if sample $\geq$ PQL/reporting limit and < 10x ICB level.
PQL Standard or LLCCV	At beginning and end of run	70-130% of true value	Do not use results for failing elements, unless PQL rec.> upper limit and sample result < PQL/reporting limit, or if sample result > 10x detected PQL concentration
Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of run.	Recovery within $\pm$ 10% of true value.	1) Do not use bracketed sample results for failing elements, unless CCV >110% and sample result < PQL/reporting limit. 2) Investigate and correct problem.
Continuing Calibration Blank (CCB)	Immediately after every CCV	Absolute value of CCB < PQL or project specific reporting limit.	Do not use sample results if $\geq$ PQL/reporting limit and < 10x CCB level.
Interference Check Solution A (ICS-A)	Before analyzing samples, and every 12 hours during a run.	Interferents: Recovery within $\pm$ 20% of true value. Analytes: No criteria established (Project specific criteria may apply)	Do not use sample results for failing elements, Unless ICSAB passes and trace metal element is > 10x absolute value of ICSA trace-metals concentration.
Interference Check Solution AB (ICS-AB)	Before analyzing samples, and every 12 hours during a run.	Recovery within $\pm$ 20% of true value.	Do not use sample results for failing elements, unless ICSAB >120% and sample result < PQL/reporting limit.
Preparation Blank (PBW/PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL (standard practice), or based on the project specific guidelines.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration $\geq$ PQL and <10x the blank conc.
Laboratory Control Sample (LCSW/LCSS/LCSO)	At least one per digestion batch of 20 or fewer samples.	Recovery within $\pm$ 20% of true value, unless vendor-supplied or statistical limits have been established.	1) Investigate source of problem. 2) Redigest and reanalyze all associated samples, unless LCS >120% and sample result < PQL.
Matrix Spike Sample (S), or Matrix Spike Duplicate	At least one per digestion batch of	Recovery within $\pm$25% of true value, if sample <4x	Flag results

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 1

QC REQUIREMENTS

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
Sample (P)	20 or fewer samples.	spike added.	
Duplicate Sample (D), Matrix Spike Duplicate (P), or LCS Duplicate (LC2W/LC2S/LC2O)	See section 8.13	1) RPD ≤ 20%, if sample > 100x IDL.	Flag results
Post-Digestion Matrix Spike (A)	When serial dilution fails and analyte concentration < 100 x MDL.	Recovery ± 20% of true value, if sample < 4x spike added.	Flag results and/or analyze sample by method of standard additions.
Serial Dilution (L)	1 per digestion batch	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.	Flag result or dilute and reanalyze sample to eliminate interference.
Internal Standard (IS)	Appropriate IS required for all analytes in all samples. Mass of IS must be <50 amu different from that of analyte.	1) For each sample, IS intensity within 30%-120% of that of initial calib. blank. 2) For ICV, ICB, CCV, and CCB, IS intensity within 80%-120% of that in initial calib. blank.	Do not use results for failing elements.
Instrument Detection Limit (IDL) Study	Quarterly.	IDL < MDL PQL at least 2-3x IDL	1) Repeat IDL study. 2) Raise PQL.
Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, “Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications”, current revision.		
Lower Limit of Quantitation Check (LLQC) Sample	Digest and analyze annually or as needed to confirm PQLs	70% - 130% of true value	Reevaluate PQLs
Method of Standard Additions	When matrix interference is suspected	r ≥ 0.995	Dilute and reanalyze sample to eliminate interference.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Linear Dynamic Range (LDR) or High-level Check Standard	Daily.	Within ±10% of true value.	Dilute samples within the calibration range, or re-establish/verify the LDR.	Flagging is not appropriate.	Data cannot be reported above the calibration range without an established/passing high-level check standard.
Tuning	Prior to ICAL.	Mass calibration = 0.1 amu from the true value; Resolution < 0.9 amu full width at 10% peak height.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Initial Calibration (ICAL) for All Analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, r2 = 0.99.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum one high standard and a calibration blank. No samples shall be analyzed until ICALhas passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes, within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level Calibration Check Standard (Low Level ICV)	Daily.	All reported analytes within ± 20% of the true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the LOQ.
Internal Standards (IS)	Every field sample, standard and QC sample.	IS intensity in the samples within 30-120% of intensity of the IS in the ICAL blank.	If recoveries are acceptable for QC samples, but not field samples, the field samples may be	Flagging is not appropriate.	Samples suffering from matrix effect should be diluted until criteria are met, or an alternate IS should be selected.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
			considered to suffer from a matrix effect. Reanalyze sample at 5-fold dilutions until criteria is met. For failed QC samples, correct problem, and rerun all associated failed field samples.		
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed. Flagging is not appropriate.	Results may not be reported without a valid calibration blank.	For CCB, failures due to carryover may not require an ICAL.
Interference Check Solutions (ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	ICS-A: Absolute value of concentration for all non-spiked project analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis, locate and correct problem, reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the failed ICS.	All analytes must be within the LDR. ICS-AB is not needed if instrument can read negative responses.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all reported analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if	Examine the project-specific requirements. Contact the client as	For the specific analyte(s) in the parent sample, apply J-flag if acceptance	If MS results are outside the limits, the data shall be evaluated to determine the

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
		project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	to additional measures to be taken.	criteria are not met and explain in the case narrative.	source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.
Dilution Test	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations > 50 X LOQ (prior to dilution). Use along with MS/MSD or PDS data to confirm matrix effects.
Post Digestion Spike (PDS) Addition	One per preparatory batch if MS or MSD fails (using the same sample as used for the MS/MSD if possible).	Recovery within 80-120%	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Criteria apply for samples with concentrations < 50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution or post digestion spike fails and if the required by project.	NA.	NA.	NA.	Document use of MSA in the case narrative.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-627-14	METHOD 6020, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		
QC - Calibration Blanks	Acceptance criteria employed for 6020: ± PQL	Acceptance criteria stated in 6020: <10% of PQL

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 4

ISOTOPES MONITORED AND CORRECTION EQUATIONS USED FOR USEPA METHOD 6020

Element Class	Element	Sym-bol	Isotopes Monitored	Correction Equations [See note 1]
Analytes	Aluminum	Al	27	
	Antimony	Sb	121, 123	
	Arsenic	As	75	$^{75}As = (75)*1 - (77)*2.95 + (82)*2.548 – (83)*2.571$ [See note 2]
	Barium	Ba	135, 137	
	Beryllium	Be	9	
	Boron	B	11	
	Cadmium	Cd	106, 108, 111, 114	$^{111}Cd = (111)*1 – (108)*1.073 + (106)*0.764$ [See note 3] $^{114}Cd = (114)*1 – (118)*0.0268 – (108)*1.84$ [See note 4]
	Calcium	Ca	44	$^{44}Ca = (44)*1 – (88)*0.0169$ [See note 7]
	Chromium	Cr	52, 53	
	Cobalt	Co	59	
	Copper	Cu	63, 65	
	Iron	Fe	54, 56, 57	$^{54}Fe = (54)*1 – (52)*0.0282$ [See note 8] $^{57}Fe = (57)*1 – (43)*0.03$ [See note 9]
	Lead	Pb	206, 207, 208	$^{208}Pb = (208)*1 + (206)*1 + (207)*1$ [See note 5]
	Magnesium	Mg	25	
	Manganese	Mn	55	
	Molybdenum	Mo	98	$^{98}Mo = (98)*1 – (99)*0.146$ [See note 10]
	Nickel	Ni	60, 61	
	Potassium	K	39	
	Selenium	Se	82	$^{82}Se = (82)*1 – (83)*1.009$ [See note 11]
	Silver	Ag	107, 109	
	Sodium	Na	23	
	Strontium	Sr	88	
	Thallium	Tl	203, 205	
	Thorium	Th	232	
	Tin	Sn	118, 120	
	Tungsten	W	182	
	Uranium	U	238	
	Vanadium	V	51	$^{51}V = (51)*1 – (53)*2.95 + (52)*0.378$ [See note 12]
	Zinc	Zn	66, 67, 68	
Internal Stan-dards.	Bismuth	Bi	209	
	Germanium	Ge	72	
	Indium	In	115	$^{115}In = (115)*1 – (118)*0.016$ [See note 6]
	Lithium	Li	6	
	Scandium	Sc	45	
	Terbium	Tb	159	
	Yttrium	Y	89	

TABLE 4 (continued)

ISOTOPES MONITORED AND CORRECTION EQUATIONS USED FOR USEPA METHOD 6020

Notes:

1) Numbers in parentheses, e.g "(51)", indicate measured counts at the indicated mass.
2) Corrects for ArCl interference, taking into account secondary interferences from Se and Kr
3) Corrects for MoO interference, taking into account secondary interference from ^{108}Cd
4) Corrects for Sn interference
5) Corrects for variations in isotopic composition of lead
6) Corrects for Sn interference
7) Corrects for interference from $^{88}Sr^{2+}$
8) Corrects for Cr interference
9) Corrects for Ca interference
10) Corrects for Ru interference
11) Corrects for Kr interference
12) Corrects for ClO, taking into account secondary interference from Cr

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 5

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Continuing Calibration Verification CCV (1.0% HNO_3 / 0.5% HCl)	CL-CAL-3	Spex Industries	0.25
	ICP-MS-MIX-Z	Lab Prepared	0.50
	ICP-MS CAL 1	Lab Prepared	1.25
Calibration Standard (1.0% HNO_3 / 0.5% HCl)	CL-CAL-3	Spex Industries	0.50
	ICP-MS-MIX-Z	Lab Prepared	1.0
	ICP-MS CAL 1	Lab Prepared	2.5
Initial Calibration Verification (ICV) (1.0% HNO_3 / 0.5% HCl)	CL-ICS-1,CL-ICS-4, CL-ICS-5	Spex Industries	0.20 of each
	CL-ICS-3	Spex Industries	2.0
	1000 mg/L Si	Inorganic Ventures	0.040
	1000 mg/L Al	Inorganic Ventures	0.038
	1000 mg/L B, W Solution (0.5mL each per 50mL and use same day only)	Inorganic Ventures	0.200
Practical Quantitation Limit Solution (PQL) (1.0% HNO_3 / 0.5% HCl)	ICP-MS PQL Intermediate	Lab Prepared	0.1
Interference Check Solution A (ICS-A) (1.0% HNO_3 / 0.5% HCl)	6020ICS-0A	Inorganic Ventures	10.0
Interference Check Solution AB (ICS-AB) (1.0% HNO_3 / 0.5% HCl)	6020ICS-0A	Inorganic Ventures	10.0
	ICP-MS-CAL 1	Lab Prepared	1.0
	ICP-MS ICSAB Intermediate	Lab Prepared	1.0
P/A Tuning Solution (1.0% HNO_3 / 0.5% HCl)	1000 mg/L Co, Cr, Mo, Mn, Pb, Sb, Sr, U, V	High Purity Standards	0.02
	10,000 mg/L Al, K, Na	High Purity Standards	0.002
Instrument Tuning Solution (1.0% HNO_3 / 0.5% HCl)	ICP-MS-TS-2	High Purity Standards	0.10
	Conc. HNO_2	Baker Instra Analyzed	4

TABLE 5

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Internal Standard Solution (5.0% HNO_3 / 0.5% HCl)	Internal Standard Mix	Spex Industries	10
Method Tuning Solution (1.0% HNO_3 / 0.5% HCl)	ICP-MS Method Tune Intermediate	Lab Prepared	1.0
	Internal Standard Mix 1	Spex Industries	1.0

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 6

PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
ICP-MS PQL Intermediate (5% HNO_2/5%HCL)	10,000 mg/L K, Na	High Purity Standards or Inorganic Ventures	2.0 of each
	10,000 mg/L Si	High Purity Standards or Inorganic Ventures	1.0
	1000 mg/L B	High Purity Standards or Inorganic Ventures	0.40
	10,000 mg/L Al, Ca, Fe, Mg 1000 mg/L Zn	High Purity Standards	0.20 of each
	1000 mg/L As, Se, V, W, Sr, Sn, Mo, Cr	High Purity Standards or Inorganic Ventures	0.10 of each
	1000 mg/L Cu	High Purity Standards	0.06
	1000 mg/L Ba, Mn, Ni	High Purity Standards	0.04 of each
	1000 mg/L U, Be, Cd, Co, Ag, Th, Tl, Pb, Sb	High Purity Standards	0.02 of each
ICP-MS CAL 1 -(5% HNO_2/5%HCL)	1000 mg/L Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Th, Tl, U, V, Zn	High Purity Standards	0.2 of each
	10,000 mg/L Al	High Purity Standards or Inorganic Ventures	0.02
	Conc. HCL	Baker Instra Analyzed	2
ICP-MS-MIX-Z (1.0% HNO_3 / 0.5% HCl)	10,000 mg/L K, Na, Fe, Mg, Ca	High Purity Standards or Inorganic Ventures	5.0 of each
	10,000 mg/L Si	High Purity Standards or Inorganic Ventures	1.0
	10,000 mg/L Al	High Purity Standards or Inorganic Ventures	0.95
	1000 mg/L B, Sn, Sr, W	High Purity Standards or Inorganic Ventures	0.50 of each
ICP-MS-MIX-Y (1.0% HNO3/ 0.5% HCl)	10,000 mg/L Al	High Purity Standards or Inorganic Ventures	0.030
	1000 mg/L As, Ba, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, V, Zn	High Purity Standards or Inorganic Ventures	0.30 of each
ICP-MS ICSAB Intermediate (1.0% HNO_3 / 0.5% HCl)	10,000 mg/L Si	High Purity	0.50
	1,000 mg/L B, Sn, Sr, W	High Purity or Inorganic Ventures	0.20 each

TABLE 6

PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
ICP-MS Method Tune Intermediate (1.0% HNO_3 / 0.5% HCl)	1000 mg/L Be, Co, Tl 10,000 mg/L Mg	High Purity Standards or Inorganic Ventures	0.1 of each
	1000mg/L Pb		0.30

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 7

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L				
	CCV	Cal. Std.	ICV	PQL	P/A Tune Soln.
Aluminum	500.0	1000.0	400.0	20.0	200
Antimony	25.0	50.0	20.0	0.2	200
Arsenic	25.0	50.0	20.0	1.0	
Barium	25.0	50.0	20.0	0.4	
Beryllium	25.0	50.0	20.0	0.2	
Boron	25.0	50.0	20.0	4.0	
Cadmium	25.0	50.0	20.0	0.2	
Calcium	5000.0	10000.0	4000.0	20.0	
Chromium	25.0	50.0	20.0	1.0	200
Cobalt	25.0	50.0	20.0	0.2	200
Copper	25.0	50.0	20.0	0.6	
Iron	5000.0	10000.0	4000.0	20.0	
Lead	25.0	50.0	20.0	0.2	200
Magnesium	5000.0	10000.0	4000.0	20.0	
Manganese	25.0	50.0	20.0	0.4	200
Molybdenum	25.0	50.0	40.0	1.0	200
Nickel	25.0	50.0	20.0	0.4	
Potassium	5000.0	10000.0	4000.0	200.0	200
Selenium	25.0	50.0	20.0	1.0	
Silicon	500.0	1000.0	400.0	100.0	
Silver	25.0	50.0	20.0	0.2	
Sodium	5000.0	10000.0	4000.0	200.0	200
Strontium	25.0	50.0	20.0	1.0	200
Thallium	25.0	50.0	20.0	0.2	
Thorium	**25.0**	**50.0**	**20.0**	**0.2**	
Tin	25.0	50.0	20.0	1.0	
Tungsten	25.0	50.0	20.0	1.0	
Uranium	25.0	50.0	20.0	0.2	200
Vanadium	25.0	50.0	20.0	1.0	200
Zinc	25.0	50.0	20.0	2.0	

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 7 (continued)

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L				
	ICSA[1]	ICSAB[1]	Internal Std Solution	Method Tune Solution	Instrument Tuning Solution
Aluminum	100000	100000			
Antimony		20			
Arsenic		20			
Barium		20		10	
Beryllium		20			
Boron		20			
Cadmium		20			
Calcium	100000	100000			
Chromium		20			
Cobalt		20		10	
Copper		20			
Iron	100000	100000			
Lead		20		10	
Magnesium	100000	100000		100	
Manganese		20			
Molybdenum	2000	2000			
Nickel		20			
Potassium	100000	100000			
Selenium		20			
Silver		20			
Sodium	100000	100000			
Strontium		20			
Thallium		20		10	10.0
Thorium		**20**			
Tin		20			
Tungsten		20			
Uranium		20			
Vanadium		20			
Zinc		20			
Bismuth			1000.0	10	
Germanium			1000.0	10	
Indium				10	
Lithium (^{6}Li)			1000.0	10	
Scandium			1000.0	10	
Terbium			1000.0	10	
Yttrium			1000.0	10	10.0

TABLE 7 (continued)

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L				
	ICSA[1]	ICSAB[1]	Internal Std Solution	Method Tune Solution	Instrument Tuning Solution
Cerium					10.0
Lithium					10.0

1) Solution also contains 1000 mg/L Chloride, 200 mg/L Carbon, and 100 mg/L Phosphorus and Sulfur, and 2mg/L Titanium.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 8

ELEMENT CONCENTRATIONS IN INTERMEDIATE STANDARDS

ELEMENT	CONCENTRATION IN SOLUTION, mg/L					
	MS-MIX-Z	ICP-MS PQL Intermediate	ICP-MS-MIX-Y	ICP-MS Method Tune Intermediate	ICP-MS CAL 1	ICP-MS ICSAB Intermediate
Aluminum	95.0	2.0	3.0		0.2	
Antimony		0.02	3.0		0.2	
Arsenic		0.10	3.0		0.2	
Barium		0.04	3.0		0.2	
Beryllium		0.02		1.0	0.2	
Boron	5.0	4.0				0.2
Cadmium		0.02			0.2	
Calcium	500	2.0				
Chromium		0.10	3.0		0.2	
Cobalt		0.02		1.0	0.2	
Copper		0.06	3.0		0.2	
Iron	500	2.0				
Lead		0.02	3.0	3.0	0.2	
Magnesium	500	2.0		10.0		
Manganese		0.04	3.0		0.2	
Molybdenum		0.10	3.0		0.2	
Nickel		0.04	3.0		0.2	
Potassium	500	20.0				
Selenium		0.10	3.0		0.2	
Silicon	100	10.0				5.0
Silver		0.02			0.2	
Sodium	500	20.0				
Strontium	5.0	0.10				0.2
Thallium		0.02		1.0	0.2	
Tin	5.0	0.10				0.2
Thorium		0.02			0.2	
Tungsten	5.0	0.10				0.2
Uranium		0.02			0.2	
Vanadium		0.10	3.0		0.2	
Zinc		0.20	3.0		0.2	

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 9

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L				
	Instrument Calibration Standard 3 (Spex)	CL-ICS-1 (Spex)	CL-ICS-3 (Spex)	CL-ICS-4 (Spex)	CL-ICS-5 (Spex)
Aluminum		10.0			
Antimony		10.0			
Arsenic		10.0			
Barium		10.0			
Beryllium		10.0			
Boron					
Cadmium		10.0			
Calcium	1000		200.0		
Chromium		10.0			
Cobalt		10.0			
Copper		10.0			
Iron	1000		200.0		
Lead		10.0			
Magnesium	1000		200.0		
Manganese		10.0			
Molybdenum				10.0	10.0
Nickel		10.0			
Potassium	1000		200.0		
Selenium		10.0			
Silver		10.0			
Sodium	1000		200.0		
Strontium					10.0
Thallium		10.0			
Thorium				10.0	
Tin					10.0
Tungsten					
Uranium				10.0	
Vanadium		10.0			
Zinc		10.0			

TABLE 9 (continued)

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L		
	6020ICS-0A[1] (Inorganic Ventures)	**Internal Standard Mix 1 (Spex)**	**ICP-MS-TS-2 (High Purity)**
Aluminum	1000		
Arsenic			
Cadmium			
Calcium	1000		
Chromium			
Cobalt			
Copper			
Iron	1000		
Magnesium	1000		
Manganese			
Molybdenum	20.0		
Nickel			
Potassium	1000		
Silver			
Sodium	1000		
Zinc			
Bismuth		1000	
Cerium			10000
Germanium		1000	
Indium		1000	
Lithium			10000
Lithium (^{6}Li)		1000	
Scandium		1000	
Terbium		1000	
Thallium			10000
Yttrium		1000	10000

1) Solution also contains 10000 mg/L Chloride, 2000 mg/L Carbon, and 1000 mg/L Phosphorus and Sulfur, and 20 mg/L Titanium.

TABLE 10

REQUIRED ANALYTICAL SEQUENCE

Sequence Number	Standard/Sample	Purpose
1	Method Tuning Solution	Verify mass calibration and resolution
2	S0 (Calibration Blank)	Initial calibration
3	S1 (Calibration Standard)	Initial calibration
7	ICV (Initial Calibration Verification)	Check calibration accuracy
8	ICB (Initial Calibration Blank)	Check calibration accuracy
9	PQL (Practical Quantitation Limit)	Check calibration accuracy at low concentration
10	ICS-A (Interference Check Solution A)	Verify accuracy of mass correction equations
11	ICS-AB (Interference Check Solution AB)	Verify accuracy of mass correction equations
12	CCV (Continuing Calibration Verification)	Check calibration stability
13	CCB (Continuing Calibration Blank)	Check calibration stability
14-23	Analyze up to 10 samples	
24	CCV (Continuing Calibration Verification)	Check calibration stability
25	CCB (Continuing Calibration Blank)	Check calibration stability
…	Continue analyzing sequences of up to 10 samples, followed by a CCV and a CCB	
…	After last analytical sample, analyze PQL , followed by a CCV and a CCB	

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TABLE 11

INSTRUMENT OPERATING CONDITIONS

Data Acquisition Program	Acquisition Mode	Spectrum
	Points per Mass	3
	Number of Replicates	3
	Detector Mode	Auto for all elements
	Integration Time per Point (for listed masses and their correction masses)	0.10 sec for Li, B, ^{29}Si, Sc, V, Cr, Mn, Ni, Cu, Zn, Y, Mo, Ag, In, Sn, Sb, Ba, Tb, W, Tl, Pb, Bi, Th, U
		0.30 sec for Be, As, Cd, Ge
		0.010 sec for Na, Al, K, ^{28}Si
		0.030 for Ca, Fe, Sr
		1.00 sec for Se
		0.050 sec for Mg, Co
	Spray Chamber Temperature	2° C
	Total Acquisition Time	105 sec for 3 replicates
Peristaltic Pump Program	Analysis Speed	0.15 rps
Before Acquisition	Uptake Speed	0.15 rps
	Uptake Time	5 sec
	Stabilization Time	15 sec
After Acquisition (Probe Rinse)	Rinse Speed	0.15 rps
	Rinse Time (sample)	5 sec
	Rinse Time (standard)	5 sec
After Acquisition (Rinse)	Rinse Vial	1
	Uptake Speed	0
	Uptake Time	0 sec
	Stabilization Time	0 sec
Calibration Curve fit	All quantitation masses	Y=ax+(blank)
	All internal standard masses	(Excluded)
	All interference correction masses	(Excluded)
Reporting Parameters	QC Reports	On-Printer
	All Other Reports	Off

TABLE 12

INSTRUMENT TUNE SPECIFICATIONS

Sensitivity	Li >5000 cts/0.1 sec/10 ppb
	Y >10,000 cts/0.1 sec/10 ppb
	Tl >5000 cts/0.1 sec/10 ppb
Precision	Li <8% RSD (0.1 sec integration time)
	Y <5% RSD (0.1 sec integration time)
	Tl <5% RSD (0.1 sec integration time)
Oxides	<1.0%
Doubly Charged (Ce^{++}/Ce^{+})	<2.0%
Background	Li <15 cps
	Y <15 cps
	Tl <15 cps
Mass Resolution	Width at 10% peak height: 0.7-0.8 amu
Mass Axis	Li ±0.1 amu of nominal mass
	Y ±0.1 amu of nominal mass
	Tl ±0.1 amu of nominal mass

TABLE 13

METHOD TUNE SPECIFICATIONS

Precision	≤5% RSD of 4 replicates
Mass Resolution	Width at 10% peak height: <0.9 amu
Mass Calibration	±0.1 amu of nominal mass

TABLE 14

REPORTED ISOTOPES AND INTERNAL STANDARDS

ELEMENT	MASS	INTERNAL STANDARD (mass)
Aluminum	27	Scandium (45)
Antimony	123	Terbium (159)
Arsenic	75	Yttrium (89)
Barium	135	Terbium (159)
Beryllium	9	Lithium (6)
Boron	11	Lithium (6)
Cadmium	114	Yttrium (89)
Calcium	44	Scandium (45)
Chromium	52	Yttrium (89)
Cobalt	59	Yttrium (89)
Copper	65	Yttrium (89)
Iron	57	Yttrium (89)
Lead	208	Bismuth (209)
Magnesium	25	Scandium (45)
Manganese	55	Yttrium (89)
Molybdenum	98	Yttrium (89)
Nickel	60	Yttrium (89)
Potassium	39	Scandium (45)
Selenium	82	Yttrium (89)
Silicon	29	Scandium (45)
Silver	107	Yttrium (89)
Sodium	23	Scandium (45)
Strontium	88	Yttrium (89)
Thallium	203	Bismuth (209)
Thorium	232	Bismuth (209)
Tin	118	Terbium (159)
Tungsten	182	Terbium (159)
Uranium	238	Bismuth (209)
Vanadium	51	Yttrium (89)
Zinc	66	Yttrium (89)

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

ATTACHMENT 1

HARDNESS BY CALCULATION

As referenced in “Standard Methods for the Examination if Water and Wastewater,” Methods 2340 A & B, Hardness Introduction and Hardness by Calculation, American Public Health Association, 18th Edition, Revised 1992, total hardness is the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in milligrams per liter.

Once the calcium and magnesium concentrations have been determined by EPA methods 6010, 6020, 200.7 or 200.8, the total hardness of an aqueous sample may be calculated as follows:

$$\text{Total Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L) + 4.118\ (Mg, mg/L)$$

The calcium hardness of an aqueous sample may also be calculated as follows:

$$\text{Calcium Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L)$$

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

Prepared By: George Brewer Date: 3/98

Approved By:

Group Supervisor: George Brewer Date: 01/24/01

Operations Manager: Jol C. Burton Date: 1/24/01

QA Officer: Deborah J. Nadeau Date: 1.24.01

General Manager: Duncan F. Hufnagle Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3050B	Format changes, added pollution prevention, added MSD, added spiking instruction tables.	DH	1.24.01	1/24/01
02 3050B	Removed all references/procedures devoted to GFAA. Added use of digestates for ICP/MS analysis. Revised standard solution names & concs. in Tables 3 & 4 to reflect current practice.	DH	8.29.02	8.29.02
03 3050B	New Title to include ICP/MS, 3. Use of digestion block and polyethylene digestion tubes added to sections 4.0, 7.0 and Table 1. PBS changed from 1.0g water to 1.0g boiling chips. H_2O_2 addition from 3.0ml then 7.0mls to 2.0ml, 2.0ml then 1.0ml. Figures and Tables updated to reflect current practices.	LAD	03/08	03/08
04	Updated Tables 3 and 4 with current spike concentrations and volumes added. Updated logbook page. Added CA-108 reference for subsampling information.	LAD	08/09	08/09
05	Updated Tables 3 and 4 to reflect current spiking procedures.	LAD	09/10	09/10

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Sect. 7 – Added wording concerning subsampling. Table 3 and 4 – Corrected standard concentrations. Attachment A - Modifications For 8330B Preparation & Digestion. Changed KAS INC. to KAS throughout	UAD	08/15	08/15
07	Update figure 2. Change title of section 5.0. Update method references for NELAC + DoD. Minor changes to Table 1 + section 8.2.	UAD	09/17	09/17
08	Updated Table 3 to include Thallium and corrected standard concentration of U + W. Updated Table 4 to include Thallium Sect. 8 - added Contingency Plan. Sect 10. updated references	UAD	01/19	01/19
09	Sect. 4 - Added PTFE boiling chips. Sect. 8 - Added PTFE boiling chips to LCS preparation.	UAD	01/20	01/20
10	Sect. 4.13 - Added balance make and model	UAD	06/20	06/20

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

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KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

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TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the Katahdin Analytical Services procedure utilized to dissolve solid matrices and solubilize metals from solid samples prior to analysis for metals by ICP-AES and ICP-MS. This SOP applies to samples prepared by EPA Method 3050, with method modifications as summarized in Table 2.

This procedure applies to all solid sample (e.g. sediments, sludges, soils, and ashes) preparations for ICP-AES and ICP-MS analyses. This method is not a total digestion technique for most samples. It is a very strong acid digestion that will dissolve almost all elements that could become "environmentally available". By design, elements bound in silicate structures are not normally dissolved by this procedure as they are not usually mobile in the environment.

1.1 Definitions

ICP-AES – Inductively Coupled Plasma Atomic Emission Spectroscopy.

ICP-MS – Inductively Coupled Plasma Mass Spectrometry.

LCSO – Laboratory Control Sample for Solids – An aqueous standard that had been brought through the sample preparation process.

LCSS – Laboratory Control Sample for Solids – A solid reference material that has been brought through the sample preparation process.

Matrix Spike – An aliquot of a sample to which a known amount of analyte has been added before digestion.

PBS – Preparation Blank for Solids – An aliquot of reagent water that has been brought through the sample preparation process.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the acid digestion of solid samples by USEPA Method 3050 for metals analysis. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Training".

It is the responsibility of all Katahdin technical personnel involved in the acid digestion of solid samples by USEPA Method 3050 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the procedure or irregularities with the

samples should also be recorded in the lab notebook and reported to the responsible Department Manager or designated qualified data reviewer.

It is the responsibility of the Department Manager to ensure that technical personnel perform acid digestions in accordance with this SOP and to confirm that their work is properly documented through periodic review of the associated logbooks.

1.3 Safety

The acids used in this procedure are highly corrosive and reactive, and spiking standards contain toxic metals. The toxicity and reactivity of client samples are usually unknown, so samples should always be assumed to present a contact hazard. To reduce or eliminate exposure to potentially harmful chemicals, lab coats, gloves, and safety glasses or goggles must be worn whenever handling samples or reagents. Additional safety apparel, including face shields, aprons, dust masks, and shoe protectors, is available in the Metals prep lab and should be worn whenever circumstances warrant.

Acids should be added to samples slowly and carefully, while watching for reactions. This should be done under a hood in case harmful fumes are evolved.

Hood sashes should be lowered as far as possible whenever beakers are being heated on a hot plate. Use caution when handling hot beakers.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from the Environmental Health and Safety Officer, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the Metals prep lab for subsequent appropriate disposal in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual.

Sample digestates should be stored for a minimum of 60 days after digestion to allow for analysis, and reanalysis if necessary. Digestates older than 60 days may be emptied into the corrosive waste carboy in the Metals prep lab for subsequent appropriate disposal in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual.

2.0 SUMMARY OF METHOD

A representative 1 to 2 g (wet weight) sample is digested with repeated additions of nitric acid and hydrogen peroxide. Hydrochloric acid is added to the initial digestate and the sample is refluxed. The digestate is then filtered and diluted to a final volume of 100 mL.

3.0 INTERFERENCES

Interferences are discussed in the applicable analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1 Digestion vessels. If digestion is performed using a hot plate, the appropriate digestion vessels are 100 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning" and CA-602, “Glassware Preparation and Sample Preservation for Trace Element Analyses”). If digestion is performed using a block digester, the appropriate digestion vessels are new 70 mL disposable graduated polyethylene digestion tubes with attached snap lids.

4.2 Ribbed watch glasses. If digestion is performed using a hot plate, 75 mm diameter glass watch glasses (pre-cleaned as above) are used. If digestion is performed using a block digester, 40 mm diameter disposable polyethylene watch glasses are used.

4.3 Adjustable volume automatic pipets covering the range from 10 uL to 1000 uL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.

4.4 Disposable graduated polystyrene specimen containers with pouring lips, 200 mL capacity.

4.5 Hot plate or block digester, griddle, or other heating source - adjustable and capable of maintaining a temperature of 95°C ± 5°C. Heating sources must be numbered for easy identification.

4.6 Device for measuring hot plate temperature, consisting of a flask or digestion vessel in which the bulb of a thermometer is immersed in sand or water. The temperature

of each hot plate used is measured and recorded each day. The hot plate identification number and the measured temperature are recorded on the sample preparation logbook sheet.

4.7 Plastic funnels, pre-cleaned as in Section 4.1.

4.8 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.

4.9 Polyethylene wash bottles for dispensing reagent water and 5% HNO_3.

4.10 Filter paper, Whatman No. 41 or equivalent. Filters are acid-washed immediately prior to use as follows. Place a pre-cleaned funnel in the funnel holder and put a disposable plastic specimen container under the funnel to collect the rinsates. Place a folded filter in the funnel and rinse three times with approximate 10 mL volumes of 5% HNO_3, making sure the entire surface of the filter is wetted each time and allowing each rinse to drain completely before continuing. Then rinse three times with approximate 25 mL volumes of reagent water, again allowing each rinse to drain completely. Discard the rinsates into the appropriate waste container. The acid-washed filter is now ready for use.

4.11 Polyethylene sample containers with screw caps or graduated polyethylene sample containers with attached snap lids, 125 mL capacity.

4.12 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, 1:1 HNO_3, and concentrated HCl.

4.13 Analytical balance capable of reading to 0.01 gram, Ohaus model SPE402, or equivalent.

4.14 Spatulas, scoops, or spoons; plastic or stainless steel, rinsed with 5% HNO_3 and reagent water. Disposable tongue depressors may be used and do not require to be rinsed.

4.15 Pre-cleaned PTFE boiling chips. These are cleaned by soaking in 5% nitric acid overnight, then rinsing thoroughly with reagent water, and air drying.

5.0 REAGENTS AND STANDARDS

5.1 Concentrated nitric acid, HNO_3 – trace metals grade.

5.2 Concentrated hydrochloric acid, HCl – trace metals grade.

5.3 Reagent water - water that meets the performance specifications of ASTM Type II water (ASTM D1193).

5.4 Nitric acid, 1:1. Add a volume of concentrated HNO_3 to an equivalent volume of reagent water and swirl gently to mix.

5.5 Nitric acid, 5% v/v. Add 25 mL concentrated HNO_3 to 475 mL reagent water in a 500 mL wash bottle. Cap, point the dispensing tip into a sink, and shake gently to mix.

5.6 30% hydrogen peroxide (H_2O_2) - spectrometric grade.

5.7 Multielement spiking solutions (see Table 3 for a list of required spiking solutions).

5.8 Solid reference material – a soil containing all the elements of interest, with empirically established method-specific recoveries and acceptance limits for all analytes. Solid reference materials are purchased with documentation of analysis provided by the vendor. See Figure 4 for an example certificate of analysis for a solid reference material.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples should be collected in clean plastic or glass containers. Samples must be refrigerated (4°C ±2°C) upon receipt by the laboratory. The holding time for solid samples is 6 months from the date of sample collection.

7.0 PROCEDURE

The procedure described below is condensed for quick reference in Table 3.

SAMPLE PREPARATION

7.1 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer spreadsheet. Print out a copy of the spreadsheet (see Figure 2 for an example). Hand label the digestate vessels

7.2 If using glass beakers as the digestion vessels, submerge previously cleaned beakers and watch glasses three times into a 10% nitric acid bath, then rinse three times with reagent water. The polyethylene digestion tubes used in conjunction with the block digester do not require acid rinsing or precleaning. Label the digestion vessels with sample numbers.

7.3 Weigh 1 to 2 g of well-mixed sample into a properly cleaned, labeled, and tared Griffin beaker or polyethylene digestion tube. Avoid rocks, roots, leaves and other

organic or inorganic foreign material. Record (hand write) the weight of each sample on the printout of the digestion spreadsheet.
Refer to Katahdin Analytical Services SOP CA-108, current revision "Basic Laboratory Technique" for more information on subsampling.

7.4 Weigh an appropriate amount of solid reference material to a clean, labeled, and tared Griffin beaker or polyethylene digestion tube to serve as a laboratory control sample.

7.5 Add spike solutions to matrix spike samples (refer to Tables 3 and 4 for spiking instructions).

7.6 Using repipetters, add 10 mL of 1:1 HNO_3, mix the slurry. Cover with a ribbed watch glass and place on heat source. Gently heat the sample to 95°C ± 5 ^{o}C and reflux for 10 to 15 minutes without boiling. Remove the digestion vessel from the heat source and cool the sample.

7.7 Add 5 mL of concentrated HNO_3 to the sample, replace the watch glass, and reflux for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO_3, repeat this step (addition of 5 mL of concentrated HNO_3) until no brown fumes are given off by the sample, indicating complete reaction by HNO_3.

7.8 Continue heating the sample at 95°C ± 5°C without boiling until the digestate has evaporated to approximately 5 to 10 mL or until two hours have elapsed, whichever occurs first. Do not allow the sample to go to dryness. Remove the digestion vessel from the heat source and cool the sample.

7.9 Add 2 mL of reagent water and 2 mL of 30% H_2O_2 to the sample, replace the watch glass, and heat gently on the heat source to start the peroxide reaction. Continue heating until effervescence subsides.

7.10 Add an additional 2 mL of 30% H_2O_2 to the sample, replace the watch glass, and heat gently on the heat source to start the peroxide reaction. Continue heating until effervescence subsides.

7.11 Add an additional 6 mL of 30% H_2O_2 in 1-mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.

7.12 Continue heating the sample at 95°C ± 5°C without boiling until the digestate has evaporated to approximately 5 to 10 mL or until two hours have elapsed, whichever occurs first. Do not allow the sample to go to dryness. Remove the sample from the heat source and cool.

7.13 Add 10 mL of concentrated HCl to the digest from 7.12, replace the watch glass, and reflux at 95°C ± 5°C for 15 minutes. Remove the sample from the heat source and cool.

7.14 Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated polystyrene specimen container or graduated polyethylene sample container with attached snap lid. Using a wash bottle, rinse the digestion vessel with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse. Using the graduations on the specimen container or snap-lid container, dilute to 100 mL with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid container has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for ICP-AES or ICP-MS analysis.

7.15 Review the ACCESS computer spreadsheet for accuracy. If any information is incorrect, make the necessary changes to the computer spreadsheet and print out a corrected copy. Do not discard the original copy of the spreadsheet. Record (hand write) reagent lot numbers, spiking information, and heat source temperature in the appropriate spaces on the spreadsheet. Record any method deviations, irregularities with the samples, or other pertinent observations at the bottom of the page, and sign and date the spreadsheet. Bind all copies of the spreadsheet in the sample preparation log. An example sample preparation logbook page (ACCESS spreadsheet) is included as Figure 2.

7.15 Reopen the electronic ACCESS spreadsheet for the digestion and transcribe the sample weights from the handwritten, bound copy into the electronic copy. The information in this electronic spreadsheet will later be imported into the ACCESS metals database and used to calculate sample concentrations on a weight basis.

7.16 Place each batch of digestates in a box labeled with the QC Batch ID, and put the box of digestates in the metals digestates storage area.

CALCULATIONS

7.17 Analytical results for solid samples are reported on a dry weight basis. Total solids are determined by the Wet Chemistry Group, and are recorded in spreadsheets that are electronically imported into the Access metals database. Final dry weight concentrations are calculated by the Access database as follows:

$$\text{Concentration (mg/kg dry weight)} = (C \times V) / (W \times S)$$

where: C = Measured concentration (mg/L)
V = Digestate final volume (L)
W = Sample wet weight (kg)
S = % Solids/100

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 3050 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

8.1 At least one preparation blank for soils (PBS) is processed concurrently with each digestion batch of 20 or fewer samples, and is used to assess contamination resulting from the digestion procedure. The PBS consists of a 1.0 g of PTFE boiling stones that is digested using the same reagents as those used to digest associated samples. Refer to the appropriate analytical SOP for PBS acceptance criteria and corrective actions.

8.2 Prepare an appropriate number of laboratory control samples. Typically, the laboratory control sample is prepared by weighing 1.0 g of PTFE boiling chips into a digestion vessel and spiking as described in Table 3. This type of laboratory control sample is identified as “LCSO” in sample preparation and analysis records. Alternatively, a laboratory control sample may be prepared by weighing an appropriate amount of a solid reference material into a digestion vessel. Laboratory control samples prepared from solid reference materials are identified as “LCSS”. Laboratory control samples are digested using the same reagents as those used to digest associated samples. The analyte concentrations of the LCSS will depend on the solid reference material used, and the mass of each aliquot. Refer to Figure 3 for an example certificate of analysis for a solid reference material.

8.3 Matrix spike samples are processed along with each digestion batch at a minimum frequency of one per digestion batch. A matrix spike sample consists of an aliquot of a sample that is fortified with known amounts of all analytes of interest prior to

digestion. Matrix spike recoveries are used to assess the biasing effects of sample matrix on digestion and analysis performance. Directions for spiking matrix spike samples are contained in Figure 2. Refer to the appropriate analytical SOP for matrix spike recovery acceptance criteria and corrective actions.

8.4 Matrix spiked duplicate samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. Matrix spiked duplicate samples are used to assess the precision of the digestion and analysis methods. Refer to the appropriate analytical SOP for matrix spike duplicate precision acceptance criteria and corrective actions.

NOTE: Clients may choose specific samples for matrix spike and duplicate analysis; otherwise, the choice is left to the person performing the digestion.

8.5 The quality control measures and frequencies described above are minimum requirements. Individual clients and analytical programs may impose additional QC requirements.

8.6 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

Refer to the applicable instrumental analysis SOP for method performance information.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste," United States Environmental Protection Agency, SW-846, Third Edition, Final Update III, 12/96, Method 3050B.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM, Current Version.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

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Table 2 Summary of Method Modifications – Method 3050
Table 3 Preparation of Matrix Spikes and Spiking Solutions
Table 4 Element Concentrations in ICP-AES Matrix Spikes and Their Component Spiking Solutions
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Figure 2 Example Page from Metals Sample Preparation Logbook
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Attachment A Modifications For 8330B Preparation & Digestion

TABLE 1

QC REQUIREMENTS – METHOD 3050

Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3050	Preparation Blank for Solids (PBS)	One per prep batch of 20 or fewer samples.	Refer to analytical method.	Refer to analytical method.
	Laboratory Control Sample for Aqueous or Solids (LCSO or LCSS)	One each per prep batch of 20 or fewer samples, if specified by project or client	Refer to analytical method.	Refer to analytical method.
	Matrix Spike Sample	One per prep batch.	Refer to analytical method.	Refer to analytical method.
	Matrix Spike Duplicate Sample	One per prep batch.	Refer to analytical method.	Refer to analytical method.
	Demonstration of analyst proficiency	One-time demonstration by each analyst performing the method.	Must pass all applicable QC for method.	Repeat analysis until able to perform passing QC; document successful performance in personal training file.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 2

SUMMARY OF METHOD MODIFICATIONS – METHOD 3050

Topic	Katahdin SOP CA-605-10	Method 3050, current revision
Apparatus /Materials	1) Digestion performed in 100 mL Griffin beaker or 70 mL polyethylene tube. 2) Graduated disposable plastic cup or 120 mL polyethylene tube used to bring digestate to final volume.	1) Digestion performed in 250 mL Griffin beaker. 2) Volumetric flask used to bring digestate to final volume.
Procedure	1) Digestate volume reduced to 5 to 10 mL prior to filtering. 2) After filtration, the filters are rinsed three times with reagent water. 3) 30% H_2O_2 is added in two 2 mL aliquots and then six 1 mL aliquots.	1) Digestate volume reduced to 5 mL prior to filtering. 2) After filtration, the filters are rinsed twice with reagent water. 3) 30% H_2O_2 is added in one 3 mL aliquot and then seven 1 mL aliquots.

TABLE 3

PREPARATION OF MATRIX SPIKES AND SPIKING SOLUTIONS FOR DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Matrix Spike for ICP-AES	CLPP-SPK-1	Inorganic Ventures(IV)	0.10
	CLPP-SPK-INT1	Lab Prepared (see below)	1.00
	CLPP-SPK-INT2	Lab Prepared (see below)	1.00

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
CLPP-SPK-INT1	1000 mg/L As,Pb,Sb,Se,Tl	High Purity Standards	1.0 each
	1000 mg/L Cd	High Purity Standards	2.5
	10000 mg/L K	High Purity Standards	10.0
	10000 mg/L Na	High Purity Standards	7.5
	10000 mg/L Mg	High Purity Standards	5.0
	10000 mg/L Ca	High Purity Standards	2.5
CLPP-SPK-INT2	1000 mg/L Mo	IV or High Purity Standards	1.0
	1000 mg/L B,Li,Sn,Sr,Ti	IV or High Purity Standards	5.0 each
	10000 mg/L Si	High Purity Standards	1.0
	1000 mg/L U	High Purity Standards	1.0
	1000 mg/L W	High Purity Standards	1.0
	1000 mg/L Th	High Purity Standards	1.0

TABLE 4

ELEMENT CONCENTRATIONS IN ICP-AES MATRIX SPIKES AND THEIR COMPONENT SPIKING SOLUTIONS FOR DIGESTION OF SOLID SAMPLES BY METHOD 3050

	CONCENTRATION IN SOLUTION, mg/L			
Element	**Matrix Spike**	**CLPP-SPK-1**	**CLPP-SPK-INT1**	**CLPP-SPK-INT2**
Aluminum	2.000	2000		
Antimony	0.100		10	
Arsenic	0.100		10	
Barium	2.000	2000		
Beryllium	0.050	50		
Boron	0.500			50
Cadmium	0.250		25	
Calcium	2.500		250	
Chromium	0.200	200		
Cobalt	0.500	500		
Copper	0.250	250		
Iron	1.000	1000		
Lead	0.100		10	
Lithium	0.500			50
Magnesium	5.000		500	
Manganese	0.500	500		
Molybdenum	0.300			10
Nickel	0.500	500		
Potassium	10.000		1000	
Selenium	0.100		10	
Silicon	5.000			100
Silver	0.050	50		
Sodium	7.500		750	
Strontium	0.500			50
Thallium	0.100		10	
Tin	0.500			50
Titanium	0.500			50
Tungsten	0.100			10
Uranium	0.100			10
Vanadium	0.500	500		
Zinc	0.500	500		
Thorium	0.100			10

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 1

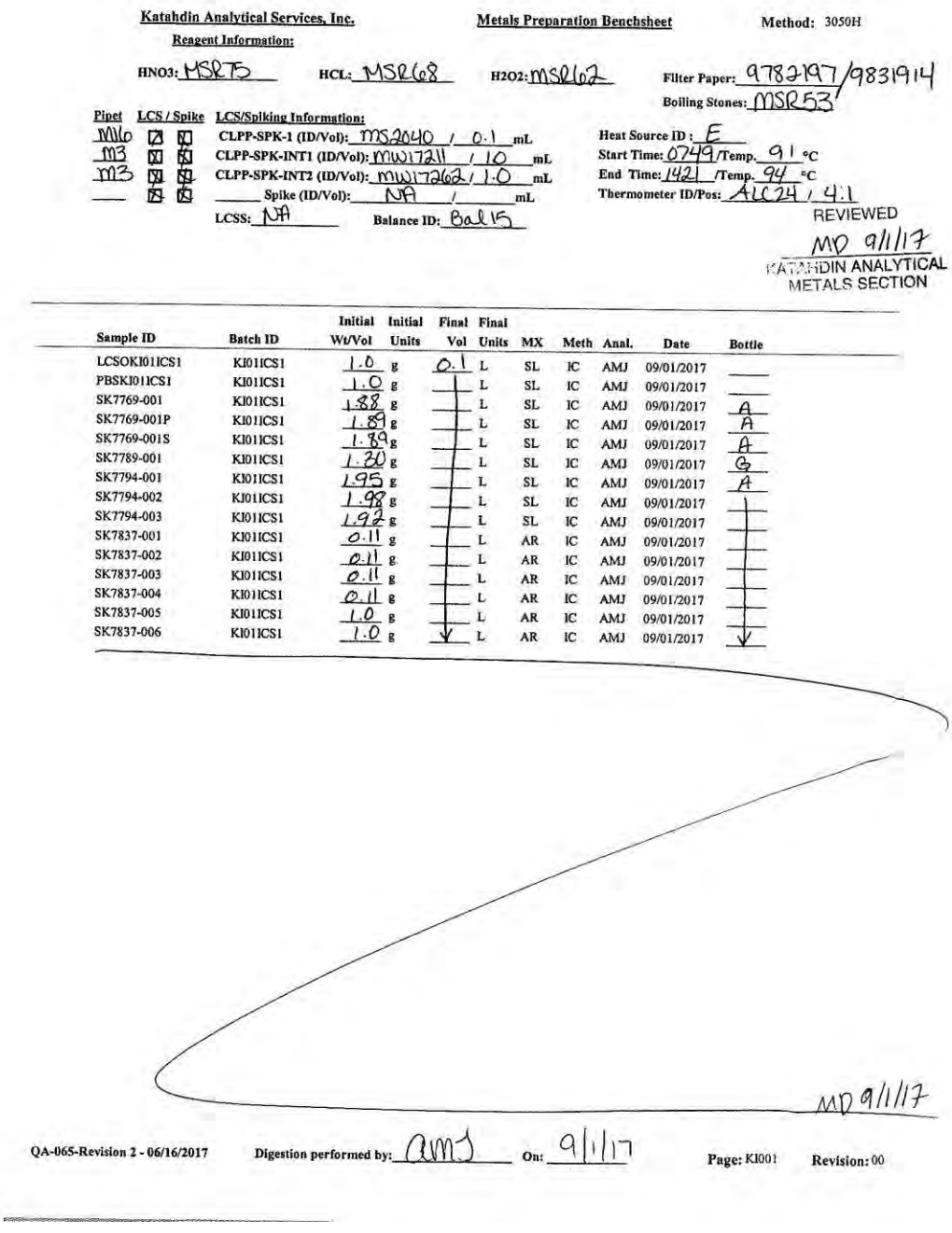
PROCEDURE CONDENSATION – METHOD 3050

1. Prepare and print out ACCESS spreadsheet.
2. If performing digestion on a hot plate, rinse 250 mL Griffin beakers and ribbed watch glasses 3 times in acid bath. Then rinse beakers and watch glasses 3 times with laboratory reagent grade water. If performing digestion with block digester, polyethylene digestion tubes do not require precleaning.
3. Label digestion vessels (beakers or polyethylene sample tubes) with sample numbers.
4. Weigh 1 to 2 g of well-mixed sample into tared digestion vessels. Record sample weights.
5. Add spike solutions to matrix spike samples.
6. Add 10 mL 1:1 HNO_3 to samples and cover with watch glasses.
7. Reflux for 10 to 15 minutes at $95^{o} \pm 5^{o}$ C. without boiling. Cool samples.
8. Add 5 mL conc. HNO_3, cover beakers, and reflux for 30 minutes.
9. Repeat Step 8 as necessary until digestion is complete.
10. Reduce sample volumes to 5 to 10 mL or heat for 2 hours, whichever occurs first.
11. Cool sample and add 2 mL reagent water and 2 mL 30% H_2O_2. Heat gently until effervescence subsides.
12. Cool sample and add 2 mL 30% H_2O_2. Heat gently until effervescence subsides.
13. Cool samples and add 6 mL of 30% H_2O_2 in 1 mL aliquots. Heat gently until effervescence subsides.
14. Reduce sample volumes to 5 to 10 mL or heat for 2 hours, whichever occurs first.
15. Add 10 mL conc. HCl and reflux for 10 to 15 minutes at $95^{o} \pm 5^{o}$ C.
16. Cool sample and filter into graduated specimen container. Bring to volume with reagent water and transfer to labeled polyethylene bottle.
17. Enter sample weights into ACCESS spreadsheet.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 2

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK



Katahdin Analytical Services, Inc. — Metals Preparation Benchsheet — Method: 3050H

Reagent Information:

HNO3: MSR75 HCL: MSR68 H2O2: MSR62 Filter Paper: 9782197/9831914

Boiling Stones: MSR53

Pipet	LCS / Spike	LCS/Spiking Information:
M16	☒ ☒	CLPP-SPK-1 (ID/Vol): MS2040 / 0.1 mL
M3	☒ ☒	CLPP-SPK-INT1 (ID/Vol): MW17211 / 1.0 mL
M3	☒ ☒	CLPP-SPK-INT2 (ID/Vol): MW17262 / 1.0 mL
___	☒ ☒	___ Spike (ID/Vol): NA / ___ mL

LCSS: NA Balance ID: Bal15

Heat Source ID: E

Start Time: 0749 /Temp. 91 °C

End Time: 1421 /Temp. 94 °C

Thermometer ID/Pos: ALC24 / 4.1

REVIEWED
MD 9/1/17
KATAHDIN ANALYTICAL
METALS SECTION

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Bottle
LCSOKI01ICS1	KI01ICS1	1.0	g	0.1	L	SL	IC	AMJ	09/01/2017	
PBSKI01ICS1	KI01ICS1	1.0	g		L	SL	IC	AMJ	09/01/2017	
SK7769-001	KI01ICS1	1.88	g		L	SL	IC	AMJ	09/01/2017	A
SK7769-001P	KI01ICS1	1.89	g		L	SL	IC	AMJ	09/01/2017	A
SK7769-001S	KI01ICS1	1.89	g		L	SL	IC	AMJ	09/01/2017	A
SK7789-001	KI01ICS1	1.80	g		L	SL	IC	AMJ	09/01/2017	G
SK7794-001	KI01ICS1	1.95	g		L	SL	IC	AMJ	09/01/2017	A
SK7794-002	KI01ICS1	1.98	g		L	SL	IC	AMJ	09/01/2017	"
SK7794-003	KI01ICS1	1.92	g		L	SL	IC	AMJ	09/01/2017	"
SK7837-001	KI01ICS1	0.11	g		L	AR	IC	AMJ	09/01/2017	"
SK7837-002	KI01ICS1	0.11	g		L	AR	IC	AMJ	09/01/2017	"
SK7837-003	KI01ICS1	0.11	g		L	AR	IC	AMJ	09/01/2017	"
SK7837-004	KI01ICS1	0.11	g		L	AR	IC	AMJ	09/01/2017	"
SK7837-005	KI01ICS1	1.0	g		L	AR	IC	AMJ	09/01/2017	"
SK7837-006	KI01ICS1	1.0	g	"	L	AR	IC	AMJ	09/01/2017	"

MD 9/1/17

QA-065-Revision 2 - 06/16/2017 Digestion performed by: AMJ On: 9/1/17 Page: KI001 Revision: 00

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 3

EXAMPLE CERTIFICATE OF ANALYSIS FOR SOLID REFERENCE MATERIAL

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

MS1475

DataPacK™
Lot No. D051-540

Trace Metals in Soil
Catalog No. 540

Certification

Method 3050 HNO3, H2O2, HCl Parameter	Total Concentration [1] (mg/Kg)	Certified Value [2] (mg/Kg)	Performance Acceptance Limits™ [3] (mg/Kg)
aluminum	55600*	7870	4630 - 11100
antimony	160	70.5	D.L. - 149
arsenic	316	289	234 - 344
barium	869	211	174 - 247
beryllium	60.9	54.4	45.2 - 63.6
boron	129	91.3	58.8 - 124
cadmium	114	101	82.9 - 119
calcium	9750*	3680	2970 - 4390
chromium	249	224	180 - 268
cobalt	113	101	82.7 - 119
copper	94.9	88.0	73.3 - 103
iron	24400*	15700	6610 - 24900
lead	184	158	129 - 187
magnesium	3780*	2260	1760 - 2750
manganese	703	420	343 - 497
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	69.6	55.5 - 83.7
nickel	137	120	99.1 - 141
potassium	33000*	3000	2200 - 3800
selenium	146	130	101 - 159
silver	127	104	68.9 - 139
sodium	15600*	1080	692 - 1470
strontium	326	113	90.5 - 135
thallium	106	94.0	72.8 - 115
tin	175	149	104 - 194
titanium	3100*	284	116 - 453
vanadium	151	111	85.1 - 137
zinc	311	272	215 - 329

Method 3050 HNO3, H2O2 Parameter	Total Concentration [1] mg/Kg	Certified Value [2] mg/Kg	Performance Acceptance Limits™ [3] mg/Kg
aluminum	55600*	7380	4440 - 10300
antimony	160	75.2	D.L. - 198
arsenic	316	284	225 - 343
barium	869	217	177 - 257
beryllium	60.9	53.6	42.7 - 64.5
boron	129	89.5	58.9 - 120
cadmium	114	103	83.6 - 122
calcium	9750*	3540	2800 - 4270
chromium	249	224	172 - 275
cobalt	113	101	82.0 - 120
copper	94.9	85.5	70.4 - 100
iron	24400*	12500	5480 - 19500
lead	184	162	132 - 192
magnesium	3780*	2160	1650 - 2670
manganese	703	415	330 - 500
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	68.8	52.7 - 84.9
nickel	137	119	98.5 - 140
potassium	33000*	2840	2160 - 3520
selenium	146	135	104 - 166
silver	127	107	49.8 - 164
sodium	15600*	1010	709 - 1310
strontium	326	111	89.0 - 133
thallium	106	99.3	76.8 - 122
tin	175	148	70.6 - 225
titanium	3100*	283	104 - 463
vanadium	151	104	70.5 - 138
zinc	311	275	222 - 328

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

ATTACHMENT 1

MODIFICATIONS FOR 8330B PREPARATION & DIGESTION

4.0 APPARATUS AND MATERIALS – additional materials

4.1 Sieves - #10 mesh (2 mm) – 8" diameter with covers and collection trays.

4.2 Aluminum drying trays with drying rack

4.3 Heavy duty aluminum foil

4.4 Stainless steel scoopulas

4.5 Dust mask

9.0 PROCEDURES – additional procedures

Prior to the digestion of samples (section 7.1 in SOP):

Spread the entire aliquot of soil onto a drying tray lined with heavy duty aluminum foil and dry in air at room temperature or colder to a constant weight (last two successive dry weights within 3% RPD). Trays should be placed in rack for drying. Record all weights in the Sample Drying Logbook.

Note: Hydric soils and sediments with high moisture content may take several days to dry to constant weight.

Remove the oversize fraction by passing it through a 10-mesh (2 mm) sieve. Be sure to break up caked up soil with a gloved hand. Weigh both fractions – oversize and <2mm. Record all weights in the Sieving & Grinding Logbook.

To obtain a subsample, the entire sample must be mixed with a stainless steel scoopula and spread out on a clean surface (aluminum tray lined with foil) so that it is only 1 or 2 cm thick - preferably in a fume hood designed to prevent the spread of dust and possible inhalation or residue losses. Using the scoopula, obtain at least 30 different increments, i.e., portions (~0.3 g) from randomly chosen locations throughout the entire sample profile for a total of ~10 g. Mix this subsample one more time with the scapula and then obtain an aliquot for metals digestion (beginning with section 7.3).

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: CA-604
Revision History
Cover Page
Page 1

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Prepared By: George Brewer Date: 11/97

Approved By:

Group Supervisor: George Brewer Date: 01/19/01

Operations Manager: John C. Burton Date: 1/22/01

QA Officer: Deborah J. Nadeau Date: 1·22·01

General Manager: Duncan F. Hepburn Date: 1/22/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3010A	Format changes, added pollution prevention, block digester; revised database references; revised and added tables.	DH	1·22·01	1/22/01
02 3010A	Added wording allowing use of digestates for ICP-MS analysis. Added use of block digester as primary heating source & adjusted volumes. Revised standard solution names & concs. in Figures 3 & 4.	DH	8·29·02	8·29·02
03	Added Uranium to spiking solutions for LCS & MS/D. Removed the Internal Custody Record for Metals Digestates figure and reference.	LAD	04/06	04/06
04	Minor changes to Section 7 to reflect current practices. Updated Figure 1 - Sample Prep Logbook. Updated Figure 2 and 3 - Spike amounts.	LAD	05/09	05/09
05	Added references. Updated Figure 2 and 3 with correct spike information. Added CA-108 reference for subsampling information.	LAD	04/10	04/10

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Updated Figures 2 and 3. Changed KAS, INC. to KAS.	LAD	06/15	06/15
07	Sect. 1 - Replaced beaker with digestion vessel. Sect. 7.5 & 7.9 - Added calibrated Pipet.	LAD	06/16	06/16
08	Update figure 1. Change title of section 5.0 to Reagents and Standards. Update method references for NELAC and DoD.	LAD	09/17	09/17
09	Added Thorium to Tables 2 and 3. Sect. 8 - Added contingency plan.	LAD	01/19	01/19

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ____ of document **SOP CA-604-09**, titled **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ____ of document **SOP CA-604-09**, titled **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**.

Recipient: _____ Date: _____

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure utilized by Katahdin Analytical Services personnel to solubilize metals in aqueous samples, wastes that contain suspended solids, and mobility-procedure extracts prior to analysis by inductively coupled plasma atomic emission spectroscopy (ICP) and inductively coupled plasma mass spectrometry (ICP-MS). This SOP applies to samples prepared by EPA Method 3010, with the method modifications mentioned in Table 2.

1.1 Definitions - none.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the acid digestion of aqueous samples by EPA Method 3010. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in the acid digestion of aqueous samples using EPA Method 3010 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their work in the appropriate lab notebook. Any deviations from the method or irregularities with the samples should also be recorded in the lab notebook and reported to the Supervisor or designated qualified data reviewer responsible for these data.

It is the responsibility of the Supervisor to ensure that technical personnel perform acid digestions in accordance with this SOP and to confirm that their work is properly documented through periodic review of the associated logbooks.

1.3 Safety

The acids used in this procedure are highly corrosive and reactive, and spiking standards contain toxic metals. The toxicity and reactivity of client samples are usually unknown, so samples should always be assumed to present a contact hazard. To reduce or eliminate exposure to potentially harmful chemicals, lab coats, gloves, and safety glasses or goggles must be worn whenever handling samples or reagents. Additional safety apparel, including face shields, rubber aprons, dust masks, and rubber shoe protectors, is available in the metals prep lab and should be worn whenever circumstances warrant.

Acids should be added to samples slowly and carefully while watching for reactions. This should be done under a hood, in case harmful fumes are evolved.

Hood sashes should be lowered as far as possible whenever digestion vessels are being heated in the hood. Use caution when handling hot digestion vessels.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Sample digestates should be stored for a minimum of 60 days after digestion to allow for analysis, and reanalysis if necessary. Digestates older than 60 days may be emptied into the corrosive waste carboy in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Any other wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Chemical Hygiene Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision.

2.0 SUMMARY OF METHOD

The aqueous sample is refluxed with nitric acid in a covered digestion vessel. Additional nitric acid is added until the color of the digestate has stabilized. After the digestate has been evaporated to a low volume, it is refluxed with hydrochloric acid and diluted to the appropriate final volume with reagent water.

Samples may be concentrated (i.e. final digestate volume less than initial sample volume) during digestion if lower detection limits are required. Volumes of reagents and spiking standards must be added in proportion to the final volume of the digestate. Because concentration of samples during digestion increases the concentrations of dissolved solids

and may exacerbate analytical interferences, concentration factors greater than 5 are not recommended.

3.0 INTERFERENCES

Interferences are discussed in the applicable analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1. 250 mL and 400 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning") for digestion using a hot plate. If digestion will be performed using a block digester, 70ml graduated, polyethylene block digester tubes (with attached snap caps) will be used instead of glass beakers.

4.2 Ribbed watch glasses. If digestion is performed using a hot plate, 75 mm diameter and 100 mm diameter glass watch glasses (pre-cleaned as above) are used. If digestion is performed using a block digester, 40mm diameter disposable polyethylene watch glasses are used.

4.3 Adjustable volume automatic pipets covering the range from 10 uL to 1000 uL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.

4.4 Disposable graduated polystyrene specimen containers with pouring lips, 200 mL capacity.

4.5 Hot plate, block digester, or other heating source - adjustable and capable of maintaining a temperature of 90-95°C. Hot plates must be numbered for easy identification.

4.6 Device for measuring hot plate temperature. This may consist of a heat-resistant 100ml beaker containing reagent water in which a thermometer is immersed. When using a block digester, a digestion tube containing reagent water in which a thermometer is immersed may be used. The temperature of one hot plate is measured each day, on a rotating basis. The hot plate identification number and the measured temperature are recorded on the sample preparation logbook sheet.

4.7 Plastic funnels, pre-cleaned as in Section 4.1.

4.8 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.

4.9 Polyethylene wash bottles for dispensing reagent water and 5% HNO_3.

4.10 Filter paper, Whatman No. 41 or equivalent. Filters are acid-washed immediately prior to use as follows. Place a pre-cleaned funnel in the funnel holder and put a disposable plastic specimen container under the funnel to collect the rinsates. Place a folded filter in the funnel and rinse three times with approximate 10 mL volumes of 5% HNO_3, making sure the entire surface of the filter is wetted each time and allowing each rinse to drain completely before continuing. Then rinse three times with approximate 25 mL volumes of reagent water. Discard the rinsates into the appropriate waste container. The acid-washed filter is now ready for use.

4.11 Polyethylene sample containers with screw caps or graduated polyethylene sample containers with attached snap lids, 125 mL capacity. These are not necessary when using the block digester since the final digestates are stored in the digestion tubes.

4.12 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid and 1:1 HCl.

5.0 REAGENTS AND STANDARDS

5.1 Concentrated nitric acid, HNO_3 – trace metals grade.

5.2 Concentrated hydrochloric acid, HCl – trace metals grade.

5.3 Reagent water - water that meets the performance specifications of ASTM Type II water (ASTM D1193).

5.4 Hydrochloric acid, 1:1. Add a volume of concentrated hydrochloric acid to an equivalent volume of reagent water and swirl gently to mix.

5.5 Nitric acid, 5% v/v. Add 25 mL concentrated HNO_3 to 475 mL reagent water in a 500 mL wash bottle. Cap, point the dispensing tip into a sink, and shake gently to mix.

5.6 Multi-element spiking solutions (as listed in Figure 3).

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for dissolved metals should be filtered through a 0.45 um membrane filter and preserved as soon as possible after collection. Samples to be analyzed for total metals should be preserved, unfiltered, as soon as possible after collection. Aqueous samples are preserved by acidification with nitric acid to a pH of <2.

Please refer to Katahdin Analytical Services SOP CA-108, “Basic Laboratory Technique”, current revision, for information on subsampling.

7.0 PROCEDURES

7.1 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer spreadsheet. Print out a copy of the spreadsheet. With a permamament marker, make sample labels and attach to the polyethylene sample containers that will contain the digestates.

7.2 If using glass beakers as the digestion vessels, submerge previously cleaned beakers three times into a 10% nitric acid bath, then rinse three times with reagent water. The polyethylene digestion tubes used in conjunction with the block digester do not require acid rinsing or precleaning. Label the digestion vessels with sample numbers.

7.3 If digestion is performed using a block digester, the sample aliquot may be measured in the digestion vessel using the graduations on the digestion tubes. Measure 50 ml of well-mixed sample into a 70 ml block digestion tube. A larger sample aliquot may be used (up to 250 mL) if concentration of the sample during digestion is desired. Sample volumes larger than 50 mL may be digested in 250 mL beakers. Measure aliquot of well-mixed sample into a graduated specimen cup and transfer into a properly cleaned 250 mL beaker. Sample volumes of more than 50ml may not be digested using the 70ml block digester tubes. The volumes of reagents and spiking solutions used must be adjusted in proportion to the final digestate volume. The reagent and spiking solution volumes listed below are based on a final volume of 50 mL.

7.4 Add spike solutions to matrix spike samples and laboratory control samples (refer to Figure 3 for spiking instructions).

7.5 Use a repipetter or calibrated pipet, to add 1.5 mL of concentrated HNO_3 (per 50 mL final volume) to the sample. Cover with a ribbed watch glass and place on heatsource. Heat cautiously, without boiling the sample, and evaporate to a low volume (10 - 15 mL).

NOTE: Do not allow any portion of the bottom of the digestion vessel to go dry during any part of the digestion. If a sample is allowed to go to dryness, low recoveries may result. Should this occur, discard the digestate and re-prepare the sample.

7.6 Cool the sample and add another 1.5 mL aliquot (per 50 mL final volume) of concentrated HNO_3. Cover and resume heating, increasing the temperature until a gentle reflux action occurs.

7.7 Continue heating, adding additional acid as necessary, until the digestate is light in color or does not change in appearance with continued refluxing.

7.8 Evaporate digestate to a low volume (10 - 15 mL).

7.9 Cool the sample and use a repipetter or calibrated pipet to add 5 mL (per 50 mL final volume) of 1:1 HCl. Cover the sample and resume heating, refluxing for an additional 15 minutes to dissolve any precipitate or residue resulting from evaporation.

7.10 Allow the sample to cool.

7.11 If the digestate contains visible particulate material, it must be filtered. Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated plastic specimen container or block digester digestion tube. Using a wash bottle, rinse the digestion vessel with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container or digestion tube, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse.

If the digestion was performed using hot plates and the digestate does not contain particulate material, simply decant the digestate into a clean graduated specimen container (or graduated sample container with attached snap lid), rinse the beaker with reagent water, and add the rinsates to the container.

If the digestion was performed using a block digester and the digestate contains no visible particulate material, the digestate may be brought to final volume and stored in the digestion tube without decanting or rinsing.

7.12 Using the graduations on the specimen container, snap-lid container or digestion tube, dilute to the required final volume with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid container or digestion tube has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for analysis.

7.13 Review the ACCESS computer spreadsheet for accuracy. If any information is incorrect, make the necessary changes to the computer spreadsheet and print out a corrected copy. Do not discard the original copy of the spreadsheet. Record (hand write) the sample bottle ID, reagent lot numbers, spiking information, initial and final

volumes, hot plate ID and hot plate temperature in the appropriate spaces on the spreadsheet. Record any method deviations, irregularities with the samples, or other pertinent observations at the bottom of the page, and sign and date the spreadsheet. Bind all copies of the spreadsheet in the sample preparation log. An example sample preparation logbook page (ACCESS spreadsheet) is included as Figure 1.

7.14 Place each batch of digestates in a box labeled with the QC Batch ID, and put the box of digestates in the metals digestates storage area.

7.15 A condensation of the procedure described above is included in this SOP as Table 3. A controlled copy of this table may be posted in the metals preparation laboratory for reference by the analyst.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

8.1 At least one preparation blank for waters (PBW) is processed concurrently with each digestion batch of 20 or fewer samples, and is used to assess contamination resulting from the digestion procedure. The PBW consists of an aliquot of reagent water that is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the PBW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the PBW must also be concentrated). Refer to the appropriate analytical SOP for PBW acceptance criteria and corrective actions.

8.2 At least one laboratory control sample for waters (LCSW) is processed concurrently with each digestion batch of 20 or fewer samples. The LCSW consists of an aliquot of reagent water that is spiked to contain all analytes of interest at known concentrations, and is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the LCSW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the LCSW must also be concentrated). Directions for spiking the LCSW are contained in Figures 3 and 4. The measured analyte recoveries for the LCSW are used to assess digestion method performance. Refer to the appropriate analytical SOP for LCSW recovery acceptance criteria and corrective actions.

8.3 Matrix spiked samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. A matrix spike sample consists of an aliquot of a sample that is spiked with known amounts of all analytes of interest. Matrix spike recoveries are used to assess the effects of sample matrix on digestion and analysis performance. Directions for spiking matrix spike samples are contained in Figures 3 and 4. Refer to the appropriate analytical SOP for matrix spike recovery acceptance criteria and corrective actions.

8.4 Matrix spiked duplicate samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. Matrix spiked duplicate samples are used to assess the precision of the digestion and analysis methods. Refer to the appropriate analytical SOP for matrix spike duplicate precision acceptance criteria and corrective actions.

NOTE: Clients may choose specific samples for matrix spike and matrix spike duplicate analysis; otherwise, the choice is left to the person performing the digestion. The sample volumes available may restrict the choice of samples used for matrix spike and duplicate digestion. Field blank samples should not be chosen for matrix spike and matrix spike duplicate analysis.

8.5 The quality control measures and frequencies described above are minimum requirements. They are summarized for reference in Table 1. Individual clients and analytical programs may impose additional QC requirements.

8.6 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOPs for method performance information.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 3010A.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1, January, 2017.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

LIST OF TABLES AND FIGURES

Table 1 QC Requirements
Table 2 Summary of Method Modifications
Table 3 Procedure Condensation
Figure 1 Example Page From Metals Sample Preparation Logbook
Figure 2 Preparation of Matrix Spikes, LCSs, and Spiking Solutions: Method 3010
Figure 3 Element Concentrations in Matrix Spikes, LCSs, and Spiking Solutions: Method 3010

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 1

QC REQUIREMENTS

Analytical Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3010	Preparation Blank for Waters (PBW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Laboratory Control Sample for Waters (LCSW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Matrix Spike Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Matrix Spike Duplicate Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-604-09	EPA METHOD 3010, current revision
Apparatus/Materials	1) Disposable plastic specimen cup used to measure sample volume. 2) Digestion performed in 250 mL, 400 mL Griffin beaker, or 70ml digestion tube to facilitate evaporation. 3) Ribbed watch glass used throughout digestion to reduce contamination.	1) Graduated cylinder used to measure sample volume. 2) Digestion performed in 150 mL Griffin beaker. 3) Ribbed and non-ribbed watch glasses alternated in digestion.
Procedures	1) Digestate may be analyzed for antimony and silver. 2) Sample aliquots larger or smaller than 100 mL may be used. 3) Sample evaporated to 10 - 15 mL.	1) Digestate may not be analyzed for antimony and silver. 2) Requires sample aliquot of 100 mL. 3) Sample evaporated to 5 mL.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 3

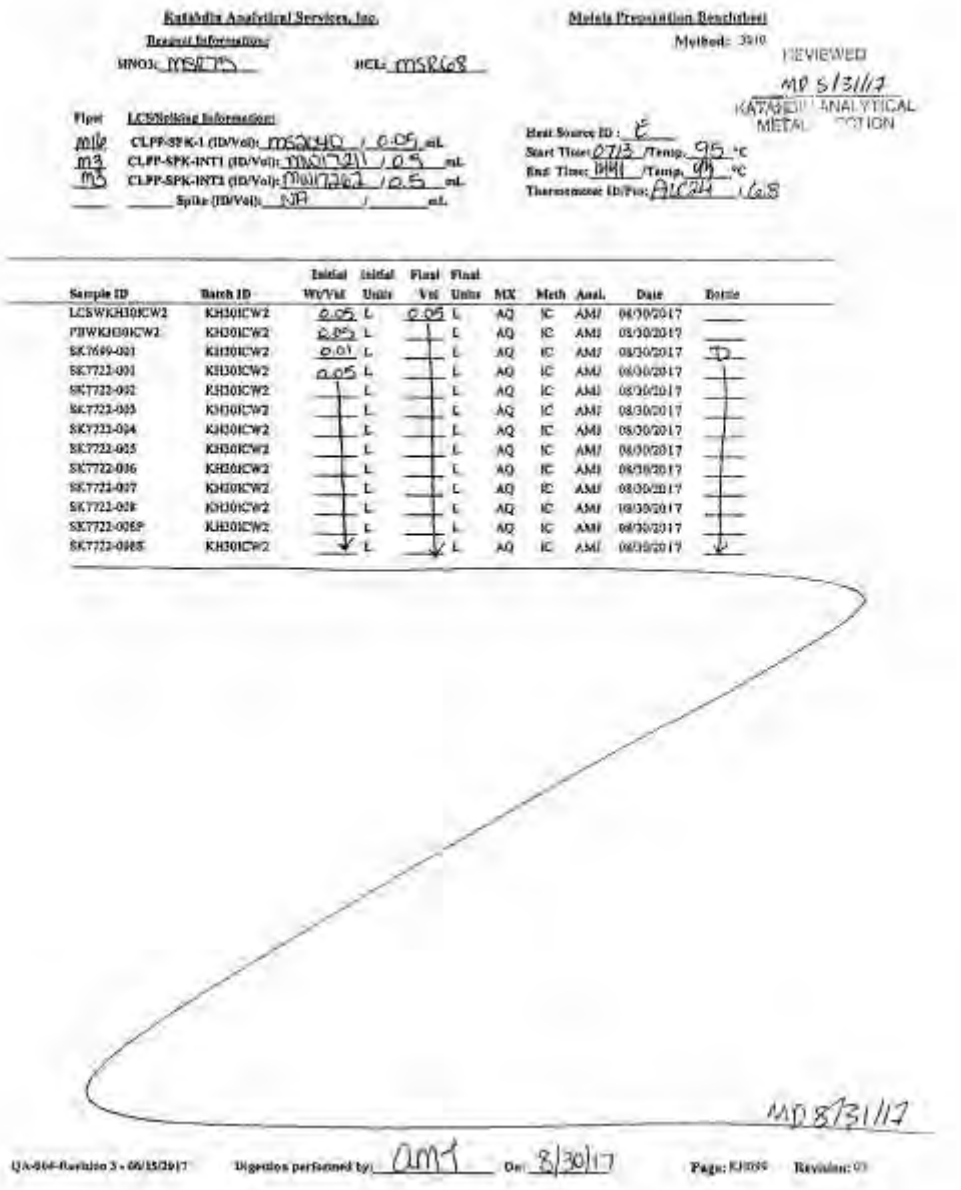
PROCEDURE CONDENSATION: EPA METHOD 3010

1. If performing digestion on a hot plate, rinse glass beakers and ribbed watch glasses 3 times in acid bath. Then rinse beakers and watch glasses 3 times with reagent water. If performing digestion with block digester, polyethylene digestion tubes do not require precleaning.
2. Label digestion vessels with sample numbers.
3. Mix sample well, measure 50 mL (or smaller or larger aliquot) into a polyethylene digestion tube. If using glass beakers, measure aliquot into graduated specimen container, and transfer to appropriate digestion vessel.
4. Add spike solutions to matrix spike samples and LCSW (refer to Figure 3 of this SOP).
5. Add 1.5 mL (per 50 mL final volume) concentrated HNO_3 to sample.
6. Cover with a ribbed watch glass.
7. Place on heating device (hotplate or block digester) and evaporate to 10 - 15 mL.
8. Cool sample and add another 1.5 mL (per 50 mL final volume) concentrated HNO_3.
9. Resume heating until gentle reflux action occurs.
10. Continue heating, adding additional HNO_3 as necessary until digestion is complete.
11. Evaporate to 10 - 15 mL.
12. Cool sample and add 5 mL (per 50 mL final volume) 1:1 HCl. Resume heating and reflux gently for 15 minutes.
13. Cool sample and filter (if necessary) or decant into a graduated polyetheyne digestion tube. Rinse beaker with reagent water and filter or decant rinsate into specimen container.
14. Dilute to appropriate final volume with reagent water.
15. Cap sample container and shake gently to mix.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 1

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK



Katahdin Analytical Services, Inc.

Metals Preparation Benchsheet

Reagent Information:

HNO3: MSR75 HCL: MSR68

Method: 3010

REVIEWED
MD 8/31/17
KATAHDIN ANALYTICAL
METAL SECTION

Pipet LCS/Spiking Information:

m16 CLPP-SPK-1 (ID/Vol): MS2040 / 0.05 mL
m3 CLPP-SPK-INT1 (ID/Vol): MW17211 / 0.5 mL
m3 CLPP-SPK-INT2 (ID/Vol): MW17262 / 0.5 mL
___ ___ Spike (ID/Vol): NA / ___ mL

Heat Source ID: E
Start Time: 0713 /Temp. 95 °C
End Time: 1441 /Temp. 95 °C
Thermometer ID/Pos: AL24 / 68

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Bottle
LCSWKH30ICW2	KH30ICW2	0.05	L	0.05	L	AQ	IC	AMJ	08/30/2017	
PBWKH30ICW2	KH30ICW2	0.05	L		L	AQ	IC	AMJ	08/30/2017	
SK7699-001	KH30ICW2	0.01	L		L	AQ	IC	AMJ	08/30/2017	D
SK7722-001	KH30ICW2	0.05	L		L	AQ	IC	AMJ	08/30/2017	
SK7722-002	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-003	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-004	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-005	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-006	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-007	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-008	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-008P	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-008S	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	

MD 8/31/17

QA-604-Revision 3 - 06/15/2017 Digestion performed by: AMJ On: 8/30/17 Page: KH099 Revision: 03

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 2

PREPARATION OF MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 50 mL Final Volume (mL)
Laboratory Control Sample (LCSW) and Matrix Spike	CLPP-SPK-1	Inorganic Ventures	0.050
	CLPP-SPK-INT1	Lab Prepared (see below)	0.50
	CLPP-SPK-INT2	Lab Prepared (see below)	0.50

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
CLPP-SPK-INT1	1000 mg/L Se	High Purity Standards	1.0
	1000 mg/L As	High Purity Standards	1.0
	1000 mg/L Pb	High Purity Standards	1.0
	1000 mg/L Cd	High Purity Standards	2.5
	1000 mg/L Sb	High Purity Standards	1.0
	10,000 mg/L K	High Purity Standards	10.0
	10,000 mg/L Na	High Purity Standards	7.5
	10,000 mg/L Mg	High Purity Standards	5.0
	10,000 mg/L Ca	High Purity Standards	2.5
	1000 mg/L Tl	High Purity Standards	1.0
CLPP-SPK-INT2	1000mg/L Sr	High Purity Standards	5.0
	1000mg/L Sn	High Purity Standards	5.0
	10,000mg/L Si	High Purity Standards	1.0
	1000mg/L B	High Purity Standards	5.0
	1000mg/L Li	High Purity Standards	5.0
	1000mg/L Ti	High Purity Standards	5.0
	1000mg/L Mo	High Purity Standards	1.0
	1000mg/L U	High Purity Standards	1.0
	1000mg/L W	High Purity Standards	1.0
	1000mg/L Th	High Purity Standards	1.0

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 3

ELEMENT CONCENTRATIONS IN MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND THEIR COMPONENT SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Element	CONCENTRATION		
	CLPP-SPK-1	CLPP-SPK-INT1	CLPP-SPK-INT2
Aluminum	2000		
Antimony		10	
Arsenic		10	
Barium	2000		
Beryllium	50		
Boron			50
Cadmium		25	
Calcium		250	
Chromium	200		
Cobalt	500		
Copper	250		
Iron	1000		
Lead		10	
Magnesium		500	
Manganese	500		
Molybdenum			10
Nickel	500		
Potassium		1000	
Selenium		10	
Silicon			100
Silver	50		
Sodium		750	
Strontium			50
Thallium		10	
Tin			50
Titanium			50
Uranium			10
Vanadium	500		
Zinc	500		
Lithium			50
Tungsten			10
Thorium			10

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Maren Crabill

Review Date: 1/14/2020

SOP Number: CA-604-09

SOP Title: Acid Digestion of Aqueous Samples by EPA method 3010 for ICP and ICP-MS Analysis of total or dissolved metals

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: K. Bruner **Date:** 03/30/20

QAO Signature: Lori Dimond **Date:** 03.30.20

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

Prepared By: Gary Brewer Date: 05/04/07

Approved By:

Department Manager: Gary Brewer Date: 05/04/07

Lab Operations Mgr: Deborah J. Nadeau Date: 5.4.07

QA Officer: Leslie Dimond Date: 5-4-07

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout to reflect current practices. Updated Figures 1-4. Added glassware modifications to Table 2. Corrected calculation. Added PQL of 0.1 umole/g. Added stability time criteria to sect. 7.	LAD	05/09	05/09
02	Updated and/or added references to Sect.s 1.2, 1.3, 9 and 10. Added LOQ/LOD to Table 1.	LAD	06/10	06/10
03	Sect. 5 - Added metals spiking solutions. Sect. 7 - Added instructions for spiking LCSs and MSs. Sect. 9 - Added MDL, LOD & LOQ information. Sect. 10 - Added and edited references. Updated Figures 1→3.	LAD	05/12	05/12
04	Sect. 1 - Added MDR safety concerns, added additional waste disposal directions. Sect. 7 - Added wording for procedural clarification. Sect. 8 - Added contingency plan. Sect 10 - Updated references	LAD	01/19	01/19

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-738-04**, titled **Determination of Acid Volatile Sulfide and Simultaneously Extractable Metals in Sediments.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-738-04**, titled **Determination of Acid Volatile Sulfide and Simultaneously Extractable Metals in Sediments.**

Recipient: _____ Date: _____

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures used by Katahdin Analytical Services technical personnel for the determination of: 1) acid volatile sulfide (AVS) and 2) the simultaneously extractable metals (SEM) that are solubilized during the acidification step. The conditions used have been reported to measure amorphous moderately crystalline monosulfides. As a precipitant of toxic heavy metals, sulfide is important in controlling the bioavailibility of metals in anoxic sediments. If the molar ratio of toxic metals measured by SEM to AVS exceeds one, the metals are potentially bioavailable. Because the relative amounts of AVS and SEM are important in the prediction of potential metal bioavailability, it is important to use the SEM procedure for sample preparation for metal analysis. This uses the same conditions for release of both sulfide and metal from the sediment and thus provides the most predictive means of assessing the amount of metal associated with sulfide.

1.1 Definitions

Method Blank - A deionized water sample that is carried through the entire analytical procedure in the same manner as a sample.

ICV - Initial Calibration Verification. One ICV per batch is prepared from a separate source from the CCV and calibration curve standards. ICV verifies the calibration curve.

LCS- Laboratory Control Standard. One LCS per batch is carried through the entire analytical procedure in the same manner as a sample. The LCS is prepared from the same source as the ICV.

CCV - Continuing Calibration Verification. The CCV is made from the same source as the calibration. One CCV is run every ten samples.

CCB - Continuing Calibration Blank. The CCB is deionized water with no reagents added. One CCB is run every ten samples.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of AVS and SEM by EPA Draft Method 821-R-91-100. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, “Personnel Training & Documentation of Capability,” current revision.

It is the responsibility of all Katahdin technical personnel involved in analysis of AVS or SEM by EPA Draft Method 821-R-91-100 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the

appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Mixed Diamine Reagent (MDR): Corrosive and toxic, a face shield should be worn in addition to standard PPE when preparing solution.

Hydrogen sulfide is a highly poisonous, gaseous compound having a characteristic odor of rotten eggs. It is detectable in air by humans at a concentration of approximately 0.002 ppm. Handling of acid samples should be performed in a hood or in a well ventilated area.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Health and Safety Manual and SOP SD-903,

"Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Place all analyzed samples, standards, rinsates, and unsaved portions of SEM samples (reacted AVS LCS and MS's) in a Satellite Waste "A" Acid for proper disposal in main waste area "A". W1, W2, & filtered portions of S1 & S2 standards, as well as leftover AVS solutions from distillation should be disposed of in Satellite Waste "N-Hi" for proper disposal in main waste area "N-Hi".

2.0 SUMMARY OF METHOD

The method measures the concentrations of AVS and SEM liberated during the acidification of a 10 g sediment sample. Hydrochloric acid is added to the sample to liberate the volatile sufides as hydrogen sulfide (H_2S) at room temperature, and then the H_2S is purged from the sample and collected into an aqueous buffer solution. The amount of AVS that has been trapped is then measured colorimetrically, by reacting sulfide with N-N-dimethyl-p-phenylenediamine to form methylene blue. SEM are determined after filtration of the sediment-acid slurry using ICP spectrometry.

3.0 INTERFERENCES

Oxidation of sulfide in samples may result in a low bias in the results obtained for AVS and SEM. For this reason, contact of the samples with oxygen must be avoided in all stages from sampling through analysis. Collecting samples in containers with minimal headspace, minimizing contact of samples with the air during sample preparation, and using deaerated reagents during sample preparation and analysis all aid in preventing oxidation of samples.

To avoid metals contamination during the SEM digestion part of the method, all reaction vessels and scrubbers should be acid washed prior to use.

4.0 APPARATUS AND MATERIALS

4.1 Polyethylene gas-washing bottles with rubber stoppers and fritted polyethylene spargers, 250 mL capacity, for use as reaction vessels and sulfide traps.

4.2 Flexible tubing for connection from the nitrogen supply to the apparatus and from the flask to the absorber unit.

4.3 High purity nitrogen gas tank with regulator

4.4 Flow meters with needle valves

4.5 Spectrophotometer capable of measuring absorbance at 670 nm

4.6 Spectrophotometer cells, 1 cm path length

4.7 Analytical balance capable of weighing to 0.001 g

4.8 10 mL buret, calibrated in 1/10 mL increments

4.9 Calibrated adjustable pipettors, 0.1 mL, 1.0 mL, and 5.0 mL capacity

4.10 250 mL snap cap graduated containers

4.11 50 mL polyethylene graduated containers

5.0 REAGENTS AND STANDARDS

5.1 Laboratory reagent grade water, deaerated by sparging with nitrogen for one hour just prior to use. Dissolved oxygen should be close to 1 mg/L.

5.2 Sodium hydroxide (NaOH) solution, 0.5 M: Dilute 50.0 mL of 10 N NaOH solution to 1 liter with laboratory reagent grade water. This solution could also be made by dissolving 20 g of NaOH in DI water and diluting to 1 liter. This solution must be deaerated by sparging with nitrogen for one hour just prior to use.

5.3 Starch solution: Make a paste consisting of 2 g lab grade soluble starch, 0.2 g of salicylic acid in a few mL of DI water. Then bring up to volume in a 100 mL volumetric flask. Or use a purchased solution.

5.4 Standard sodium thiosulfate solution, 0.0375 N: Use a purchased primary standard sodium thiosulfate solution with its concentration certified by the vendor.

5.5 Hydrochloric acid, 6 N: In a 1 L volumetric flask, add 500 mL of concentrated HCl to approximately 400 mL of laboratory reagent grade water, slowly mix and allow to cool. Bring to a final volume of 1 L with laboratory reagent grade water. This solution should be deaerated by sparging with nitrogen for one hour prior to use.

5.6 Standard iodine solution, approximately 0.0250N: Dissolve 20 - 25 g potassium iodide in 200 mL of laboratory reagent grade water and add 3.2 g iodine. After the iodine has dissolved, dilute to 1 L with laboratory reagent grade water and standardize against 0.0375 N sodium thiosulfate using the starch solution as an indicator, as explained in Sections 7.8 – 7.11.

5.7 Saturated sodium sulfide standard solutions: Because sulfide is unstable, a standard of pre-determined concentration cannot be prepared. However, a saturated sulfide standard solution can be prepared by dissolving 20 g of sodium sulfide crystals in 10 mL of deaerated laboratory reagent grade water. The sodium sulfide crystals will not all dissolve (if at any point all the crystals in this solution dissolve add more crystals). Two saturated sodium sulfide solutions are required for the analysis – one standard (labeled "S2") is used to prepare the calibration curve and the continuing calibration verification standard, and the other standard (labeled "S1") is used to prepare laboratory control samples and matrix spikes. The concentrations of these saturated standards are determined titrimetrically just prior to use as described in Sections 7.12 – 7.15.

5.8 Sulfide working standard "W1": Fill a 100 mL volumetric flask with deaerated laboratory reagent grade water to about 1 cm below the 100 mL graduation. Using a calibrated adjustable pipet, pipet 0.10 mL of saturated sodium sulfide stock standard "S1" into the flask, dispensing the standard below the surface of the water in the flask. Bring the volume in the flask to the 100 mL graduation with deaerated laboratory reagent grade water, stopper, and invert to mix. Sulfide working standard "W1" must be prepared just prior to use, and may be used to prepare an initial calibration standard for the colorimetric analysis. The sulfide concentration of this standard is $1/1000^{th}$ that of saturated sodium sulfide stock standard "S1", which is determined titrimetrically (see Sections 7.12 – 7.15).

5.9 Sulfide working standard "W2": Fill a 100 mL volumetric flask with deaerated laboratory reagent grade water to about 1 cm below the 100 mL graduation. Using a calibrated adjustable pipet, pipet 0.10 mL of saturated sodium sulfide stock standard "S2" into the flask, dispensing the standard below the surface of the water in the flask. Bring the volume in the flask to the 100 mL graduation with deaerated laboratory reagent grade water, stopper, and invert to mix. Sulfide working standard "W2" must be prepared just prior to use, and is used to prepare the calibration standards and a continuing calibration standard for the colorimetric analysis. The sulfide concentration of this standard is $1/1000^{th}$ that of saturated sodium sulfide stock standard "S2", which is determined titrimetrically (see Sections 7.12 – 7.15).

5.10 Metals spiking standards: Four different standards, named CLPP-SPK-1, CLPP-SPK-INT1, CLPP-SPK-INT2, and Intermediate Mercury Standard A, are required. Intermediate Mercury Standard A has a mercury concentration of 1000 ug/L, and instructions for its preparation are contained in Section 5.9 of the current revision of SOP CA-615 (Digestion and Analysis of Aqueous Samples for Mercury by USEPA Method 7470). Details of the compositions and preparation of the other metals spiking standards are contained in Figures 2 and 3 of the current revision of SOP CA-604 (Acid Digestion of Aqueous Samples By EPA Method 3010 for ICP and ICP-MS Analysis of Total or Dissolved Metals).

5.11 Mixed Diamine Reagent (MDR): Prepare Components A and B as follows, and then mix them together:

5.11.1 Component A - Add 660 mL concentrated sulfuric acid to 340 mL of laboratory reagent grade water. After the solution cools, dissolve 2.25 g N-N-dimethy-p-phenylenediamine oxalate in it.

5.11.2 Component B - Dissolved 5.4 g ferric chloride hexahydrate ($FeCl3 \cdot 6H2O$) in 100 mL concentrated hydrochloric acid and dilute to 200 mL with reagent water.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Sulfide ion is unstable in the presence of oxygen. Protect sediment samples from exposure to oxygen during sample collection and storage.

6.2 Samples should be collected in wide mouth jars, and tightly capped with a minimum of air space above the sediment. The jar lids must have Teflon or polyethylene liners. Alternatively, samples may be collected in syringe samplers (syringes with the tip ends cut off) by plunging the syringe into the sediment while drawing up the plunger, and then tightly capping the open end of the syringe. The syringe sampling method effectively eliminates contact with atmospheric oxygen.

6.3 Samples should be cooled to 4° C as soon as possible after collection, and stored at 4° C until analysis.

6.4 Holding time for samples should not exceed 14 days.

7.0 PROCEDURES

GENERATION OF HYDROGEN SULFIDE FROM SAMPLES

7.1 Assemble the sulfide generation apparatus as shown in Figure 5. The nitrogen gas supply must be shut off. Fill each sulfide trap with 170 mL of 0.5 M NaOH solution.

7.2 Add 100 mL of deaerated laboratory reagent grade water into each reaction vessel. Weigh out approximately 10 g of sample directly into each reaction vessel, minimizing contact with the air. If the sample has been collected in a wide mouth jar, quickly open the jar, scrape away the top centimeter of sediment, scoop up an aliquot of the sample, and transfer it to the tared, water-filled reaction vessel. If the sample has been collected in a syringe sampler, quickly uncap the syringe and extrude the contents of the syringe directly into the tared, water-filled reaction vessel. Record the

sediment weight. Stopper the gas reaction vessel and attach its outlet to the inlet of the sulfide trap.

7.3 All Acid Volatile Sulfide (AVS) spiking should be done beneath the surface of the water in the reaction vessel by penetrating the water's surface with the pipette tip containing the spike and expelling it while submerged. Prepare a Laboratory Control Sample for AVS by spiking the deaerated laboratory reagent grade water in a reaction vessel with 0.10 mL of saturated sodium sulfide stock standard "S1". Prepare AVS matrix spike samples by spiking 0.10 mL of saturated sodium sulfide stock standard "S1" into the reaction vessel containing deaerated water and a sample aliquot.

7.4 In order to prevent reactions between added AVS spiking solutions and SEM spiking solutions, Laboratory Control Samples (LCS) and matrix spikes for Simultaneously Extractable Metals (SEM) must be prepared in separate reaction vessels from the AVS Laboratory Control Sample and AVS matrix spikes. Prepare each SEM LCS and matrix spike by adding the following amounts of each spiking solution to the appropriate reaction vessels.

SEM Spiking Solution	Amount Added (mL)
CLPP-SPK-1	0.20
CLPP-SPK-INT1	2.00
CLPP-SPK-INT2	2.00
Intermediate Mercury Standard A	1.00

7.5 There are three valves in which nitrogen passes through to reach the reaction vessel. The main valve (located in the Extractions lab), the hood valve (located between the two hoods in the Wet lab, left position), and the sample valve. Turn on the main valve on the nitrogen tank and make sure the high pressure gauge reads 300 PSI or more.

7.6 Connect the inlets of the reaction vessels to the nitrogen gas supply. Make sure all tubing connections and stoppers are tight. Make sure the hood valve is in the off position (turn clockwise). Turn the sample valves to the off position (clockwise). Unstopper the reaction vessel and quickly add 20 mL of 6.0 M HCl to the water and sediment in the vessel. Quickly re-stopper the reaction vessel tightly. Some samples may effervesce with the addition of HCl. If this happens, open the sample valve and the hood valve slightly to encourage the airflow from the reaction vessel to the trap vessel. This prevents back flow. Once all samples have been acidified, slightly open all the sample valves and if it isn't already, open the hood valve. Adjust the valves so all samples are bubbling equally.

7.7 After 60 minutes, disconnect the sulfide trap, transfer the sodium hydroxide solution from the trap into a 250 mL plastic container, and shut off the main valve on the nitrogen tank. Retain the sodium hydroxide solution for colorimetric analysis of sulfide.

7.8 Filter the solution sediment / acid slurry remaining in the reaction vessel through a membrane filter (pore size 0.45um) and save the filtrate in a 250 mL snap cap container for the SEM analysis. Bring the volume of filtrate to 200 mL in each snap cap container with deionized water, cap the vessel, and shake well to mix. The filtrate is now ready for SEM analysis.

TITRATION OF STANDARD IODINE SOLUTION

7.10 Add 15 mL of standard iodine solution and 2.0 mL of 6 N HCl to 200 mL of deaerated laboratory reagent grade water in a 250 mL Erlenmeyer flask. The solution will be an orange-yellow color.

7.11 Using the 10 mL buret, titrate this solution with 0.0375 N sodium thiosulfate standard solutions until a pale straw yellow color is obtained.

7.12 Add a few drops of starch solution to produce a deep blue color in the solution. Continue the titration with sodium thiosulfate until a colorless endpoint is reached. Record the total volume of sodium thiosulfate solution that was added to reach the endpoint.

7.13 Repeat the titration two more times, recording the volume of sodium thiosulfate required to reach the endpoint each time. Enter the appropriate volumes and normalities in the AVS Calculation Spreadsheet to calculate the normality of the standard iodine solution. The spreadsheet calculates the mean normality from the three titrations.

TITRATION OF SATURATED SODIUM SULFIDE STOCK STANDARDS

7.14 Add 200 mL of deaerated laboratory reagent grade water to a 250 mL Erlenmeyer flask. Pipet 0.10 mL of one of the saturated sodium sulfide stock standards to the water in the flask, placing the pipet tip below the surface of the water while adding the sulfide standard. Add 10.0 mL portions of standard iodine solution to the flask until a persistent orange-yellow color is obtained. Record the volume of iodine solution used, and then add 2.0 mL of 6 N hydrochloric acid to the flask.

7.15 Using the 10 mL buret, titrate this solution with 0.0375 N sodium thiosulfate standard solution until a pale straw yellow color is obtained.

7.16 Add a few drops of starch solution to produce a deep blue color in the solution. Continue the titration with sodium thiosulfate until a colorless endpoint is reached. Record the total volume of sodium thiosulfate solution that was added to reach the endpoint.

7.17 Repeat the titration two more times, recording the volume of sodium thiosulfate required to reach the endpoint each time. Enter the appropriate volumes and normalities in the AVS Calculation Spreadsheet to calculate the concentration of the saturated sodium sulfide stock standard. The spreadsheet calculates the mean sulfide concentration of the standard from the three titrations. Triplicate titrations must be performed on both of the saturated sodium sulfide stock solutions.

PREPARATION OF CALIBRATION STANDARDS AND THE CONTINUING CALIBRATION VERIFICATION STANDARD

7.18 Prepare a calibration curve as follows. Add 40 mL of 0.5 M sodium hydroxide solution into each of eight 50 mL graduated polyethylene containers. Using calibrated adjustable pipets, pipet the following volumes of Sulfide Working Standard W2 into the containers, dispensing the standard below the surface of the sodium hydroxide solution: 0.00 mL, 0.04 mL, 0.10 mL, 0.30 mL, 0.50 mL, 0.70 mL, 0.90 mL, 1.00 mL. The sulfide concentrations of the calibration standards will vary depending on the actual concentration the sulfide stock standard, as determined in Sections 7.12 – 7.15. The standard that contains 0.50 mL of Sulfide Working Standard W2 is analyzed throughout the colorimetric analysis as the Continuing Calibration Verification standard.

COLORIMETRIC ANALYSIS OF SAMPLES AND STANDARDS

7.19 Standards: A face shield should be worn. Working in the fume hood, add 5.0 mL MDR to each standard, and bring the volume to the 50 mL graduation with deaerated laboratory reagent grade water. These must be mixed by inversion and vented immediately to prevent the pressure buildup causing snap cap to burst open. Solution is extremely dangerous to come in contact with. Open and close snap caps with care to prevent splashing. After 30 minutes, measure the absorbance of the standards at 670 nm. The absorbance of the reacted samples is stable for no more than two hours.

7.20 Samples: A face shield should be worn. Working in the fume hood, transfer an appropriate volume aliquot of the scrubber solution (depending on the expected sulfide concentration) into a 50 mL graduated polyethylene container. Add sufficient 0.5M sodium hydroxide solution to bring the total volume to 40 mL. Add 5.0 mL MDR to the container and bring the solution to the 50 mL graduation with deaerated laboratory reagent grade water. These must be mixed by inversion and vented immediately to prevent the pressure buildup from causing snap cap to burst open.

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

Solution is extremely dangerous to come in contact with. Open and close snap caps with care to prevent splashing. After 30 minutes, measure the absorbance of the samples at 670 nm. The absorbance of the reacted samples is stable for no more than two hours.

CALCULATIONS

7.21 Calculate the concentration of the saturated sodium sulfide stock standards as follows:

$$S^{2-}\ (mg/L) = \frac{(A \times B) - (C \times D) \times 16000}{E}$$

where:
- A = mL of I_2 solution
- B = Normality of I_2 solution
- C = mL of Thiosulfate Titrant
- D = Normality of Thiosulfate Titrant
- E = Volume of Sulfide Standard added to flask

7.20 Calculate the LCS and Spike level as follows:

$$LCS/Spike\ (mg/kg) = (F \times G) / H$$

where:
- F = mL of the sulfide
- G = concentration of the sulfide standard
- H = weight of sample

7.21 Calculate the concentration of sulfide in samples as follows:

$$\frac{umole}{g}\ S^{2-} = \frac{Final\ Vol \times umole\ S-}{analysis\ volume} \times \frac{1}{sample\ wt\ g} \times \frac{1}{\%\ Solids}$$

7.22 The above calculations can be done using a spreadsheet template (Figure 2) on which the analyst enters the sample number, date prepared, date analyzed, sample weight and final volume, volume of sample, volume of Iodine and the normality of both the iodine and thiosulfate. Manually calculated results should be reported to 2 significant figures.

7.23 All batch sheets, raw data, and supporting documents are scanned after final review and the resulting image files are saved on a Katahdin server for use in data package assembly. Image files of raw data are periodically archived by the laboratory's MIS department.

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

Refer to Table 1 for summary of QC requirements.

8.1 Distill and analyze a method blank with every batch or analytical session. The concentration of the blank is expected to be less than the Practical Quantitation Limit (.1umole/g).

8.2 Analyze a Continuing Calibration Verification standard at the beginning of every analytical session, after every 10 samples, and at the end of each analytical session. The acceptance limits for the CCV are 80% - 120% recovery.

8.3 Prepare and analyze one duplicate sample in every batch of 20 or fewer samples. The results for duplicate samples must agree within 20% relative percent difference (RPD).

8.4 Prepare and analyze one matrix spike sample with every 10 client samples. The acceptance limits for matrix spike recoveries are 75% - 125% of the added analyte concentration.

8.5 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Detection (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Inorganic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revisions of the applicable methods for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

USEPA Draft Analytical Method for Determination of Acid Volatile Sulfide in Sediment, Allen et al, 1991, Document 821/R-91-100

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Current Version.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF TABLES AND FIGURES

Table 1 QC Requirements
Table 2 Summary of Method Modifications
Figure 1 Example of Waters and Soils Runlog Page
Figure 2 Acid Volatile Sulfide Calculation Spreadsheet
Figure 3 Reactive Sulfide Logbook Page
Figure 4 Acid Volatile Sulfide Batch Sheet
Figure 5 Acid Volatile Sulfide Apparatus

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
AVS SEM	Initial Calibration – Minimum of 5 pt. plus blank	Each day of analysis	r $\geq$ 0.995	(1) Recalibrate
	Method blank	One per prep batch of 20 or fewer samples	No analyte detected >PQL	(1) Investigate source of contamination (2) Reprep and analyze method blank and all samples processed with the contaminated blank
	LCS/ICV	One of each per prep batch	80-120 %R; Statistically derived from lab data	(1) Recalibrate and reanalyze sample batch
	CCV	At beginning of run, after every 10 samples and at the end of the run. Same conc. As LCS/ICV	80-120 %R	(1) Reanalyze all samples back to last acceptable CCV recovery
	Matrix Spike	One for every set of 10 samples	75-125 %R	(1) Notate sample result in raw data with Notation I-1
	Sample Duplicate	One sample duplicate per twenty samples or per batch	RPD $\leq$20	(1) If lab QC in criteria and matrix interference suspected, flag data (2) Else, reanalyze
	Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file
	MDL and-or LOD/LOQ Verification	Refer to KAS SOP QA-806, “, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision		
	CCB	At beginning of run, after every 10 samples and at the end of the run.	No analyte detected > PQL.	Reanalyze all samples after last acceptable CCB.

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

TABLE 2

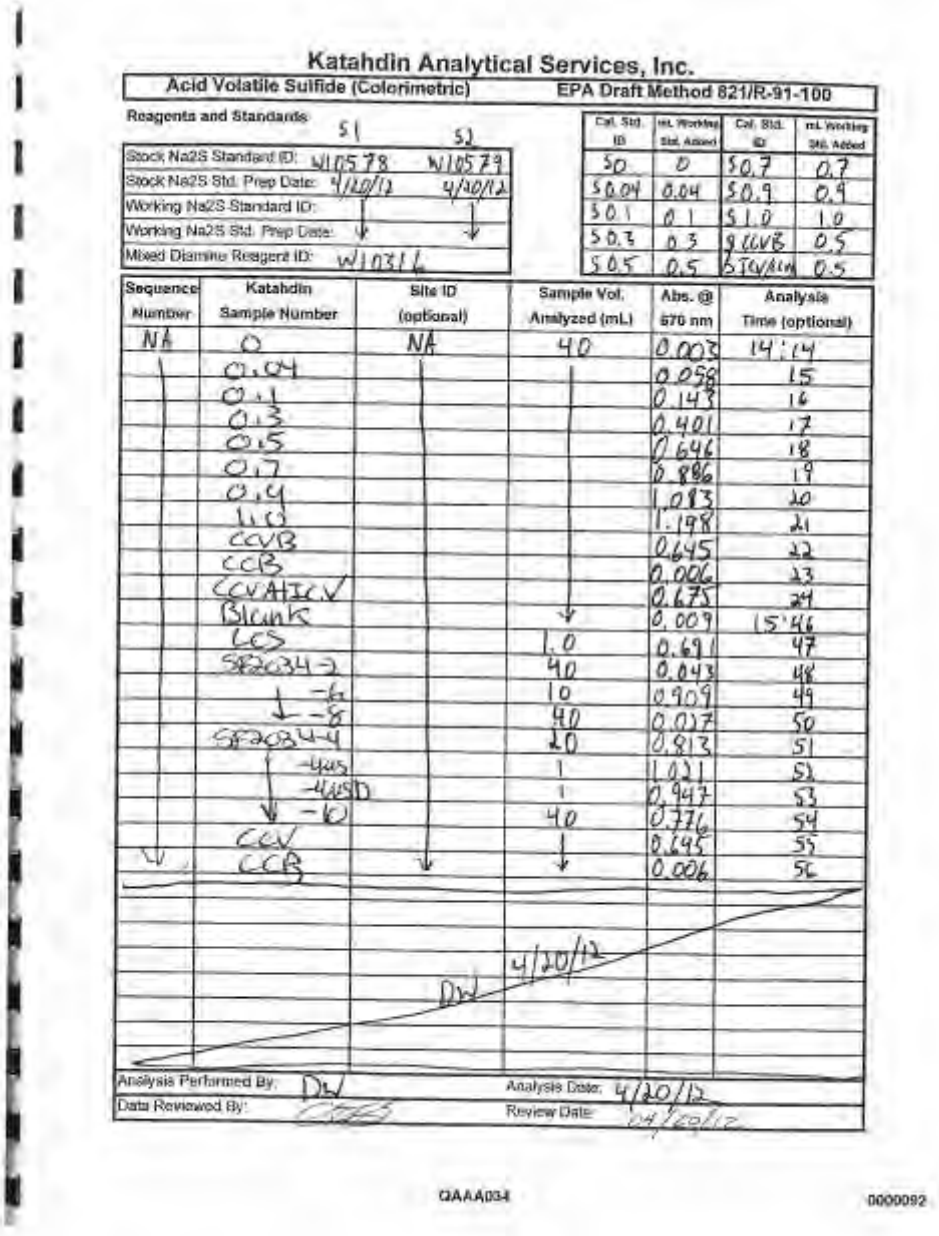
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-738-04	Draft Analytical Method for Determination of Acid Volatile Sulfide in Sediment
Apparatus/ Materials	Use gas washing bottles	Use glassware with ground glass connections
Reagents		
Sample preservation/ handling		
Procedures	170 mL 0.5N NaOH used	80 mL 0.5N NaOH used
QC – Spikes	Recovery acceptance limits = 75% - 125%	Recovery acceptance limits = 85% - 105%
QC – LCS	Recovery acceptance limits = 80% - 120%	Recovery acceptance limits = 85% - 105%
QC - Accuracy/Precision		
QC – MDL		

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

FIGURE 1

EXAMPLE OF WATERS AND SOILS RUNLOG PAGE



Katahdin Analytical Services, Inc.

Acid Volatile Sulfide (Colorimetric) — **EPA Draft Method 821/R-91-100**

Reagents and Standards

	S1	S2
Stock Na2S Standard ID:	W10578	W10579
Stock Na2S Std. Prep Date:	4/20/12	4/20/12
Working Na2S Standard ID:	↓	↓
Working Na2S Std. Prep Date:	↓	↓
Mixed Diamine Reagent ID:	W10316	

Cal. Std. ID	mL Working Std. Added	Cal. Std. ID	mL Working Std. Added
S0	0	S0.7	0.7
S0.04	0.04	S0.9	0.9
S0.1	0.1	S1.0	1.0
S0.3	0.3	8 CCVB	0.5
S0.5	0.5	5 ICV/ICB	0.5

Sequence Number	Katahdin Sample Number	Site ID (optional)	Sample Vol. Analyzed (mL)	Abs. @ 670 nm	Analysis Time (optional)
NA	0	NA	40	0.003	14:14
	0.04			0.058	15
	0.1			0.143	16
	0.3			0.401	17
	0.5			0.646	18
	0.7			0.886	19
	0.9			1.083	20
	1.0			1.198	21
	CCVB			0.645	22
	CCB			0.006	23
	CCV/ICV			0.675	24
	Blank		↓	0.009	15:46
	LCS		1.0	0.691	47
	SB2034-2		40	0.043	48
	-6		10	0.909	49
	↓ -8		40	0.027	50
	SB2034-4		20	0.813	51
	-4MS		1	1.021	52
	-4MSD		1	0.947	53
	↓ -10		40	0.776	54
	CCV			0.645	55
↓	CCB	↓	↓	0.006	56

4/20/12

DW

Analysis Performed By: DW — Analysis Date: 4/20/12

Data Reviewed By: [signature] — Review Date: 04/20/12

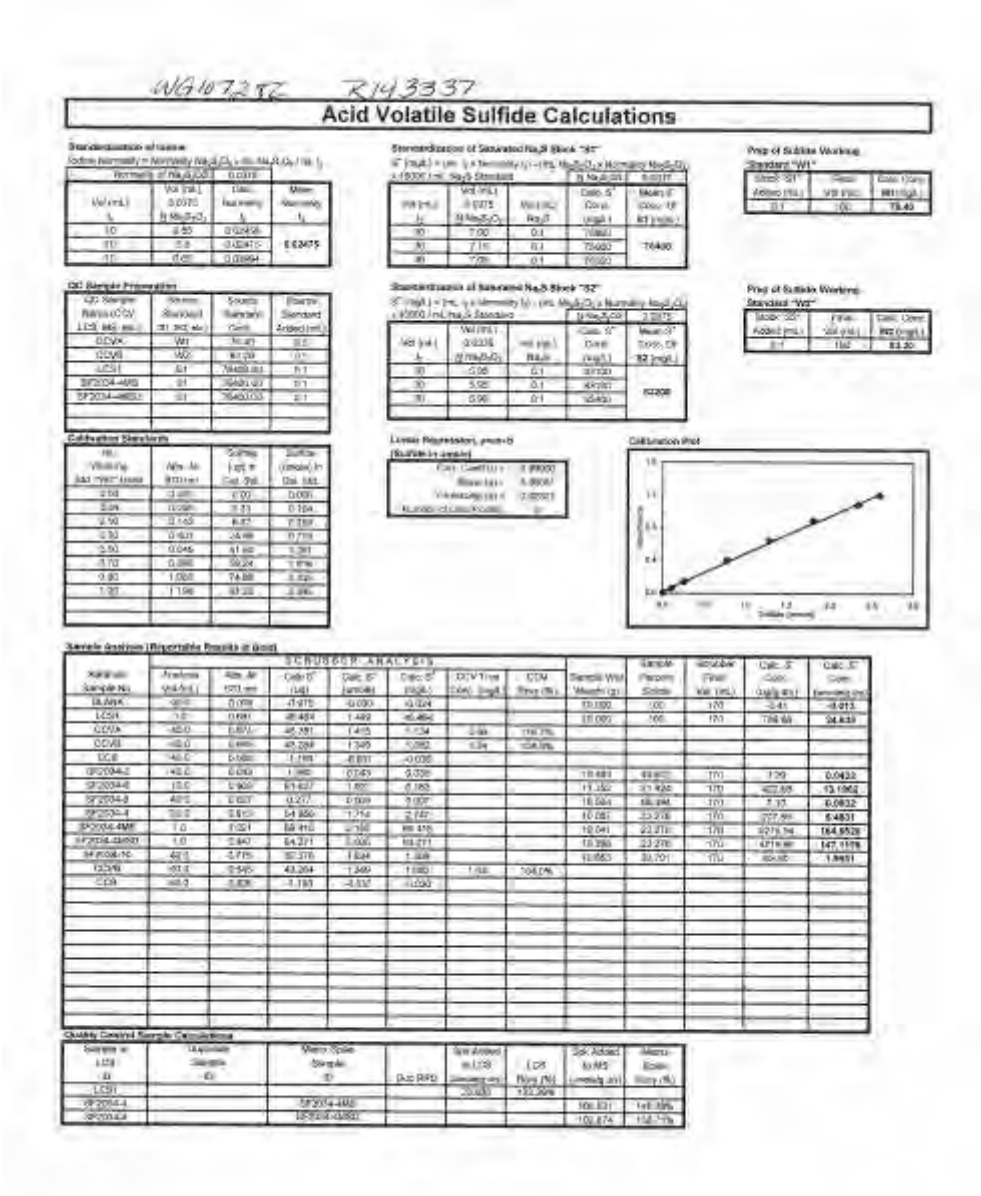
QAAA034

0000092

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

FIGURE 2

EXAMPLE OF AVS CALCULATION SPREADSHEET



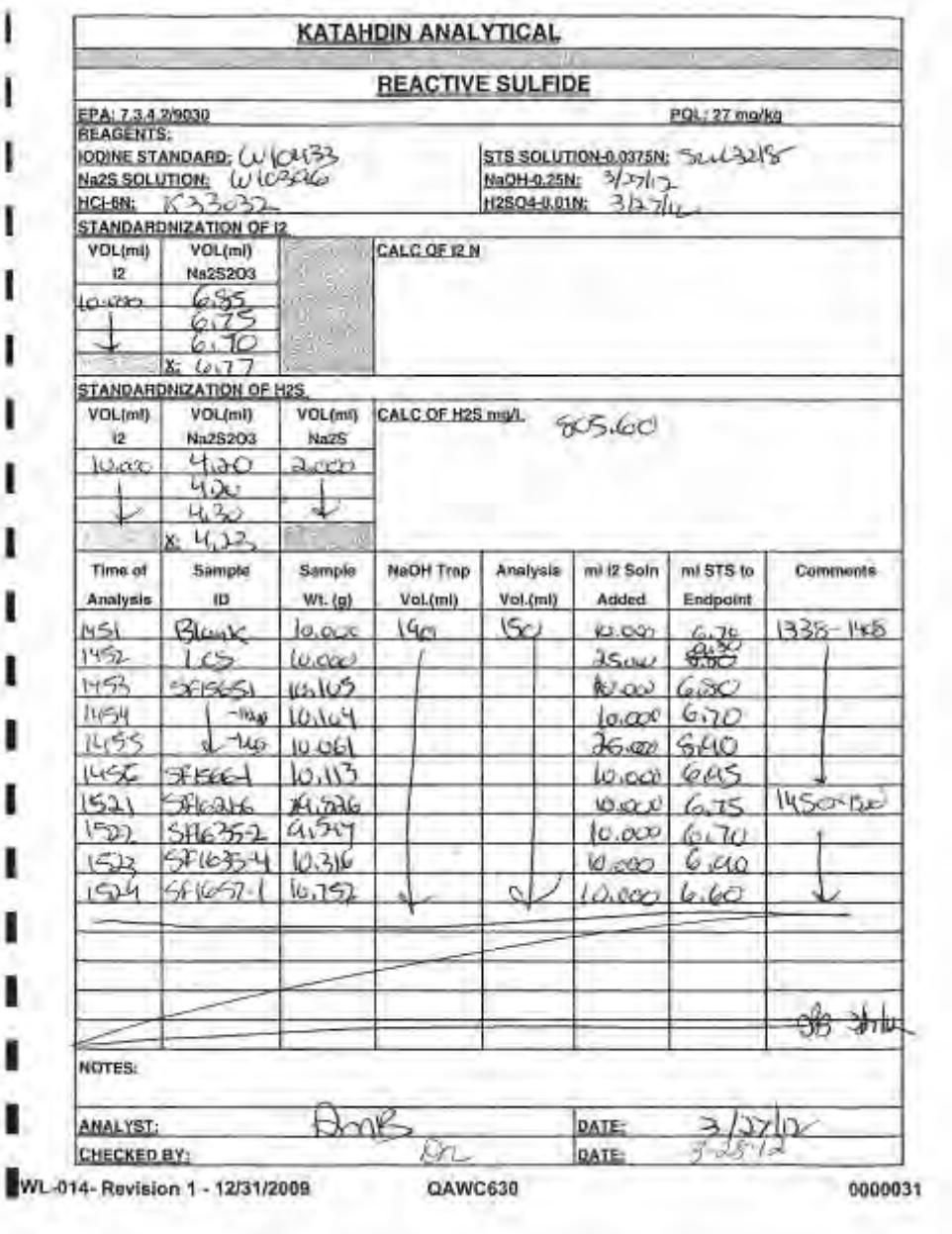
WG107275 R143337

Acid Volatile Sulfide Calculations

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

FIGURE 3

REACTIVE SULFIDE LOGBOOK PAGE



KATAHDIN ANALYTICAL

REACTIVE SULFIDE

EPA: 7.3.4.2/9030 PQL: 27 mg/kg

REAGENTS:

IODINE STANDARD: W10433
Na2S SOLUTION: W10396
HCl-6N: K33032
STS SOLUTION-0.0375N: SCL3218
NaOH-0.25N: 3/27/12
H2SO4-0.01N: 3/27/12

STANDARDNIZATION OF I2

VOL(ml) I2	VOL(ml) Na2S2O3	
10.000	6.85	
↓	6.75	
	6.70	
	X: 6.77	

CALC OF I2 N

STANDARDNIZATION OF H2S

VOL(ml) I2	VOL(ml) Na2S2O3	VOL(ml) Na2S
10.000	4.20	2.000
↓	4.20	↓
	4.30	
	X: 4.23	

CALC OF H2S mg/L 805.60

Time of Analysis	Sample ID	Sample Wt. (g)	NaOH Trap Vol.(ml)	Analysis Vol.(ml)	ml I2 Soln Added	ml STS to Endpoint	Comments
1451	Blank	10.000	140	150	10.000	6.70	1338-1408
1452	LCS	10.000	↓	↓	25.000	~~6.90~~ 6.80	↓
1453	SF1565-1	10.105			10.000	6.80	
1454	↓ -1mg	10.164			10.000	6.70	
1455	↓ -1mg	10.061			25.000	6.90	
1456	SF1566-1	10.113			10.000	6.95	↓
1521	SF1626-6	9.826			10.000	6.75	1450-1500
1522	SF1635-2	9.547			10.000	6.70	↓
1523	SF1635-4	10.316			10.000	6.90	
1524	SF1657-1	10.752	↓	↓	10.000	6.60	↓

NOTES:

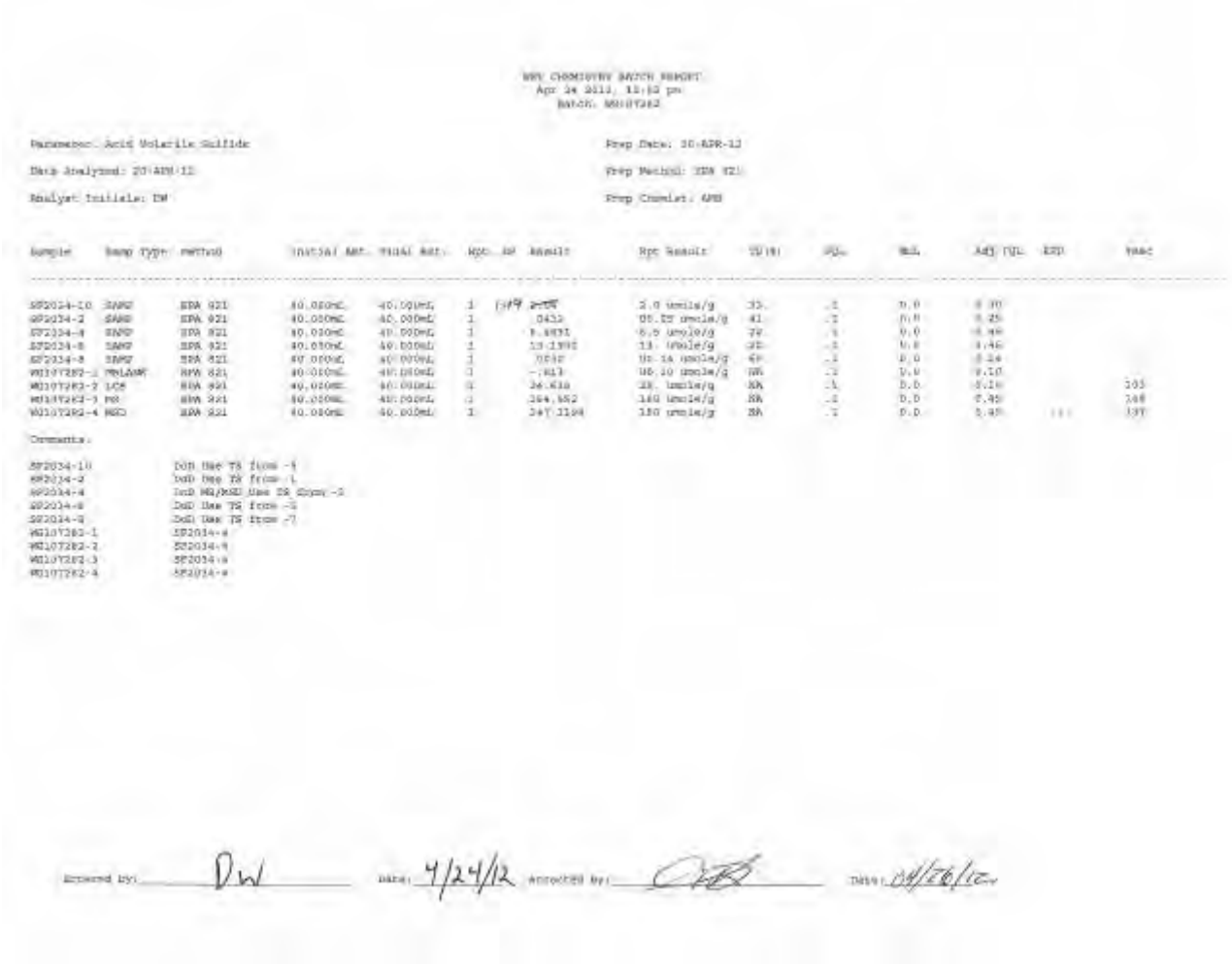
ANALYST: DMB DATE: 3/27/12
CHECKED BY: DR DATE: 3-28-12

WL-014- Revision 1 - 12/31/2009 QAWC630 0000031

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

FIGURE 4

ACID VOLATILE SULFIDE BATCH SHEET



WET CHEMISTRY BATCH REPORT
Apr 24 2012, 11:53 pm
Batch: WG107282

Parameter: Acid Volatile Sulfide — Prep Date: 20-APR-12
Date Analyzed: 20-APR-12 — Prep Method: EPA 821
Analyst Initials: DW — Prep Counter: AMB

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpc. Nr	Result	Rpc Result	%R(1)	%RL	Wt.	Adj %RL	RPD	Yac
SF2034-10	SAMP	EPA 821	40.000mL	40.000mL	1	[illegible]	2.0 umole/g	35	.1	0.0	0.20		
SF2034-2	SAMP	EPA 821	40.000mL	40.000mL	1	.0432	05.22 umole/g	41	.1	0.0	0.25		
SF2034-4	SAMP	EPA 821	40.000mL	40.000mL	1	1.4831	6.5 umole/g	78	.1	0.0	0.49		
SF2034-8	SAMP	EPA 821	40.000mL	40.000mL	1	13.1991	13. umole/g	25	.1	0.0	0.46		
SF2034-8	SAMP	EPA 821	40.000mL	40.000mL	1	.0512	00.14 umole/g	47	.1	0.0	0.24		
WG107282-1	MBLANK	EPA 821	40.000mL	40.000mL	1	-.013	00.10 umole/g	ND	.1	0.0	0.10		
WG107282-2	LCS	EPA 821	40.020mL	40.000mL	1	34.431	28. umole/g	RA	.1	0.0	0.14		105
WG107282-3	MS	EPA 821	40.020mL	40.000mL	1	264.552	140 umole/g	RA	.1	0.0	0.95		148
WG107282-4	MSD	EPA 821	40.000mL	40.000mL	1	247.2194	130 umole/g	RA	.1	0.0	0.97	11	137

Comments:

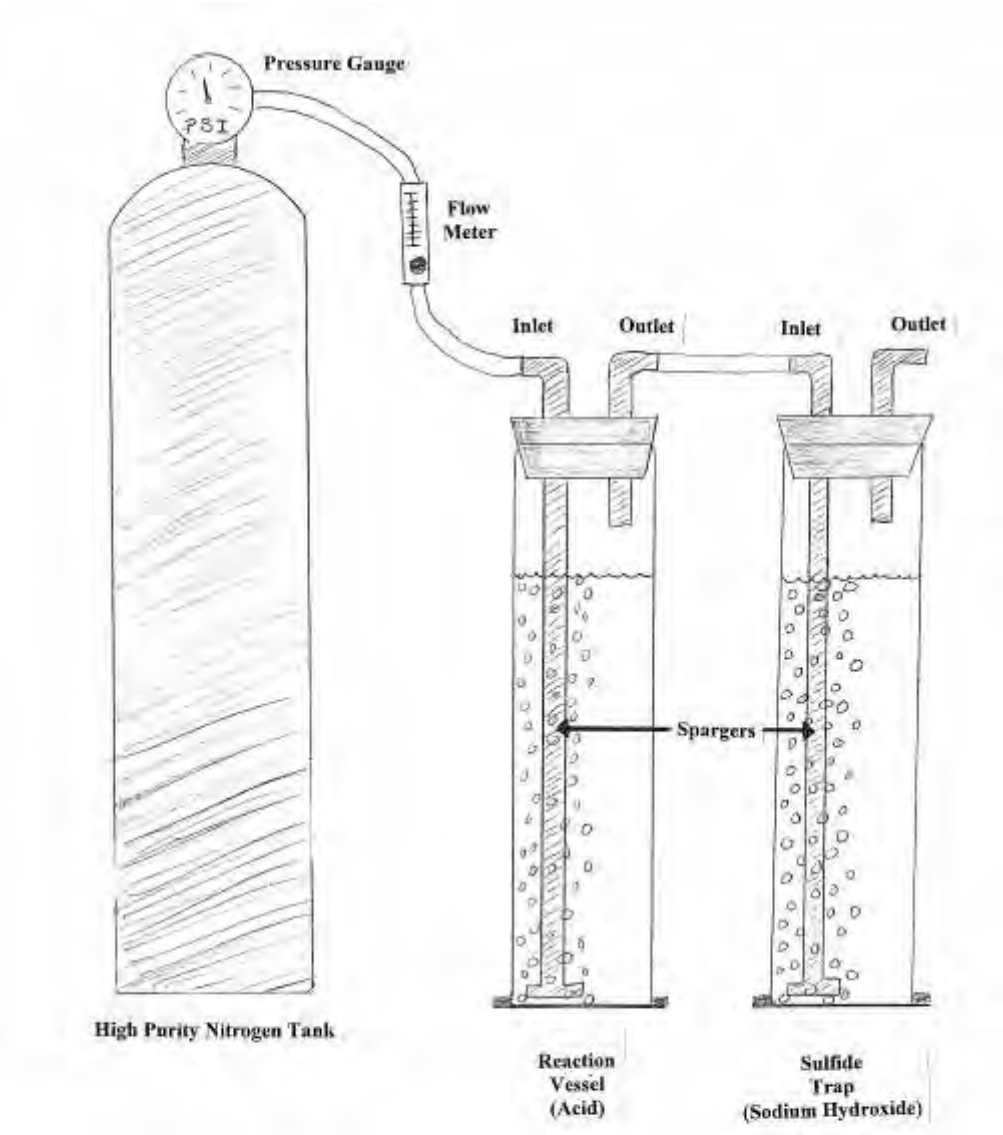
SF2034-10	DUP Use TS from -9
SF2034-2	DUP Use TS from -1
SF2034-4	LCS MS/MSD Use TS Dupe -2
SF2034-8	DUP Use TS from -5
SF2034-8	DUP Use TS from -7
WG107282-1	SF2034-a
WG107282-2	SF2034-a
WG107282-3	SF2034-a
WG107282-4	SF2034-a

Reviewed by: DW Date: 4/24/12 Approved by: [signature] Date: 04/26/12

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

FIGURE 5

ACID VOLATILE SULFIDE APPARATUS



ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Zachary Fuller

Review Date: 1/6/21

SOP Number: CA-738-04

SOP Title: Determination of Acid Volatile Sulfide and Simultaneously Extractable Metals in Sediments

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 01/08/21

QAO Signature: Leslie Dimond **Date:** 011521

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-741
Revision History
Cover Page
Page 1

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II LLOYD KAHN METHOD

Prepared By: Greg Lull Date: 7/2002

Approved By:

Group Supervisor: Keith Tanguay Date: 091102

Lab Operations Mgr: Jol C. Burton Date: 9/11/02

QA Officer: Deborah J. Nadeau Date: 9.11.02

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Sect. 1-clarified CCB, Sect. 2-clarified phosphoric acid conc. Sect. 7- reworded software and instr. startup, added TS to calc., removed steps not currently in practice. Sect. 8 - updated LCS and CCV information. Updated Table 1	LAD	01/07	01/07
02	Added method SW846 9060 to title and Table 2. Added 9060 quadruplicate sample analysis to sect. 7 and Table 1. Removed D.I. water from MB definition. 5.3-added TIC std. info. Sect. 7.0 - rewrote for clarity on instrument and software instructions. Fixed typos- sect. 2.	LAD	06/08	06/08
03	Minor changes to reflect current equipment, practices and techniques.	LAD	08/09	08/09
04	Added 9060 QC requirements to Table 1. Added method modifications to Table 2. Minor edits to Section 7 to reflect current practice, remove redundancy and for clarification. Updated and/or added references to section 7.9 and 10.	LAD	06/10	06/10
05	Removed 1:1 phosphoric acid and added 1:1 hydrochloric acid. Added MDL, LOD and LOQ information. Updated Figures 1 → 4.	LAD	02/13	02/13

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Sect. 5 – Updated for current CCV and LCS source and preparation.	LAD	05/16	05/16
07	Added Particulate Organic Carbon to Sect. 1 & 2. Sect. 8 - Added contingency plan. Added Attachment A - preparation and calculation of particulate organic carbon, (POC)	LAD	05/18	05/18
08	Sect. 1 - Added muffled sand to M. Blk and CCB definitions Sect 6 - Added 0.5ml HCl to TOC calibration std. prep., Added muffled sand and the preparation. Sect. 9 - Added requirement to dry M.Blk, LCS and CCVs. Updated Logbook Ex.	LAD	01/19	01/19
09	Sect. 2 - 2nd Paragraph - changed combusting to drying.	LAD	09/20	09/20

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP **CA-741-09**, titled **DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP **CA-741-09**

, titled **DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.**

Recipient: _____ Date: _____

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

1.0 SCOPE AND APPLICATION

This SOP describes the procedures used by Katahdin Analytical Services technical personnel to determine Total Organic Carbon (TOC) in solids and Particulate Organic Carbon (POC) in aqueous matrices in accordance with EPA Region II Lloyd Kahn method and SW846 9060 Mod.

This method is applicable to sediment, sludges, and soil samples. The detection limit for this method is 100 µg C and a method PQL of 400 µg/g.

This method may also be used to determine the Particulate Organic Carbon (POC) associated with aqueous samples. The preparation and calculation of POC is detailed in Attachment A of this SOP.

1.1 Definitions/Acronyms

TC – Total carbon

IC – Inorganic Carbon

TOC – Total Organic Carbon

POC - Particulate Organic Carbon

Method Blank – A sample boat filled with approximately halfway with muffled sand, no reagents are added and is carried through the entire analytical procedure in the same manner as a sample.

LCS/ICV - Laboratory Control Sample/ Initial Calibration Verification. One LCS/ICV per batch is prepared from a separate source from the CCV and calibration curve standards. LCS/ICV verifies the calibration curve.

CCV - Continuing Calibration Verification. The CCV is made from the same source as the calibration. One CCV is run every ten samples.

CCB - Continuing Calibration Blank. The CCB is a sample boat filled approximately half way with muffled sand, no reagents are added. One CCB is run every ten samples.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of TOC in solids by the Lloyd Kahn Method. Each analyst must demonstrate and document their ability to generate acceptable results with this

method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of TOC in solids by the Lloyd Kahn method to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method have not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their department manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in adherence with the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

2.0 SUMMARY OF METHOD

Total Carbon (TC) is measured utilizing a carbonaceous analyzer with a boat sampling module and 900°C furnace attached. The resulting combustion converts carbon-to-carbon dioxide (CO_2) in the presence of oxygen. The amount of CO_2 derived from a sample is directly proportional to the concentration of carbonaceous material in the sample and is then measured by a non-dispersive infrared detector (NDIR).

To determine **Total Organic Carbon** (TOC), however, carbonate and bicarbonate ions contributing to the TC result must be accounted for. This is achieved by adding 1:1 hydrochloric acid to the sample and drying it at 103° C for 10 minutes to remove any **Inorganic Carbon** (IC) before analyzing the sample. The **Total Carbon** result then equals the **Total Organic Carbon.**

To determine Particulate Organic Carbon (POC), an aqueous sample is filtered through a pre-combusted / pre-cleaned glass fiber filter to obtain the particulates on the filter. The filter + particulates sample is then dried. The dried filter + particulates are then milled, ground, and homogenized prior to removal of an aliquot to place in the sampling boat, treated with HCl to remove IC (using the same procedure as for TOC) and placed into the carbonaceous analyzer for combustion at 900ºC. Procedures for POC are detailed in Attachment A of this TOC SOP.

3.0 INTERFERENCES

Volatile organics in the sediment may be lost in the decarbonation step resulting in a low bias.

4.0 APPARATUS AND MATERIALS

4.1 Shimadzu model TOC-Vcph with NDIR.

4.2 SSM-5000A 900ºC furnace with boat sampling module.

4.3 Mettler AE 100 balance (accurate to 0.1 mg) or equivalent.

4.4 Ceramic boats.

4.5 Drying oven capable of maintaining 103-105°C

4.6 Oxygen gas

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

5.0 REAGENTS AND STANDARDS

5.1 TOC Calibration / CCV Standard: D(+) Glucose Reagent ACS Anhydrous, ACROS

5.1.1 Calibration / CCV Solution – Prepare using 25.0 g and bring up to 250 mL with DI water. Before reaching the 250 mL fill line, add 0.5 mL HCl for preservation. Stable for 3 months.

5.1.1.1 Using the table below, each calibration level is prepared by pipetting the appropriate amount of Calibration / CCV Solution into a ceramic boat filled with approximately 0.5 g of sand.

Calibration Level ug C	Weight (mg)	mL of stock
Blank	0	0
200	0.5	0.005
400	1.0	0.01
2000	5.0	0.05
4000	10	0.1
16000	40	0.4
24000	60	0.6

(Upper range limit for TOC is 24,000ugC)

5.1.1.2 The 16000 ug C level is also used as the CCV

5.2 LCS Stock Standard – Dextrose Anhydrous Powder – Prepare by weighing 5.0 and bringing to 50 mL with D.I. Water. Stable for three months.

5.3 TIC Calibration / CCV Standard Sodium Carbonate, anhydrous (11.3% Carbon by weight = 113,000 ug/gC)

Calibration Standards: weigh into ceramic boat.

0.0 mg - Calibration Blank
3.5 mg - 400ugC
17.7 mg - 2000ugC
35.3 mg - 4000ugC
70.7 mg - 8000ugC
212.0 mg - 24,000ugC

(Upper range limit for TIC is 25,000 ugC)

5.4 1:1 Hydrochloric acid / DI water solution

5.5 Sodium Hydrogen Carbonate (14.28% Carbon by weight = 142,857ug/gC) used as LCS run at 4000ugC level (28 mg).

5.6 Muffled Sand – Play sand muffled at 900 ºC for at least 4 hours.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Collect sediments in a glass jar with Teflon or aluminum foil. Cool and maintain at 4° (±2) C. Analyze within 14 days for Lloyd Kahn and 28 days for SW846 9060.

7.0 PROCEDURES

SET UP AND CALIBRATION

7.1. Turn on TOC-Vcph analyzer, SSM-5000Afurnace, and oxygen supply and connect to the TOC software program.

7.2. Start the TOC-V software program by double clicking the TOC-V icon on the desktop and selecting sample table editor. Enter user name, passwords and press **OK**. Click the “new” icon followed by “sample run”. Choose SSM-5000A from the pull down menu. This activates a sample field spreadsheet in which calibrations, controls, and samples can be inserted and run. Click the “connect” icon and choose “Settings on PC”. This will start the gas flows through both units and will activate the NDIR. Wait for the TC furnace to read 900ºC before beginning analysis. The gauges on the soil module for the gas lines should read 150psi and 200psi from right to left. Also place any sample boats that will be used in the furnace for several minutes to bake off any remaining residue.

7.3. To run a calibration, minimize the sample run. Click the “New” icon followed by “calibration curve”. This activates the calibration curve wizard.

7.4. Choose SSM-5000A next to system by using the pull down menu, the click **NEXT** twice. Type in the file name specifying that it is a curve in the name (i.e toccurve030510), and then click **NEXT**. Using the pull down menu select ug as the units and click **NEXT**.

7.5. A 6-point curve (for either TC or IC) must be run at least every 3 months to verify the calibration. The calibration may also be updated as necessary as demonstrated by failure of the Laboratory Control Sample (LCS) or Continuing Calibration Verification (CCV). To add the points to the curve select “(1)” then click **ADD**. In the highlighted **Conc:** box type in the concentration of the first point (24000), and then press **OK**. Click on the next point “(2)” and **ADD**. Continue with previous steps until all 6-points have been entered. Click **NEXT** twice and then **FINISH**. Now the curve needs to be run.

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

7.6. Maximize the sample run and highlight the first row. Select insert then calibration curve from the top menu. Select the calibration curve that was just saved and click **OK**. The calibration curve will be renamed with the date and time (i.e toccal030510, note: name must include the word curve or cal, whichever was not used in the original file name) when the start button is clicked.

7.7. Once all the boxes on the top of the sample run say “Ready”, click the start icon (traffic light) on the menu bar. Once the calibration has been started the instrument will prompt two windows.

7.8. The first will state which point will be running, verify the point and press **OK**. The second window will ask for the exact amount of dextrose that has been weighed out. Enter the amount and click OK. For the last point, the blank point, an empty sample boat is used and 500mg is used for the weight.

7.9. Once entered click **OK** and the instrument will prompt a message to move the sample boat to the measure position.

7.10. Open the TC sample port and insert the boat. Close the port tight and set timer for two (2) minutes. These two minutes ensures that the CO2 that entered the instrument has had enough time to go completely through the detector and not give any false positives. When the timer goes off slide the sample boat to the measurement position.

7.11. Once the instrument is done analyzing the sample, about 4-5 minutes, there will be a message prompt to slide the sample boat to the cooling position. After a few minutes it will then prompt again to move the sample boat back in to the port. As soon as the boat has been pulled all the way back the instrument will automatically start the process for the next point. Repeat steps 7.8 to 7.10 for the rest of the calibration.

7.12. When the entire curve is complete, print the sample report and check the curve and its linearity. The correlation coefficient must be greater than or equal to 0.995. The value of the calibration checks must fall within the control limits (80-120% recovery). If not rerun the sample up to two more times. If the calibration check is still out of the acceptable recovery range, recalibrate the instrument and repeat the procedure. If problem persists, remake the standards and repeat the procedure or perform instrument maintenance. If maintenance is required, record in TOC instrument maintenance notebook.

7.13. Once there is a valid calibration curve, the curve must be saved to the method. Click file, then open. Select method from the pull down menu and select tc method soils. Click the SSM-TC tab and change the calibration to the new one. Click OK. Samples are now ready to run. Select “new” file icon on the menu bar and, then click on sample run. Highlight the first row on the sample run. Click the insert sample icon. The parameter box opens, click the method box and select tc method soils. Click next and enter the sample name. Click next twice then finish.

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7.14. Using a calibrated analytical balance weigh out 100-500 mg, wet weight, of the sample into a tarred ceramic boat. Using a disposable pipet add 1:1 hydrochloric acid to the sample until there is no more effervescing, usually 2-4 drops. If a sample concentration is greater than 24000 ug/g, a smaller sample amount must be used to achieve a concentration that is within the curve. Samples that require less than 50 mg must be analyzed in quadruplicate and the standard deviations calculated.

For Method Blanks, LCSs and CCVs, 1:1 hydrochloric acid is not needed.

For TC or Percent TC analysis, 1:1 hydrochloric acid is not needed.

NOTE: Please refer to Katahdin Analytical Services SOP CA-108, “Basic Laboratory Technique”, current revision, for more information on subsampling

NOTE: Samples analyzed by method SW846 9060 must be analyzed in quadruplicate.

7.15. Dry the samples, Method Blanks, LCSs and CCVs in a 103° - 105° C oven for ten minutes. This process removes the inorganic carbon and residual moisture from the samples.

7.16. To start the analysis, click the start icon. The first sample entered will bring up a file name box. Name the file by analysis and date, click OK. Then the box to enter the weight will open. Enter weight and press OK. Follow steps 7.10 and 7.11 with the exception that each sample is now entered separately throughout the batch.

7.17. Calculate the TC or IC (whichever is being preformed) concentration using the following equation:

7.18.

$$\frac{\text{Abs C value (instrument reading) in ug}}{\text{Sample Weight (g)}} \times \frac{100}{\%TS} = \text{TC or IC result in ug/g C}$$

7.18. Workgroup samples and get run ID. Enter true values for the LCS and MS and save. Go back to the spreadsheet and enter “LLOYD”, “Percent”, or “TOC” in the comments section for the samples you wish to report. Change QC to match workgroup. Data is then exported by selecting the ASCII export option from the file menu. Select “save as” and choose parsefiles on LVSlims. Select TOC and type file name. Click save and wait for data to export, then review data in wetrev.\

7.19. A batch sheet is generated (Figure 4). Raw data, calibrations, and batch sheets are reviewed for completeness and accuracy by the Wet Chemistry supervisor or other qualified designee.

7.20. Analysts file printouts of instrument calibrations and sample data in the lab for approximately 3 months for reference. Prior calibrations are archived and all are available for retrieval.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

See below and refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed below and in Table 1, all associated samples must be evaluated against all the QC. In some cases, data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases, “qualified” data with narration may be advisable after consultation with the client.

In some cases, the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases, the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 A Method Blank is analyzed at the beginning of the run and a CCB is analyzed every ten samples thereafter and at the end of the run. The Method Blank and CCBs are ceramic boats filled approximately halfway with muffled sand. Results must not be greater than the reporting limit (PQL). The PQL for this test is 400mg/kg.

8.2 Analyze an LCS (40-50 mg of Dextrose = 16000-20000ugC for TC) / (28 mg Sodium Hydogen Carbonate = 4000 ug C for IC) with each batch of 20 samples. Acceptance criteria is 80-120% of expected value.

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8.3 Run a CCV (40-50 mg Dextrose = 16000-20000 ug C for TC) / (35.3 mg Hydrogen Carbonate = 4000 ug C for IC) every 10 samples and at the end of each batch. Acceptance criteria is 80-120% of expected value.

8.4 Run a duplicate every 20 samples. Run a matrix spike every 10 samples by weighing out the sample and adding 10 mg of dextrose to it for the TC spike or 35.3 mg of hydrogen carbonate for the IC spike. Run a matrix spike duplicate every 10 samples for SW 9060 samples. The recovery can be determined by calculating the theoretical yield from the sample result based on the weight as compared to the native result and adding 4000 ug C that was added from the spike component. The actual yield divided by the theoretical yield will give the recovery.

8.5 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

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Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of EPA SW846 9060 and the Lloyd Kahn method for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Determination of Total Organic Carbon in Sediment, Lloyd Kahn, USEPA Region II, 7/88.

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Update III, December 1996, Method 9060.
TOC-V series SSM-5000A user's manual.

Installation and Operation of Shimadzu's Solid Sample Module.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM, curent Version.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision

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TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method blank	One per prep batch	No analyte detected >PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS/ICV	One per prep batch of twenty samples (Lloyd Kahn); one per fifteen samples (SW 9060)	80-120% recovery	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.
CCV	CCV at the beginning of the analysis and one after every 10 samples: same conc. as LCS/ICV	80-120% recovery	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reanalyze all samples back to last acceptable CCV recovery
Sample Quadruplicate	One every twenty samples. SW 9060 – all samples are analyzed in quadruplicate	≤ 30% RSD	(1) If lab QC in criteria and matrix interference suspected, flag data (2) Else, reanalyze
Matrix spike/Matrix spike duplicate	One MS per ten samples (MSD required every 10 samples for SW 9060)	75-125% Recovery ≤ 30% RPD	(1) If LCS in criteria and matrix interference suspected, flag data (2) Else, reanalyze
Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file
MDL and-or LOD/LOQ Verification study	Refer to KAS SOP QA-806, “Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications”, current revision.		

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TABLE 2

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-741-09	METHOD LLOYD KAHN/SW846 9060
Apparatus/Materials		
Reagents	Dextrose solid for TC calibration (Dextrose for LCS) Sodium Carbonate for IC calibration (Sodium Hydrogen Carbonate for LCS)	Potassium Hydrogen Phthalate solution used for calibration.
Sample preservation/ handling		
Procedures	Report one of the duplicate samples using an RPD of 30%.	Report average and range of quadruplicate analysis (SW 9060) Use 3 standard deviation limit (Lloyd Kahn)
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

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FIGURE 1

EXAMPLE OF TOC SOLIDS LOGBOOK PAGE

R487211
WG234890

Katahdin Analytical Services

Carbon Analysis of Solid Samples - Shimadzu TOC-V_{CPH} / SSM-5000A

Analysis Type and Method (Check One)	
____Total Carbon (SW846 9060M)	____Total Inorganic Carbon (SW846 9060M)
____Total Organic Carbon (SW846 9060)	____Other (Specify):
✓ Total Organic Carbon (Lloyd Kahn)	____Percent Carbon (SW846 9060M)

Spiking Information			Calibration Information	
LCS Spike Source ID / Compound:	WI6776	TV: 16000 ug	Calibration Date:	8/18/18
CCV Spike Source ID / Compound:	WI6817	TV: "	Calibration Analyst:	SC
MS Spike Source ID / Compound:	WI6776	TV: 4000 ug	Calibration Source ID:	WI6817
Acid Lot Number:	WI6673		Pipette IDs:	47, 48

Katahdin Sample Number	Sample Wt. (mg)	Sample Type * (Circle One)		% Recovery
CCV	40	(Wet)	Dry	98%
CCB	500	(Wet)	Dry	
LCS	40	(Wet)	Dry	106%
LCSD	40	(Wet)	Dry	108%
SL7696-28	242.7	Wet	(Dry)	
-33	240.7	Wet	(Dry)	
-34	258.8	Wet	(Dry)	
-35	258.0	Wet	(Dry)	
SL7786-1	279.7	Wet	(Dry)	
-2	258.8	Wet	(Dry)	
-3	265.5	Wet	(Dry)	
-4	328.6	Wet	(Dry)	
CCV	40	(Wet)	Dry	103%
CCB	500	(Wet)	Dry	
SL7786-5	301.0	Wet	(Dry)	
-6	283.0	Wet	(Dry)	
-7	272.0	Wet	(Dry)	
-8	273.8	Wet	(Dry)	
-9	250.2	Wet	(Dry)	
CCV	40	(Wet)	Dry	103%
CCB	500	(Wet)	Dry	

* "Wet" = field-moist sample (as received). "Dry" = oven-dried sample. "TV" = True Value

Analyst: ZF	Analysis Date: 8/19/18
Reviewer:	Review Date:

WL-019- Revision 4 – 06/10/2017 QAWL902 0000017

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FIGURE 2

EXAMPLE OF TOC SOLIDS INSTRUMENT PRINTOUT

	Type	Analysis	Sample Name	Dilution	Result	Comment	Status	Date / Time
1	Unknown	SSM-TC	CCV	1.00	SSM-TC:15907 ug		Completed	01/23/13 09:48:06 A
2	Unknown	SSM-TC	CCB	1.00	SSM-TC:0.000 ug		Completed	01/23/13 09:55:01 A
3	Unknown	SSM-TC	LCS	1.00	SSM-TC:15615 ug		Completed	01/23/13 10:10:11 A
4	Unknown	SSM-TC	LCSD	1.00	SSM-TC:15254 ug		Completed	01/23/13 10:18:37 A
5	Unknown	SSM-TC	SG0288-1	1.00	SSM-TC:7567 ug		Completed	01/23/13 10:30:11 A
6	Unknown	SSM-TC	SG0421-1	1.00	SSM-TC:10697 ug		Completed	01/23/13 10:38:56 A
7	Unknown	SSM-TC	SG0421-2	1.00	SSM-TC:8713 ug		Completed	01/23/13 11:49:06 A
8	Unknown	SSM-TC	SG0365-1	1.00	SSM-TC:6723 ug		Completed	01/23/13 12:07:23 P
9	Unknown	SSM-TC	SG0365-1	1.00	SSM-TC:6614 ug		Completed	01/23/13 12:18:03 P
10	Unknown	SSM-TC	SG0365-6	1.00	SSM-TC:4359 ug		Completed	01/23/13 12:27:49 P
11	Unknown	SSM-TC	SG0365-6	1.00	SSM-TC:4475 ug		Completed	01/23/13 12:43:35 P
12	Unknown	SSM-TC	SG0443-1	1.00	SSM-TC:13287 ug		Completed	01/23/13 12:55:28 P
13	Unknown	SSM-TC	CCV	1.00	SSM-TC:15752 ug		Completed	01/23/13 01:07:03 P
14	Unknown	SSM-TC	CCB	1.00	SSM-TC:0.000 ug		Completed	01/23/13 01:14:02 P
15	Unknown	SSM-TC	SG0365-11	1.00	SSM-TC:0.000 ug		Completed	01/23/13 01:31:39 P
16	Unknown	SSM-TC	SG0365-11	1.00	SSM-TC:265.1 ug		Completed	01/23/13 01:43:54 P
17	Unknown	SSM-TC	SG0365-15	1.00	SSM-TC:6585 ug		Completed	01/23/13 01:52:34 P
18	Unknown	SSM-TC	SG0365-15	1.00	SSM-TC:7112 ug		Completed	01/23/13 02:05:11 P
19	Unknown	SSM-TC	SG0365-20	1.00	SSM-TC:7023 ug		Completed	01/23/13 02:19:50 P
20	Unknown	SSM-TC	SG0365-20	1.00	SSM-TC:6512 ug		Completed	01/23/13 02:29:21 P
21	Unknown	SSM-TC	SG0365-20MS	1.00	SSM-TC:11013 ug		Completed	01/23/13 02:41:57 P
22	Unknown	SSM-TC	SG0365-20MS	1.00	SSM-TC:9743 ug		Completed	01/23/13 02:54:30 P
23	Unknown	SSM-TC	SG0473-1	1.00	SSM-TC:105.2 ug		Completed	01/23/13 03:01:58 P
24	Unknown	SSM-TC	SG0473-1MS	1.00	SSM-TC:4586 ug		Completed	01/23/13 03:16:10 P
25	Unknown	SSM-TC	CCV	1.00	SSM-TC:14992 ug		Completed	01/23/13 03:26:56 P
26	Unknown	SSM-TC	CCB	1.00	SSM-TC:0.000 ug		Completed	01/23/13 03:35:06 P
27	Unknown	SSM-TC	SF0473-1MSD	1.00	SSM-TC:5927 ug		Completed	01/23/13 03:48:30 P
28	Unknown	SSM-TC	SG0488-2	1.00	SSM-TC:5709 ug		Completed	01/23/13 03:58:13 P
29	Unknown	SSM-TC	CCV	1.00	SSM-TC:15477 ug		Completed	01/23/13 04:13:04 P
30	Unknown	SSM-TC	CCB	1.00	SSM-TC:0.000 ug		Completed	01/23/13 04:21:43 P

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FIGURE 3

EXAMPLE OF TOC SOIL INSTRUMENT RESULTS PRINTOUT

KP 01/23/13 04:21:59 PM TOCSOIL012313.132

Instr.Information

System	SSM-5000A
Detector	Combustion
Catalyst	Regular Sensitivity
Cell Length	short

Sample

Sample Name:	CCV
Sample ID:	<Untitled>
Origin:	tc method SOILS.met
Chk. Result	

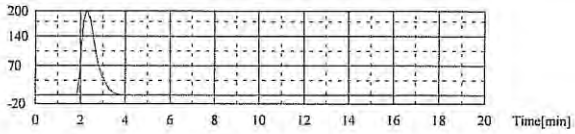
Type	Anal.	Dil.	Density	Result
Unknown	SSM-TC	1.000	1.000mg/uL	SSM-TC:15907 ug

1. Det

Anal.: SSM-TC

No.	Area	CNV	Abs C	Conc.	Weight	Volume	Ex.	Cal. Curve	Date / Time
1	891.1	891.1	15907ug	15907ug	42.20mg	42uL		tocsoilcurve120512.2012_12_05_14_36_50.cal	01/23/13 09:48:06 AM

Mean Area	891.1
Mean CNV	891.1
Mean Conc.	15907ug



Sample

Sample Name:	CCB
Sample ID:	<Untitled>
Origin:	tc method SOILS.met
Chk. Result	

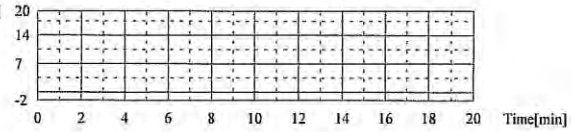
Type	Anal.	Dil.	Density	Result
Unknown	SSM-TC	1.000	1.000mg/uL	SSM-TC:0.000 ug

1. Det

Anal.: SSM-TC

No.	Area	CNV	Abs C	Conc.	Weight	Volume	Ex.	Cal. Curve	Date / Time
1	0.000	0.000	0.000ug	0.000ug	500.0mg	500uL		tocsoilcurve120512.2012_12_05_14_36_50.cal	01/23/13 09:55:01 AM

Mean Area	0.000
Mean CNV	0.000
Mean Conc.	0.000ug



1/14

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

FIGURE 4

EXAMPLE OF TOC SOLIDS BATCH SHEET

WET CHEMISTRY BATCH REPORT
Jan 31 2013, 05:09 am
Batch: WG119373

Parameter: TOC In Soil
Prep Date: N/A

Date Analyzed: 23-JAN-13
Prep Method: N/A

Analyst Initials: KP
Prep Chemist: N/A

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SG0443-1	SAMP	SW846 M9060	141.9mg	200.00ug	1	269215.35593	290000 ug/gdrywt	32.	400	260	1200		
SG0473-1	SAMP	SW846 M9060	315.4mg	200.00ug	1	414.9495	U500 ug/gdrywt	80.	400	100	500		
WG119373-1	MBLANK	SW846 M9060	500mg	200.00ug	1	0	U300 ug/gdrywt	NA	400	85.	400		
WG119373-2	LCS	SW846 M9060	42mg	200.00ug	1	371785.71428	370000 ug/gdrywt	NA	400	85.	400		93
WG119373-3	LCSD	SW846 M9060	40.6mg	200.00ug	1	375714.28571	380000 ug/gdrywt	NA	400	85.	400	1	94
WG119373-4	MS	SW846 M9060	264.9mg	200.00ug	1	21537.40051	22000 ug/gdrywt	NA	400	100	500		132
WG119373-5	MSD	SW846 M9060	282.7mg	200.00ug	1	26082.56576	26000 ug/gdrywt	NA	400	100	500		116

Comments:

SG0473-1	MS/MSD; RE103D3-SOIL-011413-725-727
WG119373-1	SG0473-1
WG119373-2	SG0473-1
WG119373-3	SG0473-1
WG119373-4	SG0473-1
WG119373-5	SG0473-1

Entered by: [signature] Date: 1/31/13 Accepted by: [signature] Date: 01/31/13

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

ATTACHMENT A

PARTICULATE ORGANIC CARBON (POC) PREPARATION AND CALCULATION

The following are the additional apparatus, materials, reagents, sample handling, procedures and quality control (in addition to those previously listed for TOC) required for preparing aqueous samples for particulate organic carbon.

4.0 APPARATUS AND MATERIALS

4.1 Oven - capable of maintaining a temperature of 104°C.

4.2 Filter Holder - Filter holders for pressure filtration are used. They are constructed of type 316 stainless steel (with or without PTFE linings) and are capable of sustaining internal pressures exceeding 50 psi. These devices have an internal capacity of 1.5 L and accommodate glass fiber filters 142 mm in diameter.

4.3 Glass fiber filter discs without organic binders, 142 mm diameter, nominal pore size 0.7 um. Pre-combusted / pre-cleaned.

4.4 Analytical balance - capable of weighing to 0.1 mg; balance must be calibrated in accordance with Katahdin SOP, CA-102, Balance Calibration, before each measurement. Weights used are 100g, 50g, 5g, 2g, 0.2g

4.5 Spex Certiprep 8000 Mixer/Mill or equivalent (Ceramic Ball Mixer)

4.6 Desiccator with conditioned indicating desiccant (desiccant is conditioned by drying at 210°C for one hour)

4.7 Graduated cylinder – 1000 mLs.

4.8 Aluminum tins to hold filters for drying

5.0 REAGENTS AND STANDARDS

5.1 Analyte-free water: Water that has been treated to remove organic carbon

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

For this POC method, holding time for aqueous samples has been established as 48 hours from time sampled to time filtered. The analytical hold time of the prepared (dried/homogenized) particulate sample is 28 days from date prepared to date analyzed.

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

This holding time can be extended to one year by freezing the prepared/dried POC sample at < 10°C. Holding time is arrested as long as the sample is maintained frozen.

Note: A project may require analysis of Total Suspended Solids (TSS) and Dissolved Organic Carbon (DOC) on the same parent sample as for POC. In such cases, TSS is analyzed first (using pre-combusted / pre-cleaned filter), then the filter + particulates generated during TSS are prepared and analyzed using the procedures in this SOP Attachment A. The Filtrate is acidified and used for DOC analysis.

7.0 PROCEDURES

7.1 Filter Preparation

7.1.1 Pre-combust filters at 500 ºC

7.1.2 Cool combusted filters to room temperature, in a desiccator

7.1.3 The filters are then weighed to 0.0001g. Record the dried filter weight on a small piece of paper. The filters are placed back into their original box with the weight placed on top of the filter. The box of filters must be stored in a desiccator until the time of use

7.2 Assembly filter apparatus

7.3 Place the filter on the support screen and secure.
7.4 Record the weight of the prepared filter in the Special Project TSS logbook

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

7.5 Measure the volume of sample using a graduated cylinder. Typically, the sample volume will be 4L, 1L or 500mL. For POC analysis on aqueous samples, note that for some projects, the entire sample bottle volume must be filtered.

7.6 Quantitatively transfer the sample to the filter holder.

7.7 Gradually apply gentle pressure of 10-30 psi until sample moves through the filter. Collect the filtrate in a 2L polyethylene container.

7.8 When the pressurizing gas begins to move through the filter, or when the liquid flow has decreased at 30 psi (i.e., filtration does not result in any additional filtrate within a 2 minute period), stop the filtration. If the project requires the entire sample volume to be used for POC analysis, multiple filters may need to be used to filter the entire sample.

7.10 Rinse the filter apparatus, graduated cylinder and filter 3 times with 50 mL aliquots of filtrate. If DOC is to be measured on the filtrate, fill 3 40 mL VOA vials preserved with H_2SO_4. This MUST be done before any rinses with analyte free water.

7.11 Rinse the filter 3 times with 100 mL aliquots of analyte-free water (this step is especially important for saline samples, where salts may be retained on the filter and affect the final filter weights).

7.12 <u>The material retained on the filter is defined as the POC.</u>

7.13 Filters are placed in an aluminum weighing dish and dried overnight at 104 ºC.

7.14 Cool the filters in a desiccator to room temperature and weigh. Repeat cycle of heating, cooling, and weighing until a constant weight is obtained or until the difference between successive weighings is 0.5mg or 4%, whichever is less. Record the weight in the Special Project TSS logbook.

7.15 Using a ceramic ball mill, grind filter and the POC retained on the filter. Homogenize sample.

7.16 Remove an approximately 0.5g aliquot of the dried, pulverized, homogenized sample and place in sample boat, treat with several drops of HCl solution to remove inorganic carbon (IC) and analyze for organic carbon as for the Lloyd Kahn Method (see TOC method procedure for analysis details).

7.17 Calculations:

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

$$\frac{\mu g\ C}{\text{Analysis Aliquot (mg)}} * \frac{\text{Total wt mg (Sample + Residue)}}{\text{Residue wt (mg)}} * \frac{\text{Residue wt (g)}}{\text{Vol. Filtered (L)}} = \text{POC } \mu g/L$$

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

8.1 A filter Blank is prepared daily for each batch of ≤ 20 samples filtered using 1 L of analyte-free water, or project-specified volume of analyte-free water, and processed in the same manner as the samples. Filter blank result should be non-detect at the sample QL (QL dependent on sample volume).

8.2 A Certified Reference Material (CRM) should be analyzed with the POC at a frequency of 1 per batch of ≤ 20 samples. Acceptance limits are ± 30% of certified value.

8.3 Additional method QC as per TOC method including Method Blank, LCS, MS/MSD or MS/LR, and CCV with same acceptance criteria as TOC (see Table 1 of TOC SOP). Quadruplicate analyses not required for POC. MS/MSD or MS/Lab replicate (LR) frequency 1 per 20 samples.

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Zachary Fuller

Review Date: 1/21/20

SOP Number: CA-741-09

SOP Title: Determination of Total Organic Carbon in Solids using the EPA Region II Method LLOYD KAHN & SW9060 MOD.

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: Z. Fuller **Date:** 01/21/21

QAO Signature: Leslie Dimond **Date:** 01/25/21

TITLE: TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) BY EPA METHOD 160.2 AND STANDARD METHODS 2540 D

Prepared By: Betsy Carbone Date: 12/97

Approved By:

Group Supervisor: Keith Tarquay Date:

Operations Manager: Jol C. Burton Date: 1/24/01

QA Officer: Deborah J. Nadeau Date: 1·22·01

General Manager: Duncan F. Hughes Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, added pollution prevention, added SM 2540D. Minor changes to sections 7+8 and Tables.	DN	1·22·01	1·22·01
02	Changed Title to reflect referenced methods. Added LIMS data entry. added new logbook. Revised text for clarity. Added wording to Sect. 8	LAD	031605	031605
03	added definitions to section 1.	LAD	02/08	02/08
04	Added 1L sample volume required for lower PQL. Corrected balance requirement typo. Added method modification to Table 2.	LAD	06/08	06/08
05	Added requirement to record oven and thermometer IDS in logbook. Added references to section 10.	DN	08/09	08/09

TITLE: TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) BY EPA METHOD 160.2 AND STANDARD METHODS 2540 D

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Updated or added references to section 10 and Table 1.	LAD	06/10	06/10
07	Sect. 7 - Added pre-weighed glass filters. Also, data archived. Sect. 8 - Updated balance Calibration and LCS acceptance criteria. Also, increased M. Bek. from 250mL to 1000 mL. Sect. 10 - Added and edited references, Updated Fig. 1 & 2.	LAD	05/12	05/12
08	Sect. 7 - Added requirement to "Seed" the filter with a small amount of DI H_2O. Sect. 8 and Table 1 - Changed duplicate acceptance Criteria to RPD ≤ 5%	LAD	12/17	12/17
09	Sect. 4.6 & 8.1 - Changed weights used from 5, 2, 0.2 to 150, 100, 50, 10, 1, 0.1 & 0.0029. Table 2 - changed DUP RPD Limit from +/- 5% to +/- 10%, changed DUP CA from 20% to 10 % Updated NELAC TNI references.	LAD	06/20	06/20

TITLE: TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) BY EPA METHOD 160.2 AND STANDARD METHODS 2540 D

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-720-09, titled TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) BY EPA METHOD 160.2 AND STANDARD METHODS 2540 D.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-720-09, titled TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) BY EPA METHOD 160.2 AND STANDARD METHODS 2540 D.

Recipient: _____ Date: _____

TITLE: TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) BY EPA METHOD 160.2 AND STANDARD METHODS 2540 D

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure used by Katahdin Analytical Services technical personnel to measure the concentration of total suspended solids (TSS) found in drinking and surface waters and domestic and industrial wastes as described in EPA Method 160.2 and SM 2540 D.

The procedure is applicable to measurement of TSS to a Practical Quantitation Limit (PQL) of 4 mg/L when 250 mL of sample is used for analysis.

1.1 Definitions

Total Suspended Solids – the portion of total solids retained by a standard glass fiber filter.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

LCS - Laboratory Control Sample - A standard or solid reference material of known value that has been brought through the sample preparation an analysis process. The LCS is used to assess the accuracy of the method.

MB – Method Blank - Reagent water that has been brought through the sample preparation and analysis process. The MB is used to assess contamination.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the MDL.

MDL - Method Detection Limit - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in the analysis of Total Suspended Solids by EPA Method 160.2 and SM 2540 D. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of TSS by 160.2 and 2540 D to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in

the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, “Sample Disposal,” current revision.

2.0 SUMMARY OF METHOD

A well-mixed sample is filtered through a glass fiber filter, and the residue retained on the filter is dried to constant weight at 103-105°C. The filtrate from this procedure may be used to determine total dissolved solids (filterable residue). See Katahdin SOP CA-719, current revision.

TITLE: TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) BY EPA METHOD 160.2 AND STANDARD METHODS 2540 D

3.0 INTERFERENCES

Filtration apparatus, filter material, pre-washing, post-washing, and drying temperature are specified because these variables have been shown to affect the results.

Samples high in total dissolved solids (filterable residue), such as saline waters, brines, and some wastes, may be subject to a positive interference from entrained dissolved solids. Washing of the filter to remove any dissolved solids entrained in the filter and suspended solids minimizes this potential interference.

Because excessive residue on the filter may form a water-entrapping crust, limit the sample size to that yielding no more than 200 mg of residue.

4.0 APPARATUS AND MATERIALS

4.1 Vacuum pump - capable of generating vacuum at 20-25 psi.

4.2 Oven - capable of maintaining a temperature between 98-105°C.

4.3 Glass fiber filter discs without organic binders, 47 mm diameter, nominal pore size 2.0 um or less. Gelman type A/E, Millipore type AP40, Whatman type 934-AH, or equivalent are acceptable.

4.4 Plastic filter funnel with filter support grid and magnetic closure, 300 mL capacity.

4.5 Filter flask with side arm, 500 mL capacity.

4.6 Analytical balance - capable of weighing to 0.1 mg; balance must be calibrated in accordance with Katahdin SOP, CA-102, Balance Calibration, before each measurement. Weights used are 150, 100, 50, 10 g, 1.0 g, 0.10, and 0.0020g and readings should be recorded in the TSS Logbook (Figure 1).

4.7 Desiccator with conditioned indicating desiccant (desiccant is conditioned by drying at 210°C for one hour)

4.8 Graduated cylinders – 10, 25, 50, 100, 250 mLs.

4.9 Aluminum tins to hold filters for drying

TITLE: TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) BY EPA METHOD 160.2 AND STANDARD METHODS 2540 D

5.0 REAGENTS AND STANDARDS

5.1 Laboratory reagent grade water: Water, which has been treated by the Culligan Water System and the Millipore Mill-Q™ Water System.

5.2 Laboratory Control Sample (LCS) solution - The LCS is a purchased non-filterable residue standard that is prepared according to the manufacturer's directions. The prepared standard has a certified reference value that is calculated for each standard from the measured weight. The LCS solution must be shaken before each use to suspend settled particulates.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples are collected in plastic or glass sample containers and to minimize microbiological decomposition of solids, stored at 4 (±2) °C until time of analysis

The holding time for this test is 7 days from the date of sampling. To obtain a PQL of 4 mg/L, 250 mL of sample is required.

7.0 PROCEDURES

7.1 Prepare glass filters as follows. If using pre-weighed glass filters disregard this section. Place the glass fiber filter on the filter funnel with the wrinkled side up. While vacuum is applied, wash the filter with three successive 20 mL volumes of laboratory reagent grade water. Remove all traces of water by continuing to apply vacuum after water has passed through. Remove filter and dry in oven at 103-105°C for one hour. After drying, place in desiccator until needed. Weigh the filter immediately before use. Record initial filter weight in TSS Logbook (Figure 1). Handle filter with forceps only.

7.2 Assemble filtering apparatus with a prepared, preweighed filter. Seat the filter with a small amount of DI water.

7.3 Shake sample vigorously and quantitatively transfer 250 mL of sample, or another measured aliquot less than 250 mL, to the filter funnel using a graduated cylinder.

7.4 Begin suction. Remove all traces of water by continuing to apply vacuum after sample has passed through. Record the initial sample volume in TSS Logbook.

Note: For clients who require a PQL of 1.0 mg/L, 1L of sample must be filtered.

TITLE: TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) BY EPA METHOD 160.2 AND STANDARD METHODS 2540 D

Note: If the measured volume does not filter completely, the sample must be discarded and reanalyzed with a fresh filter. For these cases, it is advisable to prepare several extra filters ahead of time and store them in the desiccator.

Note: Since excessive residue may form a water trapping crust, samples in excess of 200 mg of residue must be rerun at a dilution.

7.5 With suction on, wash graduated cylinder, filter, non-filterable residue and filter funnel three times with approximate 10-mL volumes of laboratory reagent grade water, allowing complete draining between washings.

Note: If the sample filtrate is to be analyzed for total dissolved solids (TDS), remove the TDS aliquot from the filter flask before rinsing the filter.

7.6 Remove filter from filter funnel, and place in an aluminum tin. Place in the oven at 103-105°C for at least one hour. Record initial and final times and temperatures in TSS Logbook. Record the oven ID and thermometer ID in the TSS logbook.

7.7 Cool in desiccator to room temperature and weigh. Repeat cycle of heating, cooling, and weighing until a constant weight is obtained or until the difference between successive weighings is 0.5 mg or 4%, whichever is less. For each sample, method blank, and LCS, record the final two weights in the TSS logbook.

7.8 The initial sample volume and initial and final sample weights are entered manually into the Katahdin Information Management System (KIMS) for calculation and reporting. After the data are entered, a batch sheet (Figure 2) is automatically printed by KIMS. Refer to the current revision of Katahdin SOP CA-732 ("Wet Chemistry Data Entry and Review Using Katahdin Information Management System") for further information.

The final concentration of TSS is calculated by the KIMS using the following formula:

$$\text{TSS (mg/L)} = \frac{[\text{Wt of filter + residue (g)}] - [\text{Wt of filter (g)}]}{\text{Sample Volume (mL)}} * \frac{1000\ \text{mg}}{\text{g}} * \frac{1000\ \text{mL}}{\text{L}}$$

7.9 After completion of each test, the TSS Logbook must be signed and dated by the person performing the test. All unused lines are to be "z-ed" out and initialed and dated.

7.10 Data Archival

All batch sheets, raw data, and supporting documents are scanned after final review and the resulting image files are saved on a Katahdin server for use in data package

assembly. Image files of raw data are periodically archived by the laboratory's MIS department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

See below and refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The Department Manager, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Due to the 7-day hold time associated with this method, samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be reported after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 The analytical balance must be calibrated in accordance with Katahdin SOP, CA-102, Balance Calibration, before each measurement. Weights used are 150, 100, 50, 10 g, 1.0 g, 0.10, and 0.0020 g and readings should be recorded in the TSS logbook.

Acceptance Criteria:

ASTM Class 1 Weight	Acceptance Criteria (g)
150 g	± 0.0005
100 g	± 0.0005
50 g	± 0.0005
10 g	± 0.0005
1 g	± 0.0005
0.1 g	± 0.0005
0.002 g	± 0.0002

8.2 A method blank, consisting of a blank filter with 1000 mL of laboratory reagent grade water filtered through it, must be run for every batch of 20 or fewer samples.

Acceptance Criteria: Measured TSS < Practical Quantitation Limit
Corrective Action for Non-Conformance: Evaluate sample results. Report results <PQL. Report results >10x blank contamination with narration. Reanalyze all other samples. If samples cannot be reanalyzed, a Corrective Action Report (CAR) must be initiated and data appropriately qualified.

8.3 A duplicate sample must be run for every 10 samples.

Calculate the mean (X) and relative percent difference (RPD) for every duplicate run as follows:

$$X = \frac{X_1 + X_2}{2}$$

$$RPD = \frac{X_1 - X_2}{X} \times 100$$

where: X_1 = non-filterable residue (TSS) measurement for sample
X_2 = non-filterable residue (TSS) measurement for sample duplicate

Acceptance Criteria: Relative Percent Difference (RPD) ≤5%.
Corrective Action for Non-Conformance: Reanalysis of sample in duplicate; if RPD is again >5%, report original result with the appropriate flag or narration.

8.4 One Laboratory Control Sample (LCS) must be prepared and analyzed with every sample batch of twenty or fewer samples.

Acceptance criteria: 80-120% recovery of the true value for USEPA Method 160.2. These nominal acceptance criteria may change dependent on the actual true value of the LCS. The acceptance criteria for the LCS for SM 2540 D are 80-120% recovery of the true value. If criteria are exceeded, samples must be evaluated and/or reanalyzed. If samples cannot be reanalyzed, a Corrective Action Report (CAR) must be initiated and data appropriately qualified.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

MDLs are filed with the Inorganic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revisions of USEPA Method 160.2 and SM 2540 D for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Method 160.2, Residue, Non-Filterable, "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, Revised March 1983.

American Public Health Association (APHA) (2012) Standard method for examination of water and wastewater, 22nd edn. APHA, AWWA, WPCF, Washington, Method Method 2540 D. Total Suspended Solids Dried at 103-105°C,

American Public Health Association (APHA) (2005) Standard method for examination of water and wastewater, 21st edn. APHA, AWWA, WPCF, Washington, Method Method 2540 D. Total Suspended Solids Dried at 103-105°C,

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM, current version.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF FIGURES & TABLES

TABLE 1 QC Requirements
TABLE 2 Summary of Method Modifications
FIGURE 1 Example of TSS Logbook Page
FIGURE 2 Example of TSS KIMS Batch Sheet

TITLE: TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) BY EPA METHOD 160.2 AND STANDARD METHODS 2540 D

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Total Suspended Solids, TSS (Non-Filterable Residue) / USEPA Method 160.2 and Standard Methods 2540 D	Method blank	One per prep batch of 20 or fewer samples	Measured TSS <PQL	1) Investigate source of contamination 2) Evaluate sample results. Report results <PQL. Report results >10x blank contamination with narration. 3) Reprep a blank and all remaining samples.
	LCS	One per prep batch of 20 or fewer samples	80-120% (EPA 160.2 and SM 2540D)	1) Investigate source of problem. 2) If the LCS recovery is high report sample results <PQL with narration. 3) Reprep a blank and all remaining samples.
	Sample duplicate	One duplicate every ten samples	RPD ≤ 10%	1) Investigate problem and reanalyze sample in duplicate 2) If RPD still >10%, report original result with notation.
	Initial Demonstration of Performance: Precision and accuracy study using 4 replicate analyses LCS	Initially once per analyst, then annually per analyst.	All recoveries within method QC acceptance limits	Investigate source of problem; rerun P & A study.
	MDL Study and/or LOD/LOQ Verification.	Refer to KAS SOP QA-806, “Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications”, current revision.		

TITLE: TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) BY EPA METHOD 160.2 AND STANDARD METHODS 2540 D

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-720-09	USEPA method 160.2 or SM 2540 D, current revisions
Apparatus/Materials	None	
Reagents	None	
Sample preservation/ handling	None.	
Procedures	7.1 ……Remove filter and dry in oven at 103-105°C for one hour. After drying, place in desiccator until needed. Weigh the filter immediately before use.	SM2540 D, 3.a. ……Repeat cycle of drying or igniting, cooling desiccating and weighing until a constant weight is obtained or until weight change is less than 4% of the previous weighing or 0.5 mg.
QC - Spikes	None	
QC - LCS	None	
QC - Accuracy/Precision	None.	
QC - MDL	None	

TITLE: TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) BY EPA METHOD 160.2 AND STANDARD METHODS 2540 D

FIGURE 1

EXAMPLE OF TSS LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES, LLC

TOTAL DISSOLVED SOLIDS/TOTAL SUSPENDED SOLIDS

Total Disolved Solids: EPA 160.1____ Total Dissolved Solids: SM 2540C____ PQL: 10mg/L

Total Suspended Solids: EPA 160.2____ Total Suspended Solids: SM 2540D____ PQL: 4mg/L

Aqueous Solids LCS Sand ID/Lot#		
ASTM Class Weights		
Balance ID:		
True WT (g)	Initial WT (g)	Final WT (g)
0.1000		
1.0000		
10.0000		
100.0000		

Notes:

Filter Lot #		
Oven Temperatures (C)		
TDS: 180 +/- 2°C, TSS: 103 - 105 °C		
Oven ID:		
In Temp.	Date:	Time:
Out Temp.	Date:	Time:
In Temp.	Date:	Time:
Out Temp.	Date:	Time:
In Temp.	Date:	Time:
Out Temp.	Date:	Time:

KAS Sample ID	Sample Volume (ml)	Time of Initial Aliquot	Filter or Dish ID	Filter or Dish Wt. (g)	Dry 1 Dish/Filter + Sample Residue Wt (g)	Dry 2 Dish/Filter + Sample Residue Wt. (g)	Time of Final Weighing (Analysis)
Blank							
LCS							

Analyst:	Date:
Checked By:	Date:

WL-012 - Revision 6 - 01/25/2016

TITLE: TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) BY EPA METHOD 160.2 AND STANDARD METHODS 2540 D

FIGURE 2

EXAMPLE OF KIMS TSS BATCH SHEET

WET CHEMISTRY BATCH REPORT
Apr 26 2012, 04:05 pm
Batch: WG107423

Parameter: Solids-Nonfilterable Residue
Prep Date: 25-APR-12

Date Analyzed: 26-APR-12
Prep Method: SM 2540D

Analyst Initials: CBT
Prep Chemist: CBT

Sample	Samp Type	Method	Tare Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SF2243-3	SAMP	SM 2540D	0.1175000g	0.1191000g	1	6.4	6.4 mg/L	NA	4	1.22	4.0		
SF2245-1	SAMP	SM 2540D	0.1198000g	0.1401000g	2.2	176.52174	180 mg/L	NA	4	1.22	8.8		
SF2245-2	SAMP	SM 2540D	0.1179000g	0.1368000g	1	75.6	76. mg/L	NA	4	1.22	4.0		
SF2245-3	SAMP	SM 2540D	0.1209000g	0.1244000g	1	14	14. mg/L	NA	4	1.22	4.0		
SF2245-4	SAMP	SM 2540D	0.1181000g	0.1328000g	2.5	147	150 mg/L	NA	4	1.22	10.		
SF2245-5	SAMP	SM 2540D	0.1165000g	0.1313000g	1	59.2	59. mg/L	NA	4	1.22	4.0		
WG107423-1	MBLANK	SM 2540D	0.1184000g	0.1186000g	1	.8	U3.0 mg/L	NA	4	1.22	4.0		
WG107423-2	LCS	SM 2540D	0.1176000g	0.2166000g	2.5	990	990 mg/L	NA	4	1.22	10.		99

Comments:

WG107423-1 SF2243-3
WG107423-2 SF2243-3

Entered by: _____ Date: 4/26/12 Accepted by: _____ Date 04/27/12

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Julie D. Gourley

Review Date: 1/12/21

SOP Number: CA-720-09

SOP Title: Total Suspended Solids (Non-Filterable Residue) by EPA Method 160.2 and Standard Methods 2540D

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 01/13/21

QAO Signature: _____ **Date:** 011521

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: CA-551
Revision History
Cover Page
Page 1

TITLE: GRAIN SIZE ANALYSIS

Prepared By: Jessi Spurlock Date: 10-1-15

Approved By:

Department Manager: CLC Date: 10-1-15

Operations Manager: Deborah J. Kadean Date: 10.1.15

QA Officer: Lisa Dimond Date: 10.01.15

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Updated title of sections 1.4 + 5.0, Updated method references for NELAC + DoD Added information about hydrometer readings for sandy samples.	LAD	09/17	09/17
02	Sect. 2 - Updated method summary. Sect. 4 - Added 1/4" sieve, removed 3/4". Sect. 17 - Updated sieve only analysis. Updated references and logbook example	LAD	04/19	04/19
03	Sect. 4 & 7 - Added wooden tongue depressors and removed "rubber-tipped" from pestle. Updated references	LAD	06/20	06/20

TITLE: **Grain Size Analysis**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-551-03,** titled **GRAIN SIZE ANALYSIS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-551-03,** titled **GRAIN SIZE ANALYSIS.**

Recipient: _____ Date: _____

TITLE: **Grain Size Analysis**

1.0 SCOPE AND APPLICATION

This SOP details the procedure used by Katahdin Analytical Services technical personnel for particle size analysis in soils. This method is applicable to ASTM D422.

1.1 Definitions

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in Grain Size Analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in Grain Size Analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, that their work is properly documented and to indicate periodic review of the associated logbooks

1.3 Safety

1.3.1 Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

1.3.2 Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation

from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Management Program for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

For Grain size with hydrometer, depending on total solids of a sample, a certain amount is soaked in a dispersing agent overnight. The sample is then split into two portions, the material retained on the #10 sieve and the material passing the #10 sieve. The material retained on the #10 sieve is dried overnight to a constant weight. The sample is then passed through a large size sieve stack. Material passing the #10 sieve may be subjected to hydrometer analysis. After wet washing the sample on a #200 sieve the sample retained is dried overnight, then passed through a small size sieve stack. The material retained on each sieve, large and small sieves are measured and recorded. All measurements, large and small sieves and hydrometer readings are used to determine the particle size distribution of the sample. If the analysis requires sieve only, the sample weight will be determined as with samples including hydrometer. The samples will then be soaked in DI overnight, than wet washed and baked overnight. They will then be sieved through large and small sieves.

3.0 INTERFERENCES

Not Applicable

4.0 APPARATUS AND MATERIAL

4.1 Sieves ASTM E-11 Specifications, Brand Advantech, of the following size(s):

4.1.1 3.0” (75.00 mm)

4.1.2 2.0” (50.00 mm)

4.1.3 1.5” (37.50 mm)

4.1.4 1.0” (25.00 mm)

4.1.5 3/4" (19.00 mm)

4.1.6 1/4 “ (6.3mm)

4.1.7 # 4 (4.75 mm)

4.1.8 #10 (2.00 mm)

4.1.9 #20 (850.0 um)

4.1.10 #40 (425 um)

4.1.11 #60 (250.0 um)

4.1.12 #80 (180 um)

4.1.13 #100 (150.0 um)

4.1.14 #200 (75.0 um)

4.2 Sedimentation Classico Cylinder(s) 1000 mL

4.3 Hydrometer: ASTM 151H Humboldt H-4242

4.4 Drying Oven with temperature range of 60-110°C

4.5 Stainless Steel Spatulas & Spoons

4.6 Metal & Bristle Brushes

4.7 Ro-Tap Sieve Shaker- Gilson Company

4.8 Timers- capable of counting up to 24hours

4.9 Balance, capable of weight measurement to 0.01 g

4.10 Mechanical Stirring Device and Dispersion Cup- Hamiliton Beach Humboldt

4.11 Thermometer: Accurate to 0.5°C

4.12 Mortar and Pestle

4.13 Glass beakers- 1000ml and 500ml

4.14 Wooden Tongue Depressors

5.0 REAGENTS AND STANDARDS

5.1 Laboratory Grade Reagent Water

5.2 Sodium Hexametaphosphate:

5.3 **Sodium Hexametaphosphate Solution**: Add 120 g of sodium hexametaphosphate and 2940 g of reagent water to a 1-gallon plastic jug with cover. Mix the solution until it is homogeneous. Assign an expiration date of 30 days from the date made unless the parent reagent expires sooner in which case use the earliest expiration date. Store the prepared solution at ambient temperature.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

7.1 The following information must be recorded in the Grainsize logbook (all that are applicable).

- Start/End Date and Time
- Date/Time placement of samples in and out of oven
- Hydrometer(s) Serial Number
- Hydromter(s) Calibration Date
- Balance ID(s)
- Reagant ID
- Initial and final weights
- Analysts Initials
- Any comments regarding the sample extraction

Samples need to be “swiped” out when removing and “swiped” in when replacing samples in storage locations to maintain the internal chain of custody. Refer to Katahdin SOP, Sample Receipt and Internal Control, current revision, for the proper procedure for removal and return samples.

Fill out the sample preparation/extraction log with the necessary information before starting the extraction.

7.2 Equipment Calibration

Calibrate the balances being used each day prior to use. Record in the logbook designated for this purpose.

Calibrate or replace the hydrometers every five years

7.3 Total Solids Determination

Refer to SOP CA-717 “Total Solids/Total Volatile Solids Determination In Solid Matrices”

7.4 Sample Preparation

7.4.1 From the calculated percent total solids and the sample characteristics for each sample, determine the amount needed for the analysis can be determined using Table 1.

7.4.2 After determining the amount of sample to be used, place a 1000ml glass beaker on the balance and tare the balance. Mix the sample thoroughly with the stainless steel spatula or wooden tongue depressor. If the sample container is full to the extent that stirring the sample is impractical, try to remove the “best representative” aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.4.3 Add 125ml sodium hexametaphosphate solution to each beaker containing sample. Stir and mix thoroughly, soak sample in solution for at least 16 hours.

7.4.4 Refer to Katahdin SOP CA-108, current revision, “Basic Laboratory Technique” for more information on subsampling.

7.5 Sample Partition

After sample and solution has soaked for a minimum of 16 hours, the sample slurry is rinsed into a dispersion cup using DI water. Fill the dispersion cup ½ full with DI water and place the cup on the blender to mix for one minute.

7.5.1 If the sample appears to have large gravel, sand, or organic material that does not appear to be amendable for using the blender notify the PM and proceed to the next step without blending.

7.5.2 After the sample slurry has been blended, pour sample through a #10 sieve with pan attached, so sample smaller than a #10 sieve is collected in pan. Transfer contents in pan to a 1000ml graduated cylinder and continue to rinse the dispersion cup through the #10 sieve until transfer is complete. After all sample in the pan is rinsed into the cylinder bring the volume of the cylinder to 1000ml using DI water. Cover the cylinder with a rubber stopper and equilibrate the sample to ambient temperature for the hydrometer analysis.

7.5.3 Weigh and label a medium aluminum pan, than transfer the contents of the material retained on the # 10 sieve to the pan. Place the aluminum pan in a drying oven set at 110 $\pm$ 5° C and dry the sample material for at least 16 hours or until constant weight set aside for sieve analysis.

7.6 Hydrometer

Prepare a hydrometer blank by adding 125ml sodium hexametaphosphate and bring to 1000 mL with DI water in a 1000 mL graduated cylinder. Be sure to take readings with a hydrometer and a thermometer while taking readings on actual hydrometer samples. This will provide us with the temperature and solution correction factors later in the procedure. Also prepare a hydrometer rinse bath, used to rinse the hydrometer between uses.

7.6.1 To shake the cylinder, rotate the flask up and down for one minute approximating at least 60 turns. One turn down and one turn up equals two turns.

7.6.2 To take a hydrometer reading, gently insert the hydrometer into the graduated cylinder and wait approximately 20 seconds. Read the hydrometer from the top of the meniscus to the nearest 0.0005. Enter the reading on the logbook. After each reading, clean the hydrometer by twisting and dropping the hydrometer into the hydrometer rinse bath.

7.6.3 Insert a temperature probe into the cylinder to the same depth used for the hydrometer reading. Read the temperature to the nearest 0.5°C and enter the temperature measurement on the logbook. Rinse the temperature probe in the hydrometer rinse bath.

7.6.4 Repeat the above process taking hydrometer readings every 2, 5, 15, 30, 60, 240 and 1440 minutes, proceed to small sieve analysis.
With samples that are mostly sand, the hydrometer reading can drop below the reading of the blank. When this occurs, we will apply the lowest hydrometer reading to the blank reading. We will include this in the technical narrative. The logic behind this issue is due to the high amount of sand, resulting in more density. As a result, less DI water is added when transferring the sample/hexametaphosphate solution to the 1000 mL cylinders. Since the blank has a higher DI water/hexametaphosphate ratio, it increases the reading.

7.7 Sieve Analysis for Large and Small Sieves

Look at the sample material in the aluminum pan and record a description of the non-soil material (such as- sticks, grass, wood, plastic), hardness of material and shape of material in the logbook.

Hardness qualifiers include hard, soft or brittle.
Shape qualifiers include well rounded, rounded, subrounded, subangular, and angular.

Large Sieves

7.7.1 Weigh the 3/4", 1/4", #4 and #10 sieves and enter the weight measurements in the logbook as the tare weight.

7.7.2 Stack the sieves then transfer the sample material from the aluminum dish to the sieve stack. If the sample material is less than 30 g, manually shake the sieve stack for 2 minutes. If the sample material is greater than 30 g, place the sieve stack into the Ro-tap machine and shake the sieve stack for 4 minutes. Weigh each sieve and record these measurements in the logbook.

Small Sieves

Completely transfer the sample from the graduated cylinder to a #200 wet wash sieve. Make sure the entire sample has been transferred to the #200 wet wash sieve by rinsing the graduated cylinder several time with DI water. Using DI water, wash the sample through the #200 sieve until the water runs clear then transfer the material retained on the sieve into a aluminum tin labeled with the sample's LAB ID.

7.7.3 Place the beaker in the drying oven and dry at a temperature of 110ºC for at least 16 hours. After 16 hours, remove the beaker from the oven and allow it to cool.

7.7.4 Samples are sieved through the #20, #40, #60, #80, #100, and #200.

7.7.5 After samples have cooled, stack the sieves then transfer the sample material from the aluminum dish to the sieve stack. If the sample material is less than 30 g, manually shake the sieve stack for 2 minutes. If the sample material is greater than 30 g, place the sieve stack into the Ro-tap machine and shake the sieve stack for 4 minutes. Weigh each sieve and record these measurements in the logbook.

7.8 Sieve Only Analysis

7.8.1 Determine the total solids of the sample. Use the Percent Solid Table (Table 1) to determine the sample size to be used. If total solids are not available depending on sample matrix we make an educated guess as to how much sample will be used. Samples are then soaked in DI overnight.

7.8.2 After sample and solution has soaked for a minimum of 16 hours, the sample slurry is rinsed into a dispersion cup using DI water. Fill the dispersion cup ½ full with DI water and place the cup on the blender to mix for one minute.

7.8.3 If the sample appears to have large gravel, sand, or organic material that does not appear to be amendable for using the blender notify the PM and proceed to the next step without blending.

7.8.4 Samples are then wet-washed through the #200 Sieve until samples run clear.

7.8.5 Samples are then completely transferred to a metal tin labeled with appropriate sample ID, and dry at a temperature of 110ºC for at least 16 hours. After 16 hours, remove the tin from the oven and allow it to cool.

7.8.6 Gently mix the dried contents of the beaker with a pestle to break any soil aggregates that may have formed during the drying stage.

7.8.7 For Large Sieve analysis follow 7.7.1-7.7.2

7.8.8 For Small Sieve analysis follow sections 7.7.2-7.7.3

7.9 Calculations

7.9.1 Sample Used (SU): total dry sample

SU =Total Sample Weight ((100-%Moisture)/100)*

HMCF = Hygroscopic moisture correction factor (we assume 1)

7.9.2 Sieve Analysis (Percent Finer = PF)
Large Sieves:

3 inch: PF = 100-100 (Sieve and Sample (3 inch) - Sieve (3 inch))/SU*

2 inch: PF = PF (3 inch) - 100(Sieve and Sample (2 inch) - Sieve (2 inch))/SU and so on through the #10 Sieve.*

Small Sieves:

#20: PF = PF(#10) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#20) - sieve(#20))/sample used*

#40: PF = PF (#20) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#40) - sieve (#40))/sample used and so on up through #10 sieve.*

7.9.3 Hydrometer Analysis

Particle size, Micron

1000*sqrt [930*viscosity/980*(SG-1))*(effective depth/time)]

Effective Depth, cm = 16.29-264.5*(actual Hydrometer reading - 1)
Time, minutes = Time of hydrometer reading from beginning of edimentation
Sqrt - square root
SG - Specific Gravity of soil (assuming a default SG)
Viscosity - is the resistance of a liquid to flow

Percent Finer (PF):

PF = Constant*(actual hydrometer reading - hydrometer correction factor - 1)

Constant = (100,000/W)*SG/(SG-1)
W = (Total sample used *sample used for hydrometer analysis*HMCF)/Amount of total sample passing #10 sieve
Hydrometer Correction = slope*sample temperature + Intercept
Slope = ((low temp. reading -1)-(high temp. reading -1)/(low temp. - high temp.))

Intercept = (low temp. reading -1) - (low temp. * slope)

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Not Applicable

TITLE: **Grain Size Analysis**

9.0 METHOD PERFORMANCE

Not Applicable

10.0 APPLICABLE DOCUMENTS/REFERENCES

ASTM Standard D 422-63 (Re-approved 2007) “Standard Test Method for Particle-Size Analysis of Soils”, ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

Katahdin SOP SOP CA-717 “Total Solids/Total Volatile Solids Determination In Solid Matrices”, current revision.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM, Current Version.

LIST OF TABLES AND FIGURES

Table 1 Percent Solids Table for Weight Determination
Table 2 Summary of Method Modifications
Figure 1 Example of Logbook Page

TITLE: **Grain Size Analysis**

TABLE 1

PERCENT SOLIDS TABLE FOR WEIGHT DETERMINATION

Percent Solid Table

Quantities of sample (in grams) to be utilized in Wet method version of ASTM D854 and D422

% Sol	Spec Grav	Hydrometer Slt/Cl	Slt/Snd	Snd	Snd/Gr
	25	50	75	100	200
1	2500	5000	7500	10000	20000
2	1250	2500	3750	5000	10000
3	833	1667	2500	3333	6667
4	625	1250	1875	2500	5000
5	500	1000	1500	2000	4000
6	417	833	1250	1667	3333
7	357	714	1071	1429	2857
8	313	625	938	1250	2500
9	278	556	833	1111	2222
10	250	500	750	1000	2000
11	227	455	682	909	1818
12	208	417	625	833	1667
13	192	385	577	769	1538
14	179	357	536	714	1429
15	167	333	500	667	1333
16	156	313	469	625	1250
17	147	294	441	588	1176
18	139	278	417	556	1111
19	132	263	395	526	1053
20	125	250	375	500	1000
21	119	238	357	476	952
22	114	227	341	455	909
23	109	217	326	435	870
24	104	208	313	417	833
25	100	200	300	400	800
26	96	192	288	385	769
27	93	185	278	370	741
28	89	179	268	357	714
29	86	172	259	345	690
30	83	167	250	333	667
31	81	161	242	323	645
32	78	156	234	313	625
33	76	152	227	303	606
34	74	147	221	294	588
35	71	143	214	286	571
36	69	139	208	278	556
37	68	135	203	270	541
38	66	132	197	263	526
39	64	128	192	256	513
40	63	125	188	250	500
41	61	122	183	244	488
42	60	119	179	238	476
43	58	116	174	233	465
44	57	114	170	227	455
45	56	111	167	222	444
46	54	109	163	217	435
47	53	106	160	213	426
48	52	104	156	208	417
49	51	102	153	204	408
50	50	100	150	200	400

% Sol	Spec Grav	Hydrometer Slt/Cl	Slt/Snd	Snd	Snd/Gr
	25	50	75	100	200
51	49	98	147	196	392
52	48	96	144	192	385
53	47	94	142	189	377
54	46	93	139	185	370
55	45	91	136	182	364
56	45	89	134	179	357
57	44	88	132	175	351
58	43	86	129	172	345
59	42	85	127	169	339
60	42	83	125	167	333
61	41	82	123	164	328
62	40	81	121	161	323
63	40	79	119	159	317
64	39	78	117	156	313
65	38	77	115	154	308
66	38	76	114	152	303
67	37	75	112	149	299
68	37	74	110	147	294
69	36	72	109	145	290
70	36	71	107	143	286
71	35	70	106	141	282
72	35	69	104	139	278
73	34	68	103	137	274
74	34	68	101	135	270
75	33	67	100	133	267
76	33	66	99	132	263
77	32	65	97	130	260
78	32	64	96	128	256
79	32	63	95	127	253
80	31	63	94	125	250
81	31	62	93	123	247
82	30	61	91	122	244
83	30	60	90	120	241
84	30	60	89	119	238
85	29	59	88	118	235
86	29	58	87	116	233
87	29	57	86	115	230
88	28	57	85	114	227
89	28	56	84	112	225
90	28	56	83	111	222
91	27	55	82	110	220
92	27	54	82	109	217
93	27	54	81	108	215
94	27	53	80	106	213
95	26	53	79	105	211
96	26	52	78	104	208
97	26	52	77	103	206
98	26	51	77	102	204
99	25	51	76	101	202
100	25	50	75	100	200

TITLE: **Grain Size Analysis**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-551-03	METHOD ASTM D 422-63
Procedures		
Apparatus/Materials		
Reagents		
Sample Preservation and handling		
QC – Accuracy/ Precision		

TITLE: **Grain Size Analysis**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

Katahdin Analytical Services, LLC.
Sediment Grain Size - Method ASTM D422

Lab Sample ID	SM2456-4	Start Date/Time	3-20-19: 11:51
Analyst:	JMS	End Date/Time	4-2-19: 12:44
Sample Description:	Mafic/organics - removed 3.1g sticks/grass/roots		
Sample Weight	Sample (g)	Date/Time in oven	See pg. 1
Sample Weight (wet)	~~1250~~ 89.9	Date/Time out of oven	↓
Sample Weight (dried)	25.98		

		Hydrometer Data	
% Moisture	71.105	Serial Number	742303
		Cal Date:	3/25/19: 11:32
Sample Split (Oven Dried)	Sample (g)	Low Temp C	16.4
Sample >=#10	0	Low Temp Reading	1.0035
Sample <=#10	25.98	High Temp	17.6
		High Temp Reading	1.0035
Sieve Only:			
Sieve Only, Wet Wash:			
		Soil Gravity	2.65

Gravel/Sand Fraction (Sieves)			
Sample Fraction	Size (um)	Pan Tare	Pan+Sample
3"	75000		
2"	50000		
1.5"	37500		
1"	25000		
3/4"	19000		
1/4"	6300		
#4	4750		
#10	2000		
#20	850	307.2	307.5
#40	425	270.0	270.6
#60	250	248.5	249.0
#80	180	241.9	242.1
#100	150	226.5	236.7
#200	75	315.2	315.8
Pan	Pan	339.9	340.3

11:45

Silt/Clay Fraction (Hydrometer Test)				
Time (min)	Proposed Read Time	Actual Time (min)	Temp C	Spec. Gravity
2	11:50	11:50 (2)	17.2	1.0155
5	11:53	11:53 (5)	17.0	1.0130
15	12:03	12:03 (15)	17.5	1.0115
30	12:18	12:17 (29)	17.3	1.0105
60	12:48	12:48 (60)	16.6	1.0100
240	15:48	15:48 (240	16.5	1.0075
1440	11:48	11:44 (1441)	17.5	1.0060

EX-028 – Revision 3 – 03/19/2019

QAEX379

0000002

SEDTREND ANALYSIS LIMITED

Laboratory Standard Operating Procedures for the Particle Size Analyses of Suspended Sediment in Water

SedTrend Analysis Limited
#73 1338 Stelly's X-Road
Brentwood Bay, B.C.
Canada, V8M 0B6
250-652-1334
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Prepared By:
Patrick McLaren, Ph.D., P.Geo.

June, 2018

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1.0 PURPOSE AND SCOPE

This document describes methods, materials, equipment, and special conditions used by SedTrend to determine the particle size distribution for suspended sediment contained in water samples using laser diffraction. Its purpose is to ensure that SedTrend consistently delivers the highest standard of grain-size analyses to its clients.

2.0 EQUIPMENT

(1) Malvern Mastersizer 2000 optical unit
(2) Malvern Hydro 2000G sample-handling unit
(3) 1 mm sieve
(4) Micro-90 concentrated cleaning solution

3.0 PROCEDURE

(1) Using the Malvern software, an electronic file is created in which all particle size data for a given sample are stored under a specific project.
(2) Samples arrive at the laboratory as "water" samples containing suspended sediment. Containers are typically 1 to 4 liters in size. The samples' ID, the date of analysis and any comments related to the sample are recorded on an Excel spreadsheet which is separately backed up.
(3) The instruments are turned on and the correct measurement SOP[1] is selected to begin the analyses. Prior to every analysis, the Mastersizer 2000 automatically aligns the laser beam, and a background measurement of the suspension medium is taken.
(4) The container of water and sediment is stirred to ensure that sediment is equally dispersed throughout the container and a portion is immediately poured into the bath of the Hydro 2000G in sufficient quantity to obtain the correct level of obscuration. In the event obscuration becomes unacceptably high for the instrument to make a measurement, tap water is added to the bath until the correct obscuration level is obtained.
(5) In addition, the sample is introduced into the bath through a 1mm sieve to ensure that larger particles or organic debris such as twigs or leaves will not interfere with the instrument's pumping mechanisms. Any material visible on the sieve is examined and appropriate notes taken of the observations. (It is unlikely that any of the suspended sediment samples will contain particles larger than 1 mm and even more unlikely that such particles would be in sufficient quantity to analyze further by sieving.)
(6) Following the completion of the measurement cycle, the instrument is flushed clean. Because of the nature of the samples, the water that is pumped through the Malvern may contain oils and other contaminants that are capable of coating the lenses of the cell. Such an occurrence is evident from spurious background measurements before the introduction of the next sample in which case the run

[1] SedTrend has developed a standard operating procedure (SOP) using the Malvern Mastersizer 2000 laser particle size analyzer software. This procedure ensures that all parameters and variables will remain constant throughout the sample analyses. Prior to every analysis, the Mastersizer 2000 automatically aligns the laser beam, and a background measurement of the suspension medium is taken.

is cancelled in order to carry out an intensive cleaning cycle using a concentrated cleaning solution (Micro-90).

(7) All digital files from the Malvern analysis are backed up on both a thumb drive and second computer..

4.0 Laboratory: Additional Notes

SedTrend's laboratory is equipped with a Malvern Mastersizer 2000 optical unit coupled with a Malvern Hydro 2000G sample-handling unit. Both of these units have passed Quality Control and Quality Assurance testing by the manufacturer and meet ISO 9001 standards (see Appendix 1). These units are operated using a licensed copy of Malvern's analysis software, which is run through a PC computer. All system parameters at the time of analysis, and data collected by these instruments are automatically stored in the computer. Proper laboratory attire is worn at all times, including latex gloves, safety glasses and respirator when necessary.

The Malvern system is tested for accuracy and proper function after every 300 analyses. This is accomplished using Malvern's Quality Audit Standard – one shot bottles of polydisperse glass-bead transfer standard (see Appendix 2). UsingThe system cell windows are also disassembled and cleaned after every 300 analyses. SedTrend will archive samples at the request of the client.

5.0 Data Reporting

Data taken from the result file generated by the Malvern software, grainsize data are converted to phi distributions where phi = $\log10^{mm}/\log10^{2}$. The data are presented in ½ phi interval bins that can range from 1mm (0.00 phi) to 0.06 microns (14 phi) depending on the range of grain sizes present in the sample. In addition, the phi mean, sorting and skewness of the distributions, together with the % sand and % mud are reported.

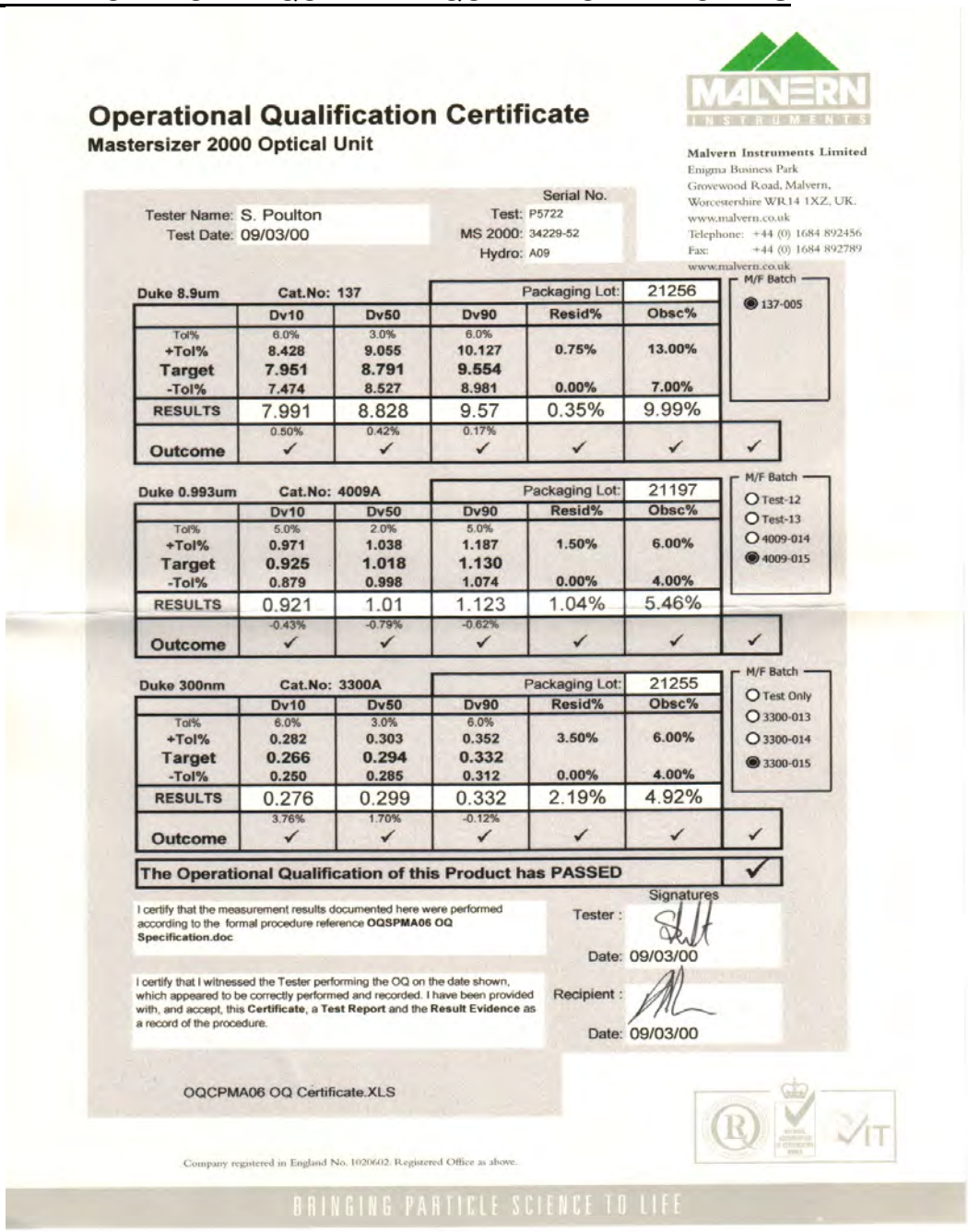
6.0 SedTrend SOP

The following table defines the parameters that will be used in the SedTrend SOP to analyse suspended sediments sediment.

Material Name	Marine sediment
Refractive Index	1.545
Absorption	0.01
Dispersant Name	Tap water
Refractive Index	1.33
Measurement Time	40s
Measurement Snaps	40000
Background Time	15s
Background Snaps	15000
Pump Speed	2500
Stirrer Speed	1000
Pre-measurement Ultra-Sonics	300s (5 min)

Tip Displacement	100%
Aliquots	1 per SOP
Measurements	1 per aliquot
Cleaning	After each aliqot - enable
Clean Mode	Automatic/Full Wash
Flush Cycles	1
Dispersant Degassing	Enable

APPENDIX 1: LABORATORY EQUIPMENT QUALITY CERTIFICATES



Operational Qualification Certificate
Mastersizer 2000 Optical Unit

MALVERN INSTRUMENTS

Malvern Instruments Limited
Enigma Business Park
Grovewood Road, Malvern,
Worcestershire WR14 1XZ, UK.
www.malvern.co.uk
Telephone: +44 (0) 1684 892456
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www.malvern.co.uk

Tester Name: S. Poulton
Test Date: 09/03/00

Serial No.
Test: P5722
MS 2000: 34229-52
Hydro: A09

Duke 8.9um Cat.No: 137 — Packaging Lot: 21256 — M/F Batch: 137-005

	Dv10	Dv50	Dv90	Resid%	Obsc%	
Tol%	6.0%	3.0%	6.0%			
+Tol%	8.428	9.055	10.127	0.75%	13.00%	
Target	7.951	8.791	9.554			
-Tol%	7.474	8.527	8.981	0.00%	7.00%	
RESULTS	7.991	8.828	9.57	0.35%	9.99%	
Outcome	0.50% ✓	0.42% ✓	0.17% ✓	✓	✓	✓

Duke 0.993um Cat.No: 4009A — Packaging Lot: 21197 — M/F Batch: Test-12 / Test-13 / 4009-014 / 4009-015

	Dv10	Dv50	Dv90	Resid%	Obsc%	
Tol%	5.0%	2.0%	5.0%			
+Tol%	0.971	1.038	1.187	1.50%	6.00%	
Target	0.925	1.018	1.130			
-Tol%	0.879	0.998	1.074	0.00%	4.00%	
RESULTS	0.921	1.01	1.123	1.04%	5.46%	
Outcome	-0.43% ✓	-0.79% ✓	-0.62% ✓	✓	✓	✓

Duke 300nm Cat.No: 3300A — Packaging Lot: 21255 — M/F Batch: Test Only / 3300-013 / 3300-014 / 3300-015

	Dv10	Dv50	Dv90	Resid%	Obsc%	
Tol%	6.0%	3.0%	6.0%			
+Tol%	0.282	0.303	0.352	3.50%	6.00%	
Target	0.266	0.294	0.332			
-Tol%	0.250	0.285	0.312	0.00%	4.00%	
RESULTS	0.276	0.299	0.332	2.19%	4.92%	
Outcome	3.76% ✓	1.70% ✓	-0.12% ✓	✓	✓	✓

The Operational Qualification of this Product has PASSED ✓

Signatures

I certify that the measurement results documented here were performed according to the formal procedure reference **OQSPMA06 OQ Specification.doc**

Tester :
Date: 09/03/00

I certify that I witnessed the Tester performing the OQ on the date shown, which appeared to be correctly performed and recorded. I have been provided with, and accept, this **Certificate**, a **Test Report** and the **Result Evidence** as a record of the procedure.

Recipient :
Date: 09/03/00

OQCPMA06 OQ Certificate.XLS

Company registered in England No. 1020602. Registered Office as above.

BRINGING PARTICLE SCIENCE TO LIFE

Performance Verification Certificate

Mastersizer 2000 Sample Handling Unit

Hydro 2000G

Malvern Instruments Limited
Enigma Business Park
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Worcestershire WR14 1XZ, UK.
www.malvern.co.uk
Telephone: +44 (0) 1684 892456
Fax: +44 (0) 1684 892789
www.malvern.co.uk

Test Number: Final Test
Test Date: 10th Feb. 00
Tester: C. Wilks

MS2000 Serial No: AB2

Acc. Serial No: 34182-21

Sample Lot No: 3124

Valid Accessory
- (●) Hydro 2000G
- ( ) Hydro 2000S
- ( ) Hydro 2000M/MU
- ( ) Scirrocco 2000
- ( ) Hydro 2000SM

QAS 0002

M/F Batch No.
- (●) -001

Test Results:

	Dv10	Dv50	Dv90
Tol%	3.0%	2.0%	3.0%
+Tol%	29.36	48.45	80.34
Target	28.50	47.50	78.00
-Tol%	27.65	46.55	75.66
RESULTS	28.758	47.627	78.901
Outcome	0.91% ✓	0.27% ✓	1.16% ✓
OBSC%	15%	10.% - 30.%	✓
RESIDUAL%	0.25%	< .75%	✓
Product Operational Qualification has PASSED			

I certify that the measurement results documented here were performed according to the Operational Qualification Procedure reference MS2000Accs Operational Qualification.DOC.

Tester Signature: C. Wilks Date: 10th Feb. 2000

Issue Level: 2

I certify that I witnessed the Tester performing the OQ on the date shown, and that the results appear to be correctly performed and recorded. I have been provided with, and accept, this **Certificate, Test Report** and the **Result Evidence** as a record of the measurement.

Recipient Signature: Date:

MS2000Accs.02 OQ Certificate.XLS

Company registered in England No. 1020602. Registered Office as above.

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APPENDIX 2: MALVERN QA FOR DISPERSION UNITS

Malvern Quality Audit Standards QAS2001, QAS2002 and QAS2003.
Malvern's Quality Audit Standards have been produced to provide users of Malvern particle size analyzers with a reliable one-shot polydisperse transfer standard to enable them to check the performance of their sample dispersion units. As noted above glass bead particles present within QAS3002 are used to the Quality Audit Standards every 300 samples with each test being required to be within the limits as shown in the table below. The output from such a test (March 17, 2018) is also shown with each of the values being within prescribed limits.

	Dv10 / µm	Dv50 / µm	Dv90 / µm
Lower Limit	35.326	59.995	85.473
Target Value	37.185	61.850	89.972
Upper Limit	39.044	63.706	94.471

MASTERSIZER 2000

Result Analysis Report

Sample Name: QA Test March 17-18- Average - Average	**SOP Name:** Glass beads (typical)	**Measured:** Saturday, March 17, 2018 11:28:36 AM	
Sample Source & type: Factory = Paris	**Measured by:** Owner	**Analysed:** Saturday, March 17, 2018 11:28:38 AM	
Sample bulk lot ref: 123-ABC	**Result Source:** Averaged		
Particle Name: Glass beads (typical)	**Accessory Name:** Hydro 2000G (A)	**Analysis model:** Single narrow mode (spherical)	**Sensitivity:** Enhanced
Particle RI: 1.520	**Absorption:** 0	**Size range:** 0.020 to 2000.000 um	**Obscuration:** 22.26 %
Dispersant Name: Water	**Dispersant RI:** 1.330	**Weighted Residual:** 0.501 %	**Result Emulation:** Off
Concentration: 0.1996 %Vol	**Span :** 0.842	**Uniformity:** 0.264	**Result units:** Volume
Specific Surface Area: 0.043 m²/g	**Surface Weighted Mean D[3,2]:** 56.982 um	**Vol. Weighted Mean D[4,3]:** 64.356 um	
d(0.1): 38.259 um	d(0.5): 63.280 um	d(0.9): 91.531 um	

Malvern's Quality Audit Standards have been produced by one of Europe's leading suppliers of glass bead standards, Whitehouse Scientific, who have used an extremely efficient and accurate riffle splitting process to ensure that each one-shot pot of the material is a representative sample of the whole bulk of sample material.

Comprehensive sampling of pots taken from the whole production run of the reference material has allowed us to establish and document confidence limits on the sample material of 0.36% on the D_v50 of the distribution.

As can be seen from the diagram below, the quality of the riffling and packing process is such that the difference between pots is truly within experimental error (It also speaks quite well for the repeatability of Malvern's laser diffraction particle size analyzers as a reliable and repeatable measurement technique)

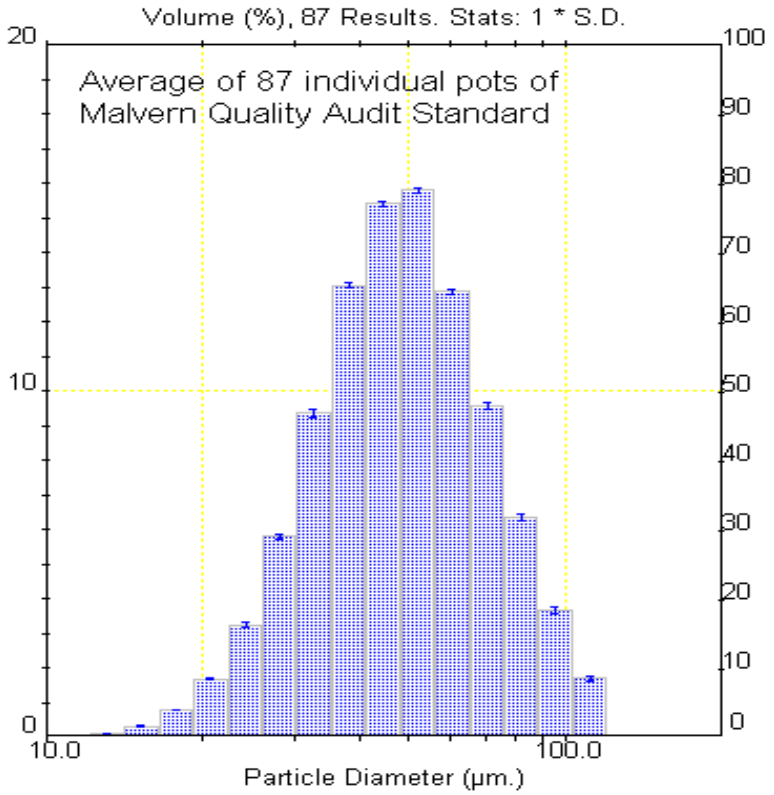

Figure 1. Statistics plot of 87 separate samples extracted from all parts of the riffling process. The error bars illustrate the consistency of the particle size distributions yielded by these samples.

An extensive programme of testing of Malvern's sample dispersion units has been carried out and is continuing to characterize the results which may be expected from them in order to establish pass/fail criteria for the purposes of OQ testing of these units.

To ensure that the full benefit is obtained from the single shot concept, it is essential that the entire contents of the bottle are consumed in a single measurement.

Three separate pack sizes of the standard material have been produced in order that users can obtain the correct level of obscuration with most Malvern sample dispersion units. These are:

QAS2001 (0.25g)	f*or MSX1, MS1, DIF2021, Hydro 2000 S and Hydro 2000 SM*
QAS2002 (2.5g)	*for MS15, DIF2012, Hydro 2000 G, Hydro 2000 M with 1000ml beaker, Hydro 2000 MU with 1000 ml beaker, Mastersizer Micro with 1000 ml beaker and Mastersizer Microplus with 1000 ml beaker.)*
QAS2003 (5g)	*for MAM 2460 and MAM 2461 Manual and QSpec Dry powder feeders on the Mastersizer S and X as well as the Scirocco Dry Powder Feeder on the Mastersizer 2000.)*

Every individual bottle of the Malvern Quality Audit Standard is uniquely numbered to aid traceability of the measurements performed.

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SGS AXYS ANALYTICAL SERVICES LTD

SGS AXYS METHOD MLA-021: ANALYTICAL METHOD FOR THE DETERMINATION OF POLYCYCLIC AROMATIC HYDROCARBONS (PAH), ALKYLATED POLYCYCLIC AROMATIC HYDROCARBONS, AND ALKANES

SCOPE

This method describes the determination of concentrations of PAHs, alkylated PAHs and alkanes in solid (sediment, soil, ash), tissue (including blood), aqueous, XAD-2 column (resin and filters), air, and oil samples and in solvent extracts such as those produced by dialysis of SPMD (Semipermeable Membrane Device) samplers. All samples are spiked with deuterated surrogate standards prior to analysis. Solid and tissue samples are normally extracted by Soxhlet extraction but may optionally be extracted by base digestion procedures. Aqueous samples are solvent extracted. Aqueous samples with a high particulate content are filtered prior to a Soxhlet extraction of the filter and solvent extraction of the filtrate. Blood samples are solvent extracted. Oil samples are diluted with solvent. XAD-2 resin and filters from water sampling columns and polyurethane foam plugs from air samplers are Soxhlet extracted. The sample extracts are fractionated on a silica gel column into polar and non-polar fractions. These fractions are analyzed by GC/MS for PAHs, alkylated PAHs and alkanes. Concentrations are reported in ng/g, ng/L, ng/column or ng/sample. Solid data are reported on a dry weight basis. Tissue data are reported on a wet weight basis.

These methods may be used for analysis of samples where USEPA Methods 1625B or 8270C/D have been requested **provided the modifications described in this document are permitted by contract**. Key attributes of methods MLA-021, EPA 8270C/D and EPA 1625B are summarized in Appendix B.

Upon client request, this method may be used to determine a suite of hopanes, as described in Appendix A, or to determine 2-chloronaphthalene, as described in Appendix F.

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Approved 26-Oct-2020

Shea Hewage, General Manager
Dale Hoover, Senior Technical Director
Rhonda Stoddard, QA Manager

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This method describes the determination of concentrations of PAHs, alkylated PAHs and alkanes in solid (sediment, soil, ash), tissue (including blood), aqueous, XAD-2 column (resin and filters), air, and oil samples and in solvent extracts such as those produced by dialysis of SPMD (Semipermeable Membrane Device) samplers. All samples are spiked with deuterated surrogate standards prior to analysis. Solid and tissue samples are normally extracted by Soxhlet extraction, but may optionally be extracted by base digestion procedures. Aqueous samples are solvent extracted. Aqueous samples with a high particulate content are filtered prior to a Soxhlet extraction of the filter and solvent extraction of the filtrate. Blood samples are solvent extracted. Oil samples are diluted with solvent. XAD-2 resin and filters from water sampling columns and polyurethane foam plugs from air samplers are Soxhlet extracted. The sample extracts are fractionated on a silica gel column into polar and non-polar fractions. These fractions are analyzed by GC/MS for PAHs, alkylated PAHs and alkanes. Concentrations are reported in ng/g, ng/L, ng/column or ng/sample. Solid data are reported on a dry weight basis. Tissue data are reported on a wet weight basis.

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Approved 03-Feb-2015 John Cosgrove, President and Senior Technical Director
Shea Hewage, Director of Operations
Dale Hoover, QA Manager

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REFERENCES

1. USEPA Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 3, Dec 1996

2. USEPA 8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 4 February 2007

3. USEPA Method 1625, Revision B, Semivolatile Organic Compounds by Isotope Dilution GC/MS

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CHANGES FROM PREVIOUS REVISIONS AND VERSIONS

This table lists the details of the changes from revision 12 version 03 of this document.

Page	Change details
15	Section B: Removed references to any particular brand of anti-bumping granules/boiling chips.
17	Table 3, footnote 4: Clarified that the <-10°C storage requirement as applicable to final extracts, not to extracts in progress.
20	Section 3.1.1: Added that the water bath temperature for the rotary evaporator is 30°C.
31-34	Table 8 (document correction): Added missing blank method acceptance limits for other matrices than solid, SPMD, aqueous and tissue. Corrected erroneous flagging of footnote 1 in the table.
36	Table 8: Clarified that the ±35% ion ratio limit applies for both the authentic as well as for the labeled compounds dibenz[ah]anthracene, indeno[1,2,3-cd]pyrene and benzo[ghi]perylene.
42	Section 5.2.2: Removed "sintered glass" from Soxhlet thimble description.

This table lists the details of the changes from revision 12 version 02 of this document.

Page	Change details
1	Modified scope to make Soxhlet extraction default and base-digestion optional for tissues.
13-14	Table 1: Reinstated Lists 1, 2 and 3 for the PAH compounds as these are used for products
27	Section 3.3: Updated shelf life for baked anhydrous sodium sulphate to 1 month.
29	Section 3.5.2: Changed the length of the silica gel column to 40 cm.
36, 38	Tables 8 and 9: Clarified the analysis duplicate and bracketing calibration specifications.
42-43	Switched sections 5.2 and 5.3 to reflect Soxhlet extraction as default tissue extraction procedure and base digest as optional procedure. Changed wording in the section to reflect the switch, Added lipid capacity of 1.25 g lipid to both Soxhlet and base digest procedures. Internal supporting documentation: http://axys/info/AAS/ProdDevImp/Concept%20Issues%20Wiki/MLA-021-R10V11-Tissue%20Extraction%20Soxhlet%20vs%20Base%20Digest.aspx

This table lists the details of the changes from revision 12 version 01 of this document.

Page	Change details
14, 22, 36, 58	Tables 1 (footnote) and 8, and sections 3.2.1.3, 3.2.1.4, 7.1.3 and 7.1.4: Reinstated single-point bracketing calibration as an optional calibration method for PAHs.

This table lists the details of the changes from revision 11 version 01 of this document.

Page	Change details
1	Scope: Document correction of default extraction methods for solid samples and tissues – Soxhlet extraction for solid samples and base digestion for tissues. (This is to correspond with clarifications entered into sections 5.1, 5.2 and 5.3 of MLA-021 Rev. 10 Ver. 03).
12-13, 21, 23, 35, 57	Sections A, 3.2.1, 7.1.3 and tables 1, 6, 8: Changed compounds analyzed with single-point bracketing calibration to be calibrated by multi-point calibration, removed references to single-point calibration. Internal supporting data in AXYS Product Development and Improvement Docs: MLA-021_LinearityValidation_Jun2014.xls and MLA-021_CalVerValidation_Jun2014.xls.
37-38	Table 9: Updated blank, OPR, and sample surrogate recovery limits for several alkane compounds. Internal supporting data in AXYS Product Development and Improvement Docs: MLA-021_ValidationAlkaneBlanks(4587)_Jun2014.xls and MLA-021_ValidationAlkaneOPRs(4587)_Jun2014.xls.

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This table lists the details of the changes from revision 10 version 12 of this document.

Page	Change details
1	Added SPMD extracts to the scope
17	Table 3: Storage of solvent extract samples changed from -20°C to < 4°C
19	Section 2: Sample pretreatment details added for SPMD
21-22	Section 3.2.1.1: Added information about a third surrogate that is used for SPMD analysis.
31	Added that SPMD samples can have only field duplicates.
32-36	Table 8: Added SPMD blank limits, typical SDLs, and PRC recovery limits for OPR samples. Added footnotes to surrogates which are used as PRCs in SPMD. Added Guidance Limits for Recovery of PRCs in Day 0 and Field/Trip Blank SPMDs.
49	Section 5.6: Added info that SPMD extracts will be dried and split prior to MLA-021 analysis.
99-100	Appendix H: Added SPMD extracts to table "Laboratory Mean + 3 S Limits for Procedural Blanks"

This table lists the details of the changes from revision 10 version 11 of this document.

Page	Change details
14, 28	Sections B and 3.3: Deleted name of supplier of anti-bumping granules.
15, 64	Sections B and 8.4: Added information about a benzo(b)fluoranthene quantification.correction to negate interference from labeled d12-benzo(k)fluoranthene
17	Table 3: Clarified the required sample weights for solid and biosolid samples
21, 28	Sections 3.1.3 and 3.3: Moved Soxhlet extractor cleaning instructions from section 3.3 to the glassware cleaning instructions in section 3.1.3.
22	Section 3.2.1.5: Added instruction to restrict MS/MSD overspiking to List 1 and List 2 compounds.
33, 35	Table 8: Added footnotes clarifying replacement options for some standards that are used in OPRs and that may be unavailable.
44, 55	Sections 5.3.3. and 5.11: Document correction updating the Biobead cutpoints to 0-150 mL discard and 150-300 mL collect.
48, 50, 51	Sections 5.5., 5.7 and 5.8: Clarified that alkanes can not be determined in XAD-2 sorbent, ambient air samples and stationary air sample trains.

This table lists the details of the changes from revision 10 version 10 of this document.

Page	Change details
	Updated blank limits for several alkanes to current practice according to QDO-021 Rev 02.

This table lists the details of the changes from revision 10 version 09 of this document.

Page	Change details
2	Corrected reference to EPA 8270D
72	Appendix B - additional details added

This table lists the details of the changes from revision 10 version 08 of this document.

Page	Change details
16	Table 3: Added sample size for analysis of alkanes in oils. Clarified that 10-50 mg is the sample size for PAH analysis (as stated in section 5.9). Added footnote that the same subsample can be used for PAH and alkane analysis if a split is performed.

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44	Section 5.4.1: Added instruction that aqueous samples must be magnetically stirred while spiked with surrogate solution.
51	Section 5.9: Added instruction on sample size and splitting for Oils for Alkane analysis
55	Section 6.1: Revised typical Biobead cut point for F1 from 0-100 mL to 0-150 mL 1:1 dichloromethane:hexane and for F2 from 100-300 mL to 150-300 mL.
55	Section 6.3: Revised typical Silica cut point from 100 mL to 20 mL DCM for F2.

This table lists the details of the changes from revision 10 version 07 of this document.

Page	Change details
21	Table 4: Increased the concentration of the recovery standard solution 5X for aqueous and tissue samples to allow spiking of 10 µL instead of 50 µL.
24, 25	Tables 6a and 6b: Deleted the “low level” recovery standards.
57	Revised extract final blow down instructions for tissue and waters to reduce to a volume of 90 µL instead of to 50 µL.

This table lists the details of the changes from revision 10 version 06 of this document.

Page	Change details
11-12	Section A.1: Renumbered tables as Table 1a, b, c, and added description of List 1, 2, or 3 to each table.
27, 28	Sections 3.3 and 3.4: Added shelf lives where applicable.
28, 55	Sections 3.4 and 6.3: Updated alumina and silica supplier information
30	Section 4: Updated name of NIST 1647e CRM
31-34	Table 8: Increased blank acceptance limits for naphthalene in tissue and for perylene. Added aqueous, solids and tissue specific blank limits with values for carcinogenic PAH adjusted downward to laboratory mean + 3S limits. Added blank acceptance limits and typical SDLs for total alkylated PAHs.
43	Section 5.3.2: Changed the “keeper” to iso-octane
47	Section 5.5: Clarified that PCDD/F and PAH co-extraction is not an approved standard procedure and that Soxhlet Dean-Stark extraction of PAH from XAD-2 is never permitted. Added reference to SLA-105 for extraction of wound glass filters.
53	Section 5.11, point 7: Added toluene keeper to the extract before rotary evaporation.
92-96	Added Appendix G: Quantification of Client, Field and Photolysis Standards
97-98	Added Appendix H: Full listing of blank Mean + 3S limits for aqueous, solids and tissue

This table lists the details of the changes from revision 10 version 05 of this document.

Page	Change details
1, 86	Scope, Appendix F: Added information for the analysis of 2-chloronaphthalene by MLA-021 and GC-HRMS.
30-32	Table 8: Aligned the typical SDLs for PAH in aqueous samples with the current aqueous final extract volume 100 µL. Corrected erroneous name for benzo(ghi)perylene in the chromatogram quality specification.
39, 49	Sections 5.3.2 and 5.11: Updated lipid determination instructions to reflect current practice.

This table lists the details of the changes from revision 10 version 04 of this document.

Page	Change details
14	Table 3: Changed aqueous holding time from 30 days to 7 days to reflect current practice.
15	Section 2: Removed % moisture determination and Chlorophenol analysis comment from this

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	sample pre-treatment and preparation section.
22, 23, 30, 59, 61, 62, 71	Table 6a, table 6b, table 8, table 10, table 13: Added d8-dibenzothiophene to be used as the quantification reference for dibenzothiophene, 2/3-metyldibenzothiophene, 2,4-dimethyldibenzo-thiophene and C1-, C2-, C3- and C4-dibenzothiophenes.
24	Table 7: Deleted the lowest calibration solution level (10 ng/mL), renamed the remaining levels to be A through D.
26, 50	Sections 3.4 and 6.3: Added Sigma-Aldrich as an alternate supplier of silica gel for analysis of PAH and/or alkanes.
33	Table 9: Corrected Instrument Sensitivity typo: Should pertain to nC_{36}.
35, 36	Section 5.1: Specified sample sizes are wet weights.
48	Section 5.11: Updated lipid determination step to current practice (referring SLA-028).
52	Section 6.5.2: Clarified that the final extract volume of aqueous samples is 100 µL.
59	Table 10: Added footnote 6 about benzo(a)anthracene coeluting with cyclopenta(cd)pyrene.

This table lists the details of the changes from revision 10 version 03 of this document.

Page	Change details
17, 36	Sections 3.1.1 and 5.1.3: Specified filters.
30	Table 8: Added footnote explaining that due to limited availability of 1,7-dimethylphenanthrene, this compound may be absent from OPRs.
71	Appendix D added: Defined identification and quantification criteria for Alkylated PAH Compound Totals.
74	Appendix E added: Typical Chromatograms of Alkylated PAH Compound Totals
various	Added references to method performance validation data for Alkylated PAH Compound Totals and removed the reference to these as tentatively identified compounds.

This table lists the details of the changes from revision 10 version 02 of this document.

Page	Change Details
2	Added References section.
9-10, 12	Table 1a, Section B: Added notes that naphthalene and alkylated naphthalenes are reported as “maximum values” when determined in XAD-2 resin.
13	Section F: Added that the method has been validated to demonstrate that it is fit for the intended use
14	Table 3: Changed hold times for solid and tissue samples to be 1 year, and frozen storage to be at -20°C .
17	Section 3.1.1: Updated filter to current make (Pall A/E).
25	Section 3.3: Clarified that anhydrous sodium sulphate is used in granulate form, not powder.
29-30	Table 8: Added note that method blank limits don’t apply for naphthalene and alkylated naphthalenes in XAD-2 resin.
35, 37,38, 53	Sections 5.1, 5.2, 5.3 and 7.1.3: Added identification of a method default procedure versus an optional procedure.
39-41	Section 5.4: Clarified that current practice for aqueous samples is that suspended solids are separated from aqueous phase by centrifugation, not by filtration. Added step to visually inspect sample for suspended solids. Added reference to SLA-124 Liquid-Liquid Supplemental Techniques. Aligned % suspended solids limits for aqueous samples to follow EPA 1668C (to be ≤1% and >1%, instead of <1% and ≥1%). Specified that suspended solids may be estimated from visual inspection where appropriate.
34, 40, 49	Sections 5, 5.4 and 5.11: Added reference to SLA-124 Liquid-Liquid Supplemental Techniques
47	Section 5.10: Replaced procedural instructions by referring to SLA-123 “Splitting of Sample Extracts”.
58	Add section 9 Reporting Criteria and Practices
60	Added masses for C4-Naphthalene and C3-Phenanthrenes/Anthracenes
various	Renumbered some sections in accordance with updated method format standard.

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This table lists the details of the changes from revision 10 version 01of this document.

Page	Change Details
1	Updated EPA method reference codes to include EPA 8270D
28	Table 8. Clarified that ± 3 second RT windows apply to target compounds
28, 47	Standardized final extract volume for alkane analysis in all matrices to 400 µL
29	Re-stated the internal laboratory sensitivity check specification on the basis of the solution concentration and on C39, the compound of lowest response. Deleted the C24/C36 peak ratio requirement as this is redundant for the current analytical instrument operation.
43-44	Corrected blood/serum/plasma extraction protocol and lipid determination to current practice: ≥10 g sample size and 1:1:1:3 extraction solvent ratio.
61	Appendix B: Added summary of key attributes of MLA-021, EPA 8270C/D and EPA 1625B
62	Appendix C Added description of MS tuning and check procedures

This table lists the details of the changes from revision 09 of this document.

Page	Change Details
11, 30	Table 3 and Section 5.0: Clarified that all extracts must be protected from light by covering with aluminum foil.
26	Table 8: Corrected d10-acenaphthylene to be d8-acenaphthylene.
30	Section 5: Updated worksheet list
35, 36	Sections 5.4.2 and 5.4.3.1: Added magnetic stirring extraction is the default option for aqueous sample extraction.
54	Table 10: Changed 2-methylfluorene, 1,7-dimethylfluorene and C1-C3-fluorenes to use d10-phenanthrene as the quantification standard instead of d8-acenaphtylene.

This table lists the details of the changes from revision 08 of this document.

Page	Change Details
General	Grouped the analytes as “multi-point calibrated PAHs”, “single-point calibrated PAHs” and “alkanes” instead of as “Level P, “Level A and “Level H”.
	Introduction, section 5, Definitions: Added definition of “TIC”.
7-8	Changed Table 1 to reflect how PAH and alkylated PAHs are analyzed by multi-point calibration or by single-point calibration. Deleted 4,5-Methylene Phenanthrene as a target
16	Section 3.2.1: Added description of “Single Point Calibration Solution”.
18-20	Tables 6a and 6b: Divided former Table 6 into two tables, one for multi point calibration and the other for single point calibration.
24	Section 4: Updated the section about Reference Samples and CRMs.
25-26	Table 8: Added QC specifications for alkylated PAHs. Changed the acceptable matrix spike specification for fluorene to be 60 – 140% (from 70 – 130%). Added QC acceptance criteria for alkylated PAHs. Added benzo(b)fluoranthene and benzo(j/k)fluoranthene instead of “benzofluoranthenes”.
27, 29	Table 8 and Table 9: Changed the Duplicate specification wording to be “within X% of the mean (applicable to concentrations > 10 times the DL)”.
52	Section 10.1: Clarified that all alkanes, pristane and phytane are present in the calibration solutions.
54-57	Table 10: Added footnotes 4. and 5.

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This table lists the details of the changes from revision 07 of this document.

Page	Change Details
	Table 1: Added 4,5-Methylene Phenanthrene as a target
	Table 8: Updated to reflect the changes in Table 10
	Table 10: A number of surrogate standards and retention standards updated to reflect current practices. New target 4,5-Methylene Phenanthrene added.
	Updated to reflect the changes in Table 10.

This table lists the details of the changes from revision 06 of this document.

Page	Change Details
	Global Change: Deleted Appendix A and all references to sterols as analysis of sterols is no longer performed as part of this method. Added statement with each Biobead cleanup procedure that it is not a suitable cleanup procedure when alkanes analysis is required.
14-19	Section 3.2 Restructured this section to separate the preparation of standards for PAH from alkanes. Added Tables 4 & 5 to summarize volumes and amounts of standards spiked into each sample.
19	Table 7 Revised amounts of recovery standard in Cal. solutions and in recovery solution. Revised the compounds in recovery standard. Added additional native compounds to solutions.
21	Section 3.4 Added specification for two kinds of silica, only one is suitable for PAHs.
21, 44	Sections 3.5.3, 6.4.2 Changed the amount of alumina in cleanup column. Added caution in Section 6.4.2 that alumina column is not suitable for alkane fraction.
24	Table 8 Changed the specification for bracketing Cal.
27	Table 9 Revised sensitivity specification to be for 100 pg injected. Added specification for additional natives in SPM.
32	Section 5.3 Added caution that the procedure is not suitable for alkane analysis.
43	Section 6.3 Added note about the silica to use when doing alkanes.
47	Section 8.1.4 Added reference to PAH instrument method MIN-021.
48	Section 8.2.3 Revised final hold time for GC temp. program for alkanes. Added information about placing foil on autosampler vial.
54	Table 11 Revised recovery standard. Revised quantification references for odd numbered alkanes and for Norpristane. Added confirmation ion mz=71 for odd numbered alkanes.
57	Added Appendix A for analysis of hopanes.

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INTRODUCTION

A. ANALYTES OF INTEREST

The procedures described in this document are used to determine the concentrations of PAHs, alkylated PAHs and alkanes in environmental samples. PAHs, alkylated PAHs and alkanes are determined from a single extraction procedure. These compounds are isolated in different fractions collected during the chromatographic clean-up of the extract. The grouping of analysis, as described below, indicates to the analyst the group of target analytes to be analyzed and dictates the standards to be used and fractions to be collected.

The list of target analytes can be modified to meet specific client requests. Any changes to the routine target analytes list are documented by a Project QC Chemist, who communicates the details to the analytical staff.

The analytes are grouped in two groups:

Analysis of PAHs and alkylated PAHs using multi-point calibration
Analysis of Alkanes

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A.1 PAHs and Alkylated PAHs

The analysis of PAHs and alkylated PAHs is carried out using the same extraction and clean-up procedures, and is carried out by a single GC/MS analysis of the extract.

The analysis of PAHs and alkylated PAHs includes determination of some or all of the compounds listed below:

Table 1. PAHs and alkylated PAHs - determined by multi-point calibration [2]

List 1 Compounds

Naphthalene [1]	Benzofluoranthenes
Acenaphthylene	Benzo(e)pyrene
Acenaphthene	Benzo(a)pyrene
Fluorene	Perylene
Phenanthrene	Dibenzo(ah)anthracene
Anthracene	Indeno(1,2,3-cd)pyrene
Fluoranthene	Benzo(ghi)perylene
Pyrene	Dibenzothiophene
Benz(a)anthracene	2-Methylnaphthalene [1]
Chrysene	2,6-Dimethylnaphthalene [1]
Benzo(b)fluoranthene	2,3,5-Trimethylnaphthalene [1]
Benzo(j/k)fluoranthenes	1-Methylphenanthrene

List 2 Compounds

1-Methylnaphthalene [1]	C1-Phenanthrenes/Anthracenes
C1-Naphthalenes [1]	1,7-Dimethylphenanthrene
1,2-Dimethylnaphthalene [1]	1,8-Dimethylphenanthrene

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C2-Naphthalenes [1]
2,3,6-Trimethylnaphthalene [1]
C3-Naphthalenes [1]
1,4,6,7-Tetramethylnaphthalene [1]
C4-Naphthalenes [1]
2-Methylphenanthrene
3-Methylphenanthrene
9/4-Methylphenanthrenes
2-Methylanthracene
2,6-Dimethylphenanthrene
3,6-Dimethylphenanthrene
C2-Phenanthrenes/Anthracenes
1,2,6-Trimethylphenanthrene
C3-Phenanthrenes/Anthracenes
Retene
C4-Phenanthrenes/Anthracenes
Biphenyl

List 3 Compounds

C1-Biphenyls
C2-Biphenyls
C1-Acenaphthenes
2-Methylfluorene
C1-Fluorenes
1,7-Dimethylfluorene
C2-Fluorenes
C3-Fluorenes
2/3-Methyldibenzothiophenes
C1-Dibenzothiophenes
2,4-Dimethyldibenzothiophene
C2-Dibenzothiophenes
C3-Dibenzothiophenes
C4-Dibenzothiophenes
3-Methylfluoranthene/Benzo(a)fluorene
C1-Fluoranthenes/Pyrenes
C2-Fluoranthenes/Pyrenes
C3-Fluoranthenes/Pyrenes
C4-Fluoranthenes/Pyrenes
1-Methylchrysene
5/6-Methylchrysenes
C1-Benz(a)anthracenes/Chrysenes
5,9-Dimethylchrysene
C2-Benz(a)anthracenes/Chrysenes
C3-Benz(a)anthracenes/Chrysenes
C4-Benz(a)anthracenes/Chrysenes
7-Methylbenzo(a)pyrene
C1-Benzofluoranthenes/Benzopyrenes
C2-Benzofluoranthenes/Benzopyrenes

[1] Maximum value when determined in XAD-2 adsorbent.

[2] Optionally calibration may be performed by single-point bracketing calibration.

A.2 Alkanes

The analysis of alkanes is carried out using the same extraction procedures as for PAHs and alkylated PAHs, but requires different surrogate, recovery, and calibration standards, and the collection of a different fraction during the chromatographic cleanup of the extract. The analysis of alkanes is carried out by a separate GC/MS analysis. Note that alkane analysis can't be performed in the following matrices: XAD-2 adsorbent, stack gas samples and ambient air samples.

The analysis of alkanes includes the determination of some or all of the compounds listed below:

Table 2. Alkanes

Dodecane (nC12)
2,6-Dimethyl Undecane
Norfarnesane
Tridecane (nC13)
Heneicosane (nC21)
Docosane (nC22)
Tricosane (nC23)
Tetracosane (nC24)

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Farnesane
Tetradecane (nC14)
2,6,10-Trimethyl tridecane
Pentadecane (nC15)
Hexadecane (nC16)
Norpristane
Heptadecane (nC17)
Pristane
Octadecane (nC18)
Phytane
Nonadecane (nC19)
Eicosane (nC20)
Pentacosane (nC25)
Hexacosane (nC26)
Heptacosane (nC27)
Octacosane (nC28)
Nonacosane (nC29)
Triacontane (nC30)
Untriacontane (nC31)
Dotriacontane (nC32)
Tritriacontane (nC33)
Tetratriacontane (nC34)
Pentatriacontane (nC35)
Hexatriacontane (nC36)

B. CONTAMINATION AND INTERFERENCES

There is evidence to suggest that the alkane fraction (F1) is contaminated by contact with the autosampler vial cap liner. In order to eliminate the contamination, foil is used to cover the vial mouth before capping. Also, the cap is removed prior to injection of the extract onto the GC/MS, thus eliminating a source of contamination due to piercing of the cap. After GC/MS analysis, the foil cover is replaced with new foil and the vial recapped.

Contact with acid may result in protonation of the perdeuterated surrogate standards. Care must be taken to avoid contact with acid once the surrogate standard has been added to the sample.

Anti-bumping granules must be used in order to prevent “bumping” during Soxhlet extraction procedures, resulting in the lost of some volatile analytes.

The use of nitrile gloves during all procedures described in this method is prohibited due to evidence suggesting they are a source of background contamination for PAHs.

When this method is used to determine a non-routine list of PAHs that have been requested by the client, the presence of cyclopenta(c,d)pyrene and benzo(c)phenanthrene in samples may interfere with the quantification of benz(a)anthracene and benzo(ghi)fluoranthene respectively. Alternate quantification procedures, described in Section 10, may be used when this occurs.

Under the used GC conditions benzo[j]fluoranthene co-elutes with benzo[k]fluoranthene. These two analytes are reported together as benzo[j,k]fluoranthene.

Native benzo(b)fluoranthene and labeled d_{12}-benzo(k)fluoranthene elute at the same retention time, and there is a contribution from the labeled compound to the quantification ion (m/z 252) used for the native compound. This causes over-estimation of the native compound in the raw data, which can be significant in low-level samples. The routine data processing procedure uses responses observed in m/z 252 and 264 in the procedural blank to estimate labeled compound response in m/z 252 in each sample, which are subtracted from each native benzo(b)fluoranthene concentration calculation.

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Notwithstanding thorough cleaning procedures XAD-2 adsorbent from sampling equipment may contain significant residues of naphthalene and alkylated naphthalenes. Naphthalene and alkylkated naphthalene results from XAD-2 adsorbents are therefore reported as “maximum values”, i.e., as the estimated maximum possible concentrations/amounts in the sample.

The presence of cyclopenta(cd)pyrene will result in enhancement of the benz(a)anthracene confirming ion which may result in ratio failures for benz(a)anthracene.

C. SAFETY

Refer to SAF-001 “Safety Manual” for safety procedures.

D. POLLUTION PREVENTION AND WASTE MANAGEMENT

AXYS Analytical Services Ltd. complies with all federal, provincial and municipal regulations governing waste management, including land disposal restrictions and sewage discharge regulations. AXYS’ waste disposal procedures have been developed to comply with all pollution prevention regulations.

All standards are prepared in volumes consistent with volumes required by the method to minimize the disposal of standards.

Refer to SAF-001 “Safety Manual” and SAD-014 “Sample Disposal” for procedures for disposing of laboratory wastes.

E. DEFINITIONS

Refer to QDO-001 “AXYS Analytical Services QA/QC Policies and Procedures Manual” for definitions of terms used in this document.

F. METHOD PERFORMANCE

This method has been validated to demonstrate that it is fit for the intended use and the validation data is retained on file.

The measurement procedure for the Alkylated PAH Compound Total groups is inherently less certain than for individual compounds and a definitive reference sample with all possible components characterized does not exist. Detection limit, trueness and measurement uncertainty general estimates for Alkylated PAH Compound Totals, were derived from data for the associated alkylated PAH compounds.

Method performance is validated through analysis of matrix-specific reference samples, including spikes and certified reference materials. Ongoing method performance is monitored through QC samples analyzed alongside samples. The parameters monitored include percent recovery of labelled surrogates, blank concentrations, native compound recoveries and percent difference between duplicate tests.

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ANALYSIS PROCEDURE

1. SAMPLE PRESERVATION AND STORAGE

All samples must be received and stored under the conditions presented in Table 3. Where adherence to USEPA methods hold times is required, refer to the hold times summarized in SLA-003 "*Sample receipt and Log-in*".

Table 3. Sample Storage, Sample Receipt and Hold Time Requirements

Matrix	Sample size (per analysis)	Sample container [1]	Condition upon receipt	Storage conditions [2]	Sample holding time [3]	Extract holding time [4]	Preservation
Ambient air	PUF + filter	Glass	0-4ºC	-20°C, dark	Not defined	40 days	None required
Sample train (stack gas)	Containers A-F	Glass	0-4°C	4°C, dark	30 days	40 days	None required
Oil [5]	10-50 mg for PAH 0.25-2.5 mg for alkanes	Glass	Ambient	Ambient, dark	Not defined	40 days	None required
Sediment/ soil/ash	Up to 10 g dry (max. 20 g wet)	Glass	< 4°C	-20°C, dark	1 year	40 days	None required
Biosolids [6]	Up to 2 g dry (max. 20 g wet)	Glass	< 4°C	-20°C, dark	1 year	40 days	None required
Tissue/ vegetation	10 g wet 20-30 g wet	Glass	< 4°C	-20°C, dark	1 year	40 days	None required
Aqueous samples	1 L	Brown glass	0-4°C	4°C, dark	7 days	40 days	50 mg/L of sodium azide (field preserv.)
XAD-2 column	1 column Resin Particulate filter Wound glass filter	As is Glass Foil wrapped Foil wrapped	0-4°C 0-4°C < 4°C < 4°C	4°C, dark 4°C, dark -20°C, dark -20°C, dark	Not defined	40 days	None required
Solvent extract	As received		< 4°C	< 4°C, dark	Not defined	40 days	None required

[1] All glass containers should be organically clean, i.e., baked, solvent rinsed or purchased "certified" clean. All containers must be tightly sealed with screw cap lids (PTFE or foil-lined) to prevent loss of volatiles or contamination from volatiles. If samples are received in clear glass containers, they must be protected from light.

[2] Storage temperatures quoted are nominal temperatures. Samples stored at a nominal temperature of 4°C are permitted a variance of ±2°C, samples stored at -20 ºC are permitted a variance of ±4 ºC.

[3] Sample hold times are from time of sample receipt. There is no evidence to indicate that properly stored samples are not stable for longer periods of time. Client negotiated hold times or other method-specific hold times are adhered to.

[4] Holding times for extracts are from time of extraction, stored at <-10°C (the <-10°C storage requirement applies to final extracts, not to extracts in progress.) There is no evidence to indicate that properly stored samples are not stable for longer periods of time. Client negotiated hold times or other method-specific hold times are adhered to. All extracts must be protected from light by covering with aluminum foil.

[5] When both PAH and alkane analyses are required a single subsample may be used if a suitable split as approved by the Project Manager is performed.

[6] Significant judgment based on prior experience with particular samples and/or sample appearance may apply to sample size selection for biosolids due to the potential to overwhelm routine method clean-up capabilities.

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2. SAMPLE PRETREATMENT AND PREPARATION

Refer to the following Standard Operating Procedures:

Document ID	Title
SLA-011	Compositing Samples
SLA-012	Dissection of Samples
SLA-013	Procedures for Homogenization of Solid and Tissues
SLA-014	Thawing of Samples
SLA-015	Moisture Determination
SLA-043	Removing Sampling Media from Field Sampling Equipment
SLA-084	Preparation of Aqueous Samples for Extraction

Particulate Filter Papers

Handle particulate filter papers while frozen. Take care not to lose any of the particulate from the filter. Fold the filter paper to fit into a Soxhlet thimble.

Ambient Air (PUF and Filter)

If received in the sampling apparatus, use solvent rinsed forceps to withdraw the PUF from the sampler. The associated filter is typically received in a Petri dish or wrapped in foil. Use solvent rinsed forceps to handle the filter.

Solid (Sediment/Soils/Ash) Samples

Allow a solid sample to thaw prior to homogenization and subsampling procedures following standard operating procedure SLA-014. Homogenize the entire sample before subsampling for analysis following standard operating procedure SLA-013.

Tissue Samples

Allow a tissue sample to thaw prior to homogenization and subsampling procedures following standard operating procedure SLA-014. If the tissue sample is received as a homogenate, stir the homogenate well with a spatula prior to subsampling. Otherwise, homogenize tissue sample before subsampling for analysis following standard operating procedure SLA-013.

If required, determine the percent moisture of an accurately weighed subsample of (1 g) according to standard operating procedure SLA-015.

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Aqueous Samples

Refer to the standard operating procedure SLA-084 *Preparation of Aqueous Samples for Extraction* for sample preparation procedures.

XAD-2 Columns and Filters

Refer to the standard operating procedure SLA-043, *Removing Sampling Media from Field Sampling Equipment,* for details of removing the resin from the column. Filters are partially thawed prior to extraction.

SPMD Extracts

Refer to the standard operating procedure SLA-118, *Extraction (Dialysis) of SPMDs,* for details on preparing and dialyzing an SPMD to produce an extract.

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3. MATERIALS & REAGENTS

Refer to the following Standard Operating Procedures:

Document ID	Title
SLA-001	Cleaning Laboratory Items
SLA-002	Glassware and Laboratory Equipment Proofs
SLA-009	Preparation of Standards
SLA-018	Solvent Rinsing of Glassware for Organic Analysis
SLA-022	Use of Drying Ovens and Muffle Furnace
SLA-023	Use of Balances
SLA-036	Cleaning GC/MS and GC/ECD Microvials
SLA-041	Reagent Preparation
SLA-044	Activation of Copper Foil
SLA-093	Baking of Anhydrous Sodium Sulphate
SLA-094	Proofing Laboratory Equipment, Sampling Media, Solvents and Sample Containers
SQA-003	Standard Solution Validation
SQA-009	Storage and Control of Standards

3.1 Equipment List

3.1.1 Extraction

Separatory funnels (125 mL, 500 mL, 1000 mL, 2000 mL)
Erlenmeyer flasks (250 mL, 500 mL, 1000 mL, 2000 mL)
“Stirlenmeyer” flasks (2 L), with attached stopcock (Erlenmeyer flasks modified to allow for efficient extraction of aqueous samples by magnetic stirring)
Round bottom flasks (100 mL, 250 mL, 500 mL, 1000 mL)
Kuderna-Danish flasks (250 mL, 500 mL)
Beakers
Graduated Cylinders
Class A volumetric flasks, pipettes
Disposable pipettes
Disposable centrifuge tubes (15 mL)
Syringes (5 µL, 25 µL, 50 µL, 100 µL, 250 µL)
Autosampler vials (amber glass, 800 µL)
Silanized glass wool
Filter paper – PALL, glass fiber, 1.0 µm, type A/E, 102 mm, or equivalent
Filter paper – Ahlstrom, glass fiber, 1.1 µm, grade 161, 42.5 mm, or equivalent, baked before use.
Teflon tape
Spatula – stainless steel
Rotary evaporator, with water bath at 30°C

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Magnetic stirring plate
Soxhlet apparatus w/heating mantle
Dean-Stark Soxhlet apparatus/heating mantle
Water bath capable of maintaining up to 55ºC
Balance – Top loading and analytical (2, 3, and 4-place)
Glass chromatographic columns
Glassware ovens
Drying ovens
Muffle furnace
Nitrogen source with manifold apparatus

3.1.2 Instruments

Hewlett Packard 5890 Series II or Agilent 6890N Gas Chromatograph, or equivalent
Agilent 5973 Network mass selective detector (MSD), or equivalent
GC Columns: RT_X-5 (30 m, 0.25 mm i.d., 0.25 µm film thickness)

3.1.3 Glassware Cleaning

All glassware used in the preparation of reagents and in the analytical procedures must be organically clean. Glassware, including Soxhlet extraction equipment, must be washed, baked and solvent-rinsed according to standard operating procedures SLA-001 and SLA-018.

If baked glassware is not available, glassware must be washed and solvent rinsed according to standard operating procedure SLA-018.

Allow glassware to dry.

3.2 Standard Solutions

The analyses of PAHs and alkanes by GC/MS require the use of the surrogate, recovery, native and calibration standards described in Sections 3.2.1 and 3.2.2. Details of the preparation, composition and validation of concentrations of these standards are described on Standard Data Sheets. The chemist must refer to the Batch List for the names of the standard solutions used and the volumes added to each sample. Tables 4 and 5 present typical spiking volumes and amounts added for PAH and alkane analyses.

3.2.1 Standards for Analysis of PAHs

3.2.1.1 Surrogate Standard Solution

Samples are spiked with a suite of perdeuterated surrogate standards prior to extraction procedures. The surrogate standards for the analysis of PAHs are in a spiking solution with the typical concentrations presented in Tables 6a and 6b. There are two surrogate spiking solutions, a low-level solution typically used for tissue and clean water samples and a high-level solution is used for all other matrices. The amount of surrogate added is dependent upon sample size, final extract volume, and concentration of analytes in the sample and may be

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adjusted accordingly. A third surrogate spiking solution is used for SPMD analysis (see Appendix G). This surrogate does not contain the two labeled PAH that are used as PRC standards (field standards) in this matrix that are in the regular surrogate solution.

3.2.1.2 Recovery Standard

Sample extracts are spiked with perdeuterated recovery standards prior to instrumental analysis. The recovery standard solution is prepared at concentrations to contain the nominal amounts when spiking the volumes specified in Table 4. The amount of recovery standard added is designed to match the amount of surrogate in the final extract.

3.2.1.3 Multi Level Calibration Solutions

A series of five calibration solutions containing native analytes, surrogates standards and recovery standards is used to establish linearity of the analytical instrument. The concentration of the native analytes in the solutions varies to encompass the working range of the instrument, while the concentrations of the surrogates and recovery standards remain constant. A mid-level solution is analyzed every 12 hours. The solutions are prepared to have the nominal concentrations of native and labelled compounds shown in Table 6.

3.2.1.4 Single Point Calibration Solution

A single point calibration solution containing native analytes, surrogates standards and recovery standards may be used as an optional single-point calibration method of the analytical instrument. The single point calibration solution is analyzed at least once every 12 hours, before and after the GC/MS analysis of a batch of samples, and the mean RRF of the bracketing runs is used to quantify samples. The solution is prepared to have the same nominal concentrations of native and labelled compounds as the Mid-Level (Level E) calibration solution in Table 6.

3.2.1.5 Native Spiking Standard

An aliquot of native spiking solution is added to an unspiked reference material (USM) to prepare a spiked reference sample as a QC sample. The standards are prepared to have the nominal concentrations of native and labelled compounds shown in Table 4.

The native spiking standard solution is also used to prepare MS/MSD (Matrix Spike/Matrix Spike Duplicate) samples as requested by clients. Any MS/MSD overspiking (i.e., spiking at higher levels than the OPR) should be restricted to only List 1 and List 2 compounds.

Table 4. Typical Standard Spiking Levels for PAH Analysis

	High Level (Solids, Other matrices)		**Low Level (Tissue, water)**	
	Volume of Std (µL)	Amount Added (ng)	Volume of Std (µL)	Amount Added (ng)
Native Std	100	2000	100	200
Surrogate Std	100	2000	100	200
Recovery Std	100	2000	10	200

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3.2.2 Standards for Analysis of Alkanes

3.2.2.1 Surrogate Standard

The analysis of alkanes requires the use of the surrogate standards presented in Table 7. Samples are spiked with an amount of surrogate standard solution that is matrix dependent. The amount of surrogate added is dependent upon sample size, final extract volume, and concentration of analytes in the sample and may be adjusted accordingly.

3.2.2.2 Recovery Standard

Sample extracts are spiked with perdeuterated recovery standard prior to instrumental analysis. The recovery standard solution is prepared to have the nominal concentration presented in Table 5.

3.2.2.3 Calibration Solutions

A series of four calibration solutions (Level B through Level E) containing native analytes, surrogates standards and recovery standards is used to establish linearity of the analytical instrument. The concentration of the native analytes in the solutions varies to encompass the working range of the instrument, while the concentrations of the surrogates and recovery standards remain constant. A mid-level solution is analyzed every 12 hours. The standards are prepared to have the nominal concentrations of native and labelled compounds shown in Table 7.

3.2.2.4 Native Spiking Standard

An aliquot of native spiking solution is added to an unspiked reference material (USM) to prepare a spiked reference sample as a QC sample. The standards are prepared to have the nominal concentrations of native and labelled compounds shown in Table 5.

Table 5. Typical Standard Spiking Levels for Alkane Analysis

	High Level (Solids, Other Matrices)		**Low Level (Tissue)**	
	Volume of Std (µL)	Amount Added (ng)	Volume of Std (µL)	Amount Added (ng)
Native Std	250	1000	100	400
Surrogate Std	250	1000-2000	100	400-800
Recovery Std	25	500	10	200

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Table 6. Analysis of PAHs/Alkylated PAHs, Nominal Concentrations of Calibration Standard Solutions (ng/mL)

TARGET ANALYTE	Level A (Sens. Std) (ng/mL)	Concentration of Calibration Standard Solutions					Conc. of Native Std (Low Level) (ng/mL)	Conc. of Native Std (High Level) (ng/mL)
		Level B (ng/mL)	Level C (ng/mL)	Level D (ng/mL)	Level E (Mid-level) (ng/mL)	Level F (ng/mL)		
Acenaphthene	10	50	100	500	2000	5000	2000	20 000
Acenaphthylene	10	50	100	500	2000	5000	2000	20 000
Anthracene	10	50	100	500	2000	5000	2000	20 000
Benz[a]anthracene	10	50	100	500	2000	5000	2000	20 000
Benzo[b]fluoranthene	10	50	100	500	2000	5000	2000	20 000
Benzo[k]fluoranthene	10	50	100	500	2000	5000	2000	20 000
Benzo[ghi]perylene	10	50	100	500	2000	5000	2000	20 000
Benzo[a]pyrene	10	50	100	500	2000	5000	2000	20 000
Benzo[e]pyrene	10	50	100	500	2000	5000	2000	20 000
Biphenyl	10	50	100	500	2000	5000	2000	20 000
Chrysene	10	50	100	500	2000	5000	2000	20 000
Dibenzo[ah]anthracene	10	50	100	500	2000	5000	2000	20 000
2,6-Dimethylnaphthalene	10	50	100	500	2000	5000	2000	20 000
Fluoranthene	10	50	100	500	2000	5000	2000	20 000
Fluorene	10	50	100	500	2000	5000	2000	20 000
Indeno[1,2,3-cd]pyrene	10	50	100	500	2000	5000	2000	20 000
1-Methylnaphthalene	10	50	100	500	2000	5000	2000	20 000
2-Methylnaphthalene	10	50	100	500	2000	5000	2000	20 000
1-Methylphenanthrene	10	50	100	500	2000	5000	2000	20 000
Naphthalene	10	50	100	500	2000	5000	2000	20 000
Perylene	10	50	100	500	2000	5000	2000	20 000
Phenanthrene	10	50	100	500	2000	5000	2000	20 000
Pyrene	10	50	100	500	2000	5000	2000	20 000
2,3,5-Trimethylnaphthalene	10	50	100	500	2000	5000	2000	20 000
Dibenzothiophene	10	50	100	500	2000	5000	2000	20 000
3,6-Dimethylphenanthrene	10	50	100	500	2000	5000	2000	20 000
Retene	10	50	100	500	2000	5000	2000	20 000
2-Methylanthracene	10	50	100	500	2000	5000	2000	20 000
1,2-Dimethylnaphthalene	10	50	100	500	2000	5000	2000	20 000
2-Methylphenanthrene	10	50	100	500	2000	5000	2000	20 000
1,2,6-Trimethylphenanthrene	10	50	100	500	2000	5000	2000	20 000
2,3,6-Trimethylnaphthalene	10	50	100	500	2000	5000	2000	20 000
1,7-Dimethylphenanthrene	10	50	100	500	2000	5000	2000	20 000
1,4,6,7-Tetramethylnaphthalene	10	50	100	500	2000	5000	2000	20 000
2-Methylfluorene	10	50	100	500	2000	5000	2000	20 000
1,7-Dimethylfluorene	10	50	100	500	2000	5000	2000	20 000
2-Methyldibenzothiophene	10	50	100	500	2000	5000	2000	20 000
2,4-Dimethyldibenzothiophene	10	50	100	500	2000	5000	2000	20 000
5,9-Dimethylchrysene	10	50	100	500	2000	5000	2000	20 000
7-Methylbenzo(a)pyrene	10	50	100	500	2000	5000	2000	20 000
3-Methylfluoranthene	10	50	100	500	2000	5000	2000	20 000
6-Methylchrysene	10	50	100	500	2000	5000	2000	20 000
1-Methylchrysene	10	50	100	500	2000	5000	2000	20 000

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Table 6 (cont'd)

LABELLED SURROGATE STANDARDS	Level A (Sens. Std) (ng/mL)	Concentration of Calibration Standard Solutions					Conc. of Surrogate Std (Low Level) (ng/mL)	Conc. of Surrogate Std (High Level) (ng/mL)
		Level B (ng/mL)	Level C (ng/mL)	Level D (ng/mL)	Level E (Mid-level) (ng/mL)	Level F (ng/mL)		
d_8-Naphthalene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{10}-2-Methylnaphthalene	2000	2000	2000	2000	2000	2000	2000	20 000
d_8-Acenaphthylene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{10}-Phenanthrene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{10}-Fluoranthene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Benz[a]anthracene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Chrysene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-2,6-Dimethylnaphthalene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Benzo[b]fluoranthene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Benzo[k]fluoranthene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Benzo[a]pyrene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Perylene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Indeno[1,2,3-cd]pyrene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{14}-Dibenzo[ah]anthracene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Benzo[ghi]perylene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{10}-Biphenyl	2000	2000	2000	2000	2000	2000	2000	20 000
d_8-Dibenzothiophene	2000	2000	2000	2000	2000	2000	2000	20 000
LABELLED RECOVERY STANDARDS							**Conc. of Recovery Std (ng/mL)**	
d_{10}-Acenaphthene	2000	2000	2000	2000	2000	2000	20 000	
d_{10}-Pyrene	2000	2000	2000	2000	2000	2000	20 000	
d_{12}-Benzo[e]pyrene	2000	2000	2000	2000	2000	2000	20 000	

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Table 7. Analysis of Alkanes - Nominal Concentrations of Standard Solutions (ng/mL)

TARGET ANALYTES	Concentration of Calibration Standard Solutions				Conc. of Native Std (ng/mL)
	Level A (Sens. Std) (ng/mL)	Level B (Mid-level) (ng/mL)	Level C (ng/mL)	Level D (ng/mL)	
Decane (nC10)	100	1000	5000	10 000	4000
Undecane (nC11)	100	1000	5000	10 000	4000
Dodecane (nC12)	100	1000	5000	10 000	4000
Tridecane (nC13)	100	1000	5000	10 000	4000
Tetradecane (nC14)	100	1000	5000	10 000	4000
Pentadecane (nC15)	100	1000	5000	10 000	4000
Hexadecane (nC16)	100	1000	5000	10 000	4000
Pristane [1]	100	1000	5000	10 000	4000
Heptadecane (nC17)	100	1000	5000	10 000	4000
Octadecane (nC18)	100	1000	5000	10 000	4000
Phytane [1]	100	1000	5000	10 000	4000
Nonadecane (nC19)	100	1000	5000	10 000	4000
Eicosane (nC20)	100	1000	5000	10 000	4000
Heneicosane (nC21)	100	1000	5000	10 000	4000
Docosane (nC22)	100	1000	5000	10 000	4000
Tricosane (nC23)	100	1000	5000	10 000	4000
Tetracosane (nC24)	100	1000	5000	10 000	4000
Pentacosane (nC25)	100	1000	5000	10 000	4000
Hexacosane (nC26)	100	1000	5000	10 000	4000
Heptacosane (nC27)	100	1000	5000	10 000	4000
Octacosane (nC28)	100	1000	5000	10 000	4000
Nonacosane (nC29)	100	1000	5000	10 000	4000
Triacontane (nC30)	100	1000	5000	10 000	4000
Untriacontane (nC31)	100	1000	5000	10 000	4000
Dotriacontane (nC32)	100	1000	5000	10 000	4000
Tritriacontane (nC33)	100	1000	5000	10 000	4000
Tetratriacontane (nC34)	100	1000	5000	10 000	4000
Pentatriacontane (nC35)	100	1000	5000	10 000	4000
Hexatriacontane (nC36)	100	1000	5000	10 000	4000
LABELLED SURROGATE STANDARDS					**Conc. of Surrogate Std (ng/mL)**
d_{22}-Decane (nC10) [2]	2000	2000	2000	2000	4000
d_{26}-Dodecane (nC12) [2]	2000	2000	2000	2000	4000
d_{34}-Hexadecane (nC16) [2]	2000	2000	2000	2000	4000
d_{50}-Tetracosane (nC24)	2000	2000	2000	2000	4000
d_{74}-Hexatriacontane (nC36) [2]	4000	4000	4000	4000	8000
LABELLED RECOVERY STANDARDS					**Conc. of Recovery Std (ng/mL)**
d_{10}-Pyrene	1000	1000	1000	1000	20000

[2] Monitored as a quality control measure, not used for quantification.

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3.3 Preparation of Reagents

Activated Copper is freshly prepared as described in SLA-044.

Anhydrous Sodium Sulphate (Na_2SO_4, granular, 12-60 mesh, J.T. Baker, or demonstrated equivalent approved for use by Operations management and QA/QC) is cleaned by baking as described in SLA-093. Shelf life 1 month, after expiry rebake and re-use.

Anti-Bumping Granules are baked for 8 hours at a minimum of 300°C prior to use.

Hydrochloric Acid (HCl, 1M) is prepared by adding hydrochloric acid (HCl, conc. 100mL) to 1L ultra pure water. Shelf life 2 years, discard when expired.

Potassium Hydroxide Solution (KOH, 50% w/w) is prepared by mixing equal quantities (by weight) of potassium hydroxide (Fisher, certified grade) and ultra pure water. The solution is extracted by shaking with dichloromethane (2 x 100 mL) and hexane (100 mL) before use. Shelf life 1 week, discard after expiry.

Sodium Hydroxide Solution (NaOH, 0.1M) is prepared by dissolving sodium hydroxide pellets (4 g, AR grade) in ultra pure water (1L). The solution is extracted by shaking with dichloromethane (2 x 100 mL) and hexane (100 mL) before use. Shelf life 3 months, after expiry re-extract and re-use.

Sodium Hydroxide Solution (NaOH, 6N) is prepared by dissolving sodium hydroxide pellets (240g, AR graded) in ultra pure water (1L). The solution is extracted by shaking with dichloromethane (2 x 100 mL) and hexane (100 mL) before use. Shelf life 3 months, discard after expiry.

Solvents: All solvents used are high purity, distilled in glass solvents, either HPLC or pesticide residue grade. Each lot number of solvent must be checked for impurities by performing a solvent proof prior to use. All solvent mixtures are prepared on a volume:volume basis by mixing the appropriate proportions of each solvent. Shelf life 2 years for solvent mixtures, discard after expiry.

Ultra pure Water (Seastar Chemicals, contaminant-free) is used as received. Indefinite shelf life.

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3.4 Preparation of Chromatography Materials

Refer to the following Standard Operating Procedures:

Document ID	Title
SCH-001	Deactivation/Activation Procedures
SCH-002	Column Packing Procedures
SCH-003	Column Cutpoint Procedures
SCH-004	Layered Silica Gel Chromatography Procedures
SCH-005	Preparation of Carbon/Celite Column
SCH-006	Preparation and Maintenance of Biobead Columns

Biobeads (SX-3, Bio-Rad, 200-400 mesh) are soaked in 1:1 dichloromethane:hexane for 24 hours prior column preparation, as described in SCH-006.

Silica gel (Mallinckrodt, SilicAR, 100-200 mesh, or alternatively Sigma-Aldrich, high-purity grade, 70-230 mesh, catalogue no. 60741) is heated for 8 hours at 450°C, cooled, and deactivated with ultra pure water (5% by weight), as described in SCH-001. Cutpoints are determined prior to use (SCH-003). **This silica is suitable for the analysis of PAHs and/or alkanes.** Shelf life 3 months, after expiry redetermine cutpoints.

Silica gel (SiliCycle G60, 60-200 µm 60 Å or equivalent) is heated for 8 hours at 450°C, cooled, and deactivated with ultra pure water (5% by weight), as described in SCH-001. Cutpoints are determined prior to use (SCH-003). **This silica is suitable for the analysis of PAHs only. Do not use if alkanes are required.** Shelf life 3 months, after expiry redetermine cutpoints.

Alumina (Fisher, Basic, 60-325 mesh, or equivalent) is baked for a minimum of 8 hours at 450°C and then deactivated with ultra pure water (typically 2% by weight) as described in SCH-001. The degree of activation may vary depending upon the batch of alumina. Cutpoints are determined prior to use (SCH-003). Shelf life 6 months, after expiry redetermine cutpoints.

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3.5 Preparation of Chromatography Columns

3.5.1 Biobead Column (Suitable for PAH Analysis only)

A Biobead (SX-3, 60 g) column is prepared and cutpoints determined as described in SCH-006. The column is cleaned with 20% methanol:dichloromethane and 1:1 dichloromethane:hexane prior to use, as described in SCH-006.

3.5.2 Silica Gel Column

A silica gel column is prepared by dry packing silica gel (10 g, 5% deactivated) into a glass chromatographic column (1 cm x 40 cm with 100 mL reservoir) with pentane, as described in SCH-002.

3.5.3 Alumina Column

Basic alumina (8 g, 2% deactivated) is dry packed into hexane in a glass chromatographic column (1 cm x 12 cm with 50 mL reservoir) as described in SCH-002. The column is packed with a 1 cm bed of anhydrous granular sodium sulphate and eluted with hexane 10 mL.

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4. QUALITY ASSURANCE/QUALITY CONTROL

Refer to the following Standard Operating Procedures:

Q-Pulse ID	Title
SQA-013	Preparation, Storage, and Control of Reference Materials
SLA-016	Preparation of Quality Control Samples

All samples are analyzed in batches. The composition of a batch is detailed on the Batch List. Each batch has the following composition:

- Batch Size - Each batch consists of up to twenty test samples and additional QA/QC samples.
- Blanks - One procedural blank is analyzed with each batch.
- Duplicates – Sample duplicates are analyzed (provided sufficient sample is available) for batches with 7-20 test samples, or as required by contract. For some sample matrices (XAD-2 columns, air samples, SPMD extracts) only field duplicates can be analyzed.
- Reference Samples – Reference samples are relied on to demonstrate the accuracy of the data. A reference sample is either a spiked matrix sample (SPM) or certified reference material (CRM). One reference sample is analyzed with each batch. A spiked matrix sample is prepared by adding an aliquot(s) of authentic spiking solution to a sample matrix known to contain low background levels of target analytes. The type of reference material to be analyzed is assigned to the analyst when the batch is assigned.
- CRMs are used to periodically verify accuracy, examples include:

  NRC HS-4/5/6, NIST 1941/1944 (Sediment)
  NIST 1647e (Solvent)
  NIST 2977 Mussels

- Matrix Spike/Matrix Spike Duplicate – these samples are analyzed upon client request.
- Surrogate/Authentic/Recovery (SAR) solution is an optional diagnostic test that may be prepared and analyzed with a batch.

The batch composition may vary should batch or quality control requirements be specified by a client.

Elements of the QA/QC program at AXYS Analytical Services are documented in the most recent version of the Quality Manual, "QA/QC Policies and Procedures Manual" (QDO-001).

The quality control limits for duplicate samples, procedural blanks, reference samples, surrogate recoveries, and detection limits are specified in Tables 8 and 9.

Note: Due to the difficulty in recovering PAHs from some ash samples, it is not always possible to meet method specifications for this matrix.

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Table 8. PAHs - QC Acceptance Criteria

MATRIX	Typical Sample Specific * Detection Limits							Procedural Blank Level				Acceptable Matrix Spike
	Solid	Aqueous	Tissue	XAD-2 Column	PUF & SPMD Extract	Filter	Oil	Solid & SPMD Extract	Aqueous	Tissue	All other matrices	
Analyte:	ng/g	ng/L	ng/g	ng	ng	ng	ng/g	ng (tot)	ng (tot)	ng (tot)	ng (tot)	% Recovery
Naphthalene	0.5	1	0.1	5	5	5	50	≤ 10	≤ 10	≤ 50	≤ 10 [1]	70-130
Acenaphthylene	0.5	1	0.1	5	5	5	50	≤ 5	≤ 5	≤ 5	≤ 5	70-140
Acenaphthene	0.5	1	0.1	5	5	5	50	≤ 5	≤ 5	≤ 5	≤ 5	70-130
Fluorene	0.5	1	0.1	5	5	5	50	≤ 5	≤ 5	≤ 5	≤ 5	60-140
Phenanthrene	0.5	1	0.1	5	5	5	50	≤ 10	≤ 10	≤ 10	≤ 10	70-130
Anthracene	0.5	1	0.1	5	5	5	50	≤ 5	≤ 5	≤ 5	≤ 5	70-130
Fluoranthene	0.5	1	0.1	5	5	5	50	≤ 5	≤ 5	≤ 5	≤ 5	70-130
Pyrene	0.5	1	0.1	5	5	5	50	≤ 5	≤ 5	≤ 5	≤ 5	70-130
Benz(a)anthracene	0.5	1	0.1	5	5	5	50	≤ 1.9	≤ 1	≤ 0.9	≤ 5	70-130
Chrysene	0.5	1	0.1	5	5	5	50	≤ 3.0	≤ 1	≤ 1.3	≤ 5	70-130
Benzo(b)fluoranthene	0.5	1	0.1	5	5	5	50	≤ 4.2	≤ 0.6	≤ 1.6	≤ 5	70-130
Benzo(j/k)fluoranthenes	0.5	1	0.1	5	5	5	50	≤ 2.1	≤ 0.4	≤ 1.2	≤ 5	70-130
Benzo(e)pyrene	0.5	1	0.1	5	5	5	50	≤ 5	≤ 5	≤ 5	≤ 5	70-130
Benzo(a)pyrene	0.5	1	0.1	5	5	5	50	≤ 3.6	≤ 0.9	≤ 0.7	≤ 5	70-130
Perylene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	70-130
Dibenzo(ah)anthracene	1.0	2	0.2	10	10	10	100	≤ 2.8	≤ 0.7	≤ 0.7	≤ 5	70-130
Indeno(1,2,3-cd)pyrene	1.0	2	0.2	10	10	10	100	≤ 2.8	≤ 0.8	≤ 1.5	≤ 5	70-130
Benzo(ghi)perylene	1.0	2	0.2	10	10	10	100	≤ 2.4	≤ 0.9	≤ 0.7	≤ 5	70-130
Biphenyl	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	70-130
Dibenzothiophene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	60-140
1-Methylnaphthalene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5 [1]	70-130
2-Methylnaphthalene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5 [1]	70-130
2,6-Dimethylnaphthalene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10 [1]	70-130
1,2-Dimethylnaphthalene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10 [1]	60-140
2,3,5-Trimethylnaphthalene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10 [1]	50-150

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2,3,6-Trimethylnaphthalene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10 [1]	50-150
1,4,6,7-Tetramethyl-naphthalene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10 [1]	50-200
2-Methylanthracene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
3-Methylphenanthrene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	N.A.
2-Methylphenanthrene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	50-150
9/4-Methylphenanthrenes	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	N.A.
1-Methylphenanthrene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	50-150
3,6-Dimethylphenanthrene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	50-150
2,6-Dimethylphenanthrene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	N.A.
1,7-Dimethyl-phenanthrene [2]	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	50-150
1,8-Dimethylphenanthrene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	N.A.
1,2,6-Trimethyl-phenanthrene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	50-150
Retene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
2-Methylfluorene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
1,7-Dimethylfluorene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
2/3-Methyldibenzo-thiophenes	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	50-150
2,4-Dimethyl-dibenzothiophene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
3-Methylfluoranthene [3]/ Benzo(a)fluorene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
5/6-Methylchrysenes	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
1-Methylchrysene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
5,9-Dimethylchrysene [4]	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
7-Methylbenzo(a)pyrene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
C1-Biphenyls	1.0	2	0.2	10	10	10	100	≤ 50	≤ 50	≤ 50	≤ 50	
C2-Biphenyls	1.0	2	0.2	10	10	10	100	≤ 250	≤ 250	≤ 250	≤ 250	
C1-Naphthalenes	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10 [1]	
C2-Naphthalenes	1.0	2	0.2	10	10	10	100	≤ 50	≤ 50	≤ 50	≤ 50 [1]	
C3-Naphthalenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15 [1]	
C4-Naphthalenes	1.0	2	0.2	10	10	10	100	≤ 25	≤ 25	≤ 25	≤ 25 [1]	
C1-Acenaphthenes	0.5	1	0.1	5	5	5	50	≤ 10	≤ 10	≤ 10	≤ 10	
C1-Fluorenes	1.0	2	0.2	10	10	10	100	≤ 25	≤ 25	≤ 25	≤ 25	

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C2-Fluorenes	1.0	2	0.2	10	10	10	100	≤ 50	≤ 50	≤ 50	≤ 50	
C3-Fluorenes	1.0	2	0.2	10	10	10	100	≤ 20	≤ 20	≤ 20	≤ 20	
C1-Dibenzothiophenes	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	
C2-Dibenzothiophenes	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	
C3-Dibenzothiophenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
C4-Dibenzothiophenes	1.0	2	0.2	10	10	10	100	≤ 25	≤ 25	≤ 25	≤ 25	
C1-Phenanthrenes/ Anthracenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
C2-Phenanthrenes/ Anthracenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
C3-Phenanthrenes/ Anthracenes	1.0	2	0.2	10	10	10	100	≤ 25	≤ 25	≤ 25	≤ 25	
C4-Phenanthrenes/ Anthracenes	1.0	2	0.2	10	10	10	100	≤ 25	≤ 25	≤ 25	≤ 25	
C1-Fluoranthenes/ Pyrenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
C2-Fluoranthenes/ Pyrenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
C3-Fluoranthenes/ Pyrenes	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	
C4-Fluoranthenes/ Pyrenes	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	
C1-Benz(a)anthracenes/ Chrysenes	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	
C2-Benz(a)anthracenes/ Chrysenes	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	
C3-Benz(a)anthracenes/ Chrysenes	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	
C4-Benz(a)anthracenes/ Chrysenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
C1-Benzofluoranthenes/ Benzopyrenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
C2-Benzofluoranthenes/ Benzopyrenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
Typical Sample Size	10 g	1 L	10 g	1 col	1 sample	1 filter	0.1 g					
Typical Final Volume (µL)	500	100	100	500	500	500	500					

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* Detection limits quoted are those routinely achieved. Detection limits for alkylated PAH Compound Totals are based on detection of a single component.

[1] Procedural blank level limits don't apply for naphthalene and alkylated naphthalene from XAD-2 resin.

[2] Due to limited availability of 1,7-dimethylphenanthrene standard, this compound may be absent from OPR samples.

[3] Due to limited availablilty of 3-methylfluoranthene standard, this compound may be absent from OPR samples. It may be substituted by 2-methylfluoranthene as the exemplar alkylfluoranthene in the OPR.

[4] Due to limited availablilty of 5,9-dimethylchrysene standard, this compound may be absent from OPR samples. It may be substituted by other alternative dimethylchrysene isomers as the exemplar C2/C3/C4-benz(a)anthracene/chrysene in the OPR; in such cases the recovery specification for 5,9-dimethylchrysene would not apply.

NOTE: Reference samples are unavailable for Alkylated PAH Compound Totals, data acceptability evaluation is based on recoveries of the associated individual alkylated PAH compounds

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Table 8 (cont'd)

SURROGATE STANDARD RECOVERIES:	% RECOVERY RANGES ALL MATRICES
d_8-naphthalene	15 – 130
d_8-acenaphthylene	20 – 130
d_{10}-phenanthrene	30 – 130
d_{10}-fluoranthene *	30 – 130
d_{12}-benz[a]anthracene	30 – 130
d_{12}-chrysene	30 – 130
d_{12}-benzo[b]fluoranthene	30 – 130
d_{12}-benzo[k]fluoranthene	30 – 130
d_{12}-benzo[a]pyrene	30 – 130
d_{12}-perylene	30 – 130
d_{14}-dibenz[ah]anthracene *	30 – 130
d_{12}-indeno[1,2,3-cd]pyrene	30 – 130
d_{12}-benzo[ghi]perylene	30 – 130
d_{10}-2-methylnaphthalene	20 – 130
d_{12}-2,6-dimethylnaphthalene	20 – 130
d_{10}-biphenyl	15 – 130
d_8-dibenzothiophene	30 – 130

* Compound not used as surrogate standard for SPMD analysis. Surrogate recovery specifications do not apply for SPMD samples.

Performance Reference Compound (PRC) and Photolysis Standard Recovery Range Specifications for SPMD OPR samples: These ranges can also be used as guidance limits for any Client Day 0 and Field or Trip Blank Samples.

PRC	%RECOVERY RANGES
d_{10}-fluoranthene	70 – 130
d_{14}-dibenz[ah]anthracene	70 – 130
d_{10}-anthracene	70 – 130

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Table 8 (cont'd)

Parameter	Acceptance Specification
Procedural Blank	Refer to Table 8 above, or 5 times lower than analogous analyte value detected in the samples.
Analysis Duplicate	Duplicates must fall within ±20% of the mean (applicable to concentrations >10 times the DL) These are guidelines – departures based on professional judgement allowed. (Note that ±20% of the mean is equivalent to 40 relative percent difference)
Instrument Sensitivity	S/N 3:1 for 10 pg of acenaphthene, dibenzo(a,h)anthracene.
Instrument Resolution	Calibration gas PFTBA (FC43) unit mass resolution at m/e 69/70 and 219/220, Unit mass resolution is demonstrated by the presence of a resolved peak at m/z 70 and m/e 220.
Instrument Linearity	Linearity is demonstrated by a 5-point calibration over the working concentration range with a relative standard deviation of the RRFs ≤20% for targets with a labelled analog present and all labelled compounds, ≤35% for targets with no labelled analog present.
Continuing Cal Ver	Opening Cal Ver: Concentrations of native compounds and labelled surrogates must be within ±25% of expected values for all targets. Closing Cal Ver: Concentrations of native compounds must be within ±25% of expected values. Concentrations of labelled surrogates must be within ±25% of expected values, with any two (2) values allowed to be within ±40%
Bracketing Cal (optional)	RRFs for the opening and closing calibrations over a 12 hour period must agree to within ±20% of the mean (i.e., ≤40 RPD between RRFs and for the opening and closing calibrations, which is equivalent to ≤28.3% RSD).
GC Resolution	Benzo[b] & [k]fluoranthene valley height must be ≤75% for equal concentrations. Phenanthrene/anthracene valley height must be ≤30% for equal concentrations.
Chromatogram Quality	Maximum peak width must be ≤15 seconds for benzo[ghi]perylene peak at 10% peak height.
Retention Time Window for Target Compounds	RT within ± 3 seconds of the predicted retention time determined from the calibration standard and adjusted relative to the peak retention time reference (i.e. labelled surrogate). A second requirement is that an authentic elute after its labelled analog.
Ion Abundance Ratios	CAL VER: Ion ratios for authentic and labelled dibenz[ah]anthracene, indeno[1,2,3-cd]pyrene and benzo[ghi]perylene must be within ±35% of the mid-point of the I-CAL. All other native analytes and labelled surrogates must be within ±20% of the mid-point of the I-CAL. Samples: Ion ratios for authentic and labelled dibenz[ah]anthracene, indeno[1,2,3-cd]pyrene and benzo[ghi]perylene must be within ±35% of the 12 hour CAL VER (or bracketing) calibration standard. All other native analytes and labelled surrogates must be within ±20% of the 12 hour CAL VER (or bracketing) calibration standard.

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Table 9. Alkanes - QC Acceptance Criteria

	Typical Sample Specific Detection Limits[1]								
MATRIX	Solid	Aqueous	Tissue	XAD-2 Column	PUF	Filter	Oil	Procedural Blank Level ng	Acceptable Matrix Spike % Recovery[2] GC/MS
Analyte:	ng/g	ng/L	ng/g	ng	ng	ng	ng/g		
Dodecane (nC12)	1.0	10	1.0	10	10	10	100	<100	10-130
2,6 Dimethyl Undecane	1.0	10	1.0	10	10	10	100	<10	-
Norfarnesane	1.0	10	1.0	10	10	10	100	<10	-
Tridecane (nC13)	1.0	10	1.0	10	10	10	100	<15	20-130
Farnesane	1.0	10	1.0	10	10	10	100	<10	-
Tetradecane (nC14)	1.0	10	1.0	10	10	10	100	<15	20-130
2,6,10 Trimethyl Tridecane	1.0	10	1.0	10	10	10	100	<10	-
Pentadecane (nC15)	1.0	10	1.0	10	10	10	100	<200	20-130
Hexadecane (nC16)	1.0	10	1.0	10	10	10	100	<200	30-130
Norpristane	1.0	10	1.0	10	10	10	100	<100	-
Heptadecane (nC17)	1.0	10	1.0	10	10	10	100	<100	30-130
Pristane	1.0	10	1.0	10	10	10	100	<200	30-130
Octadecane (nC18)	1.0	10	1.0	10	10	10	100	<100	40-130
Phytane	1.0	10	1.0	10	10	10	100	<100	40-130
Nonadecane (nC19)	1.0	10	1.0	10	10	10	100	<100	40-130
Eicosane (nC20)	1.0	10	1.0	10	10	10	100	<40	60-130
Heneicosane (nC21)	1.0	10	1.0	10	10	10	100	<40	60-130
Docosane (nC22)	1.0	10	1.0	10	10	10	100	<60	70-150
Tricosane (nC23)	1.0	10	1.0	10	10	10	100	<150	70-150
Tetracosane (nC24)	1.0	10	1.0	10	10	10	100	<400	80-150
Pentacosane (nC25)	1.0	10	1.0	10	10	10	100	<600	80-150
Hexacosane (nC26)	1.0	10	1.0	10	10	10	100	<800	80-150
Heptacosane (nC27)	1.0	10	1.0	10	10	10	100	<800	60-150
Octacosane (nC28)	1.0	10	1.0	10	10	10	100	<800	60-150
Nonacosane (nC29)	1.0	10	1.0	10	10	10	100	<800	40-150
Triacontane (nC30)	1.0	10	1.0	10	10	10	100	<800	40-150
Untriacontane (nC31)	1.0	10	1.0	10	10	10	100	<800	30-200
Dotriacontane (nC32)	1.0	10	1.0	10	10	10	100	<500	30-200
Tritriacontane (nC33)	1.0	10	1.0	10	10	10	100	<250	10-200
Tetratriacontane (nC34)	1.0	10	1.0	10	10	10	100	<250	10-200
Pentatriacontane (nC35)	1.0	10	1.0	10	10	10	100	<150	10-200
Hexatriacontane (nC36)	1.0	10	1.0	10	10	10	100	<150	10-200
Typical Sample Size	10 g	1 L	10 g	1 col.	1 PUF	1 filter	0.1 g		
Typical Final Volume (µL)	400	400	400	400	400	400	400		

[1] Detection limits quoted are those routinely achieved. Lower detection limits can be achieved if required.

[2] Recoveries are guidelines only and vary according to matrix.

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Table 9 (cont'd)

SURROGATE STANDARD RECOVERIES[1]	% RECOVERY RANGES ALL OTHER MATRICES	TISSUE
Dodecane (nC12) -d26	10-120	30-120
Hexadecane (nC16) -d34	20-120	50-120
Tetracosane (nC24) -d50	40-130	50-120
Hexatriacontane (nC36) -d74	10-150	10-120

Parameter	Acceptance Specifications
Procedural Blank	See above table.
Analysis Duplicate	Must agree to within ±30% of the mean (applicable to concentrations >10 times the DL). (Note that ±30% of the mean is equivalent to 60 relative percent difference)
Instrument Sensitivity	S/N ratios should be ≥3:1 for a 100 pg/µL solution of n-C_{36} (m/e 57) injected.
Instrument Resolution	Calibration gas PFTBA (FC43) unit mass resolution at m/e 69/70 and 219/220, Unit mass resolution is demonstrated by the presence of a resolved peak at m/z 70 and m/e 220.
Instrument Linearity	Linearity determined by a 4-point calibration over the range of 0.1 to 10 ng/µL of all parent alkanes with a rel. std. deviation of the RRFs ±30%.
Bracketing Cal	RRFs for the opening and closing calibrations over a 12 hour period must agree to within ±20% of the mean (i.e., ≤40 RPD between RRFs and for the opening and closing calibrations, which is equivalent to ≤28.3% RSD).
Chromatogram Quality	**Resolution**: nC_{24} - d50 and nC_{24} separation must be ≥20 sec.
Analyte Responses	Must be within linear range of the instrument. Coders may use data from more than one chromatogram to get all responses within linear range.
Ion Abundance Ratios	Ion ratios for all native analytes and labelled surrogates must be ±20%.

[1] All analytes are quantified against tetracosane. The other surrogates are monitored as an indication of overall data quality only.

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5. EXTRACTION PROCEDURES

Samples are analyzed in batches. The samples to be analyzed, the batch QC samples, the name and volume of the surrogate, recovery, and authentic standard solutions required, and any additional information concerning the analysis are documented on a Batch List given to an analyst.

Each analyst performs the extractions according to the following written extraction procedures and completes an analysis worksheet for each sample during the sample extraction. The analyst is referred to the following Standard Operating Procedures for details of routine laboratory techniques.

All extracts must be protected from light by covering with aluminum foil.

Document ID	Title
SLA-003	Sample Receipt and Login
SLA-004	Sample Control Procedures
SLA-005	Maintenance and Use of Rotary Evaporation Equipment
SLA-006	Nitrogen Blowdown Concentration Technique
SLA-007	Kuderna-Danish Concentration Technique
SLA-008	Preparing Extracts for Instrumental Analysis
SLA-017	Spiking Procedure
SLA-020	Gravimetric Determination of Lipid Content
SLA-027	Completing a Worksheet
SLA-028	Independent Gravimetric Determination of Lipid Content
SLA-033	Procedures for Making an Analysis Batch
SLA-045	Removal of Sulphur From Extracts Using Activated Copper
SLA-067	Use of Nitrogen Cylinders
SLA-072	Computer Preparation of Labels
SLA-078	Spike Witness Program
SLA-081	Labelling Protocols and Sample Extract Transfer Procedures
SLA-084	Preparation of Aqueous Samples for Extraction
SLA-118	Extraction of SPMDs
SLA-085	Subsampling Procedure for Solids and Tissues
SLA-124	Liquid-Liquid Extraction Supplemental Techniques

The following worksheets are available for use:

FWO-305	Sample Labeling Information
FWO-306	Sample Weight and Moisture
FWO-307	Aqueous Samples
FWO-308	Lipid Sheet
FWO-309	Standards Spiking into C-Tube
FWO-310	Sample Spiking Sheet
FWO-312	Cleanup Information
FWO-314	Notes
FWO-317	Extraction – Blood, Milk, Nonylphenols

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5.1 Soxhlet Extraction of Solids (default procedures)

5.1.1 Sediment/Soils Samples

Matrix	Typical Sample Size
Sediment/Soil	10 g wet weight

Accurately weigh a homogenized subsample into a beaker containing anhydrous, granular sodium sulphate (80-100 g). Mix with a clean spatula. Cover the beaker with clean foil and allow the mixture to dry to a free flowing powder (minimum 30 minutes).

Quantitatively transfer the mixture with dichloromethane rinses to a Soxhlet thimble. Add an aliquot of surrogate standard solution(s) to the mixture in the thimble. Place the thimble in a Soxhlet apparatus. Allow the sample to reflux with dichloromethane (300 mL) for 16 to 18 hours; adjust as necessary to achieve a reflux rate of a minimum of 4 cycles per hour. Allow the extract to cool. Transfer the extract to a Kuderna-Danish flask with dichloromethane rinses, add iso-octane (1 mL), and concentrate to 1-2 mL in a 55°C water bath. The extract is ready for chromatographic cleanup procedures (Section 6.0).

5.1.2 Ash Samples

Matrix	Typical Sample Size
Ash	5 g wet weight

Accurately weigh a homogenized subsample into a beaker containing anhydrous, granular sodium sulphate (30 g). Mix with a clean spatula. Cover the beaker with clean foil and allow the mixture to dry to a free flowing powder (minimum 30 minutes).

Quantitatively transfer the mixture with toluene rinses to a Soxhlet thimble. Add an aliquot of surrogate standard solution(s) to the mixture in the thimble. Allow the sample to reflux with toluene (300 mL) for 16 to 24 hours; adjust as necessary to achieve a reflux rate of a minimum of 4 cycles per hour. Allow the extract to cool. Concentrate the extract by rotary evaporation. The extract is ready for chromatographic cleanup procedures (Section 6.0).

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5.1.3 Fly Ash Samples

Matrix	Typical Sample Size
Fly Ash	5 g wet weight

Accurately weigh a homogenized subsample of fly ash into a beaker. Add hydrochloric acid (50 mL, 1M HCl). Digest for 30 minutes in an ultrasonic bath. Using pH paper, check that the pH is <2. If not, add hydrochloric acid (HCl, 1M) drop wise until the pH is <2, and repeat the sonication step. Pour the mixture through a Millipore filtration apparatus containing a baked glass fiber filter paper (Ahlstrom, 1.1 µm, grade 161, 42.5 mm).

CAUTION: To avoid loss of volatile compounds when filtering, **DO NOT** draw air through the filter.

Wash the filter and beaker with rinses of ultra pure water (2 x 100 mL). Check that the pH of the final volume of filtrate passing through the filter is neutral (based on pH of ultra pure water). Process the filtrate as described in Section 5.1.3.1 and the filter with particulate as described in Section 5.1.3.2.

5.1.3.1 Filtrate

Adjust the pH of the filtrate to 6.5 -7.5 with sodium hydroxide (6N, NaOH). Transfer the filtrate with dichloromethane rinses to a separatory funnel (500 mL) and extract by shaking with dichloromethane (2 x 100 mL). Dry the dichloromethane extract with anhydrous sodium sulphate. Combine the dichloromethane extract with the toluene extract from the particulate/filter obtained in Section 5.1.3.2, as described in Section 5.1.3.3.

5.1.3.2 Particulate/Filter:

Remove the filter with particulate and place in clean Petri dish in a fume hood, place in a beaker and mix with granular sodium sulphate. Transfer the filter or filter/sodium sulphate mixture to a Soxhlet thimble. Add an aliquot of the surrogate standard(s) into the thimble. Place the thimble in a Soxhlet apparatus. Allow the sample to reflux with toluene (300 mL) for 16 to 24 hours; adjust as necessary to achieve a reflux rate of a minimum of 4 cycles per hour. Allow the extract to cool. Concentrate the extract by rotary evaporation. Combine the extract with the filtrate extract from Section 5.1.3.1, as described in Section 5.1.3.3.

5.1.3.3 Combining Filter and Filtrate Extracts

1. Transfer the dichloromethane filtrate extract to the round bottom flask containing the concentrated toluene filter extract and concentrate by rotary evaporation.

OR

2. Transfer the dichloromethane filtrate extract to a KD flask and concentrate to 1 mL. Combine the concentrated dichloromethane extract with the concentrated toluene extract.

The extract is ready for chromatographic cleanup procedures (Section 6.0).

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5.2 Soxhlet Extraction of Tissue (default procedure)

This extraction procedure is applicable to the matrix and sample size listed below and suitable for PAH analysis only. **This procedure is not suitable for the analysis of alkanes, as the Biobead column cannot be used effectively to separate the lipids from the alkane analytes.** The analyst is referred to the base digest extraction procedure (Section 5.2) if alkane analysis is required.

Matrix	Typical Sample Size
Tissue	1 – 25 g wet weight (up to 1.25 g lipid).

5.2.1 Sample Preparation

Accurately weigh a wet homogenized tissue sample (muscle: 10 g, liver: 5 g, fat: 1 g) into a beaker and add anhydrous sodium sulphate (75 g). Stir the mixture well with a spatula, cover with foil and allow the mixture to dry to a free flowing powder (30 minutes minimum).

5.2.2 Soxhlet Extraction

Quantitatively transfer the dried sample to a Soxhlet thimble. Add an aliquot of the surrogate standard solution to the sample in the thimble. Place the Soxhlet thimble in the Soxhlet apparatus. Allow the sample to reflux for 16 to 20 hours with dichloromethane (300 mL); adjust as necessary to achieve a reflux rate of a minimum of 4 cycles per hour. Allow the mixture to cool. Transfer the extract to a 500 mL Kuderna-Danish flask. Refer to the Batch List to determine whether a lipid determination is required. If so, carry out the lipid determination according to SLA-020 "*Gravimetric Determination of Lipid Content*". If lipid analysis is not required, add iso-octane (1 mL) as a "keeper", and concentrate the extract to 2 mL in a 55°C water bath.

5.2.3 Gel Permeation Cleanup

Load the extract onto a Biobead SX-3 gel permeation column with 1:1 dichloromethane:hexane and elute with 1:1 dichloromethane:hexane at 5 mL/min. Refer to the most recent Biobead cutpoint determination for the elution volume. Typical cutpoints are as follows:

F1	0	- 150 mL
F2	150	- 300 mL

Collect the second fraction. Add toluene (1 mL) to the extract and concentrate to a small volume by rotary evaporation. If the lipid content for the sample is greater than 2 g absolute (i.e. 10% of a 20 g sample), repeat the gel permeation cleanup procedure.

The extract is ready for chromatographic cleanup procedures (Section 6.0).

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5.3 Base Digest Extraction of Tissue, Soil and Sediment (optional procedure)

The base digest of tissues, soil and sediments is suitable for the analysis of PAHs, alkylated PAHs and alkanes. If a lipid determination is required, it must be performed on a separate extract, as described in SLA-028 *Independent Gravimetric Determination of Lipid Content.*

Matrix	Typical Sample Size
Tissue	10 g wet (up to 1.25 g lipid)
Vegetation	20-30 g wet
Sed/Soil (Coarse)	10-20 g wet

Accurately weigh a wet, homogenized sample into a round-bottom flask (500 mL), add methanol (100 mL) and swirl well. Add an aliquot of the appropriate surrogate standard solution(s) to the mixture. Add freshly prepared potassium hydroxide solution (KOH, 50% w/w, 10 mL for solids, 20 mL for tissues) while swirling the mixture, and add anti-bumping granules (~4-5 chips). Heat the mixture under reflux for 1 hour then allow it to cool 5-10 minutes. Add additional anti-bumping granules to the flask. Add ultra pure water (100 mL) through the condenser and allow to reflux again for another 1½ hours. With tissue samples, watch for excessive foaming. Turn down the heat to control foaming, while ensuring a complete boil. Make sure the solution continues to boil. Allow the solution to cool to lukewarm to settle suspended solids. (A cold water bath may be used to aid cooling.)

Decant the liquid into a separatory funnel (1 L). Rinse the remaining sample in the flask with methanol (2 x 40 mL) and add these rinses to the separatory funnel. Add pentane (150 mL) to the separatory funnel containing the methanol and shake the resultant mixture vigorously for 2 minutes. Collect the aqueous (lower) layer in the digestion flask and the pentane in an Erlenmeyer flask. Pour the aqueous layer back into the separatory funnel. Repeat the extraction of the aqueous layer once more, using 100 mL of pentane, shaking for 1 minute.

Combine the pentane layers in the same separatory funnel (1 L). Wash the combined pentane layers by shaking with ultra pure water (2 x 100 mL) for 5-10 seconds, discarding the lower aqueous layer each time. This removes any methanol from the pentane. Collect the pentane layer in an Erlenmeyer flask and dry over anhydrous granular sodium sulphate for 30 minutes. Decant the extract from the Erlenmeyer flask into a 500 mL Kuderna-Danish flask with pentane rinses (20-30 mL each). Add iso-octane (1 mL) as a "keeper". Concentrate the extract to 1 mL in a 55°C water bath.

The extract is ready for chromatographic cleanup procedures (Section 6.0).

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5.4 Aqueous Sample Extraction Procedure

This extraction procedure is suitable for water and effluent samples.

Matrix	Typical Sample Size
Aqueous	500 mL - 1 Litre *

* Always use one litre unless there is only one litre of sample. In this case, use 500 mL in order to preserve backup sample.

5.4.1 Sample Preparation

Prior to extraction aqueous samples are homogenized, subsampled, and spiked with an aliquot of surrogate standard solution by the analyst. Add a pre-cleaned PTFE magnetic stir bar to the sample and stir while spiking the aliquot of surrogate standard solution. The analyst must refer to SLA-084 "*Preparation of Aqueous Samples for Extraction*" for complete details of sample homogenization, subsampling, rinsing, surrogate spiking and centrifugation procedures.

Note: Aqueous samples for PAH and/or alkane analysis must never be filtered.

The aqueous sample extraction procedure depends on the percentage of suspended solids in the sample. Estimate the percent suspended solids by visual inspection. If in doubt of a reasonably accurate visual solid percent estimation, determine the percent suspended solids on a subsample of the sample according to SLA-092 "*Determination of Suspended Solids (SS) in a Sample*". Samples with ≤1% suspended solids are not centrifuged prior to extraction (Section 5.4.2). Samples with >1% suspended solids are centrifuged and extracted (Section 5.4.3).

5.4.2 Extraction of Aqueous Samples with ≤1% Suspended Solids

5.4.2.1 Extraction by Magnetic Stirring (default procedure)

Add dichloromethane (300 mL) and a pre-cleaned PTFE magnetic stir bar to the sample in an Erlenmeyer flask and extract by stirring the solution (with vortex) for a minimum of 30 minutes. Quantitatively transfer the solution to a separatory funnel and draw off the dichloromethane layer. Discard the aqueous layer. Dry the extract by filtration into a Kuderna-Danish flask (500 mL) through a solvent rinsed glass funnel with a glass wool plug and approximately half full of granular anhydrous sodium sulphate. Rinse the filter with dichloromethane to ensure quantitative transfer. Add iso-octane (1 mL) as a "keeper" and concentrate the extract to 1 mL (water bath 55°C). The extract is ready for chromatographic cleanup procedures (Section 6.0).

5.4.2.2 Extraction by Shaking in a Separatory Funnel (optional procedure)

Extract the sample by adding dichloromethane (100 mL) to the separatory funnel containing the sample and shaking vigorously for two minutes. Collect the dichloromethane layer in an Erlenmeyer flask. Repeat the extraction twice more. Combine the dichloromethane layers and dry over anhydrous sodium sulphate for at least 30 minutes. Transfer the extract to a Kuderna-Danish flask (500 mL) with dichloromethane. Add iso-octane (1 mL) as a "keeper" and

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concentrate the extract to 1 mL (water bath 55°C). The extract is ready for chromatographic cleanup procedures (Section 6.0).

5.4.3 Extraction of Samples with >1% Suspended Solids

The analyst must refer to SLA-084 for complete details of sample homogenization, subsampling, rinsing, surrogate spiking and centrifugation procedures. Note that aqueous samples for PAH/alkane determination must not be filtered. The procedures are summarized below:

Transfer a portion of the sample to a clean 500 mL stainless steel vessel with a clean baked 10 cm filter paper (Pall, type A/E, 1.0 µm) in the bottom of the jar to assist with capturing of the solids. Spin the sample in the centrifuge at 1500 rpm until the solid has settled. Decant the supernatant into an Erlenmeyer flask (if proceeding according to the default procedure in Section 5.4.3.1) or a separatory funnel (if proceeding according to the optional procedure in Section 5.4.3.1). Repeat the procedure until the entire sample has been centrifuged, leaving the filter paper in place throughout the process. Once the entire sample has been processed, dry the particulate and filter in the jar by mixing with granular anhydrous sodium sulphate. Extract the solids as described for particulate in Section 5.4.3.2.

Rinse the jar with ultra pure water and dichloromethane. Add the rinses to the Erlenmeyer flask/separatory funnel containing the supernatant. Extract the supernatant as described for centrifugates in Section 5.4.3.1.

5.4.3.1 Extraction of Centrifugate

Default extraction procedure for the centrifugate: Transfer the centrifugate (supernatant) and rinses to a clean flask. Add dichloromethane (300 mL) and a pre-cleaned PTFE magnetic stir bar to the flask, and extract by stirring the solution (with vortex) for a minimum of 30 minutes. Quantitatively transfer the solution to a separatory funnel and draw off the dichloromethane layer. Discard the aqueous layer. Dry the extract by filtration into a Kuderna-Danish flask (500 mL) through a solvent rinsed glass funnel with a glass wool plug and approximately half full of granular anhydrous sodium sulphate. Rinse the filter with dichloromethane to ensure quantitative transfer.

Optional extraction procedure for the centrifugate: Extract the centrifugate (supernatant) by adding dichloromethane (100 mL) to the separatory funnel and shaking vigorously for two minutes. Collect the dichloromethane layer in an Erlenmeyer flask. Repeat the extraction twice more. Combine the dichloromethane layers and dry over anhydrous sodium sulphate for at least 30 minutes.

> **Optional:** The centrifugate extract can be used in the Soxhlet extraction of the filter by transferring the extract with dichloromethane rinses to the round bottom flask (500 mL) used in the Soxhlet apparatus.

Should problems be encountered for liquid-liquid extraction of complex samples consult AXYS SOP SLA-124 *Liquid-Liquid Extraction Supplemental Techniques.*

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5.4.3.2 Extraction of Particulate

Transfer the dried particulate to a Soxhlet thimble. Place the Soxhlet thimble in the Soxhlet apparatus. Heat the sample under reflux for 16-20 hours using dichloromethane (300 mL) as the solvent; adjust as necessary to achieve a reflux rate of 4 cycles per hour. Allow the solution to cool.

5.4.3.3 Combining Extracts to Make a Final Extract

Combine the centrifugate and particulate extracts if separate. Transfer the extract to a Kuderna-Danish flask (500 mL) with dichloromethane. Add iso-octane (1 mL) as a “keeper” and concentrate the extract to 1 mL (water bath 55°C). The extract is ready for chromatographic cleanup procedures (Section 6.0).

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5.5 XAD-2 Columns

XAD-2 resin and associated filters from an XAD-2 water sampling column are typically analyzed for a variety of target analytes. Each class of target analyte is determined by a different analysis procedure, carried out on an organic extract obtained from the extraction of the XAD-2 resin or of the filter(s). The dried organic extract is typically split into several equal portions, one portion for each requested analysis and one portion as backup.

The XAD-2 resin is extracted using a Soxhlet extraction procedure as described in the following document:

MLA-010 *Analytical Method for the Determination of 209 PCB Congeners by EPA Method 1668A, EPA Method 1668C or EPA Method CBC01.2*

Note 1: Co-extraction of PCDD/F and PAH from XAD-2 is **not** an approved standard application; extraction of PAH from XAD-2 using Soxhlet Dean-Stark/toluene extraction is **not** permitted under any circumstances.

Note 2: Alkanes can **not** be determined in XAD-2 adsorbent.

The filters are separately extracted using the procedures as described in the following document:

SLA-105 *Ambient Temperature Extraction of Wound Glass Filters*

5.5.1 XAD-2 Resin or Filter Extract

Obtain the extract from the extraction of the XAD-2 resin or filter and proceed with the cleanup procedures described in Section 6.0.

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5.6 Samples Submitted as Extracts

This extraction procedure is suitable for samples submitted as solvent extracts, where only extract splitting (optional) and chromatographic cleanup procedures are required prior to instrumental analysis.

5.6.1 Sample Preparation

Determine the weight of the extract as received, as follows:

- Weigh to a minimum of 3 significant figures, the extract in the original container (usually jar or ampoule).
- Transfer the extract to a centrifuge tube (if received in an ampoule) or to an Erlenmeyer flask with solvent rinses; use the same solvent as in the sample.
- Reweigh the original container once it has dried.
- Record all weights on the worksheet.
- Calculate the weight of the extract.

Determine from the Batch List if the sample requires a single analysis or multiple analyses. Check the Batch List for extract splitting and surrogate spiking instructions.

5.6.2 Extract Drying Procedures

1. Inspect the extract for the presence of water.

   - If less than 3 mL water is visible, transfer the extract to an Erlenmeyer flask with solvent rinses (the same solvent as sample).

   - If greater than 3 mL of water is visible, transfer the extract to a separatory funnel with solvent rinses (same solvent as sample) and drain the water from the solvent. Transfer the solvent layer to a clean Erlenmeyer flask.

2. Dry the extract with anhydrous granular sodium sulphate for a minimum of 30 minutes. Quantitatively transfer the extract to a clean round bottom flask with solvent rinses. Concentrate the extract to 1-2 mL by rotary evaporation in preparation for extract splitting procedures.

3. SPMD extracts will not need to be dried as they will have been dried before splitting and submission for the required analyses.

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5.7 Ambient Air (PUF and Filter) Extraction and Particulate Filter Extraction Procedure

This method is applicable to the analysis of ambient air samples consisting of a polyurethane foam (PUF) and associated filter(s). The PUF and filter(s) are extracted together as one sample. The method is also applicable to particulate filters that are received dry.

Note: Alkanes can **not** be determined in ambient air samples.

5.7.1 Sample Preparation

Particulate Filter

Handle particulate filters while frozen. Fold the filter paper(s) to fit into a Soxhlet thimble for extraction. Using clean solvent-rinsed forceps place the filter(s) into a Soxhlet thimble.

PUF/Filter

Using clean solvent-rinsed forceps, transfer the PUF and filter(s) to a Soxhlet thimble.

5.7.2 Soxhlet Extraction Procedure

Place the Soxhlet thimble containing the PUF and filter(s) in the Soxhlet apparatus. If a single analysis is required, spike the surrogate standard solution onto the sample prior to starting the extraction. If multiple analyses are required, refer to the Batch List for surrogate spiking instructions. Add anti-bumping granules (4-5 granules) to the round bottom flask. Heat the sample under reflux for 16-20 hours using dichloromethane (300 mL) as the solvent; adjust as necessary to achieve a reflux rate of 4 cycles per hour. Allow the solution to cool. Transfer the extract to a Kuderna-Danish flask (500 mL) with dichloromethane. Add iso-octane (1 mL) as a “keeper” and concentrate the extract to 1 mL (water bath 55°C). The extract is ready to be gravimetrically spit. Refer to the Batch List for spiking and extract splitting instructions.

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5.8 Stationary Air (Sample Trains) Samples

The several containers obtained from a stationary air sampling event are typically analyzed for a variety of target analytes. Each class of target analyte is determined by a different analysis procedure, carried out on an organic extract obtained from the extraction of the contents of the containers. The dried organic extract is typically split into several equal portions, one portion for each requested analysis and one portion as backup.

Note: Alkanes can **not** be determined in stationary air samples.

The processing of each of the containers and the extraction procedure is described in the following documents. In order to prevent protonation of the perdeuterated surrogates, the procedures described in those documents must be modified so that **the filtrate that results from the acidification of particulate must be adjusted to pH 6.5-7.5 with sodium hydroxide (6N, NaOH) prior to extraction with dichloromethane.**

Consult a Supervisor should the sample require only PAH analysis.

MLA-010 *Analytical Method for the Determination of 209 PCB Congeners by EPA Method 1668A*

MLA-017 *Analytical Method for the Determination of Polychlorinated Dibenzodioxins and Dibenzofurans by EPA Method 1613B, EPA Method 8290A or Env. Canada EPA 1/RM/19*

5.8.1 Sample Train Extract

Obtain the extract from the extraction of the contents of the sample train and proceed with the cleanup procedures described in Section 6.0.

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5.9 Oil Extraction Procedure

This extraction procedure is suitable for the following matrix and sample size:

Matrix	Typical Sample Size
Oil	10 - 50 mg

For PAHs the whole extract may be used. For alkanes, a gravimetric split of the extract (as approved by the Project Manager) containing 0.25-2.5 mg of oil is used. If both PAH and alkane analysis are required, the gravimetric split for alkanes is done after the silica column cleanup and separation of the alkanes and PAHs (Section 6.3). If only alkane analysis is required the split may be done at any point after extraction.

Accurately weigh a sample of oil (10 - 50 mg) into a centrifuge tube and dissolve in pentane (2 mL). Transfer the sample to a 125 mL separatory funnel with pentane. Make the volume to 50 mL with pentane. Add an aliquot of the appropriate surrogate standard(s) and shake the funnel briefly. Allow the solution to equilibrate for 15 minutes. Backwash the solution by shaking with ultra pure water (20 mL). Draw off the organic layer and dry over anhydrous granular sodium sulphate. Transfer the dried extract with rinses to a Kuderna-Danish (KD) flask and add iso-octane (1 mL). Concentrate to 1 mL in a water bath at 55°C.

The extract is ready for chromatographic cleanup procedures (Section 6.0).

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5.10 Extract Splitting Procedures

For detailed splitting instructions refer to SLA-123 “Splitting of Sample Extracts”.

Some extracts must be gravimetrically split into two equal portions, one for the PAH analysis and one as backup. If multiple analyses are required, the extract may be spilt into many portions. Refer to the Batch List for extract splitting instructions.

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5.11 Analysis of Blood Samples

This extraction procedure is applicable to the matrix and sample size listed below.

Matrix	Sample Size
Whole Blood/Serum/Plasma	10 - 20 g

Refer to the standard operating procedure SLA-082, *Handling of Human Biohazards*, for details of safety precautions when handling blood samples. Use caution when using bleach to decontaminate equipment used in the analysis of human biohazards and **avoid contact between acid and bleach.** Note that ammonium sulphate, used in the extraction below, is an acidic solution.

1. Accurately weigh to a minimum of 3 significant figures, a sample into a round bottom flask (250 mL). Add an aliquot of surrogate standard solution, cover the flask with clean aluminum foil and allow the solution to equilibrate for at least 30 minutes.
2. Add ethanol, hexane and saturated ammonium sulphate to the sample and extract by shaking on the shaker table for 30 minutes. Refer to the Batch List for the volume of reagents to use.

   NOTE: The proportion of reagents used is critical to complete extraction of analytes. The minimum sample size required is 10 g and the volumes of reagents are scaled to sample size in a ratio of sample:ethanol:saturated ammonium sulphate:hexane of 1:1:1:3. An alternate procedure for sample sizes less than 10 g, using 10 mL ethanol, 50 mL hexane and 10 mL saturated ammonium may be applied on a custom basis to meet client or historical project requirements - this option requires written pre-approval of the Project Manager including confirmation that the client has been made aware of potential solvent-system effects on gravimetric determination of lipids in blood matrices. Decant the hexane layer into a separatory funnel (500 mL).
3. Add additional hexane (100 mL) to the aqueous layer in the round bottom flask and repeat the extraction step. Add the hexane layer to the hexane in the separatory funnel. Discard the aqueous phase.
4. Wash the hexane extracts by shaking with ultra pure water (2 x 50 mL) to remove residual ethanol. Discard the aqueous layer.
5. Transfer the hexane extract to an Erlenmeyer flask and dry over anhydrous sodium sulphate for a minimum of 30 minutes.
6. Refer to the Batch List to determine whether a lipid determination is required. If so, carry out the lipid determination according to SLA-020 "*Gravimetric Determination of Lipid Content*".
7. Quantitatively transfer the extract with hexane rinses to a clean round bottom flask. Add toluene (~0.5 mL) to the extract and concentrate the extract by rotary evaporation to about 1 mL. Add dichloromethane (1 mL) to the extract.

   Should problems be encountered for liquid-liquid extraction of complex samples consult AXYS SOP *SLA-124 Liquid-Liquid Extraction Supplemental Techniques*

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Note: The following cleanup procedure may not be used if alkane analysis is required, as the higher chain alkanes elute into the discard fraction.

8. Load the extract onto a Biobead SX-3 column with 1:1 dichloromethane:hexane and elute with1:1 dichloromethane:hexane at 5 mL/min. Refer to the most recent Biobead cutpoint determination for the elution volume. Typical cutpoints are as follows:

F1	0 - 150 mL
F2	150 - 300 mL

Discard the first fraction. Collect the second fraction. Add toluene (1 mL) to the extract and concentrate to a small volume by rotary evaporation. The extract is ready for chromatographic procedures (Section 6.0).

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6. CLEAN-UP PROCEDURES

The following cleanup procedures are for the analysis of PAHs, alkylated PAHs, and alkanes. For extracts that have not previously undergone a Biobead cleanup step, an optional Biobead cleanup (Section 6.1) may be carried out on an extract prior to cleanup on silica (6.3). Additional cleanup procedures, carried out after the silica column procedures are described in Section 6.4.

6.1 Optional Biobead Column Cleanup

Note: This cleanup procedure may not be used if alkane analysis is required, as the higher chain alkanes elute into the discard fraction.

Ensure the extract is in 1:1 dichloromethane:hexane. Load the extract onto a Biobead SX-3 gel permeation column with 1:1 dichloromethane:hexane and elute with 1:1 dichloromethane: hexane at 5 mL/min. Refer to the most recent Biobead cutpoint determination for the elution volume. Typical cutpoints are as follows:

F1	0	- 150 mL
F2	150	- 300 mL

Collect the second fraction, add toluene (1 mL), and concentrate to a small volume by rotary evaporation.

6.2 Solvent Exchange

For cleanup on silica, the extract must be in hexane. Carry out the following solvent exchange procedure if the extract is not in hexane.

Transfer the extract to a centrifuge tube with hexane rinses. Concentrate to 300 µL under a gentle stream of nitrogen. Add hexane (1 mL) to the extract. Add activated copper to the extract (except for tissue extracts) to remove sulphur.

6.3 Silica Column Cleanup

Note: PAH and/or Alkane analysis: Use silica, Mallinckrodt 100-200 mesh or Sigma-Aldrich 70-230 mesh (Section 3.4), that has been cutpointed for PAH and alkanes.

PAH only analysis: Use silica, Mallinckrodt 100-200 mesh, Sigma-Aldrich 70-230 mesh or SiliCycle G60 60-200 µm (Section 3.4), that has been cutpointed for PAHs.

Load the sample extract with pentane rinses onto a silica gel column (10 g, 5% deactivated). Refer to the most recent cut-point determination for elution volumes. Typical cut-points are as follows:

F1	pentane	25 mL
F2	dichloromethane	20 mL

Elute the column with pentane (F1). This fraction contains the alkanes. If analysis of alkanes has been requested, collect the eluate in a small Kuderna-Danish flask and concentrate to 1 mL in a 55°C water bath. Otherwise discard this fraction.

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Elute the column with dichloromethane (F2). This fraction contains PAHs and alkylated PAHs. If analysis of PAHs and/or alkylated PAHs have been requested, collect the eluate in a small Kuderna-Danish flask and concentrate the F2 fraction to 1 mL in a 55°C water bath. Otherwise discard this fraction. Prepare the extracts for analysis by GC/MS.(Section 6.5)

6.4 Additional (Optional) Cleanup Procedures

The following cleanup procedures may be carried out on an extract to remove interferences. An optional base wash procedure (Section 6.4.1), followed by cleanup on alumina or just an alumina cleanup procedure may be carried out.

6.4.1 Optional Base wash

Transfer the F2 fraction to a separatory funnel, and add dichloromethane to make the volume up to 100 mL. Base wash the F2 fraction by shaking with sodium hydroxide solution (0.1 M NaOH, 50 mL) for 2 minutes. Repeat the base wash procedure if the organic layer appears cloudy. Wash the organic layer by shaking with ultra pure water (50 mL). Drain the organic layer into an Erlenmeyer flask, dry over granular anhydrous sodium sulphate and decant with rinses into a Kuderna-Danish flask. Concentrate the extract to 1 mL in a 55°C water bath. The extract is ready for cleanup on alumina (Section 6.4.2).

6.4.2 Alumina Cleanup

GC/MS analysis of the F2 fraction containing PAHs and alkylated PAHs may show that the samples require further clean up due to the presence of interferences. The extracts are cleaned up on an additional alumina column as described below. **This cleanup procedure is not suitable for the F1 fraction containing alkanes.**

Solvent Exchange

Transfer the extract to a centrifuge tube with hexane rinses. Concentrate the extract to 300 µL under a gentle stream of nitrogen. Make the volume to 1 mL with hexane.

Alumina Column

Load the sample extract (in hexane) onto the alumina column (8 g, 2% deactivated, Section 3.4). Refer to the most recent cut-point determination for elution volumes. Typical cut-points are as follows:

Hexane	10 mL
Dichloromethane	50 mL

Elute first with hexane (discard) followed by dichloromethane (retain). Concentrate the fraction to 1 mL in a Kuderna-Danish flask (55°C water bath). Prepare the extract for analysis by GC/MS (Section 6.5).

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6.5 Preparation for GC/MS Analysis

6.5.1 Fraction 1 (F1) Alkanes

Transfer the F1 fraction to a centrifuge tube with hexane. Concentrate the extract under a gentle stream of nitrogen to 400 µL. Mix the extract by vortex mixing.

Transfer the extract to an autosampler vial with hexane rinses. Refer to the Batch List for the desired final volume of extract. Concentrate the extract to 375 µL under a gentle stream of nitrogen. Add an aliquot of recovery standard solution (25 µL, 10 µL to tissue extracts) and vortex well. The final volume should be 400 µL.

Cover the mouth of the autosampler vial with aluminum foil. Cap the autosampler vial. The extract is ready for analysis by GC/MS (Section 8.2).

6.5.2 Fraction 2 (F2) PAHs

Transfer the F2 fraction to a centrifuge tube with dichloromethane. Concentrate the extract to 400 µL by evaporation under a gentle stream of nitrogen.

Transfer the extract to an autosampler vial containing toluene (volume equal to 20% of final extract volume) using dichloromethane rinses. Refer to the Batch List for the desired final volume of extract. Concentrate the extract to 400 µL (90 µL for aqueous and tissue extracts) under a stream of nitrogen. Add an aliquot of recovery standard (100 µL, 10 µL to aqueous and tissue extracts). The final volume should be 500 µL (100 µL for aqueous and tissue extracts). Cap the autosampler vial. The extract is ready for analysis by GC/MS (Section 8.1).

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7. GC/MS ANALYSIS

7.1 GC/MS Analysis of PAHs

See Table 8 in Section 4.0 for specific acceptance criteria.

7.1.1 Tuning and Sensitivity

Tuning and mass calibration is performed as described in SIN-029 every 3 days or as required to meet daily tuning check requirements. The PFTBA (FC43) procedure is used. Tuning is checked daily by verifying mass calibration, mass resolution and peak relative response as described in Appendix C

Prior to the analysis of samples the sensitivity of the GC/MS is checked by running a low-level calibration solution (Table 6a, Level A), which is less concentrated than the corresponding lowest level calibration solution used in the initial calibration.

7.1.2 GC Resolution

The GC resolution is checked with every bracket of samples by monitoring the valley height (expressed in terms of the smaller peak in the pair) between benzo(b)fluoranthene and benzo(k)fluoranthene pair and the valley height between phenanthrene and anthracene in the calibration solution.

7.1.3 Instrument Multi-Level Calibration (default)

The initial calibration is performed using a series of solutions that encompass the working concentration range of the instrument. Initial calibration solutions contain the suite of labelled surrogate and recovery standards and native PAHs. Calibration is verified at least once every twelve hours by analysis of a mid-level calibration solution (Table 6, Level E). Continuing calibration procedures use the mean RRFs determined from the initial calibration to calculate analyte concentrations.

The nominal concentration range of target analytes in the calibration solutions is as follows:

50 ng/mL – 5000 ng/mL

7.1.4 Single-Level Bracketing Calibration (optional)

Upon the request of the client single-point bracketing calibration protocols may be followed. Bracketing calibration procedures use mean RRFs from the analysis of the mid-level calibration solution to calculate analyte concentrations

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7.1.5 GC/MS Analysis

HRGC/LRMS analysis is conducted on a low-resolution mass spectrometer (MS) equipped with a gas chromatograph, running manufacturer's software. The MS is operated at a unit mass resolution in the electron impact (EI) ionization mode using multiple ion detection (MID) acquiring at least one characteristic ion for each target analyte and surrogate standard. The ions acquired are listed in Table 10. A Restek Rt_x-5 chromatography column (30 m, 0.25 mm i.d., 0.25 µm film thickness) is coupled directly to the MS source. A splitless/split injection sequence is used.

Typical GC/MS operating conditions for the analysis of PAHs are presented in the following table.

GC Temperature Program		**General GC Conditions**	
Temp (°C)	60	Injector Temp (°C)	270
Hold time (min)	2	Injector	Split/Splitless
Rate (C° min^{-1})	40	Carrier Gas	Helium, variable
Temp (°C)	100	Maximum Temp (°C)	325
Hold time (min)	2		
Rate (C° min^{-1})	10	**MS Conditions**	
Temp (°C)	265	Source Temp (°C)	286
Hold time (min)	7	Electron Energy (eV)	70 ± 5
Rate (C° min^{-1})	10	Trap Current (µA)	N/A
Temp (°C)	300	Mass Resolution	Unit
Hold time (min)	0	Detector Voltage (V)	Variable
Rate (C° min^{-1})	3		
Temp (°C)	325		
Hold time (min)	3		

Additional instrumental analysis details are documented in instrument method MIN-021.

7.1.6 Analysis Sequence

The sequence in which a batch of samples is analyzed, after successful calibration, is as follows:

Calibration Sample
Reference Sample
Instrument Blank
Procedural Blank
Samples
Calibration Sample

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7.2 GC/MS Analysis of Alkanes

7.2.1 Sensitivity

Prior to the analysis of samples the sensitivity of the GC/MS is checked by running a low level calibration solution Table 7, Level A).

7.2.2 Instrument Calibration

The initial calibration is performed using a series of solutions that encompass the working concentration range of the instrument. Initial calibration solutions contain the suite of labelled surrogate and recovery standards and native alkanes. Calibration is verified at least once every twelve to fourteen hours by analysis of a mid-level calibration (Table 7, Level B) solution. Bracketing calibration protocols are followed.

The nominal concentration range of target analytes in the calibration solutions is as follows:

100 – 10,000 ng/mL

7.2.3 GC/MS Analysis

HRGC/LRMS analysis is conducted on a low-resolution mass spectrometer (MS) equipped with a high-resolution gas chromatograph, running manufacturer's software. The MS is operated at a unit mass resolution in the electron impact (EI) ionization mode using multiple ion detection (MID) acquiring two characteristic ions for each target analyte and surrogate standard. The ions acquired are listed in Table 11. A Restek Rt_x-5 chromatography column (30 m, 0.25 mm i.d., 0.25 µm film thickness) is coupled directly to the MS source. A splitless/split injection sequence is used.

Typical operating conditions for the GC/LRMS analysis of alkanes are presented below.

GC Temperature Program		**General GC Conditions**	
Temp (°C)	60	Injector Temp (°C)	250
Hold time (min)	3	Injector	Split/splitless
Rate (C° min^{-1})	10	Carrier Gas	Helium
Temp (°C)	110	Maximum Temp (°C)	325
Hold time (min)	2	**MS Conditions**	
Rate (C° min^{-1})	7.5	Source Temp (°C)	286
Temp (°C)	320	Electron Energy (eV)	70 ±5
Hold time (min)	13	Trap Current (µA)	NA
		Mass Resolution	Unit
		Detector Voltage (V)	Variable

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Note: To avoid possible contamination of the extract from the autosampler vial cap, carry out the following procedure immediately prior to GC/MS analysis:

Uncap the vial, replace the foil covering the mouth of the vial with new foil, and place the vial in the autosampler, without the cap. After GC/MS analysis, replace the pierced foil with a layer of clean foil and cap the vial.

7.2.4 Analysis Sequence

The sequence in which a batch of samples is analyzed, after successful calibration, is as follows:

Calibration Sample
Retention Time Standard (Alberta Sweet Crude)**
Reference Sample
Instrument Blank
Procedural Blank
Samples
Calibration Sample

**The retention time standard is run after the initial calibration or once in every date directory. This standard contains both alkanes and hopanes that are not in the calibration standard.

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8. QUALITATIVE AND QUANTITATIVE DETERMINATION

8.1 Compound Identification

An individual chromatographic peak is identified as a target compound if the following criteria are met for the quantification and confirmation ions (where confirmation ions are available):

- Peak responses must be at least three times the background noise level.
- The retention time (RT) must be within three seconds of that predicted from the calibration run and the sample retention time reference (labelled compound).
- Peak maxima for the quantification and confirmation ions must coincide within two seconds.
- The relative ion abundance ratios must be within 20% of the opening calibration values.

Identification of Alkylated PAH Compound Totals is based comparison of the sample chromatogram retention times and peak patterns to those of a qualitative reference sample; further details are given in Appendix D.

8.2 Analyte Quantification

Concentrations of target PAHs and alkanes are calculated using the isotope dilution method of quantification. Compounds are quantified by comparing the area of the quantification ion to that of the corresponding deuterium-labelled standard and correcting for response factors. Response factors are determined daily using authentic PAHs and alkanes. Calculations are carried out using HP EnviroQuant and Prolab MS-Extended for targeting and quantification.

$$\text{Concentration of Target (ng/g or ng/L)} = \left(\frac{\text{area of Target}}{\text{area of Surr Std}}\right) \times \left(\frac{\text{weight of Surr Std (ng)}}{\text{RRF}}\right) \times \left(\frac{1}{\text{weight of sample (g or L)}}\right)$$

where $$\text{RRF} = \left(\frac{\text{area of Target}}{\text{area of Surr Std}}\right) \times \left(\frac{\text{concentration of Surr Std}}{\text{concentration of Target}}\right)$$

and the Surr Std is either the surrogate or the internal standard

In the current HC linearity series, all of the Parent Alkanes (i.e. odd and even numbers of carbon atoms) and Pristane and Phytane are present.

Refer to Tables 10 and 11 for further details on the quantification references used.

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8.3 Non-Recovery Corrected Concentrations

Isotope dilution quantification yields concentrations that are recovery corrected. If non-recovery corrected concentrations are required for more direct comparison to standard EPA 8270 data these are back-calculated using the percent recovery of the labelled compound against which the target compound is quantified as follows:

$$C_{NRC} = C_{RC} * (\%R_L/100)$$

Where: C_{NRC} = the non-recovery corrected concentration of the target compound

C_{RC} = the recovery corrected concentrations from isotope dilution quantification of the target compound

$\% R_L$ = the percent recovery of the labelled compound used for isotope dilution quantification of the target compound

8.4 Benzo(b)fluoranthene Correction to Remove Surrogate Interference

At low concentrations native benzo(b)fluoranthene results are affected by an interference from the surrogate standard D_{12}-benzo(k)fluoranthene. D_{12}-benzo(k)fluoranthene has the same chromatographic retention time as benzo(b)fluoranthene and produces an artifactual response in the quantifying ion channel m/z 252. To remedy this interference the estimated peak area contribution from D_{12}-benzo(k)fluoranthene is subtracted from the benzo(b)fluoranthene peak area before quantification.

The peak area of d_{12}-benzo(b)fluoranthene in ion channel *m/z* 252 of the procedural blank is divided by the area of the same compound in ion channel *m/z* 264 to calculate the ion abundance ratio, usually around 0.003. (D_{12}-benzo(b)fluoranthene is used rather than d_{12}-benzo(k)fluoranthene because it doesn't coelute with any known native compound.) The corrected peak area of the native benzo(b)fluoranthene is then calculated as:

$$A^{corr.}_{benzo(b)fluoranthene\,(252)} = A^{sample}_{benzo(b)fluoranthene\,(252)} - A^{sample}_{d12\text{-}benzo(k)fluoranthene\,(264)} \left(\frac{A^{blank}_{d12\text{-}benzo(b)fluoranthene\,(252)}}{A^{blank}_{d12\text{-}benzo(b)fluoranthene\,(264)}} \right)$$

where $A^{corr.}_{benzo(b)fluoranthene\,(252)}$ = Corrected peak area for benzo(b)fluoranthene in the sample

$A^{sample}_{benzo(b)fluoranthene\,(252)}$ = Measured peak area for benzo(b)fluoranthene in the sample

$A^{sample}_{d12\text{-}benzo(k)fluoranthene\,(264)}$ = Peak area for d_{12}-benzo(k)fluoranthene in the sample

$A^{blank}_{d12\text{-}benzo(b)fluoranthene\,(252)}$ = Area for d_{12}-benzo(b)fluoranthene (m/z 252) in the blank

$A^{blank}_{d12\text{-}benzo(b)fluoranthene\,(264)}$ = Area for d_{12}-benzo(b)fluoranthene (m/z 264) in the blank

This correction is performed by the instrument quantification software and the LIMS software.

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9. REPORTING CRITERIA AND PRACTICES

Sample concentrations and detection limits are reported. The isotope dilution/internal standard method of quantification, used to determine concentrations of target analytes, corrects the concentrations based on the percent recovery of the surrogate standards. Typical reporting units for all data are ng/g, ng/L, or ng/sample. Concentrations for solids are reported on a dry weight basis. Concentrations in tissues (including blood/serum/plasma) are reported on a wet weight basis and/or on a lipid weight basis when requested. Concentrations in aqueous samples are reported on a volume basis. Concentrations in XAD-2 resin, filters and stack gas samples are reported on a per sample basis or a per volume basis. Concentrations in particulate filters are reported on a per sample basis. Results may be expressed in other units if specified by contract.

Sample specific detection limits (SDL's) are determined from the analysis data by converting the minimum detectable area to a concentration following the same quantification procedures used to convert target peak responses to concentrations. The estimated minimum detectable area is determined as 3 times the height of the noise in the m/z channel of interest, converted to an area using the area:height ratio of the corresponding labelled surrogate peak.

Percent moisture is reported for sediments, soils, and ash samples and for tissues when percent moisture is requested.

Percent lipid is reported if determined on a sample.

10. MODIFICATIONS TO REFERENCE METHODS

See Appendix B

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Table 10. Analyte Ions Monitored, Surrogates Used and RRF Determination for PAH

TARGET ANALYTES	Quantification Ion (m/z)	Confirmation Ions (m/z)	Typical Ion Ratio (Conf./Quant.)	SURROGATE	Typical Retention Time (minutes)	RRF DETERMINED FROM
Naphthalene	128	102	0.064	d_8-Naphthalene	6.84	Naphthalene
Acenaphthylene	152	151	0.222	d_8-Acenaphthylene	10.83	Acenaphthylene
Acenaphthene	154	153	1.18	d_8-Acenaphthylene	11.33	Acenaphthene
Fluorene	166	165	1.01	d_{10}-Phenanthrene	12.63	Fluorene
Phenanthrene	178	176	0.202	d_{10}-Phenanthrene	15.04	Phenanthrene
Anthracene	178	176	0.196	d_{10}-Phenanthrene	15.15	Anthracene
Fluoranthene	202	200	0.214	d_{10}-Fluoranthene	18.06	Fluoranthene
Pyrene	202	200	0.219	d_{10}-Fluoranthene	18.60	Pyrene
Benz[a]anthracene[6]	228	226	0.281	d_{12}-Benz[a]anthracene	21.68	Benz[a]anthracene
Chrysene[1]	228	226	0.312	d_{12}-Chrysene	21.79	Chrysene
Benzo[b]fluoranthene	252	253	0.218	d_{12}-Benzo[b]fluoranthene	25.21	Benzo[b]fluoranthene
Benzo[j,k]fluoranthenes	252	253	0.215	d_{12}-Benzo[k]fluoranthene	25.30	Benzo[k]fluoranthene
Benzo[e]pyrene	252	253	0.213	d_{12}-Benzo[a]pyrene	26.36	Benzo[e]pyrene
Benzo[a]pyrene	252	253	0.217	d_{12}-Benzo[a]pyrene	26.58	Benzo[a]pyrene
Perylene	252	253	0.212	d_{12}-Perylene	27.00	Perylene
Dibenzo[ah]anthracene[2]	278	139	0.144	d_{14}-Dibenzo[ah]anthracene	31.86	Dibenz[ah]anthracene
Indeno[1,2,3-cd]pyrene	276	138	0.179	d_{12}-Indeno[1,2,3,cd]pyrene	31.71	Indeno[1,2,3-cd]pyrene
Benzo[ghi]perylene	276	138	0.194	d_{12}-Benzo[ghi]perylene	32.53	Benzo[ghi]perylene
Biphenyl[3]	154	152	0.304	d_{10}- Biphenyl	9.81	Biphenyl
Dibenzothiophene[3]	184	152	0.073	d_8-Dibenzothiophene	14.72	Dibenzothiophene
1-Methylnaphthalene[3]	142	141	0.962	d_{10}-2-Methylnaphthalene	8.81	1-Methylnaphthalene

[1] Coelutes with Triphenylene

[2] Coelutes with Dibenz[ac]anthracene

[3] These compounds are in addition to the regular suite of analytes, and are analyzed by client request only.

[4] Secondary ion confirmation procedures do not apply

[5] RRT ranges apply to alkylated PAH Totals

[6] Benz(a)anthracene coelutes with cyclopenta(cd)pyrene, which may contribute response in the second ion to cause a failing ion abundance ratio. Therefore quantification of benz(a)anthracene should use response from the first ion only.

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TARGET ANALYTES	Quantification Ion (m/z)	Confirmation Ions (m/z)	Typical Ion Ratio (Conf./Quant.)	SURROGATE	Typical Retention Time (minutes)	RRF DETERMINED FROM
2-Methylnaphthalene[3]	142	141	0.930	d_{10}-2-Methylnaphthalene	8.55	2-Methylnaphthalene
C1-Naphthalenes[3]	142	[4]	[4]	d_{10}-2-Methylnaphthalene	[5]	1- & 2-Methylnaphthalene
2,6-Dimethylnaphthalene[3]	156	141	0.666	d_{12}-2,6 Dimethylnaphthalene	10.17	2,6-Dimethylnaphthalene
1,2-Dimethylnaphthalene	156	141	1.26	d_{12}-2,6 Dimethylnaphthalene	10.90	1,2-Dimethylnaphthalene
C2-Naphthalenes[3]	156	[4]	[4]	d_{12}-2,6 Dimethylnaphthalene	[5]	2,6- & 1,2-Dimethylnaphthalene
2,3,5-Trimethylnaphthalene[3]	170	155	0.873	d_{12}-2,6 Dimethylnaphthalene	12.35	2,3,5- Trimethylnaphthalene
2,3,6-Trimethylnaphthalene	170	155	0.876	d_{12}-2,6 Dimethylnaphthalene	12.17	2,3,6- Trimethylnaphthalene
C3-Naphthalenes[3]	170			d_{12}-2,6 Dimethylnaphthalene	[5]	2,3,5- & 2,3,6-Trimethylnaphthalene
1,4,6,7-Tetramethylnaphthalene	184	139	0.027	d_{12}-2,6 Dimethylnaphthalene	13.89	1,4,6,7-Tetramethylnaphthalene
C4-Naphthalene	184	[4]	[4]	d_{12}-2,6 Dimethylnaphthalene	[5]	1,4,6,7-Tetramethylnaphthalene
2-Methylanthracene	192	191	0.531	d_{10}-Phenanthrene	16.45	2-Methylanthracene
3-Methylphenanthrene	192	191	0.608	d_{10}-Phenanthrene	16.27	1- & 2-Methylphenanthrene & 2-Methylanthracene
2-Methylphenanthrene	192	191	0.608	d_{10}-Phenanthrene	16.36	2-Methylphenanthrene
9/4-Methylphenanthrenes	192	191	0.634	d_{10}-Phenanthrene	16.59	1- & 2-Methylphenanthrene & 2-Methylanthracene
1-Methylphenanthrene[3]	192	191	0.634	d_{10}-Phenanthrene	16.64	1-Methylphenanthrene
C1-Phenanthrenes/Anthracenes[3]	192	[4]	[4]	d_{10}-Phenanthrene	[5]	1- & 2-Methylphenanthrene & 2-Methylanthracene
3,6-Dimethylphenanthrene[3]	206	191	0.342	d_{10}-Fluoranthrene	17.46	3,6-Dimethylphenanthrene
2,6-Dimethylphenanthrene	206	191	0.342	d_{10}-Fluoranthrene	17.54	3,6- & 1,7-Dimethyl-phenanthrenes
1,7-Dimethylphenanthrene	206	191	0.332	d_{10}-Fluoranthrene	17.89	1,7-Dimethylphenanthrene
1,8-Dimethylphenanthrene	206	191	0.332	d_{10}-Fluoranthrene	18.13	3,6- & 1,7-Dimethyl-phenanthrenes
C2-Phenanthrenes/Anthracenes[3]	206	[4]	[4]	d_{10}-Fluoranthrene	[5]	3,6- & 1,7-Dimethyl-phenanthrenes
1,2,6-Trimethylphenanthrene	220	205	0.581	d_{10}-Fluoranthrene	19.41	1,2,6-Trimethylphenanthrene
C3-Phenanthrenes/Anthracenes	220	[4]	[4]	d_{10}-Fluoranthrene	[5]	1,2,6-Trimethylphenanthrene
Retene[3]	234	219	1.63	d_{10}-Fluoranthene	19.53	Retene
C4-Phenanthrenes/Anthracenes	234	[4]	[4]	d_{10}-Fluoranthrene	[5]	Retene
C1-Biphenyls	168	[4]	[4]	d_{10}- Biphenyl	[5]	Biphenyl
C2-Biphenyls	182	[4]	[4]	d_{10}- Biphenyl	[5]	Biphenyl
C1-Acenaphthenes	168	[4]	[4]	d_8-Acenaphthylene	[5]	Acenaphthene

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TARGET ANALYTES	Quantification Ion (m/z)	Confirmation Ions (m/z)	Typical Ion Ratio (Conf./Quant.)	SURROGATE	Typical Retention Time (minutes)	RRF DETERMINED FROM
2-Methylfluorene	180	165	1.23	d_{10}-Phenanthrene	14.06	2-Methylfluorene
C1-Fluorenes	180	[4]	[4]	d_{10}-Phenanthrene	[5]	2-Methylfluorene
1,7-Dimethylfluorene	194	177	0.092	d_{10}-Phenanthrene	15.49	1,7-Dimethylfluorene
C2-Fluorenes	194	[4]	[4]	d_{10}-Phenanthrene	[5]	1,7-Dimethylfluorene
C3-Fluorenes	208	[4]	[4]	d_{10}-Phenanthrene	[5]	1,7-Dimethylfluorene
2/3-Methyldibenzothiophenes	198	197	0.738	d_8-Dibenzothiophene	16.07	2/3-Methyldibenzothiophenes
C1-Dibenzothiophenes	198	[4]	[4]	d_8-Dibenzothiophene	[5]	2/3-Methyldibenzothiophenes
2,4-Dimethyldibenzothiophene	212	197	0.514	d_8-Dibenzothiophene	17.08	2,4-Dimethyldibenzothiophene
C2-Dibenzothiophenes	212	[4]	[4]	d_8-Dibenzothiophene	[5]	2,4-Dimethyldibenzothiophene
C3-Dibenzothiophenes	226	[4]	[4]	d_8-Dibenzothiophene	[5]	2,4-Dimethyldibenzothiophene
C4-Dibenzothiophenes	240	[4]	[4]	d_8-Dibenzothiophene	[5]	2,4-Dimethyldibenzothiophene
3-Methylfluoranthene/Benzo(a)fluorene	216	215	0.880	d_{10}-Fluoranthrene	19.53	3-Methylfluoranthene
C1-Fluoranthenes/Pyrenes	216	[4]	[4]	d_{10}-Fluoranthrene	[5]	3-Methylfluoranthene
C2-Fluoranthenes/Pyrenes	230	[4]	[4]	d_{10}-Fluoranthrene	[5]	3-Methylfluoranthene
C3-Fluoranthenes/Pyrenes	244	[4]	[4]	d_{10}-Fluoranthrene	[5]	3-Methylfluoranthene
C4-Fluoranthenes/Pyrenes	258	[4]	[4]	d_{10}-Fluoranthrene	[5]	3-Methylfluoranthene
5/6-Methylchrysenes	242	[4]	[4]	d_{12}-Chrysene	23.15	6-Methylchrysene
1-Methylchrysene	242	[4]	[4]	d_{12}-Chrysene	23.32	1-Methylchrysene
C1-Benz(a)anthracenes/Chrysenes	242	[4]	[4]	d_{12}-Chrysene	[5]	1- & 6-Methylchrysenes
5,9-Dimethylchrysene	256	[4]	[4]	d_{12}-Chrysene	24.49	5,9-Dimethylchrysene
C2-Benz(a)anthracenes/Chrysenes	256	[4]	[4]	d_{12}-Chrysene	[5]	5,9-Dimethylchrysene
C3-Benz(a)anthracenes/Chrysenes	270	[4]	[4]	d_{12}-Chrysene	[5]	5,9-Dimethylchrysene
C4-Benz(a)anthracenes/Chrysenes	284	[4]	[4]	d_{12}-Chrysene	[5]	5,9-Dimethylchrysene
7-Methylbenzo(a)pyrene	266	[4]	[4]	d_{12}-Benzo[a]pyrene	29.35	7-Methylbenzo(a)pyrene
C1-Benzofluoranthenes/Benzo-pyrenes	266	[4]	[4]	d_{12}-Benzo[a]pyrene	[5]	7-Methylbenzo(a)pyrene
C2-Benzofluoranthenes/Benzo-pyrenes	280	[4]	[4]	d_{12}-Benzo[a]pyrene	[5]	7-Methylbenzo(a)pyrene

(Table 10 continues...)

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LABELLED SURROGATE STANDARDS	Quantification Ion (m/z)	Confirmation Ions (m/z)	Typical Ion Ratio (Conf./Quant.)	RECOVERY CALCULATED AGAINST	Typical Retention Time (minutes)
d_8-Naphthalene	136	134	0.095	d_{10}-Acenaphthene	6.80
d_{10}-2-Methylnaphthalene	152	151	0.195	d_{10}-Acenaphthene	8.47
d_{10}-Biphenyl	164	[4]	[4]	d_{10}-Acenaphthene	9.75
d_{12}-2,6-Dimethylnaphthalene	168	150	0.747	d_{10}-Acenaphthene	10.07
d_8-Acenaphthylene	160	158	0.159	d_{10}-Acenaphthene	10.80
d_8-Dibenzothiophene	192	160	0.085	d_{10}-Pyrene	14.67
d_{10}-Phenanthrene	188	184	0.143	d_{10}-Pyrene	14.97
d_{10}-Fluoranthene	212	208	0.173	d_{10}-Pyrene	18.02
d_{12}-Benz[a]anthracene	240	236	0.250	d_{10}-Pyrene	21.63
d_{12}-Chrysene	240	236	0.278	d_{10}-Pyrene	21.73
d_{12}-Benzo[b]fluoranthene	264	260	0.216	d_{12}-Benzo[e]pyrene	25.11
d_{12}-Benzo[k]fluoranthene	264	260	0.208	d_{12}-Benzo[e]pyrene	25.23
d_{12}-Benzo[a]pyrene	264	260	0.216	d_{12}-Benzo[e]pyrene	26.47
d_{12}-Perylene	264	260	0.256	d_{12}-Benzo[e]pyrene	26.88
d_{12}-Indeno[1,2,3,cd]pyrene	288	284	0.192	d_{12}-Benzo[e]pyrene	31.63
d_{14}-Dibenzo[ah]anthracene	292	288	0.260	d_{12}-Benzo[e]pyrene	31.75
d_{12}-Benzo[ghi]perylene	288	284	0.205	d_{12}-Benzo[e]pyrene	32.45
LABELLED RECOVERY STANDARDS					
d_{10}-Acenaphthene	164	160	0.464		11.24
d_{10}-Pyrene	212	208	0.176		18.56
d_{12}-Benzo[e]pyrene	264	260	0.269		26.25

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Table 11. Analyte Ions Monitored, Surrogate Used and RRF Determination for Alkanes

TARGET ANALYTES	Quantification Ion (m/z)	Confirmation Ion (m/z)	Surrogate	RRF Determined From
Decane (nC10)	57	71	d_{50}-Tetracosane (nC24)	Decane (nC10)
Undecane (nC11)	57	71	d_{50}-Tetracosane (nC24)	Undecane (nC11)
Dodecane (nC12)	57	71	d_{50}-Tetracosane (nC24)	Dodecane (nC12)
2,6-Dimethyl Undecane	57	-	d_{50}-Tetracosane (nC24)	Dodecane (nC12)
Norfarnesane	57	-	d_{50}-Tetracosane (nC24)	Dodecane (nC12)
Tridecane (nC13)	57	71	d_{50}-Tetracosane (nC24)	Tridecane (nC13)
Farnesane	57	-	d_{50}-Tetracosane (nC24)	Tetradecane (nC14)
Tetradecane (nC14)	57	71	d_{50}-Tetracosane (nC24)	Tetradecane (nC14)
2,6,10-Trimethyl Tridecane	57	-	d_{50}-Tetracosane (nC24)	Tetradecane (nC14)
Pentadecane (nC15)	57	71	d_{50}-Tetracosane (nC24)	Pentadecane (nC15)
Hexadecane (nC16)	57	71	d_{50}-Tetracosane (nC24)	Hexadecane (nC16)
Norpristane	57	-	d_{50}-Tetracosane (nC24)	Mean of nC16, nC17
Heptadecane (nC17)	57	71	d_{50}-Tetracosane (nC24)	Heptadecane (nC17)
Pristane	57	71	d_{50}-Tetracosane (nC24)	Pristane
Octadecane (nC18)	57	71	d_{50}-Tetracosane (nC24)	Octadecane (nC18)
Phytane	57	71	d_{50}-Tetracosane (nC24)	Phytane
Nonadecane (nC19)	57	71	d_{50}-Tetracosane (nC24)	Nonadecane (nC19)
Eicosane (nC20)	57	71	d_{50}-Tetracosane (nC24)	Eicosane (nC20)
Heneicosane (nC21)	57	71	d_{50}-Tetracosane (nC24)	Heneicosane (nC21)
Docosane (nC22)	57	71	d_{50}-Tetracosane (nC24)	Docosane (nC22)
Tricosane (nC23)	57	71	d_{50}-Tetracosane (nC24)	Tricosane (nC23)
Tetracosane (nC24)	57	71	d_{50}-Tetracosane (nC24)	Tetracosane (nC24)
Pentacosane (nC25)	57	71	d_{50}-Tetracosane (nC24)	Pentacosane (nC25)
Hexacosane (nC26)	57	71	d_{50}-Tetracosane (nC24)	Hexacosane (nC26)
Heptacosane (nC27)	57	71	d_{50}-Tetracosane (nC24)	Heptacosane (nC27)
Octacosane (nC28)	57	71	d_{50}-Tetracosane (nC24)	Octacosane (nC28)
Nonacosane (nC29)	57	71	d_{50}-Tetracosane (nC24)	Nonacosane (nC29)
Triacontane (nC30)	57	71	d_{50}-Tetracosane (nC24)	Triacontane (nC30)
Untriacontane (nC31)	57	71	d_{50}-Tetracosane (nC24)	Untriacontane (nC31)
Dotriacontane (nC32)	57	71	d_{50}-Tetracosane (nC24)	Dotriacontane (nC32)
Tritriacontane (nC33)	57	71	d_{50}-Tetracosane (nC24)	Tritriacontane (nC33)
Tetratriacontane (nC34)	57	71	d_{50}-Tetracosane (nC24)	Tetratriacontane (nC34)
Pentatriacontane (nC35)	57	71	d_{50}-Tetracosane (nC24)	Pentatriacontane (nC35)
Hexatriacontane (nC36)	57	71	d_{50}-Tetracosane (nC24)	Hexatriacontane (nC36)

LABELLED SURROGATE STANDARD	Quantification Ion (m/z)	Confirmation Ion (m/z)	Recovery Calculated Against
d_{50}-Tetracosane (nC24)	66	82	d_{10}-Pyrene

LABELLED RECOVERY STANDARD	Quantification Ion (m/z)	Confirmation Ion (m/z)
d_{10}-Pyrene	212	208

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APPENDIX A

ANALYSIS OF HOPANES

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APPENDIX A: ANALYSIS OF HOPANES

The analysis of a suite of hopanes may be carried out on the F1 extract by GC/MS analysis, concurrently with the analysis of alkanes. The suite of hopanes determined, the ions monitored and the quantification references are listed in Table 12 below.

Table 12. Analyte Ions Monitored, Surrogate Used and RRF Determination for Hopanes

TARGET ANALYTES	Quantification Ion (m/z)	Confirmation Ion (m/z)	Surrogate	RRF Determined From
18(H)trisnorhopane	191	-	d_{50}-Tetracosane (nC24)	Tetracosane (nC24)
17(H)trisnorhopane	191	-	d_{50}-Tetracosane (nC24)	Tetracosane (nC24)
17(H),21(H)norhopane	191	-	d_{50}-Tetracosane (nC24)	Tetracosane (nC24)
17(H),21(H)hopane	191	-	d_{50}-Tetracosane (nC24)	Tetracosane (nC24)
S-17(H),21(H)homohopane	191	-	d_{50}-Tetracosane (nC24)	Tetracosane (nC24)
R-17(H),21(H)homohopane	191	-	d_{50}-Tetracosane (nC24)	Tetracosane (nC24)
LABELLED SURROGATE STANDARD	**Quantification Ion (m/z)**	**Confirmation Ion (m/z)**	**Recovery Calculated Against**	
d_{50}-Tetracosane (nC24)	66	82	d_{10}-Pyrene	
LABELLED RECOVERY STANDARD	**Quantification Ion (m/z)**	**Confirmation Ion (m/z)**		
d_{10}-Pyrene	212	208		

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APPENDIX B

Summary of Key Attributes of Methods MLA-021

EPA 8270C, EPA 8270D and EPA 1625B

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APPENDIX B: SUMMARY OF KEY ATTRIBUTES OF METHODS MLA-021, EPA 8270C, 8270D AND 1625B

Analysis by GC/LRMS, Key Attributes of AXYS MLA-021, EPA 8270C/D and EPA 1625B

	MLA-021	EPA 8270C	EPA 8270D	EPA 1625B
MS acquisition mode	SIM[1]	Full Scan or SIM[1]	Full Scan or SIM[1]	Full Scan[1]
Qualitative Identification Criteria	Retention time & ratio of 2 ions	Retention time & ratio of 3 [2] ions	Retention time & ratio of 3 [2] ions	Retention time & ratio of characteristic ions
MS Ion Ratio Criteria	20 %	30 %	30 %	-50 % to +200 %
MS Tuning Type and Check Frequency	PTFBA, daily	DFTTP[1], 12 hrs	DFTTP[1], 12 hrs	DFTTP[1], 8 hrs
Quantification References	Isotopically labeled standards added prior to extraction	Internal standards added prior to instrumental analysis	Internal standards added prior to instrumental analysis	Isotopically labeled standards added prior to extraction
Recovery correction of results	YES	NO	NO	YES
Initial Calibration, # levels	5 levels	5	5	5
Initial Calibration Limit (% RSD)	20 % (35 % if no labeled analog)	15 %	20 %	20 % (35 % if no labeled analog)
Calibration Verification Frequency	12 hrs	12 hrs	12 hrs	8 hrs
Calibration Verification Relative Response Limit (% diff.)	< 25 % of I-CAL	< 20 % of I-CAL	< 20 % of I-CAL	Various; most stringent is -20% to +25% of I-CAL
Calibration Verification IS area (% of I-CAL midpoint)	50-200 %	50-200 %	50-200 %	n.a.
Calibration verification IS RT (diff. from I-CAL midpoint)	n.a.	30 sec.	30 sec.	n.a.
Extraction	DCM (L/L (aqueous) DCM, Soxhlet (solids)	Options specified externally	Options specified externally	DCM, L/L (aqueous), pH>11 or pH other [3]

Notes:

1 SIM (Selected Ion Monitoring) acquisition protocol is permitted by EPA8270 and Federal Register Vol.77 Iss. 97 (May 18, 2012) Part 136

2 Based on availability, use of fewer ions is permitted

3 Modifications are permitted under Federal Register Vol.77 Iss.97 (May 18, 2012) Part 136.6

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APPENDIX C

MASS SPECTROMETER DAILY TUNING CHECK USING PFTBA

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APPENDIX C: MASS SPECTROMETER DAILY TUNING CHECK USING PFTBA

Using the Chem Station Software follow the following steps to check the mass spectrometer mass calibration, mass resolution and peak relative response:

1) Click the “View” option in Chem Station and select “Manual Tune”. Then select “AdjParam” and then “Edit MS Params”. Select “Prof” for profile scan and let the tune gas stabilize for 1 minute. Then select “File” and “Print”. Press “Stop”. All evaluation will be done from the printout.

2) A. Check the instrument “Mass Resolution” as per SIN-029 Rev. 03.

   B. Check “Mass Calibration” by measuring the amount of peak drift from the expected mass for M/Z 69, 219 and 502. If the peak apex has shifted more than approx 0.4 (or approx 2 mm on the calibration printout) then the instrument will need to be re-calibrated.

   C. Check “Relative Peak Response” of m/z 219 and 502 against m/z 69 by dividing the abundance of the desired peak, listed on the calibration check printout, by the abundance of the m/z 69 peak. If the relative peak intensities are outside a 50 - 150 % range compared to the reference tune, corrective action is required.

Corrective actions

3) A. If the mass resolution fails against SIN-029 Rev. 03 then return to step one and print out another calibration test. If the resolution fails a second time the instrument will need to be re-tuned.

   B. If the mass calibration is outside the specification, then in the “Manual Tune” page select “Calibrate” and then “Mass Axis”. Then return to step 1 above and re-check all specifications.

   C. If the relative intensities are outside the given specification then re-attempt step 1 and re-evaluate the printout. If the relative mass intensities are still out of spec, a full tune may be required.

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APPENDIX D

IDENTIFICATION AND QUANTIFICATION OF ALKYLATED PAH COMPOUND TOTALS

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APPENDIX D: IDENTIFICATION AND QUANTIFICATION OF ALKYLATED PAH COMPOUND TOTALS

RT windows for Alkylated PAH Compound Totals are determined from a retention time reference extract run at the beginning of the instrumental run list; assignment of the sample RT windows is made based on comparison of the peak retention times and peak patterns in samples to that of the retention time reference standard. Table 13 lists estimated 'typical' values.

Table 13. Analyte Ions Monitored, Surrogate Used and RRF Determination for Alkylated PAH Compound Totals

Alkylated PAH total parameter	Compounds/peaks	Typical peak/ window RT, min	Quant. ion	Conf. ion	Typical ion ratio	RRF reference	RT reference	Typical RT ref., min
C1-Biphenyls	(2 peaks)	11.22 - 11.44	168			Biphenyl	d10-Biphenyl	9.73
C2-Biphenyls	(6 or 7 peaks)	12.42 - 12.96	182			Biphenyl	d10-Biphenyl	9.73
C1-Naphthalenes	1-methylnaphthalene	8.80	142	141	0.965	1-methylnaphthalene	d_{10}-2-Methylnaphthalene	8.46
	2-methylnaphthalene	8.53	142	141	0.930	2-methylnaphthalene		
C2-Naphthalenes	Ethyl/dimethylnaphthalenes	9.96 - 10.75	156	141		Average of 1,2-dimethylnaphthalene and 2,6-dimethylnaphthalene	d12-2,6-dimethyl-naphthalene	10.05
	1,2-dimethylnaphthalene	10.88	156	141	1.320	1,2-dimethylnaphthalene		
C3-Naphthalenes	(≥2 peaks)	11.65 - 12.95	170			Average of 2,3,5-Trimethylnaphthalene and 2,3,6-Trimethylnaphthalene	d12-2,6-dimethyl-naphthalene	10.05
C4-Naphthalenes	(≥2 peaks)	13.05 - 14.65	184			1,4,6,7-Tetramethylnaphthalene	d12-2,6-dimethyl-naphthalene	10.05
C1-Acenaphthenes	(≥2 peaks)	11.60 - 11.70	168			Acenaphthene	d10-Acenaphthene	11.23
C1-Fluorenes	(≥2 peaks)	13.73 - 14.23	180			2-Methylfluorene	d10-Phenanthrene	14.96
C2-Fluorenes	(≥2 peaks)	14.96 - 16.01	194			1,7-Dimethylfluorene	d10-Phenanthrene	14.96
C3-Fluorenes	(≥2 peaks)	16.25 - 17.70	208			1,7-Dimethylfluorene	d10-Phenanthrene	14.96
C1-Dibenzothiophenes	C1-dibenzothiophenes-1	15.84	198			2/3-Methyldibenzothiophenes	d8-Dibenzothiophene	14.67
	2/3-methyldibenzo-thiophenes	16.05	198					
	C1-dibenzothiophenes-2	16.32	198					
C2-Dibenzothiophenes	(≥2 peaks)	16.35 - 17.95	212			2,4-Dimethyldibenzothiophene	d8-Dibenzothiophene	14.67
C3-Dibenzothiophenes	(≥2 peaks)	17.70 - 19.20	226			2,4-Dimethyldibenzothiophene	d8-Dibenzothiophene	14.67
C4-Dibenzothiophenes	(≥2 peaks)	18.80 - 20.50	240			2,4-Dimethyldibenzothiophene	d8-Dibenzothiophene	14.67
C1-Phenanthrenes/ Anthracenes	3-methylphenanthrene	16.27	192	191	0.618	Average of 1-Methylphenanthrene, 2-Methylphenanthrene and 2-	d10-Phenanthrene	14.96

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Alkylated PAH total parameter	Compounds/peaks	Typical peak/ window RT, min	Quant. ion	Conf. ion	Typical ion ratio	RRF reference	RT reference	Typical RT ref., min
						Methylanthracene		
	2-methylphenanthrene	16.33	192	191	0.610	2-methylphenanthrene		
	2-methylanthracene	16.44	192	191	0.531	2-methylanthracene		
	9/4-methylphenanthrenes	16.56	192	191	0.621	Average of 1-Methylphenanthrene, 2-Methylphenanthrene and 2-Methylanthracene		
	1-methylphenanthrene	16.61	192	191	0.629	1-methylphenanthrene		
C2-Phenanthrenes/ Anthracenes	Ethyl/dimethyl-phenanthrenes/anthracenes	17.22 - 17.95	206			Average of 3,6-Dimethylphenanthrene and 1,7-Dimethylphenanthrene	d10-Fluoranthene	17.99
	1,8-dimethylphenanthrene	18.10	206	191	0.365	Average of 3,6-Dimethylphenanthrene and 1,7-Dimethylphenanthrene		
C3-Phenanthrenes/ Anthracenes	(≥2 peaks)	18.40 - 19.40	220			1,2,6-Trimethylphenanthrene	d10-Fluoranthene	17.99
C4-Phenanthrenes/ Anthracenes	(≥2 peaks)	19.45 - 21.55	234			Retene	d10-Fluoranthene	17.99
C1-Fluoranthenes /Pyrenes	C1-Fluoranthenes/Pyrenes-1 (2 peaks)	19.17 - 19.58	216			3-Methylfluoranthene	d10-Fluoranthene	17.99
	C1-Fluoranthenes/Pyrenes-2 (3 or 4 peaks)	19.70 - 20.07	216			3-Methylfluoranthene		
C2-Fluoranthenes/ Pyrenes	(≥2 peaks)	20.50 - 21.50	230			3-Methylfluoranthene	d10-Fluoranthene	17.99
C3-Fluoranthenes/ Pyrenes	(≥2 peaks)	21.80 - 23.15	244			3-Methylfluoranthene	d10-Fluoranthene	17.99
C4-Fluoranthenes/ Pyrenes	(≥2 peaks)	23.20 - 24.50	258			3-Methylfluoranthene	d10-Fluoranthene	17.99
C1-Benz(a)anthracenes/ Chrysenes	(7 or more peaks)	22.60 - 23.38	242			Average of 5/6-methylchrysenes and 1-methylchrysene	d12-Chrysene	21.70
C2-Benz(a)anthracenes/ Chrysenes	(≥2 peaks)	23.70 - 25.00	256			5,9-Dimethylchrysene	d12-Chrysene	21.70
C3-Benz(a)anthracenes/ Chrysenes	(≥2 peaks)	25.20 - 26.20	270			5,9-Dimethylchrysene	d12-Chrysene	21.70
C4-Benz(a)anthracenes/ Chrysenes	(≥2 peaks)	26.20 - 27.90	284			5,9-Dimethylchrysene	d12-Chrysene	21.70
C1-Benzofluoranthenes/ Benzopyrenes	(≥2 peaks)	27.26 - 29.80	266			7-Methylbenzo(a)pyrene	d12-Benzo(a)pyrene	26.40
C2-Benzofluoranthenes/ Benzopyrenes	(≥2 peaks)	29.80 - 30.85	280			7-Methylbenzo(a)pyrene	d12-Benzo(a)pyrene	26.40

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APPENDIX E

TYPICAL CHROMATOGRAMS OF

ALKYLATED PAH COMPOUND TOTALS

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APPENDIX E: TYPICAL CHROMATOGRAMS OF ALKYLATED PAH COMPOUND TOTALS

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Sample    : WG33981-107,,CRM                           Inst    : Instrumen
Misc      : 1,WG33981,1.0/500uL                        Multiplr: 1.00
MS Integration Params: BENZbk.P
Quant Time: May 23 13:32 2012              Quant Results File: temp.res

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Title        : PAH LIN (c28B-F\CV4\s35A36A\r22A23A) ERA 12DEC08
Last Update  : Wed Nov 17 16:59:38 2010
Response via : Single Level Calibration
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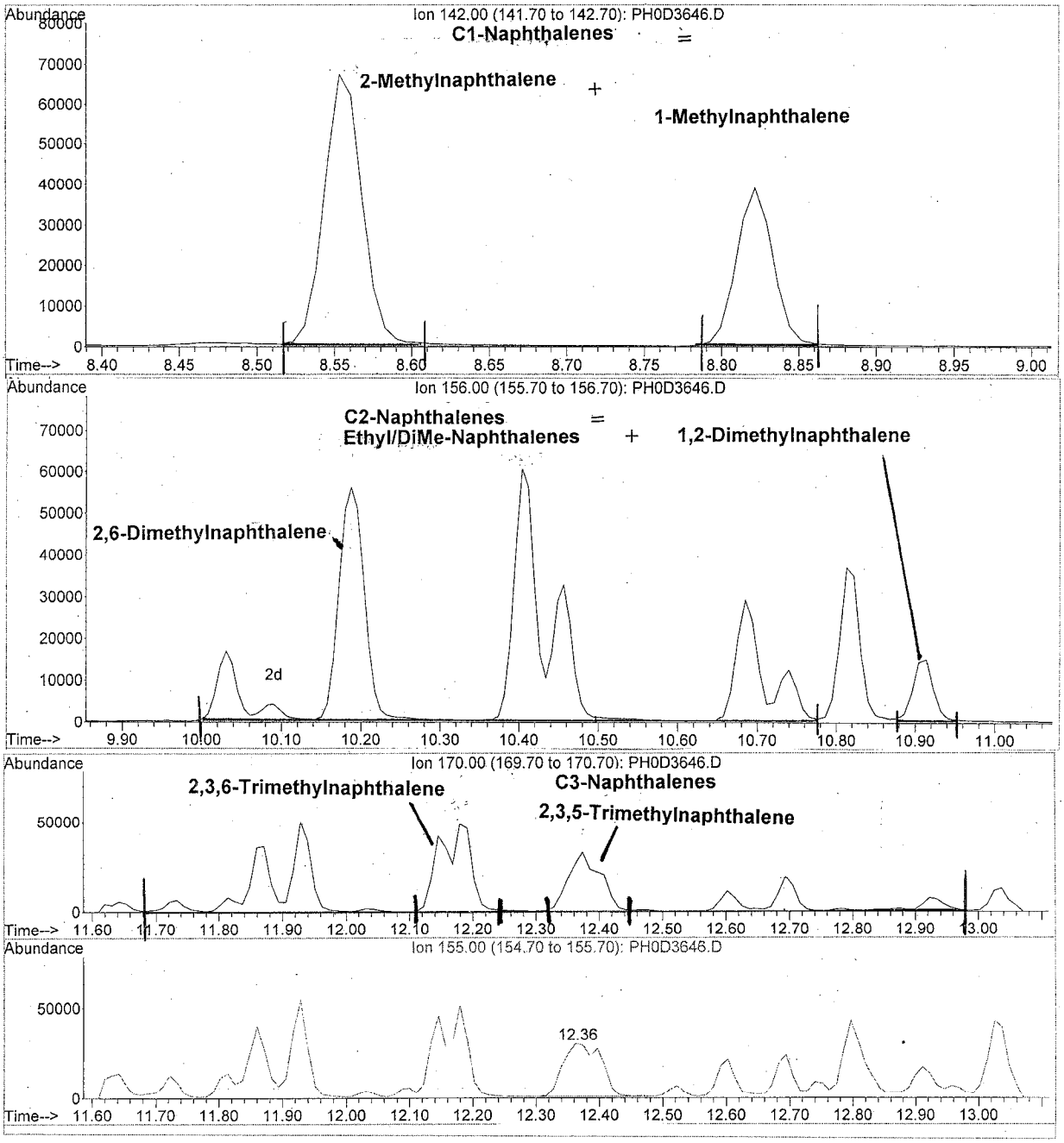

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MS Integration Params: BENZbk.P
Quant Time: May 18 17:44 2012              Quant Results File: temp.res

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Last Update  : Wed Nov 17 16:59:38 2010
Response via : Multiple Level Calibration
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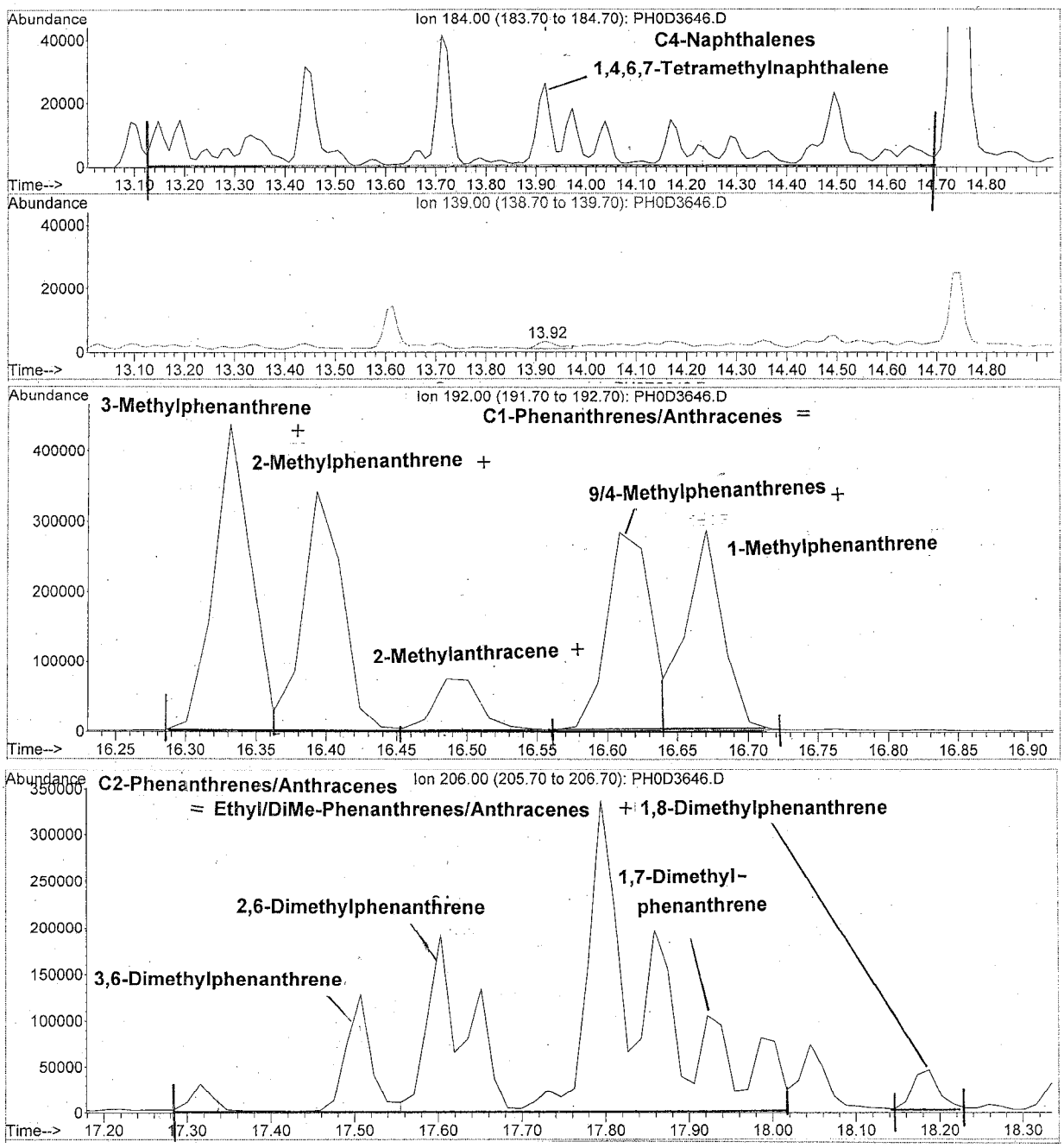

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MS Integration Params: BENZbk.P
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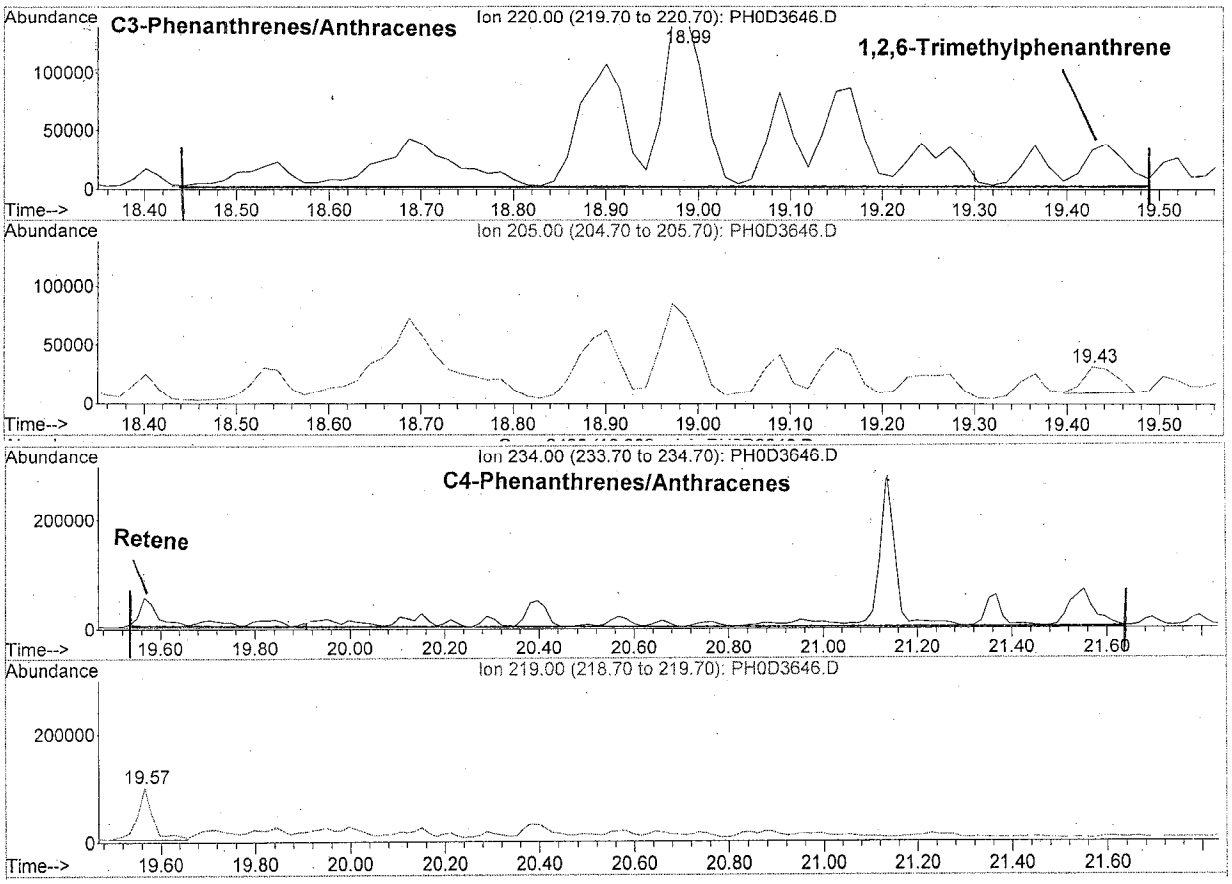

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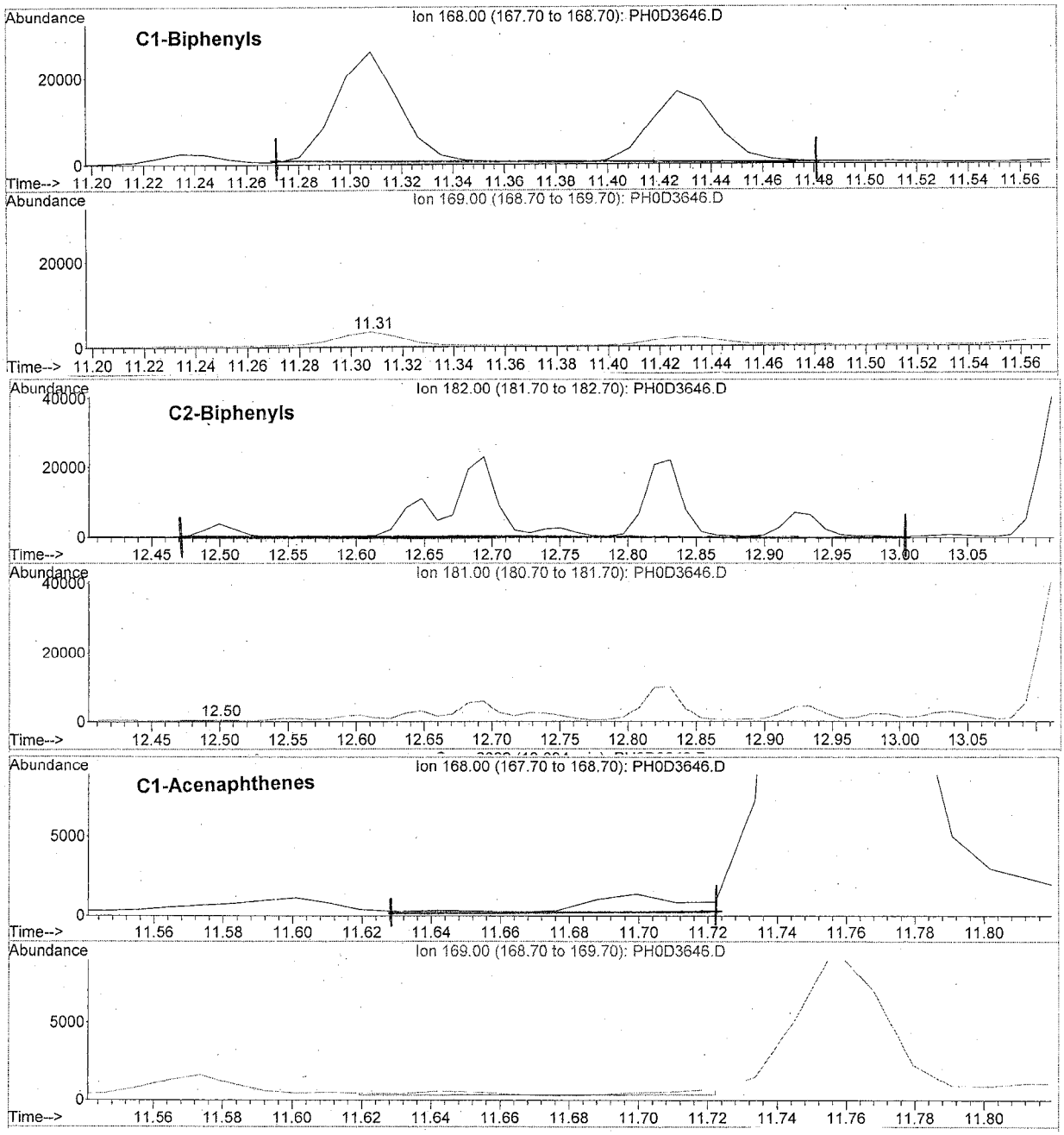
Last Update : Fri May 18 18:18:17 2012
Response via : Multiple Level Calibration



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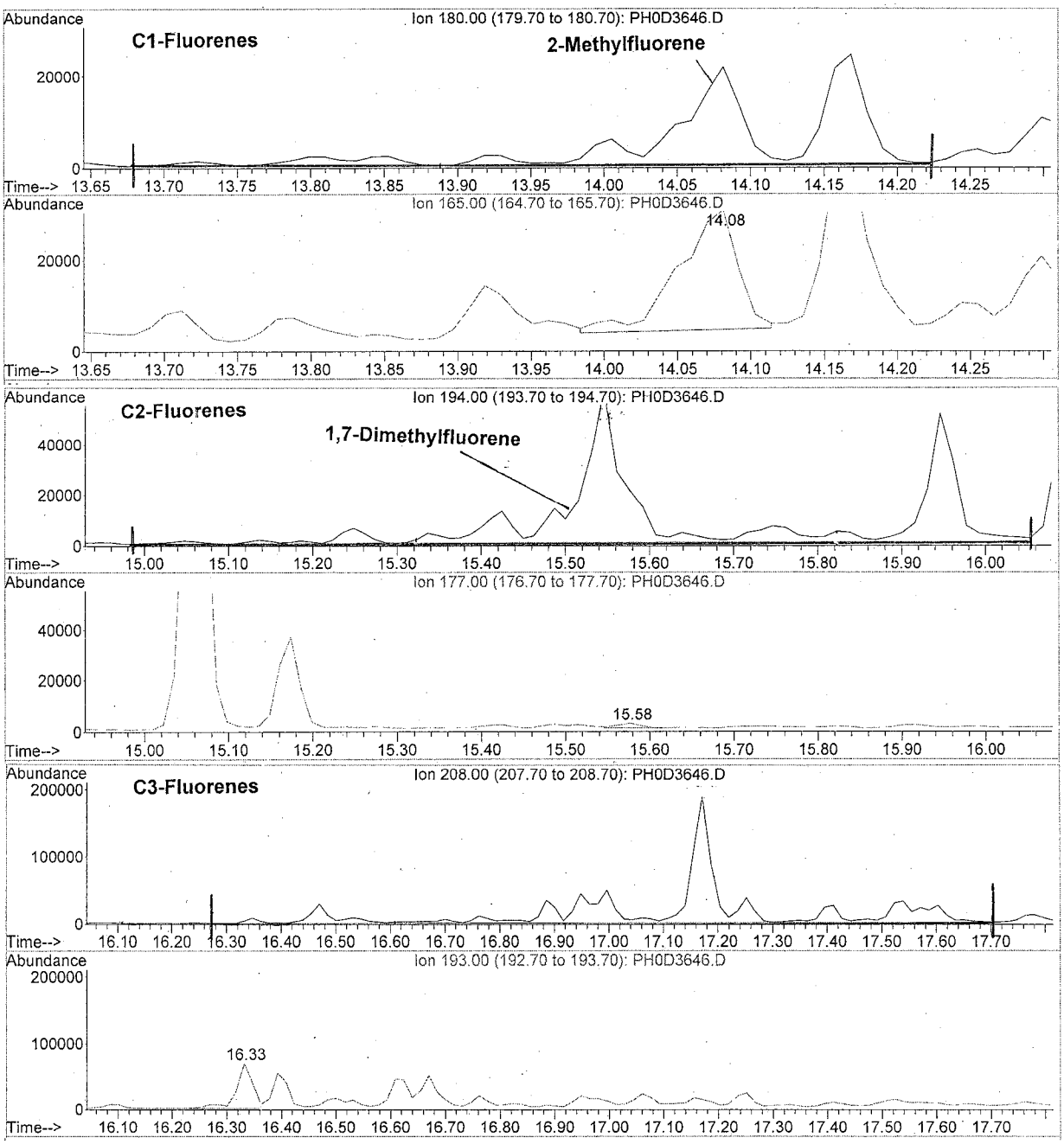

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MS Integration Params: BENZbk.P
Quant Time: May 23 14:03 2012              Quant Results File: temp.res

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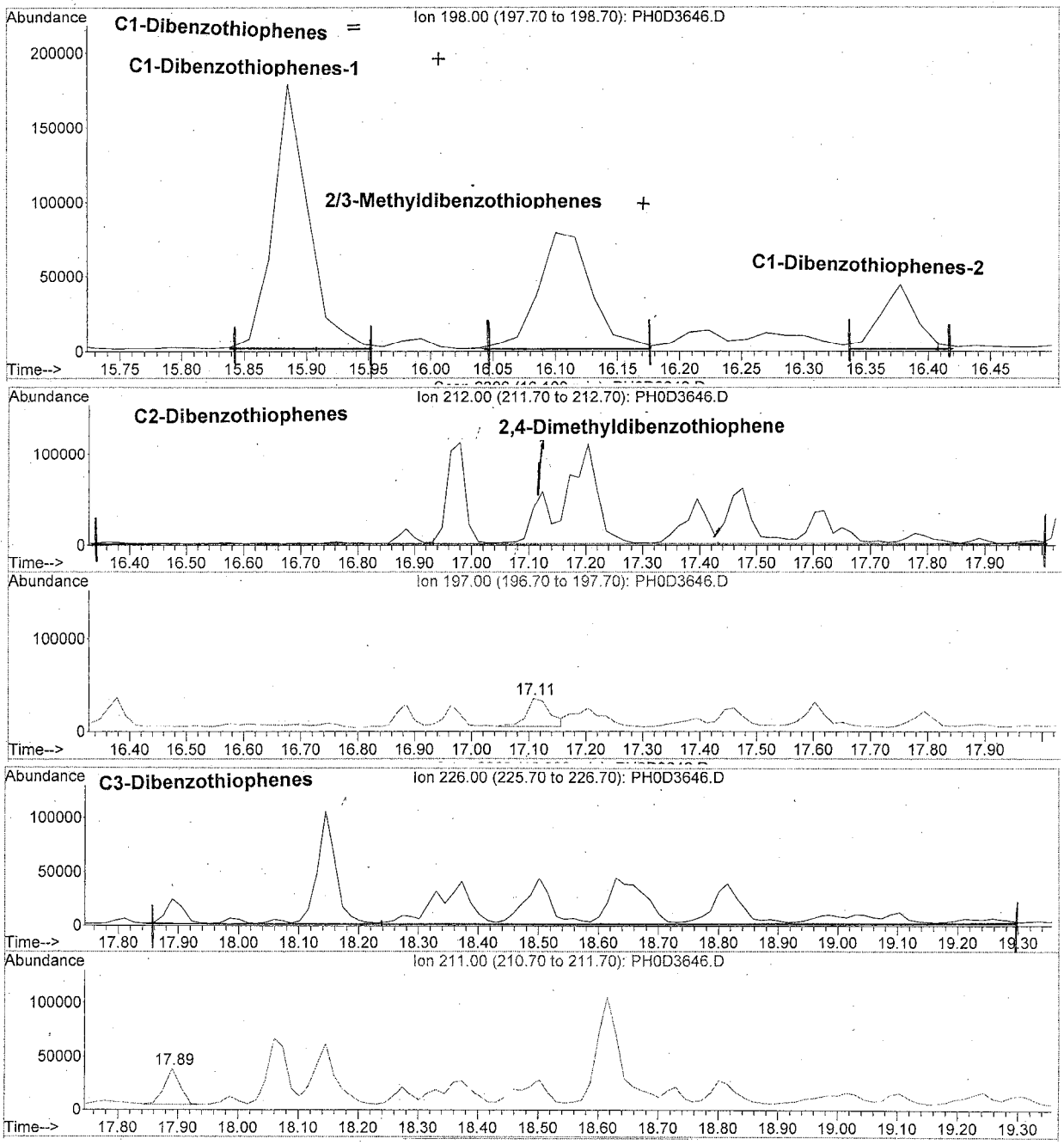

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Misc      : 1,WG33981,1.0/500uL                        Multiplr: 1.00
MS Integration Params: BENZbk.P
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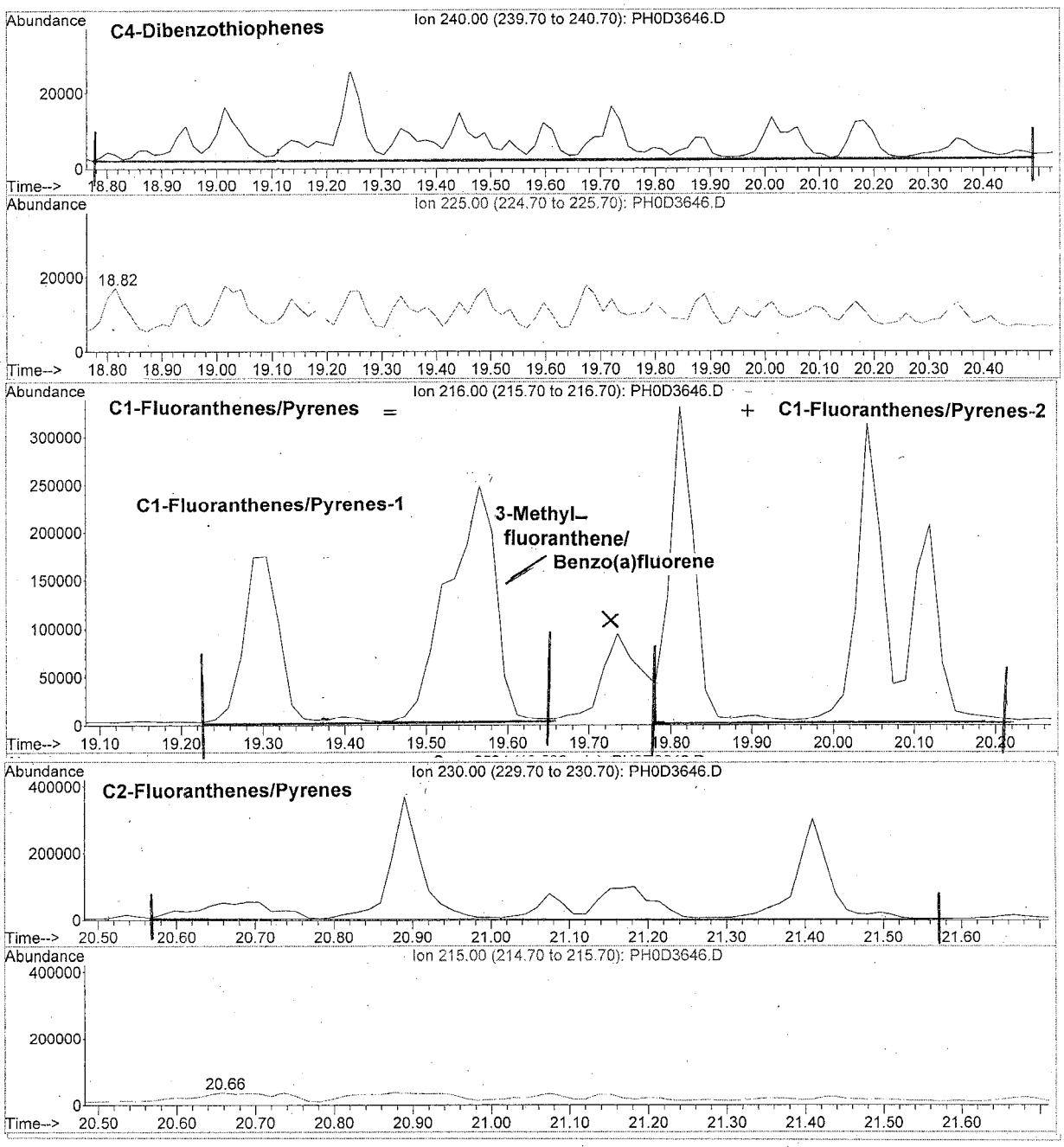

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Sample    : WG33981-107,,CRM                           Inst    : Instrumen
Misc      : 1,WG33981,1.0/500uL                        Multiplr: 1.00
MS Integration Params: BENZbk.P
Quant Time: May 23 14:07 2012              Quant Results File: temp.res

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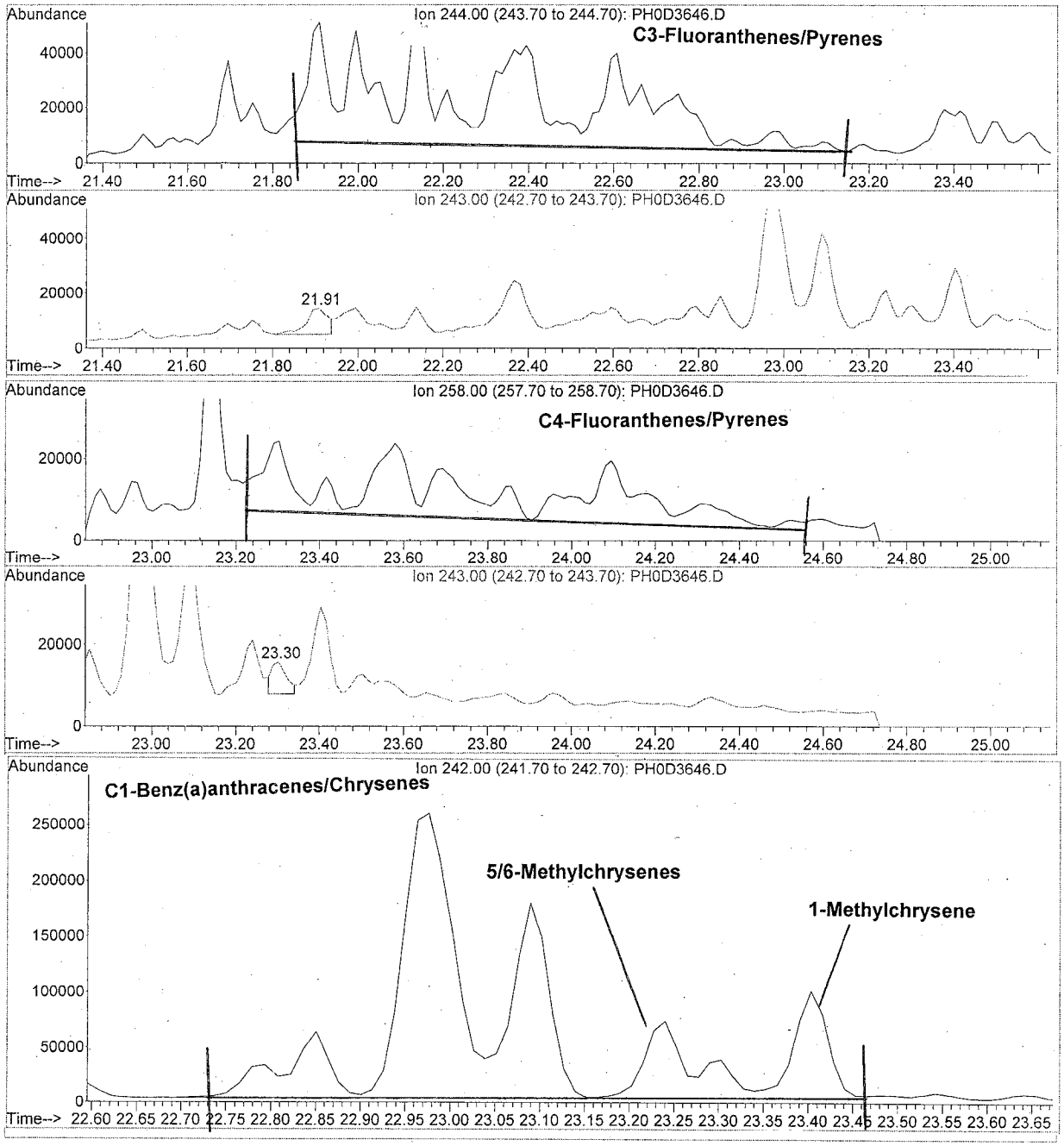

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```
Data File : G:\PROLAB\INST_D\_2010\101002PH\PH0D3646.D   Vial: 12
Acq On    :  2 Oct 2010  20:40                          Operator: RTX-5 30M S/N 958856 BW
Sample    : WG33981-107,,CRM                           Inst    : Instrumen
Misc      : 1,WG33981,1.0/500uL                        Multiplr: 1.00
MS Integration Params: BENZbk.P
Quant Time: May 23 14:08 2012              Quant Results File: temp.res

MethodPH100.M G:\PROLAB\INST_D\_2010\101002PH\088IH-B.M (RTE Integrator)
Title         : PAHEX(c28E/CV2/s35A36A/r22A23A) 14OCT08
Last Update   : Fri May 18 18:18:17 2012
Response via  : Multiple Level Calibration
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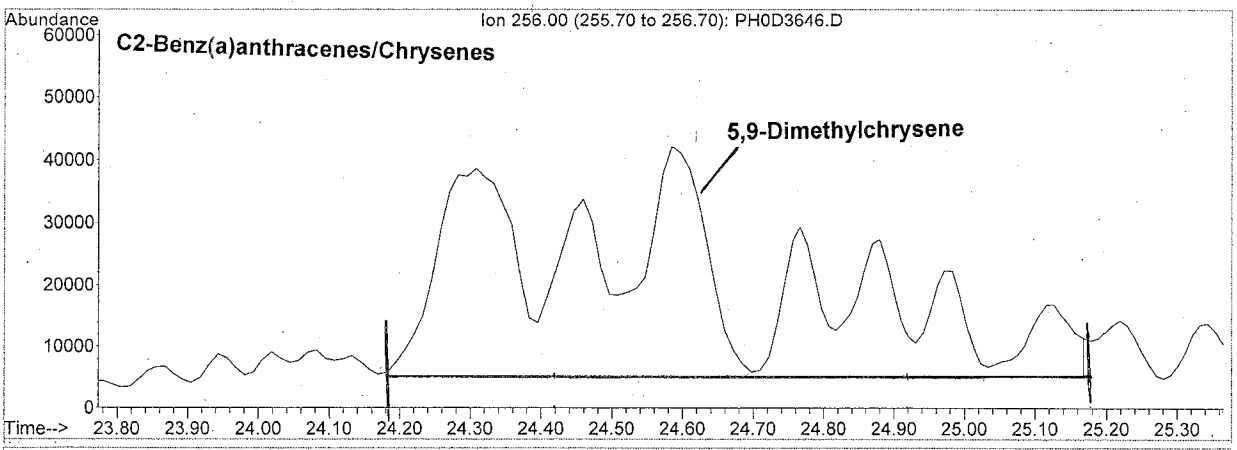

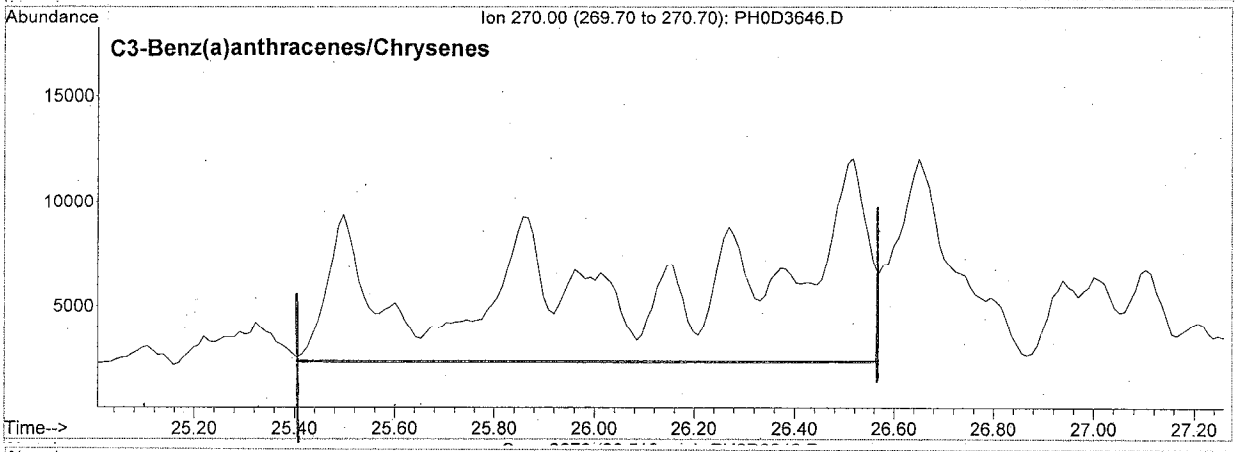

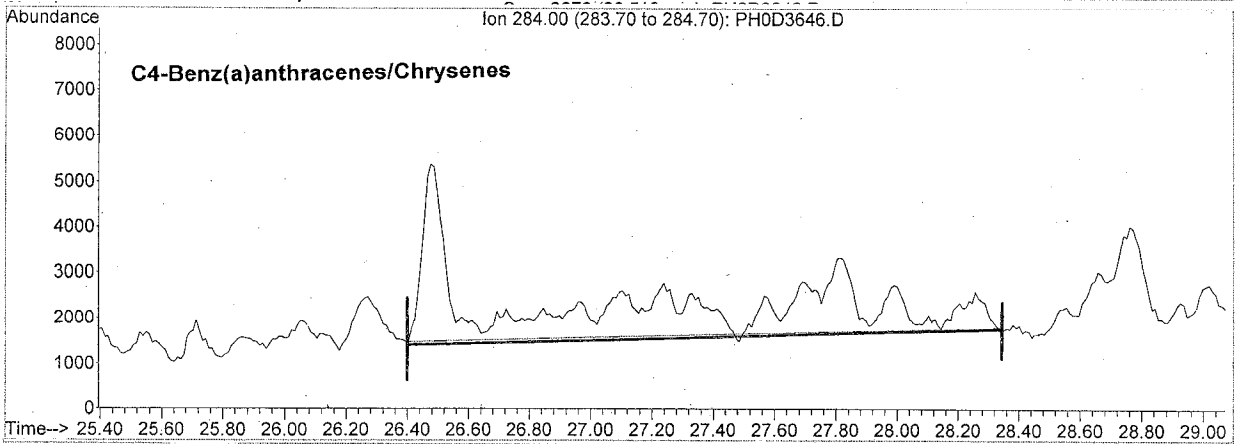

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```
Data File : G:\PROLAB\INST_D\_2010\101002PH\PH0D3646.D   Vial: 12
Acq On    :  2 Oct 2010  20:40                          Operator: RTX-5 30M S/N 958856 BW
Sample    : WG33981-107,,CRM                           Inst    : Instrumen
Misc      : 1,WG33981,1.0/500uL                        Multiplr: 1.00
MS Integration Params: BENZbk.P
Quant Time: May 23 14:10 2012              Quant Results File: temp.res

Method PH100.M G:\PROLAB\INST_D\_2010\101002PH\088IH-B.M (RTE Integrator)
Title        : PAHEX(c28E/CV2/s35A36A/r22A23A) 14OCT08
Last Update  : Fri May 18 18:18:17 2012
Response via : Multiple Level Calibration
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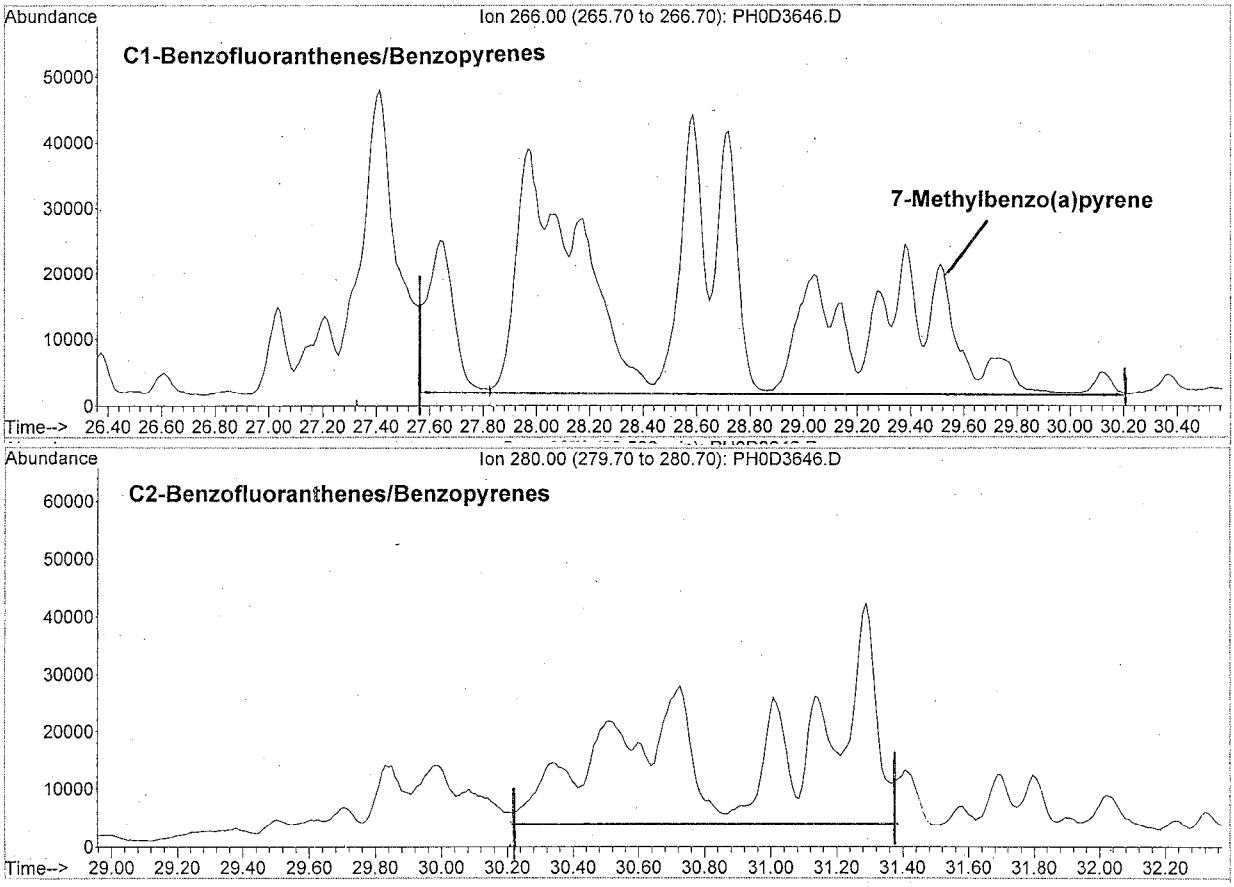

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APPENDIX F

ANALYSIS OF 2-CHLORONAPHTHALENE

BY MLA-021 AND GC-HRMS

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APPENDIX F: ANALYSIS OF 2-CHLORONAPHTHALENE BY GC-HRMS

The analysis of 2-chloronaphthalene in solid and ash samples may be carried out on the F2 extract by GC-HRMS analysis. The modifications below apply for 2-chloronaphthalene analysis:

Sample Pretreatment and Preparation: Refer to section 2, Solid (Sediment/Soils/Ash) Samples.

Surrogate Standard Solution: Typically add 50 µL of surrogate standard solution containing 1500 ng/mL of the surrogate standard d7-2-chloronaphthalene dissolved in hexane.

Recovery Standard Solution: Typically add 100 µL of recovery standard solution containing 20000 ng/mL of the recovery standard d10-acenaphthene dissolved in dichloromethane.

Native Spiking Standard Solution for OPR and SAR: Typically use 50 µL of native standard solution containing 1000 ng/mL of 2-chloronaphthalene dissolved in hexane.

Quality Assurance/Quality Control: Refer to section 4 with the following modifications:

- Prepare an SAR solution with each analysis batch to be used for bracketing calibration. Use 50 µL native standard solution, 50 µL surrogate standard solution and 100 µL recovery standard solution diluted to 500 µL with dichloromethane.
- Prepare and analyze an OPR sample with each analysis batch. Spike 50 µL native standard solution into a sample matrix known to contain low background levels of target analytes.
- Refer to Table 8 and Table 14 for QC acceptance criteria.

Table 14. QC Acceptance Criteria for 2-Chloronaphthalene

Compound	Procedural Blank Level (ng/sample)	OPR and Sample Recovery Range (%)
2-Chloronaphthalene	< 1	50 – 150
d7-2-Chloronaphthalene	n.a.	30 – 150

Extraction Procedures: Refer to Section 5 and Section 5.1.

Cleanup procedures: Refer to Section 6, using fraction F2 from the silica column cleanup.

GC-HRMS Analysis: High resolution gas chromatography/high resolution mass spectrometry analysis of the F2 fraction is performed using a capillary gas chromatograph

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coupled to a high-resolution mass spectrometer. A J&W DB-5 chromatography column (60 m, 0.25 mm i.d., 0.10 µm film thickness)) is coupled directly to the HRMS source. The HRMS is operated at a static (8000) mass resolution (10% valley) in the electron ionization (EI) mode using multiple ion detection (MID) acquiring two characteristic ions for each target analyte and surrogate standard. A splitless/split injection sequence is used. Typical operating conditions for the GC-HRMS are presented in Table 15. The typical extract volume is 500 µL; 1.0 µL is injected. The ions acquired and the quantification references are listed in Table 16. Sample concentrations are determined by bracketing calibration using the SAR solution.

Table 15. Typical GC-HRMS Operating Conditions for 2-Chloronaphthalene Analysis

GC Temperature Program		General GC Conditions	
Temp (°C)	50	Injector Temp (°C)	180
Hold time (min)	2	Injector	Split/splitless, 2 min
Rate (C° min^{-1})	30	Carrier Gas	Helium, 200 kPa (new column) Variable (depending on column length)
Temp (°C)	120		
Hold time (min)	0		
Rate (C° min^{-1})	7	**MS Conditions**	
Temp (°C)	180	Source Temp (°C)	280
Hold time (min)	0	Electron Energy (eV)	~35
Rate (C° min^{-1})	50	Trap Current (µA)	500 – 900
Temp (°C)	310	Mass Resolution	8000
Hold time (min)	3	Detector Voltage (V)	340 -400

Table 16. Analyte Ions Monitored, Surrogates Used and RRF Determination for 2-Chloronaphthalene Analysis

Compound	Quantification and RRT Standard	Typical Retention Time (min.)	Quant. Ion (m/z)	Conf. Ion (m/z)	Typical Ion Ratio (Quant./Conf.)	Ion Ratio Tolerance
2-Chloronaphthalene	d7-2-Chloronaphthalene	11:53	162.0236	164.0207	3.12	20 %
d7-2-Chloronaphthalene	d10-Acenaphthene	11:50	169.0676	171.0646	3.12	20 %
d10-Acenaphthene		13:21	162.1269	164.1410	1.1	20 %
Lock-mass			168.9888			

Qualitative and Quantitative Determination: Refer to Section 8.

Reporting Criteria and Practices: Refer to Section 9.

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APPENDIX G: QUANTIFICATION OF CLIENT, FIELD (PRC) AND PHOTOLYSIS STANDARDS

This section is applicable for determination of PAHs in PUF (polyurethane foam) and in SPMD (semipermeable membrane device) samples.

Because PUF and SPMD samples require the use of d_{10}-anthracene and d_{10}-fluoranthene as client or field standards, and SPMD samples require the use of d_{14}-dibenzo(ah)anthracene as photolytic standard, the default quantification scheme (Table 10) must be modified as per Table 17 below.

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Table 17. Analyte Ions Monitored, Surrogates Used and RRF Determination for PAH using Client, Field, PRC and Photolysis Standards

TARGET ANALYTES	Quantification Ion (m/z)	Confirmation Ions (m/z)	Typical Ion Ratio (Conf./Quant.)	SURROGATE	Typical Retention Time (minutes)	RRF DETERMINED FROM
Naphthalene	128	102	0.064	d_8-Naphthalene	6.84	Naphthalene
Acenaphthylene	152	151	0.222	d_8-Acenaphthylene	10.83	Acenaphthylene
Acenaphthene	154	153	1.18	d_8-Acenaphthylene	11.33	Acenaphthene
Fluorene	166	165	1.01	d_{10}-Phenanthrene	12.63	Fluorene
Phenanthrene	178	176	0.202	d_{10}-Phenanthrene	15.04	Phenanthrene
Anthracene	178	176	0.196	d_{10}-Phenanthrene	15.15	Anthracene
Fluoranthene	202	200	0.214	d_{10}-Phenanthrene	18.06	Fluoranthene
Pyrene	202	200	0.219	d_{10}-Phenanthrene	18.60	Pyrene
Benz[a]anthracene	228	226	0.281	d_{12}-Benz[a]anthracene	21.68	Benz[a]anthracene
Chrysene	228	226	0.312	d_{12}-Chrysene	21.79	Chrysene
Benzo[b]fluoranthene	252	253	0.218	d_{12}-Benzo[b]fluoranthene	25.21	Benzo[b]fluoranthene
Benzo[j,k]fluoranthenes	252	253	0.215	d_{12}-Benzo[k]fluoranthene	25.30	Benzo[k]fluoranthene
Benzo[e]pyrene	252	253	0.213	d_{12}-Benzo[a]pyrene	26.36	Benzo[e]pyrene
Benzo[a]pyrene	252	253	0.217	d_{12}-Benzo[a]pyrene	26.58	Benzo[a]pyrene
Perylene	252	253	0.212	d_{12}-Perylene	27.00	Perylene
Dibenzo[ah]anthracene	278	139	0.144	d_{12}-Indeno[1,2,3,cd]pyrene	31.86	Dibenz[ah]anthracene
Indeno[1,2,3-cd]pyrene	276	138	0.179	d_{12}-Indeno[1,2,3,cd]pyrene	31.71	Indeno[1,2,3-cd]pyrene
Benzo[ghi]perylene	276	138	0.194	d_{12}-Benzo[ghi]perylene	32.53	Benzo[ghi]perylene
Biphenyl	154	152	0.304	d_{10}- Biphenyl	9.81	Biphenyl
Dibenzothiophene	184	152	0.073	d_8-Dibenzothiophene	14.72	Dibenzothiophene
1-Methylnaphthalene	142	141	0.962	d_{10}-2-Methylnaphthalene	8.81	1-Methylnaphthalene
2-Methylnaphthalene	142	141	0.930	d_{10}-2-Methylnaphthalene	8.55	2-Methylnaphthalene
C1-Naphthalenes	142	[4]	[4]	d_{10}-2-Methylnaphthalene	[5]	1- & 2-Methylnaphthalene
2,6-Dimethylnaphthalene	156	141	0.666	d_{12}-2,6 Dimethylnaphthalene	10.17	2,6-Dimethylnaphthalene
1,2-Dimethylnaphthalene	156	141	1.26	d_{12}-2,6 Dimethylnaphthalene	10.90	1,2-Dimethylnaphthalene
C2-Naphthalenes	156	[4]	[4]	d_{12}-2,6 Dimethylnaphthalene	[5]	2,6- & 1,2-Dimethylnaphthalene
2,3,5-Trimethylnaphthalene	170	155	0.873	d_{12}-2,6 Dimethylnaphthalene	12.35	2,3,5- Trimethylnaphthalene
2,3,6-Trimethylnaphthalene	170	155	0.876	d_{12}-2,6 Dimethylnaphthalene	12.17	2,3,6- Trimethylnaphthalene
C3-Naphthalenes	170			d_{12}-2,6 Dimethylnaphthalene	[5]	2,3,5- & 2,3,6-Trimethylnaphthalene
1,4,6,7-Tetramethylnaphthalene	184	139	0.027	d_{12}-2,6 Dimethylnaphthalene	13.89	1,4,6,7-Tetramethylnaphthalene

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C4-Naphthalene	184	[4]	[4]	d_{12}-2,6 Dimethylnaphthalene	[5]	1,4,6,7-Tetramethylnaphthalene
2-Methylanthracene	192	191	0.531	d_{10}-Phenanthrene	16.45	2-Methylanthracene
3-Methylphenanthrene	192	191	0.608	d_{10}-Phenanthrene	16.27	1- & 2-Methylphenanthrene & 2-Methylanthracene
2-Methylphenanthrene	192	191	0.608	d_{10}-Phenanthrene	16.36	2-Methylphenanthrene
9/4-Methylphenanthrenes	192	191	0.634	d_{10}-Phenanthrene	16.59	1- & 2-Methylphenanthrene & 2-Methylanthracene
1-Methylphenanthrene	192	191	0.634	d_{10}-Phenanthrene	16.64	1-Methylphenanthrene
C1-Phenanthrenes/Anthracenes	192	[4]	[4]	d_{10}-Phenanthrene	[5]	1- & 2-Methylphenanthrene & 2-Methylanthracene
3,6-Dimethylphenanthrene	206	191	0.342	d_{10}-Phenanthrene	17.46	3,6-Dimethylphenanthrene
2,6-Dimethylphenanthrene	206	191	0.342	d_{10}-Phenanthrene	17.54	3,6- & 1,7-Dimethyl-phenanthrenes
1,7-Dimethylphenanthrene	206	191	0.332	d_{10}-Phenanthrene	17.89	1,7-Dimethylphenanthrene
1,8-Dimethylphenanthrene	206	191	0.332	d_{10}-Phenanthrene	18.13	3,6- & 1,7-Dimethyl-phenanthrenes
C2-Phenanthrenes/Anthracenes	206	[4]	[4]	d_{10}-Phenanthrene	[5]	3,6- & 1,7-Dimethyl-phenanthrenes
1,2,6-Trimethylphenanthrene	220	205	0.581	d_{10}-Phenanthrene	19.41	1,2,6-Trimethylphenanthrene
C3-Phenanthrenes/Anthracenes	220	[4]	[4]	d_{10}-Phenanthrene	[5]	1,2,6-Trimethylphenanthrene
Retene	234	219	1.63	d_{10}-Phenanthrene	19.53	Retene
C4-Phenanthrenes/Anthracenes	234	[4]	[4]	d_{10}-Phenanthrene	[5]	Retene
C1-Biphenyls	168	[4]	[4]	d_{10}- Biphenyl	[5]	Biphenyl
C2-Biphenyls	182	[4]	[4]	d_{10}- Biphenyl	[5]	Biphenyl
C1-Acenaphthenes	168	[4]	[4]	d_8-Acenaphthylene	[5]	Acenaphthene
2-Methylfluorene	180	165	1.23	d_{10}-Phenanthrene	14.06	2-Methylfluorene
C1-Fluorenes	180	[4]	[4]	d_{10}-Phenanthrene	[5]	2-Methylfluorene
1,7-Dimethylfluorene	194	177	0.092	d_{10}-Phenanthrene	15.49	1,7-Dimethylfluorene
C2-Fluorenes	194	[4]	[4]	d_{10}-Phenanthrene	[5]	1,7-Dimethylfluorene
C3-Fluorenes	208	[4]	[4]	d_{10}-Phenanthrene	[5]	1,7-Dimethylfluorene
2/3-Methyldibenzothiophenes	198	197	0.738	d_8-Dibenzothiophene	16.07	2/3-Methyldibenzothiophenes
C1-Dibenzothiophenes	198	[4]	[4]	d_8-Dibenzothiophene	[5]	2/3-Methyldibenzothiophenes
2,4-Dimethyldibenzothiophene	212	197	0.514	d_8-Dibenzothiophene	17.08	2,4-Dimethyldibenzothiophene
C2-Dibenzothiophenes	212	[4]	[4]	d_8-Dibenzothiophene	[5]	2,4-Dimethyldibenzothiophene
C3-Dibenzothiophenes	226	[4]	[4]	d_8-Dibenzothiophene	[5]	2,4-Dimethyldibenzothiophene

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C4-Dibenzothiophenes	240	[4]	[4]	d_8-Dibenzothiophene	[5]	2,4-Dimethyldibenzothiophene
3-Methylfluoranthene/Benzo(a)fluorene	216	215	0.880	d_{10}-Phenanthrene	19.53	3-Methylfluoranthene
C1-Fluoranthenes/Pyrenes	216	[4]	[4]	d_{10}-Phenanthrene	[5]	3-Methylfluoranthene
C2-Fluoranthenes/Pyrenes	230	[4]	[4]	d_{10}-Phenanthrene	[5]	3-Methylfluoranthene
C3-Fluoranthenes/Pyrenes	244	[4]	[4]	d_{10}-Phenanthrene	[5]	3-Methylfluoranthene
C4-Fluoranthenes/Pyrenes	258	[4]	[4]	d_{10}-Phenanthrene	[5]	3-Methylfluoranthene
5/6-Methylchrysenes	242	[4]	[4]	d_{12}-Chrysene	23.15	6-Methylchrysene
1-Methylchrysene	242	[4]	[4]	d_{12}-Chrysene	23.32	1-Methylchrysene
C1-Benz(a)anthracenes/Chrysenes	242	[4]	[4]	d_{12}-Chrysene	[5]	1- & 6-Methylchrysenes
5,9-Dimethylchrysene	256	[4]	[4]	d_{12}-Chrysene	24.49	5,9-Dimethylchrysene
C2-Benz(a)anthracenes/Chrysenes	256	[4]	[4]	d_{12}-Chrysene	[5]	5,9-Dimethylchrysene
C3-Benz(a)anthracenes/Chrysenes	270	[4]	[4]	d_{12}-Chrysene	[5]	5,9-Dimethylchrysene
C4-Benz(a)anthracenes/Chrysenes	284	[4]	[4]	d_{12}-Chrysene	[5]	5,9-Dimethylchrysene
7-Methylbenzo(a)pyrene	266	[4]	[4]	d_{12}-Benzo[a]pyrene	29.35	7-Methylbenzo(a)pyrene
C1-Benzofluoranthenes/Benzo-pyrenes	266	[4]	[4]	d_{12}-Benzo[a]pyrene	[5]	7-Methylbenzo(a)pyrene
C2-Benzofluoranthenes/Benzo-pyrenes	280	[4]	[4]	d_{12}-Benzo[a]pyrene	[5]	7-Methylbenzo(a)pyrene
LABELLED CLIENT/FIELD STANDARDS	**Quantification Ion (m/z)**	**Confirmation Ions (m/z)**	**Typical Ion Ratio (Conf./Quant.)**	**RECOVERY CALCULATED AGAINST**	**Typical Retention Time (minutes)**	**RRF DETERMINED FROM**
d_{10}-Anthracene	188	184		d_{10}-Phenanthrene	15.1	
d_{10}-Fluoranthene	212	208	0.173	d_{12}-Benz(a)anthracene	18.02	
LABELLED PHOTOLYSIS STANDARD	**Quantification Ion (m/z)**	**Confirmation Ions (m/z)**	**Typical Ion Ratio (Conf./Quant.)**	**RECOVERY CALCULATED AGAINST**	**Typical Retention Time (minutes)**	**RRF DETERMINED FROM**
d_{14}-Dibenzo[ah]anthracene	292	288	0.260	d_{12}-Indeno[1,2,3-cd]pyrene	31.75	
LABELLED SURROGATE STANDARDS	**Quantification Ion (m/z)**	**Confirmation Ions (m/z)**	**Typical Ion Ratio (Conf./Quant.)**	**RECOVERY CALCULATED AGAINST**	**Typical Retention Time (minutes)**	
d_8-Naphthalene	136	134	0.095	d_{10}-Acenaphthene	6.80	
d_{10}-2-Methylnaphthalene	152	151	0.195	d_{10}-Acenaphthene	8.47	
d_{10}-Biphenyl	164			d_{10}-Acenaphthene	9.75	
d_{12}-2,6-Dimethylnaphthalene	168	150	0.747	d_{10}-Acenaphthene	10.07	
d_8-Acenaphthylene	160	158	0.159	d_{10}-Acenaphthene	10.80	
d_8-Dibenzothiophene	192	160	0.085	d_{10}-Pyrene	14.67	
d_{10}-Phenanthrene	188	184	0.143	d_{10}-Pyrene	14.97	

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d_{12}-Benz[a]anthracene	240	236	0.250	d_{10}-Pyrene	21.63
d_{12}-Chrysene	240	236	0.278	d_{10}-Pyrene	21.73
d_{12}-Benzo[b]fluoranthene	264	260	0.216	d_{12}-Benzo[e]pyrene	25.11
d_{12}-Benzo[k]fluoranthene	264	260	0.208	d_{12}-Benzo[e]pyrene	25.23
d_{12}-Benzo[a]pyrene	264	260	0.216	d_{12}-Benzo[e]pyrene	26.47
d_{12}-Perylene	264	260	0.256	d_{12}-Benzo[e]pyrene	26.88
d_{12}-Indeno[1,2,3,cd]pyrene	288	284	0.192	d_{12}-Benzo[e]pyrene	31.63
d_{12}-Benzo[ghi]perylene	288	284	0.205	d_{12}-Benzo[e]pyrene	32.45

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APPENDIX H: LABORATORY MEAN + 3 S LIMITS FOR PROCEDURAL BLANKS

	Aqueous	Solid	Tissue	SPMD Extract
	Average+3stdev	Average+3stdev	Average+3stdev	Average+3stdev
	ng/sample	ng/sample	ng/sample	ng/sample
2-METHYLFLUORENE	1.8	3.8	3.0	3.5
C2-PHENANTHRENE/ANTHRACENE	3.5	11.5	11.5	3.3
C1-PHENANTHRENE/ANTHRACENE	1.6	11.3	2.3	1.0
BENZOFLUORANTHENES	1.1	6.0		54.4
BENZO[B]FLUORANTHENE	0.6	4.2	1.6	1.0
BENZO[J/K]FLUORANTHENES	0.4	2.1	1.2	1.5
2-METHYLNAPHTHALENE	2.2	4.6	3.3	5.3
1-METHYLNAPHTHALENE	1.9	4.5	1.6	4.6
C1-NAPHTHALENE	3.8	8.3	4.6	7.6
C1-BIPHENYL	31.9	26.6	43.3	
C2-BIPHENYL	165	196	237	
C2-NAPHTHALENE	9.3	40.1	52.0	37.4
1,2-DIMETHYLNAPHTHALENE	1.8	8.8	1.1	5.7
2,6-DIMETHYLNAPHTHALENE	1.3	6.3	1.9	4.1
C3-NAPHTHALENE	4.8	13.6	5.4	15.2
2,3,6-TRIMETHYLNAPHTHALENE	0.7	3.4	0.8	2.8
2,3,5-TRIMETHYLNAPHTHALENE	0.9	2.9	1.5	2.5
C4-NAPHTHALENE	18.5	22.6	11.5	36.3
C1- ACENAPHTHENEs	3.5	8.5	1.5	
C1-FLUORENES	8.1	22.7	11.0	28.7
1,7-DIMETHYLFLUORENE	0.5	2.4	0.6	2.4
C2-FLUORENES	4.5	7.2	41.2	9.1
C3-FLUORENES	6.8	15.3	18.1	16.2
C1-DIBENZOTHIOPHENES	0.5			1.4
2/3-METHYLDIBENZOTHIOPHENES	0.7	2.2	0.5	1.4
C2-DIBENZOTHIOPHENES(P)	2.3	10.2	3.9	3.1
2,4-DIMETHYLDIBENZOTHIOPHENE	0.5	1.0	0.3	1.0
C3-DIBENZOTHIOPHENE(P)	2.9	14.7	6.7	7.9
C4-DIBENZOTHIOPHENE(P)	6.3	23.1	6.9	15.6
2-METHYLPHENANTHRENE	0.5	2.4	0.7	1.3
2-METHYLANTHRACENE	0.4	1.6	0.5	5.1
1-METHYLPHENANTHRENE	3.0	1.9	0.9	1.0
3,6-DIMETHYLPHENANTHRENE	0.4	1.3	1.3	1.1
1,7-DIMETHYLPHENANTHRENE	0.2	1.6	1.3	29.0
C3-PHENANTHRENE/ANTHRACENE	12.1	5.7	24.0	3.1
1,2,6-TRIMETHYLPHENANTHRENE	0.3	1.1	2.0	1.5
RETENE	0.7	4.7	1.4	2.4
C4-PHENANTHRENE/ANTHRACENE	8.8	24.8	17.0	9.1
C1-FLUORANTHENES/PYRENES	2.6	9.4	11.9	2.0
3-METHYLFLUORANTHENE/BENZO(A)FLUOREN	0.5	2.5	2.1	1.8

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	Aqueous	**Solid**	**Tissue**	**SPMD Extract**
	Average+3stdev	Average+3stdev	Average+3stdev	Average+3stdev
	ng/sample	ng/sample	ng/sample	ng/sample
E				
C2-FLUORANTHENE/PYRENE(P)	9.3	14.1	8.2	6.5
C3-FLUORANTHENE/PYRENE(P)	1.9	5.1	4.3	11.2
C4-FLUORANTHENE/PYRENE(P)	1.4	4.0	5.1	
C1-BENZ(A)ANTHRACENE/CHRYSENE	0.8	5.0	1.4	1.0
5/6-METHYLCHRYSENE	0.2	1.0		0.6
1-METHYLCHRYSENE	0.2	1.4		0.8
C2-BENZ(A)ANTHRACENE/CHRYSENE	1.0	4.8	8.9	1.7
5,9-DIMETHYLCHRYSENE	0.2			1.3
C3-BENZ(A)ANTHRACENE/CHRYSENE	6.4	4.1	2.0	
C4-BENZ(A)ANTHRACENE/CHRYSENE	2.3	4.9	11.8	
C1-BENZOFLUORANTHENES/BENZOPYRENES	2.4	10.7	10.4	4.5
C2-BENZOFLUORANTHENES/BENZOPYRENES	2.3	8.3	14.4	2.6
1,4,6,7-TETRAMETHYLNAPHTHALENE	6.5	4.7	2.4	6.6
DIBENZOTHIOPHENE	0.9	3.1	1.1	1.4
NAPHTHALENE	6.8	9.5	54.9	35.8
ACENAPHTHYLENE	0.5	1.7	0.6	1.7
ACENAPHTHENE	1.7	3.8	2.2	4.9
FLUORENE	5.4	3.1	2.2	1.2
PHENANTHRENE	2.9	3.5	2.9	2.0
ANTHRACENE	0.4	1.6	2.5	1.4
FLUORANTHENE	1.5	2.0	1.4	1.8
PYRENE	1.6	1.8	2.4	1.1
BENZO(A)ANTHRACENE	1.2	1.9	0.9	1.0
CHRYSENE	1.3	3.0	1.3	1.2
BENZO(E)PYRENE	0.9	2.5	2.3	
BENZO(A)PYRENE	0.9	3.6	0.7	1.6
PERYLENE	1.1	9.0	1.0	
DIBENZO[AH]ANTHRACENE	0.7	2.8	0.7	2.2
INDENO[123CD]PYRENE	0.8	2.8	1.5	2.4
BENZO(GHI)PERYLENE	0.9	2.4	0.7	1.9
BIPHENYL	2.9	4.4	3.3	8.1
3-METHYLPHENANTHRENE	0.4	2.2	0.7	1.2
9/4-METHYLPHENANTHRENE	0.3	2.2	0.6	1.0
2,6-DIMETHYLPHENANTHRENE	0.4	1.9	1.5	1.2
1,8-DIMETHYLPHENANTHRENE	3.6	1.1	5.2	1.2

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KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-717
Revision History
Cover Page
Page 1

TITLE: **TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES**

Prepared By: Wet Chemistry Date: 12/97

Approved By:

Group Supervisor: Keith Tanguay Date: 02/08/01

Operations Manager: John C Dinton Date: 2/8/01

QA Officer: Deborah J. Nadeau Date: 2.8.01

General Manager: Dennis C Hufnagle Date: 2/08/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, added pollution prevention and standard method reference. Other minor changes to sections 7, 8 and QA Table.	DN	2.8.01	2.8.01
02	Added ASTM D2216 reference to the SOP.	DN	5.22.02	5.22.02
03	Revised section 7.3.15 to refer to KIMS. Removed references to supervisor - changed to Department Manager. Replaced Figures 1 and 2 with updated ones.	HRC	05.04.04	05.04.04
04	Added wording to Section 8, added Laboratory reagent grade water, changed formatting.	LAD	02.07	02.07
05	Corrected formulas in Sect.s 7.3.15 and 7.4.5.1.	LAD	06/08	06/08

TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added the requirement to record the oven ID and thermometer ID in logbook. Added references to section 10.	EN	08/09	08/09
07	Updated and/or added references to sections 7, 9 and 10. updated Logbook page.	LAD	06/10	06/10
				LAD 050112
08	Sect. 7 - Edited for new balance with direct upload to KIMS. Added scanning and archival of raw data. Added Blank prep. Other minor changes to reflect current practice and for clarity. Sect. 8 - Added calibration acceptance criteria and blank prep. Sect. 9 - Added MDL, LOD and LOQ info. Sect. 10 - Added and edited references. Table 2 - Added only 2 weighings are performed. Updated Fig. 1 & 2.	LAD	05/12	05/12
09	Throughout - changed KAS, INC to KAS. Sect. 7 - changed 0.001g to 0.0001g, changed approx. 5g to 25-50g, added sections to describe additional final weighings; Sect. 8 - Changed duplicate RPD from < 10 to <5. Sect. 10 - updated references. Table 2 - Removed modification for multiple weighings. Updated Fig.s 1 and 2.	LAD	05/15	05/15
10	Sect. 6 - changed H.T. to 7 days. Changed 500 +/- 50°C to 550°C throughout SOP. Updated figures 1 and 2.	LAD	12/17	12/17
11	Added TS-ME reference and changed drying time to overnight throughout SOP. Sect. 1 added method numbers and waste disposal instructions. Sect. 4 - added woven tongue depressors. Sect. 7 - Many changes to reflect current practices. Sect. 8 & Table 1 - Updated method blank criteria. Added	LAD Figures 2→13	02/19	02/19

TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ____ of document **CA-717-11**, titled **Total Solids/Total Volatile Solids Determination in Solid Matrices.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ____ of document **CA-717-11**, titled **Total Solids/Total Volatile Solids Determination in Solid Matrices.**

Recipient: _____ Date: _____

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures used by Katahdin Analytical Services technical personnel to determine the Total Solids (percent solids) and Total Volatile Solids in soil, sediment, sludges and solid matrices in accordance with Standard Methods 2540 G or SW846 3540C (Total Solids only).

1.1 Definitions

Total Solids - The term applied to the "as received" sample where material residue left in the vessel after drying in an oven at 103-105°C.

Decanted total Solids - The term applied to the "decanted" sample where material residue left in the vessel after drying in an oven at 103-105°C.

TS - Total Solids (SW846 3540C)

TS-ME – Total Solids (SM2540G)

Fixed Solids - The residue of total, suspended, or dissolved solids after heating to dryness for a specified time at a specified temperature.

Volatile Solids - The weight loss on ignition of fixed solids.

Total Volatile Solids - The weight loss on ignition of total solids.

TVS - Total Volatile Solids

Batch - Twenty or fewer samples prepared and analyzed together with handling that is similar to all.

LCS - Laboratory Control Sample

PQL - Practical Quantitation Limit

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the determination Total Solids and Total Volatile Solids. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the determination of Total Solids and Total Volatile Solids to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in

the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data. It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to perform periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in adherence with the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

Wastes generated by this method can be disposed of using general incineration practices, unless knowledge of the sample matrix determines it to be hazardous waste.

TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

2.0 SUMMARY OF METHOD

A quantity of soil, sediment, or solid matrix sample is weighed out, dried at 103-105°C overnight and then re-weighed. The difference in the weights before and after drying represents the "total solids" portion of the sample as defined by this procedure. The percent solids determined by this method are further used in the final calculations for other analyses to convert the analytical value of the sample to a "dry weight basis". The PQL for this procedure is 0.1 wt% for an initial weight of approximately 25-50g.

To determine Total Volatile Solids, the total fixed solids after drying at 103-105°C are ignited at 550 °C. The residue is brought to constant weight and weighed. The loss on ignition between 103-105°C to 550 °C constitutes the measurement of Total Volatile Solids.

3.0 INTERFERENCES

3.1 Total Solids

Residues dried at 103-105°C may retain water of crystallization as well as some mechanically occluded water. Because removal of occluded water is marginal at this temperature, attainment of constant weight may be very slow. The time of drying will be maintained consistently overnight. Loss of CO_2 will result in the conversion of bicarbonate to carbonate. Loss of organic material at this temperature by volatilization may be very slight, however, this is dependent upon the material. The loss of ammonium carbonate and volatile organics may be significant for sediments, sludges, and sludge cakes. After drying some residues are often hygroscopic and rapidly absorb moisture from the air.

3.2 Total Volatile Solids

Negative errors may be present due to loss of volatile constituents at the initial temperature of drying, 103-105°C. The mass of recovered organic matter from sludges and sediments may require a longer ignition time than that for wastewaters. Determination of low concentrations of volatile solids in the presence of high fixed solids may be subject to considerable error. In such instances where volatile constituents are suspected and analytes of interest alternative techniques should be sought for their determination. After drying some residues are often hygroscopic and rapidly absorb moisture from the air.

4.0 APPARATUS AND MATERIALS

4.1 Drying oven - capable of maintaining drying temperature of 103-105°C

4.2 Muffle Furnace for operation at 550 ° C

4.3 Aluminum tins, porcelain crucibles or evaporating dishes

4.4 Analytical Balance accurate to 0.0001g

4.5 Scoopulas or wooden tongue depressors

4.6 Dessicator provided with a desiccant containing a colored indicator of moisture content - with conditioned desiccant; condition by drying at 210°C for one hour (per the manufacturer's directions).

5.0 REAGENTS AND STANDARDS

5.1 Laboratory Reagent Grade Water

5.2 Sand: Muffled at 550°C for 1 hour

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples are maintained under storage conditions dictated by analytes of interest. This normally constitutes storage at 4°C. The analytical holding time for Total Solids by Method SM2540G is 7 days from sample collection. SW-846 3540C does not specify a holding time; Katahdin applies14 days as a conservative limit.

The laboratory initiates sample analysis as quickly as possible for both methods to facilitate data availability for use in final calculation of other analytical parameters.

7.0 PROCEDURES

7.1 In accordance with the current revision of Katahdin SOP CA-102, "Balance Calibration", verify balance calibration at the beginning of the day or prior to beginning analysis. Refer to Section 8.0 for acceptance criteria.

7.2 The oven ID must be recorded in the Total Solids/Total Volatile Solids logbook (or on the Work group Report). When preparing for TS exclusively, a Work Group Report may be used in place of a logbook and data may be entered directly using KIMS. See section 7.4.15 through 7.4.41 for instructions to generate a Work Group Report and export data.

7.3 PREPARATION OF CRUCIBLES, EVAPORATING DISHES, OR TINS FOR TVS

7.3.1 Verify that crucible/dish/tin IDs are unique and legible. IDs may be applied to crucibles using porcelain ink or china marker. Tin ID's should be visibly etched

into the tin. Freshly labeled crucibles/dishes should be ignited at 550°C prior to use.

7.3.2 If *total volatile solids* are to be measured, ignite crucible/evaporating dishes in muffle furnace at 550°C for 1 hour. Tins do not need to be muffled.

7.3.3 Check desiccant to assure that it has sufficient blue color. Replace with fresh material if needed prior to removing crucible/evaporating dishes from the oven or muffle.

7.3.4 Place in desiccator with desiccant that has indicator beads showing a blue color. Cool for a minimum of 1 hr. Clean dried/ignited crucible/evaporating dishes may be maintained in a desiccator prior to use. Weigh to nearest 0.0001 g and record dish ID and weight in the solids logbook (Figure 1).

7.4 TOTAL SOLIDS (TS) ANALYSIS

7.4.1 Label and weigh each tin/crucible/evaporating dish before use and record initial (tare) weight in Total Solids logbook (Figure 1). When using tins for weighing total solids, the average weight of the tins may be used for all samples. To obtain an accurate average weight of tins, at least one tin from each box per case of tins must be weighed and the average calculated using a spreadsheet (found on Server A: \\SERVER_A\wetchem\Spreadsheets\Total Solids\TIN TARE WEIGHT AVG\NEW DISH LOT AVG) (Figure 2). Once the average is calculated, a Wet Chem Lab supervisor must be notified to enter the average tare amount in KIMS.

NOTE: It is necessary to obtain a new average tin weight for each new case of total solids tins received, the lot number of the tins should be recorded with the spreadsheet.

7.4.2 Samples will be analyzed under this procedure as "total solids" or "decanted total solids". "Total Solids" will be performed on a well-homogenized sample as received where any aqueous phase is incorporated into the solid matrix prior to aliquotting. "Decanted total Solids" will be performed on sample aliquots designated for extractable organic parameters where it has been determined that the standing aqueous layer must be decanted prior to aliquotting for the organic extractable preparation. Sample login decants the samples during the login process. Decanted samples will be marked with a "DEC" on the cap. The decanted sample aliquot will constitute the "original" sample for total solids determination.

7.4.3 Whether the sample has been decanted or not, remove any obvious non-representative material such as large sticks and rocks. Thoroughly mix the sample with a scoopula (or wooden tongue depressor). Weigh 25-50 g of a

representative sample aliquot into a labeled weighing dish. For samples that are analyzed "as received" and contain a significant standing aqueous layer, a larger aliquot may be used. In order to facilitate drying and minimize moisture entrainment, the sample may be spread out across the surface of the pan/dish/crucible. Please refer to Katahdin SOP, CA-108, Basic Laboratory Technique, current revision, for more information on subsampling.

NOTE: For samples with a low density (i.e., ash), the minimum weight of 25 g may not be attainable. Also, for samples with limited volume available, it may be necessary to communicate with lab supervisors, project managers, and clients to determine how much volume should be used for TS.

7.4.4 Record the weight (to the nearest 0.0001g) of the sample plus dish by uploading the weight directly into KIMS. Please refer to sections 7.4.15 through 7.4.41 for instructions.

7.4.5 A Method Blank and LCS are prepared at a minimum frequency of 1/20 field samples and a Duplicate is prepared at a minimum frequency of 1/10 field samples or per preparation batch if fewer samples. When the batch contains TVS samples the duplicate for TS should be selected from a sample requesting TVS.

7.4.6 A Method Blank consists of a weighing tin/dish/crucible containing about 4.5 grams of muffled dry sand carried through the entire analytical process. The Method Blank result must be 100% TS +/-0.5%. If acceptance criteria are not met, all associated samples must be reanalyzed if possible.

7.4.7 The LCS is prepared by spiking muffled dry sand with a known amount of laboratory reagent grade water. Weigh out 4.5 g of sand and 0.50 mL (0.50 g) of laboratory reagent grade water and carry LCS through entire analytical process. Recovery for the LCS must be within 90-110% of the expected true value of 90% solids. If acceptance criteria are not met, all associated samples must be reanalyzed if possible.

7.4.8 A Duplicate is prepared by taking a duplicate sample aliquot, 25-50 g, through the procedure as described for the sample. NOTE: The duplicate should be aliquotted at the same time as the original sample.

7.4.9 Transfer samples to drying oven maintained at 103-105°C. Record "date in", "time in", and "temperature in" in appropriate logbook or on the Work Group Report.

7.4.10 Dry samples for overnight. Remove the samples from the oven and cool to ambient temperature in a desiccator. Record "date out", "time out", and "temp out" in logbook or on the Work Group Report.

7.4.11 Weigh the final sample plus dish weights to the nearest 0.0001g.

7.4.12 Record the final weighing in the solids logbook, or export it directly to KIMS.

7.4.13 Repeat the drying (1 hour), cooling, desiccating, and weighing until the weight change is less than 4% or 50 mg, whichever is less.

7.4.14 The raw data (batch number, sample number, pan weight, original sample + pan, and final sample + pan weights) are entered into KIMS for calculation. The calculation performed is as follows:

$$\% \text{ Total Solids} = \frac{(B - \text{pan, g})}{(A - \text{pan, g})} \times 100$$

Where: A = weight of original sample+ pan, g, and
B = weight of dried sample + pan, g

EXPORTING TOTAL SOLIDS DATA INTO KIMS

CREATING A TS WORK GROUP AND GENERATING A WORK GROUP REPORT

7.4.15 Samples must be work grouped prior to analysis in order to use a Work Group Report instead of a logbook. To work group total solids, one must go to Sample Management on the menu and then to Workstat. A new window opens up and you must make sure the mode is set to Schedule Work (Figure 3).

7.4.16 In the Work Group for Scheduling section, the Work Profile needs to be set to SOLIDS. Click on the new button to get a new work group number the system automatically generates (Figure 3). Click the SAVE and then OK buttons. It may be necessary to record the generated work group number on paper for reference.

7.4.17 In the Query Criteria section, the department number (100), new status section (WIP), product (TS%), matrix (SL), and QC profile (TOTAL SOLIDS) will be already filled in. The OK button may now be selected to show all available samples. To select the samples for the batch, in the available space check with an 'x' (Figure 4). Click save to put them in the work group.

7.4.18 Once all the samples are selected, the add QC button must be hit to add all the pertinent QC for the batch. In the reference column the sample that the QC is associated with must be entered (Figure 5). Click save to save changes. After all this is done, all the windows may be closed.

7.4.19 To print out a Work Group Report, go to Reports on the menu, then Worklist and then Print Work group. A window opens and you must enter the work

group number (unless it automatically generates, this happens when the work group is printed IMMEDIATELY after work grouping samples), the department number the work group is associated with should automatically fill in (Figure 6). Press the OK button and the Work Group Report will generate. Double check the work group to make sure all QC and samples are in the batch, and print.

7.4.20 On the left side of the Work Group Report, there is a column labeled 'Tin' next to 'Sample'. Label the corresponding tins and samples before weighing them out (Figure 7).

COLLECTING TARE WEIGHTS

7.4.21 Total Solid samples to be electronically entered into KIMS must be weighed at all steps on a balance connected to a computer with the WinWedge program (currently serial number 1124016031) and have a connection to KIMS.

7.4.22 From the desktop of the associated computer open up the WinWedge program and the file entitled Ohaus_discovery, in that order. Proceed to open KIMS

7.4.23 Log into KIMS. Work group samples. From the pull-down menu, select Data ► Enter Data.

7.4.24 Enter TARE in the list pointer Field. Select Query by work group and enter the work group number. Under the Run ID section, click New. Record the generated Run ID on either the logbook page or the Work Group Report. Enter the instrument ID (NONE) and analyst. (Figure 8). Click save. Close out the window.

7.4.25 Click OK. Select Modes► All Samples. Click Close. Click the Header button. Enter in the prep date and time in Prep Date 1 and the operator's initials in Comments. Check the box titled Save Measure Date. (Figure 9). Click Close. Click OK to start entering data.

7.4.26 Enter in prep chemist initials in the appropriate column. Record the start of prep time on the logbook page or on the Work Group Report. If the average tin weights are used (see section 7.4.1), the weights should automatically be filled-in in KIMS (Figure 10). If not, take the appropriately labeled empty weighing dish and place it on the balance. Once the weight has settled click the Print key located on the balance. The weight data should be entered in the selected box. Check to make sure both the weight in KIMS and weight displayed on the balance are the same. This is the initial weight of the weighing dish. Do this for all samples in the work group. Click Save. Close all Windows.

COLLECTING WET WEIGHTS

7.4.27 From the pull down menu, select Data ▶ Enter Data.

7.4.28 Enter WET-WT in the list pointer field. Select Query by work group and enter the work group number. Enter in the Run ID previously assigned to the work group. Click the ok button

7.4.29 Click the Header button. Enter in the prep date in Prep Date 1 and the operator's initials in comments. Check the box titled Save Measure Date. Click Close. Click OK.

7.4.30 Take the appropriately labeled weighing dish filled with sample and place it on the balance. Once the weight has settled click the Print key located on the balance. The weight data should be entered in the selected box. Check to make sure both the weight in KIMS and weight displayed on the balance are the same. Do this for all samples in the work group. Click save. Close all Windows.

COLLECTING DRY WEIGHTS

7.4.31 Heat and cool the samples for the appropriate amount of time.

7.4.32 From the pull down menu, select Data ▶ Enter Data.

7.4.33 Samples logged in for TS-ME require two dry weight measurements. To collect the first dry weight: Enter TS-FINAL in the list pointer field. Select Query by work group and enter the work group number. Enter in the Run ID previously assigned to the work group. Click the ok button.

7.4.34 Click the header button. Enter in the prep date in Prep Date 1 and the operator's initials in comments. Check the box titled Save Measure Date. Click Close. Click OK.

7.4.35 For all samples, to collect the final dry weight: Enter DRY-WT in the list pointer field. Select Query by work group and enter the work group number. Enter in the Run ID previously assigned to the work group. Click the ok button.

7.4.36 Click the header button. Enter in the prep date in Prep Date 1 and the operator's initials in comments. Check the box titled Save Measure Date. Click Close. Click OK.

7.4.37 Take the appropriately labeled weighing dish filled with dried sample and place it on the balance. Once the weight has settled click the Print key located on the balance. The weight data should be entered in the selected

box. Check to make sure both the weight in KIMS and weight displayed on the balance are the same. Do this for all samples in the work group. Click save. Close all Windows.

DATA APPROVAL

7.4.38 Once the analytical data has been sent to KIMS, the analyst must review the calculated results for completeness and accuracy. In order to do this, the analyst must be logged into KIMS

7.4.39 From the pull-down menu, select Sample Data ▶ Approval. The process ID is WETREV. Click OK. Enter in the work group number. Click OK. (Figure 11)

7.4.40 When approving, if a result is not going to be reported, the analyst must make sure the report flag is set to N.

7.4.41 Once all data is approved a batch sheet is automatically printed which contains all QC and samples within the work group. The results, recoveries, and RPD's are printed on the batch sheet. The analyst must sign and date the batch sheet (Figure 12). Before turning in the data, the analyst must make a copy of the Work Group Report (unless a logbook was used), and put the copied report in to the Total Solids 3 ring binder kept in the Wet Chemistry Lab. Use a sharpie marker to black out client names on the Work List Report if it contains multiple client names when turning in data.

7.5 TOTAL VOLATILE SOLIDS (TVS) ANALYSIS

7.5.1 A Method Blank is prepared at a minimum frequency of 1/20 field samples and a Duplicate is prepared at a minimum frequency of 1/10 field samples or per preparation batch if fewer samples utilizing the aliquots prepared for the TS determination.

7.5.2 For the determination of TVS ignite the residue in the TS crucible/dish/tin in a muffle furnace at 550°C for 1 hour. Start the muffle oven temperature at 200°C, and increase the temp by 50°C every 15-30 minutes, checking to make sure that the sample does not start to smolder/burn. If this happens, reduce the temp increase and change temp at a slower rate. Once the muffle oven temp reaches 550°C, start a 1 hour timer. At the end of one hour, reduce the oven temp and leave the door partly ajar to cool samples before removing them from the oven.

7.5.3 Transfer the dish to a desiccator, cool to room temperature and weigh.

7.5.4 Record the final weighing in the solids logbook.

7.5.5 Total Volatile Solids Calculation

The raw data (batch number, sample number, dish/crucible weight, dried 103-105 °C sample + dish/crucible, and ignited sample + dish/crucible weights) are entered into KIMS (see Current version of Katahdin SOP CA-762, Wet Chemistry Data Entry And Review Using Katahdin Information Management System (KIMS)) for calculation. The calculation performed is as follows:

$$TVS(\%) = \frac{(A - dish, g) - (B - dish, g)}{(A - dish, g)} \times 100$$

Where: A = weight of residue + dish before ignition, g, and
B = weight of residue + dish after ignition, g

7.6 Data Archival

All batch sheets, raw data, and supporting documents are scanned after final review and the resulting image files are saved on a Katahdin server for use in data package assembly. Image files of raw data are periodically archived by the laboratory's MIS department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of

the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 The analytical balance used must be calibrated using ASTM Class 1 weights (1.0g, 10.0g, and 50.0g) prior to beginning analysis. Acceptance criteria are as follows:

ASTM Class 1 Weight	Acceptance Range
1.0g	0.9995-1.0005g
10.0g	9.9995g-10.0005g
50.0g	49.9995g-50.0005g

Refer to TABLE 1 for summary of QC requirements, frequency, and acceptance criteria.

8.2 One method blank must be analyzed for every batch of twenty or fewer samples. A method blank consists of a weighing dish containing about 4.5 grams of muffled dry sand carried through the entire analytical process. The method blank result must be 100% TS +/-0.5%. If acceptance criteria are not met, all associated samples must be reanalyzed if possible.

Refer to TABLE 1 for summary of QC requirements, frequency, and acceptance criteria.

8.3 One laboratory control sample (LCS) must be prepared and analyzed for every batch of twenty or fewer samples. The LCS is prepared by spiking muffled dry sand with a known amount of laboratory reagent grade water. Weigh out 4.5g of sand and 0.5 mL of laboratory reagent grade water and carry LCS through entire analytical process. Recovery for the LCS must be within 90-110% of the expected true value. If acceptance criteria are not met, all associated samples must be reanalyzed if possible.

8.4 One in every ten (10) samples is analyzed in duplicate. The precision of duplicate analyses, as measured by Relative Percent Difference (RPD), must be less than 5%. If acceptance criteria are not met, examine sample for physical characteristics that may lead to lack of homogeneity or other matrix related interferences. If no matrix related issues are apparent all associated samples should be reanalyzed if possible.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Inorganic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO.

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revisions of Standard Method 2540 G for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

American Public Health Association (APHA) (2012) Standard method for examination of water and wastewater, 22nd edn. APHA, AWWA, WPCF, Washington, Method 2540G.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 3540C.

Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, 1979, Method 160.4

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1.1, 2018.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

Katahdin SOP CA-762, Wet Chemistry Data Entry and Review Using Katahdin Information Management System (KIMS)

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TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

TABLE 1

QC REQUIREMENTS

Analytical Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
SM 2540G / ASTM D2216-98	Total Solids Determination	Method blank	One per batch of 20 or fewer	Blank result is 100% TS +/- 0.5%	(1) Investigate source of contamination (2) Reprep method blank and reanalyze along with all associated samples
		LCS	One LCS per batch of 20	90-110%	Reprep and reanalyze all affected QC and field samples
		Sample Duplicate	One sample duplicate per ten samples	RPD ≤ 5%	(1) If lab QC in criteria and matrix interference suspected, flag data (2) Or else, reanalyze
		Demonstration of analyst proficiency – 4 replicates	Initially and once annually thereafter	P&A meet method criteria	Repeat P&A study
		MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-717-11	METHOD 2540G current revision or ASTM D2216-98, current revision
Apparatus/Materials		
Reagents	1) Muffled Sand for Blank and LCS	1) No LCS listed
Sample preservation/ handling		
Procedures	Use Tins for TVS	Crucible and Glass used for TVS
QC - Spikes		
QC - LCS	1) Muffled Sand spiked with 0.5 g laboratory reagent grade water for LCS	1) No LCS listed
QC - Accuracy/Precision	1) Duplicate Frequency 1/10	1) Duplicate Frequency 1/20
QC - MDL		

TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

FIGURE 1

EXAMPLE OF TOTAL SOLIDS LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES

WG213301 R437786

Total Solids: SM2540 G / ASTM D2216			PQL: 0.10%	Total Volatile Solids: SM2540 G / E160.4			PQL: 0.10%	Balance Calibrated?
TS/TVS	In 1	In 2	TS Only	Out 1	Out 2	TVS Only		Y / N
ANALYST IN:	[illegible]	[illegible]	ANALYST OUT:	[illegible]	[illegible]	ANALYST OUT:	[illegible]	In: Y
DATE IN:	9-7-17	9-8-17	DATE OUT:	9-8-17	9-8-17	DATE OUT:	9-11-17	Out (TS): Y
TIME IN:	16:08	12:15	TIME OUT:	10:00	13:47	TIME OUT:	15:50	Out (TVS): Y
TEMP IN:	104°C	104°C	TEMP OUT:	104°C	104°C	TEMP OUT:	550°C	Balance ID:
Oven ID: 107N0042			Muffle Oven ID: 030607 (assigned)					S/N 1124016031

SAMPLE ID	DISH ID	DISH WT (g)	DISH WET WT (g)	1st DISH DRY WT (g)	2nd DISH DRY WT (g)	DISH/ASH WT (g)	TIME
SK7950-1	20	1.3251	29.9835	23.9650	23.9644	~~27.7780~~ 28.0437	9-13-17 10:00
Blank	21	1.2649	5.8652	5.8628	5.8632	5.8559	
SK7950-1 DUP	24	1.3068	36.4084	28.8677	28.8413	27.7880	

9-13-17

WL-009 – Revision 5 – 04/29/2016

Reviewed By: [signature] QAWL820

Reviewed Date: 09/13/17

0000089

FIGURE 2

EXAMPLE OF A TIN TARE WEIGHT / NEW DISH LOT AVERAGE SPREADSHEET

Sample	Wt. (mg)	Tin ID
	1.0464	
	1.0049	
	1.0244	
	1.0437	
	1.0037	
	1.0206	
	1.0146	
	1.0134	
	1.0295	
	1.0469	
AVERAGE WT.	**1.02481**	

FIGURE 3

WORK GROUPING TOTAL SOLIDS IN KIMS

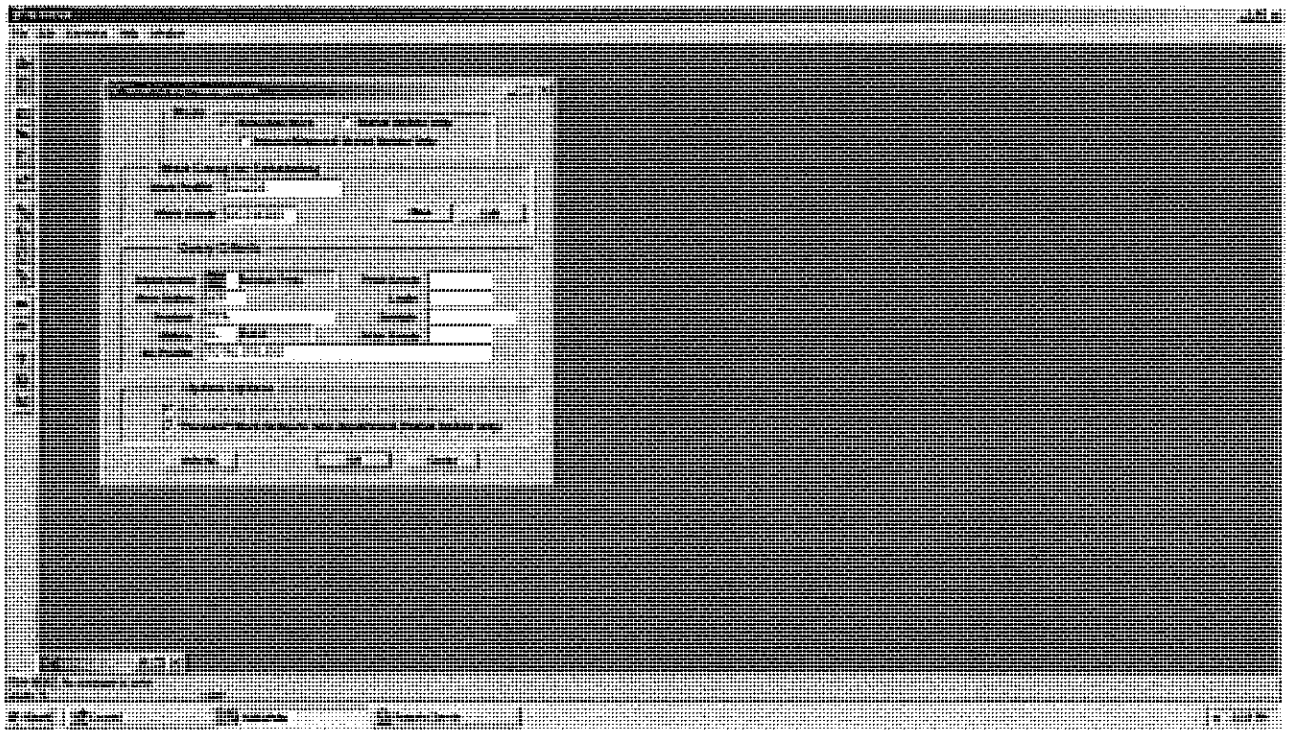


FIGURE 4

ADDING SAMPLES TO A WORKGROUP

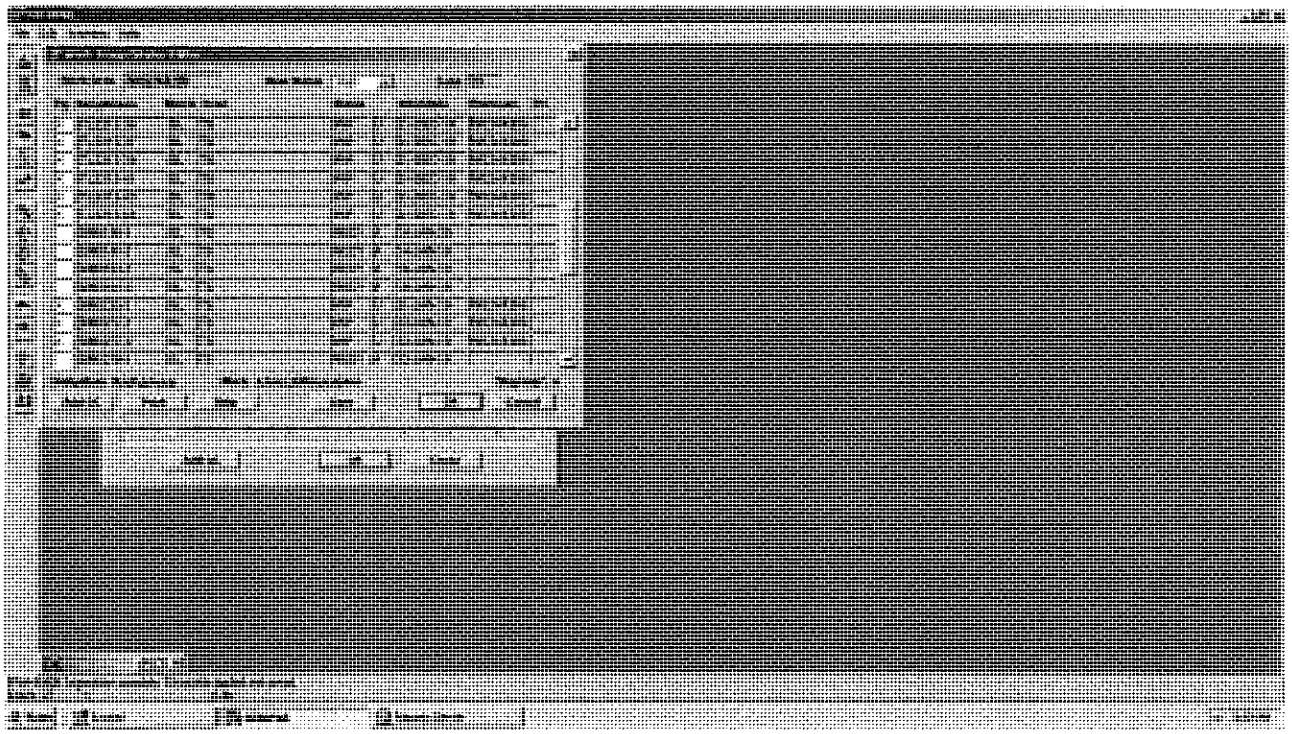
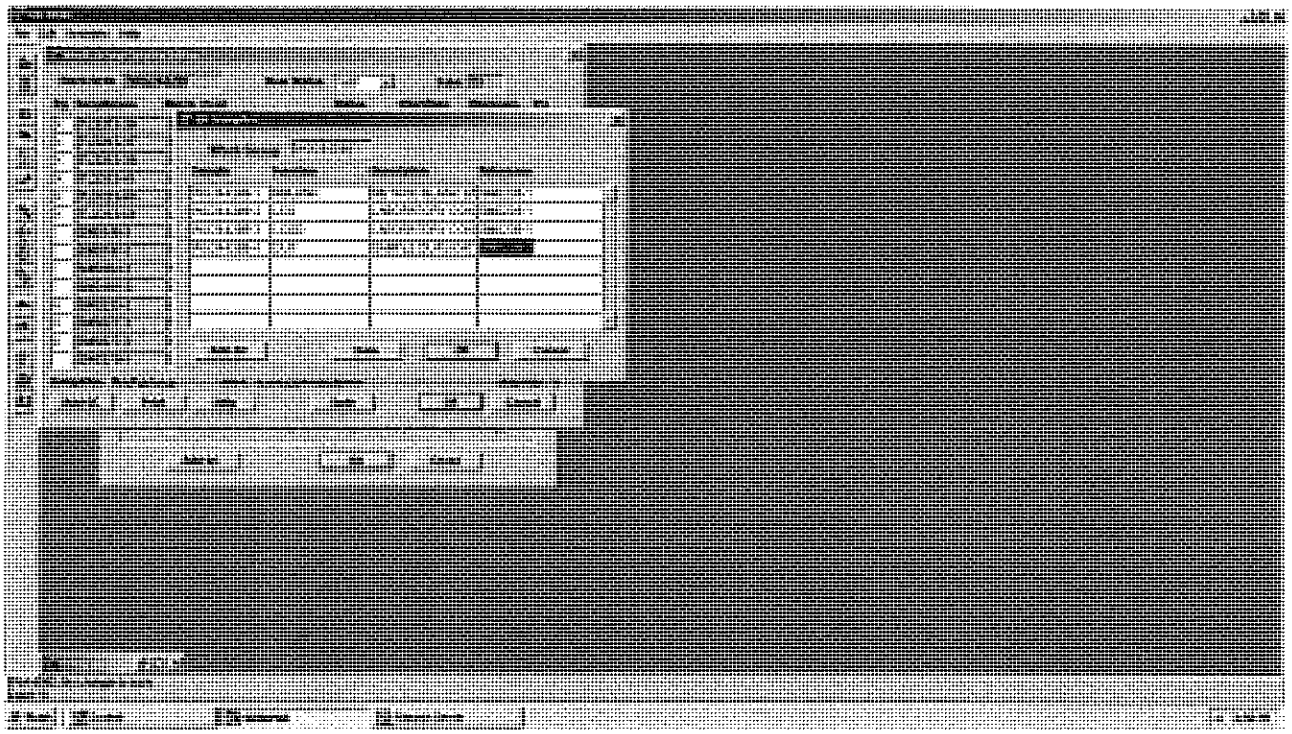


FIGURE 5

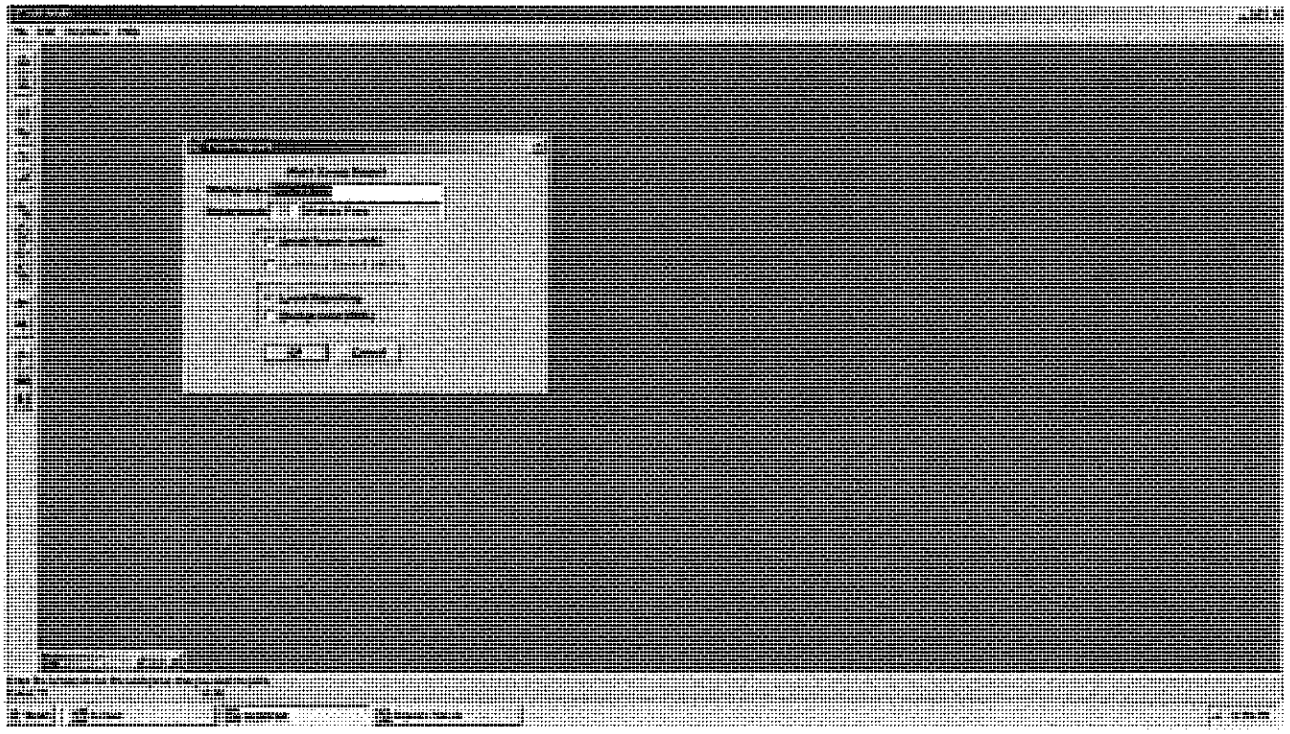
ADDING QC TO A WORKGROUP



TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

FIGURE 6

PRINTING A WORKGROUP REPORT



TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

FIGURE 7

EXAMPLE OF A WORK GROUP REPORT

Katahdin
ANALYTICAL SERVICES

Katahdin Analytical Services

Work Group Report (wk02)
24-DEC-18 11:28 AM

Page 1 of 1

Work Group: WG243473 R497188 **Created:** 24-DEC-18
Department: 100 Wetlab Prep **Operator:** **Due:**

Tin	Sample	Account Name	Product	Matrix	Status	UA	Workdate	PR Location
1	TL2535-1	ecoMaine	TS-ME	Solid	WIP	U	30-DEC-18	
2	TL2560-1	Sanford Sewer District	TS-ME	Solid	WIP	U	31-DEC-18	
3	TL2698-1	Clean Harbors	TS-ME	Solid	WIP	U	28-DEC-18	
4	TL2698-2	Clean Harbors	TS-ME	Solid	WIP	U	28-DEC-18	
5	TL2698-3	Clean Harbors	TS-ME	Solid	WIP	U	28-DEC-18	
6	TL2698-4	Clean Harbors	TS-ME	Solid	WIP	U	28-DEC-18	
7	TL2698-5	Clean Harbors	TS-ME	Solid	WIP	U	28-DEC-18	
8	TL2698-6	Clean Harbors	TS-ME	Solid	WIP	U	28-DEC-18	
9	TL2707-1	Twin Rivers Paper Compan	TS-ME	Solid	WIP	U	02-JAN-19	1L N-Amber Glass
10	WG243473-1	MBLANK	TS-ME	Solid	WIP	U	24-DEC-18	
11	WG243473-2	LCS	TS-ME	Solid	WIP	U	24-DEC-18	
12	WG243473-3	DUP	TS-ME	Solid	WIP	U	24-DEC-18	

Comments:
WG243473-1 TL2707-1
WG243473-2 TL2707-1
WG243473-3 TL2707-1

Prep Time: 11:31

Total Solids: SM2540 G / ASTM D2216			PQL: 0.10%	Oven ID: 107N0042		Balance Calibrated?
TS	In 1	In 2	TS	Out 1	Out 2	(Y) N
ANALYST IN:	ZF	ZF	ANALYST OUT:	BP	ZF	In: ✓
DATE IN:	12/24/18	12/26/18	DATE OUT:	12/26/18	12/26/18	Out (TS): ✓
TIME IN:	11:45	10:42	TIME OUT:	0855	11:52	Balance ID: Bal 01
TEMP IN:	104°C	104°C	TEMP OUT:	104	104°C	S/N 1124016031

FIGURE 8

GENERATING A RUN ID

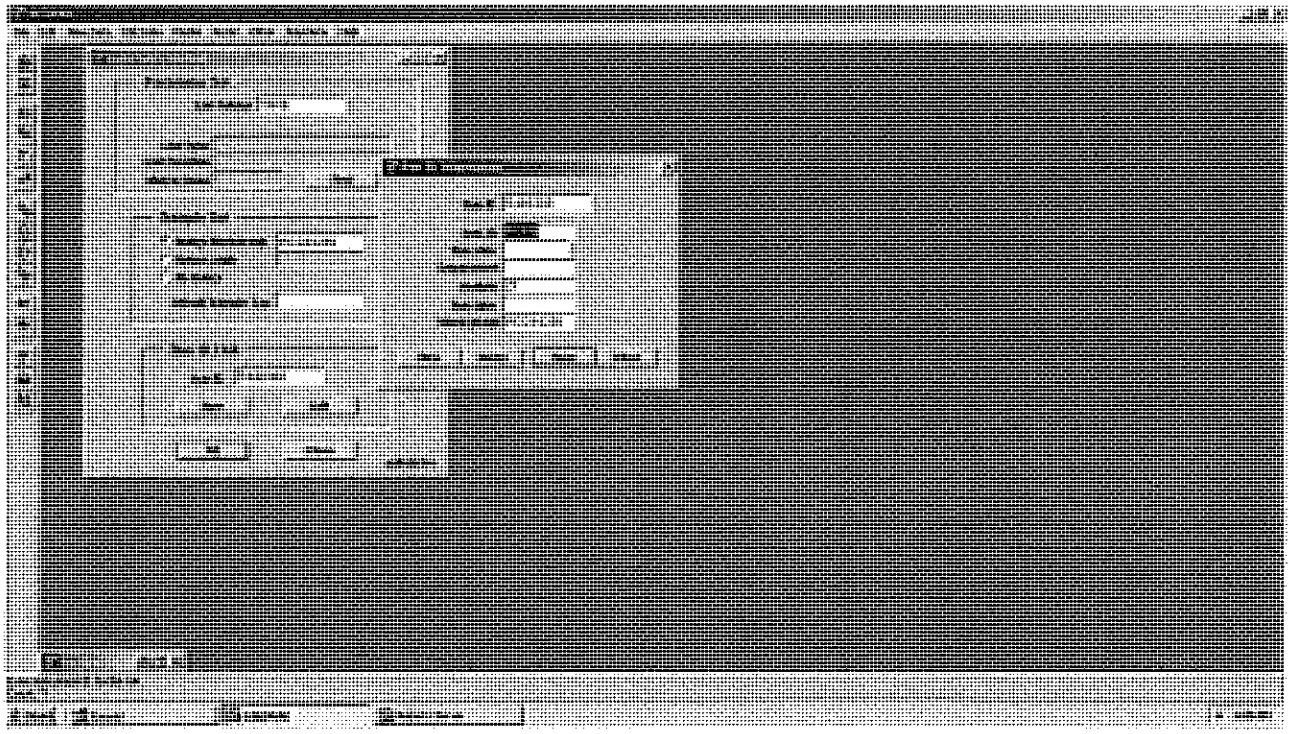


FIGURE 9

ENTERING HEADER INFORMATION

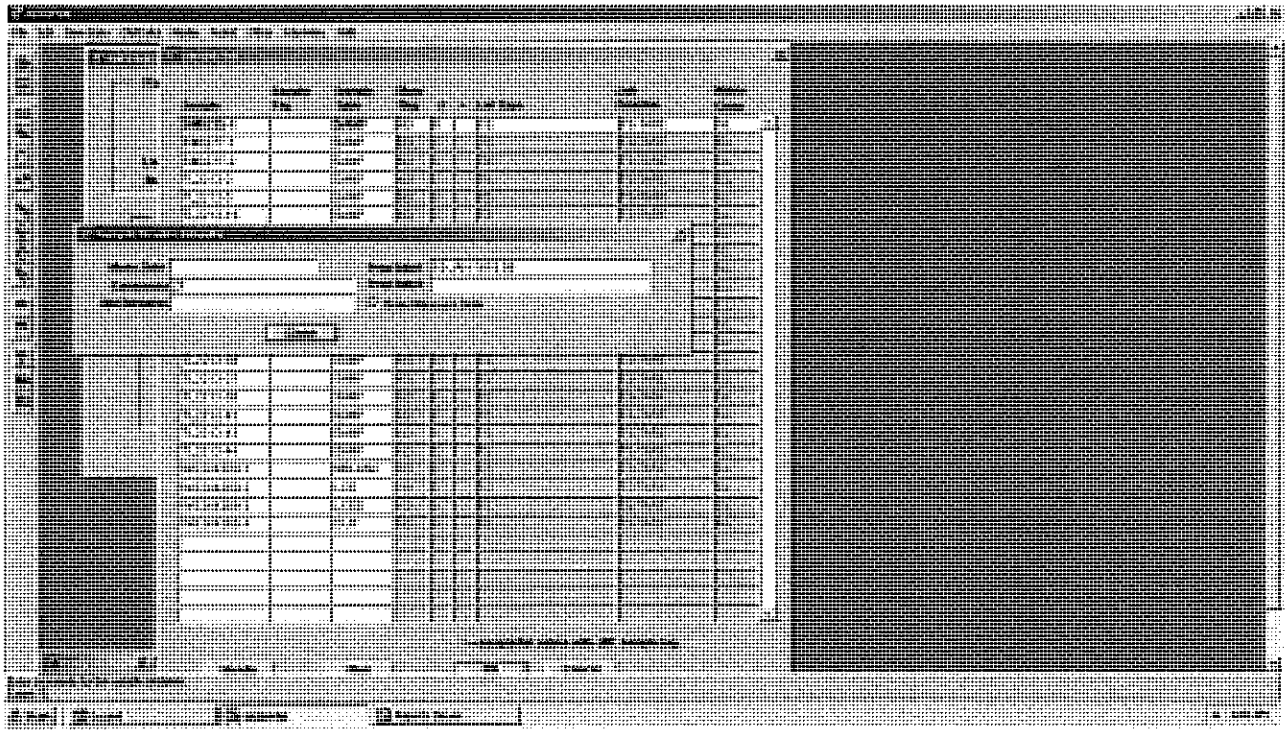


FIGURE 10

ENTERING TARE WEIGHTS

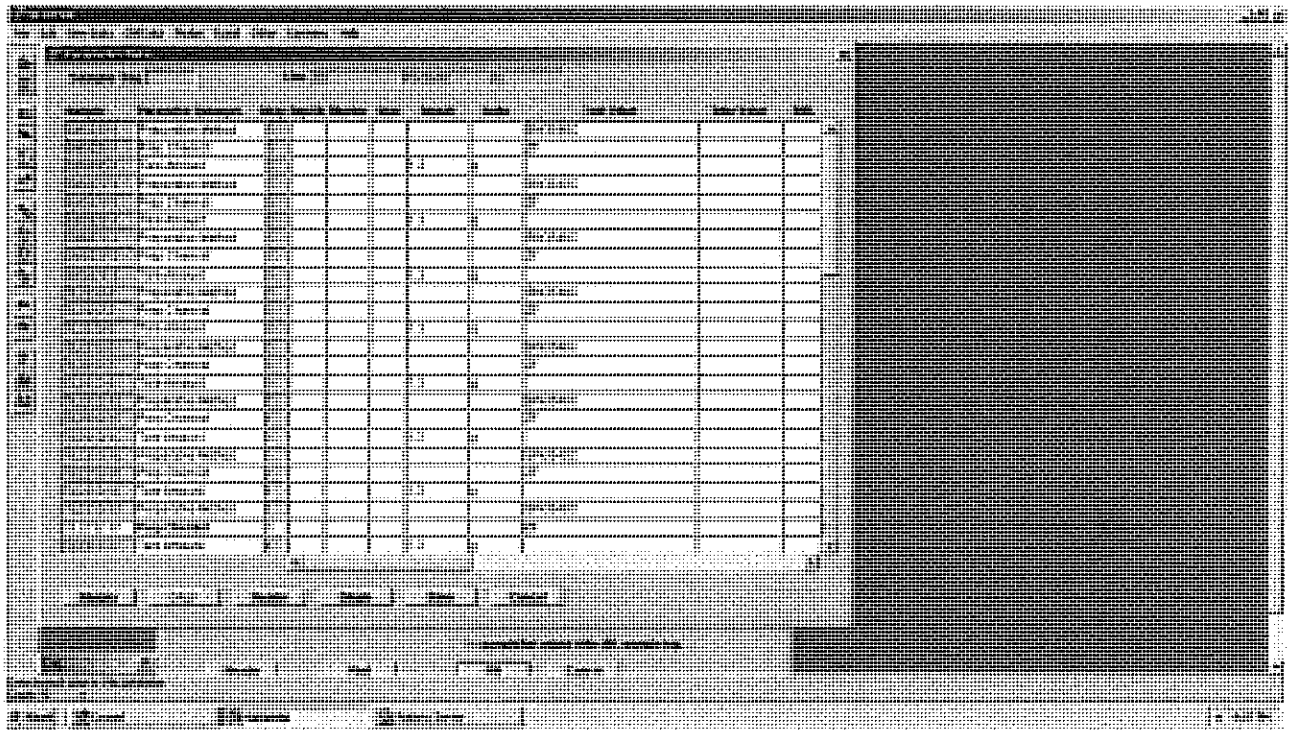
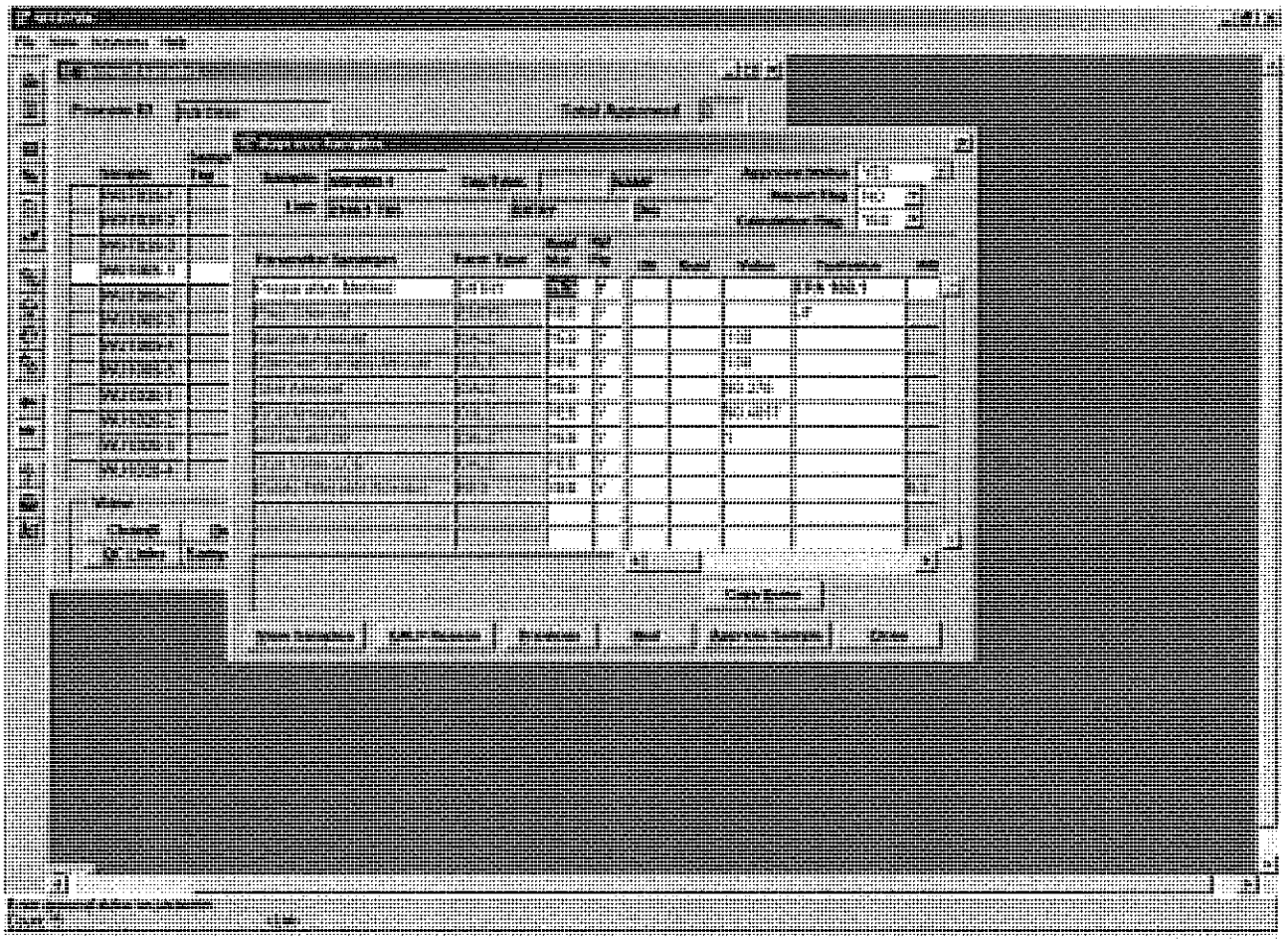


FIGURE 11

DATA APPROVAL SCREEN



TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

FIGURE 12

EXAMPLE OF A TOTAL VOLATILE SOLIDS BATCH REPORT

WET CHEMISTRY BATCH REPORT
Sep 13 2017, 10:48 am
Batch: WG213301

Parameter: Solids-Loss On Ignition
Prep Date: 07-SEP-17

Date Analyzed: 12-SEP-17
Prep Method: SM 2540G

Analyst Initials: AZ
Prep Chemist: AZ

Sample	Samp Type	Method	Tare Amt.	Dry Wt.	Ash Wt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SK7950-1	SAMP	STDM 2540	1.325100g	23.96440g	23.04370g	1	4.06682	4.1 wt %	79.	.1	.1	.1		
WG213301-1	MBLANK	STDM 2540	1.264900g	6.863200g	5.855900g	1	.15875	0.16 wt %	NA	.1	.1	.1		
WG213301-2	DUP	STDM 2540	1.306800g	28.84130g	27.78800g	1	3.82538	3.8 wt %	NA	.1	.1	.1	6	

Comments:

WG213301-1 SK7950-1
WG213301-2 SK7950-1

Entered by: _____ Date: 9-13-17 Accepted by: _____ Date: 09/13/17 ✓

TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

FIGURE 13

EXAMPLE OF A TOTAL SOLIDS BATCH REPORT

TOTAL SOLIDS BATCH REPORT
Jan 11 2019, 03:47 pm
Batch: WS244289

Sample	Matrix	Type	Prep Date	Tare	Initial	Final1	Final	by	Date	Raw TS	Rep TS	Recovery	RPD
SM0235-1	SL	SAMP	10-JAN-19	1.3 g	25.7992 g	24.3957 g	24.3957 g	ZF	11-JAN-19	96.2710	96. %		
SM0999-1	SL	SAMP	10-JAN-19	1.3 g	29.6725 g	28.5602 g	28.5602 g	ZF	11-JAN-19	96.0809	96. %		
SM0277-2	SL	SAMP	10-JAN-19	1.3 g	26.3975 g	24.4552 g	24.4552 g	ZF	11-JAN-19	92.6820	92. %		
TL2212-33	SL	SAMP	10-JAN-19	1.3 g	30.1565 g	26.6185 g	26.6185 g	ZF	11-JAN-19	87.7382	88. %		
TL2212-36	SL	SAMP	10-JAN-19	1.3 g	24.7298 g	22.2857 g	22.2857 g	ZF	11-JAN-19	89.5680	90. %		
TL2212-37	SL	SAMP	10-JAN-19	1.3 g	26.6145 g	24.0331 g	24.0331 g	ZF	11-JAN-19	89.6030	90. %		
TL2212-2	SL	SAMP	10-JAN-19	1.3 g	23.4974 g	21.2688 g	21.2688 g	ZF	11-JAN-19	94.2048	94. %		
TL2212-24	SL	SAMP	10-JAN-19	1.3 g	26.9606 g	23.6604 g	23.6604 g	ZF	11-JAN-19	87.0378	87. %		
TL2212-27	SL	SAMP	10-JAN-19	1.3 g	29.457 g	25.3544 g	25.3544 g	ZF	11-JAN-19	85.4388	85. %		
TL2212-28	SL	SAMP	10-JAN-19	1.3 g	27.6814 g	24.3839 g	24.3839 g	ZF	11-JAN-19	87.8018	88. %		
TL2212-31	SL	SAMP	10-JAN-19	1.3 g	33.1882 g	26.8658 g	26.8658 g	ZF	11-JAN-19	80.1740	80. %		
TL2212-35	SL	SAMP	10-JAN-19	1.3 g	27.0438 g	25.6426 g	25.6426 g	ZF	11-JAN-19	92.2260	92. %		
TL2212-36	SL	SAMP	10-JAN-19	1.3 g	23.4091 g	19.8743 g	19.8743 g	ZF	11-JAN-19	84.4870	84. %		
TL2212-37	SL	SAMP	10-JAN-19	1.3 g	28.2184 g	25.0815 g	25.0815 g	ZF	11-JAN-19	88.3470	88. %		
TL2212-38	SL	SAMP	10-JAN-19	1.3 g	28.1898 g	23.7956 g	23.7956 g	ZF	11-JAN-19	82.6580	84. %		
TL2212-41	SL	SAMP	10-JAN-19	1.3 g	22.3134 g	18.7359 g	18.7359 g	ZF	11-JAN-19	82.9750	83. %		
TL2212-43	SL	SAMP	10-JAN-19	1.3 g	24.8061 g	21.4508 g	21.4508 g	ZF	11-JAN-19	85.7260	86. %		
TL2212-44	SL	SAMP	10-JAN-19	1.3 g	23.5359 g	19.7048 g	19.7048 g	ZF	11-JAN-19	82.7660	83. %		
TL2212-5	SL	SAMP	10-JAN-19	1.3 g	26.5824 g	22.8425 g	22.8425 g	ZF	11-JAN-19	82.0430	82. %		
WS244289-1	SL	MSLAB1	10-JAN-19	1.3 g	5.4432 g	5.6424 g	5.6424 g	ZF	11-JAN-19	99.9620	100 %		
WS244289-2	SL	LCS	10-JAN-19	1.3 g	6.1363 g	5.6327 g	5.6327 g	ZF	11-JAN-19	89.6060	90. %	100	
WS244289-3	SL	LCSD	10-JAN-19	1.3 g	6.1915 g	5.6883 g	5.6883 g	ZF	11-JAN-19	89.7130	90. %	100	0
WS244289-4	SL	DUP	10-JAN-19	1.3 g	31.5352 g	29.8459 g	29.8459 g	ZF	11-JAN-19	94.4130	94. %		0

Comments:

TL2212-43	MS/lab DUP
WS244289-1	SM0235-1
WS244289-2	SM0235-1
WS244289-3	SM0235-1
WS244289-4	SM0235-1

Entered by:_____ Date:_____ Accepted by:_____ Date:_____

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: BP

Review Date: 1-10-20

SOP Number: CA-717-07

SOP Title: Total Solids / Total Volatile Solids in Aq Matrices

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 01/13/20

QAO Signature: Leslie Dimond **Date:** 01.13.20

KATAHDIN ANALYTICAL SERVICES
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: John D. Gourley

Review Date: 1/11/21

SOP Number: CA-717-11

SOP Title: Total Solids/Total Volatile Solids Determination in Solid Matrices

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: K Brewer **Date:** 01/13/21

QAO Signature: Leslie Dimond **Date:** 01.15.21

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Prepared By: George Brewer Date: 01/01

Approved By:

Group Supervisor: George Brewer Date: 01/29/01

Operations Manager: Joel C. Burton Date: 1/29/01

QA Officer: Deborah J. Nadeau Date: 1·29·01

General Manager: Dennis P. Hoffman Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
00 ~~7470A~~	NA	DH	1·29·01	1/29/01
01	Revised Sect. 4, 5 and 7 to reflect current practice. Revised Sect. 8 to reflect current QC limits. Revised sect. 10 to reflect current Applicable Documents and references. Removed figure 2. Update table 1 to reflect current QC limits. Minor changes throughout	LAD	02·16·05	02·16·05
02	Updated Fig. 1 - new prep Logbook page	LAD	04/08	04/08
03	Updated Figure 1 - Example of a mercury Preparation logbook page.	LAD	03/09	03/09
04	Added LOD definition. Updated sections 8, 9, 10 and Table 1 for DOD QSM version 4.1 compliance.	DH	08/09	08/09

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
05	Added Table 2 - DoDQSM Version 4.1 QC Requirements.	LAD	04/10	04/10
06	Sect. 4.6 - Changed thermometer type. Sect. 7.3 - Changed type of marker used. Table 1 - Added PQL standard corrective action. Table 2 - added comments for calibration blank. Sect. 9 - Added MDL, LOD and LOQ information	LAD	05/11	05/11
07	Sect. 7 - Calibration prep from digesting all to digesting high STD. and diluting down. Added serial dilution and PDS to sect. 8. Added more MDL, LOD & LOQ information to Sect. 9. Updated and added references to Sect. 10	LAD	04/12	04/12
08	DoDQSM 5.0 References added. Sect. 7.4 and Table 3 - Updated Calibration Standard prep - removing digesting all Standards. Added to digest high point	LAD	06/14	06/14
09	Updated figure 1. Change title of section 5.0. Update method references for NELAC and DoD. Minor additions to sections 4.1, 4.2, 4.6, 7.1, 7.10, 7.12.	LAD	09/17	09/17
10	Removed DoDQSM 4.2 QC Requirement Table. Added DoDQSM 5.0/5.1 QC Requirement Table. Updated references. Updated Logbook Example	LAD	11/18	11/18
11	Sect. 7 - Updated to reflect current calibration and Independent Calibration Verification Standards	LAD	01/19	01/19
12	Removed use of Pyrex media bottles and VOA vials. Changed water bath to heating Block. Added the addition of 6 ml sodium chloride-hydroxylamine hydrochloride solution to standards. Table 1 - Added serial dilution and post digestion spike.	LAD	06/20	06/20
13	Changed instructions for making 5.0 mg/L calibration std., in Sect 7.4, to double the final volume.	LAD	02/21	02/21

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

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KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-615-13,** titled **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470.**

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TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure used by Katahdin Analytical Services personnel for the digestion and analysis of aqueous samples for mercury using cold vapor atomic absorption spectrophotometry.

This method is applicable to the determination of mercury in groundwaters, aqueous wastes, and mobility-procedure extracts under USEPA Method 7470 (Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods, SW-846, 2nd edition, 1982 (revised 1984), 3rd edition, 1986, and Updates I, II, IIA, and III 1996, Office of Solid Waste and Emergency Response, U.S. EPA.

1.1 Definitions

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified laboratory grade reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified laboratory grade reagent water used to verify calibration accuracy.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

PB - Preparation Blank - Laboratory grade reagent water that has been brought through the sample preparation process.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence by the instrument.

MDL - Method Detection Limit - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of mercury by USEPA Method 7470. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of mercury by USEPA Method 7470 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, that their work is properly documented, and to indicate periodic review of the associated logbooks.

1.3 Safety

Many of the samples and reagents used in cold vapor atomic absorption are toxic or corrosive. Rubber gloves, safety glasses, lab coats, and other protective clothing should be worn whenever these materials are handled. Because of the toxic nature of mercury vapor, care must be taken to avoid its inhalation. The instrument exhaust fan must be in operation whenever the mercury analyzer is in use (the fan should never be shut off).

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this

method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and follow appropriate procedures such as wearing safety glasses and gloves when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location and use of all safety equipment.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in cold vapor atomic absorption may contain high concentrations of acids, mercury, and other toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested mercury samples and standards and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled “Waste Stream A”) that is located in the Metals Prep lab. Further information regarding waste classification and disposal may be obtained by consulting the laboratory's Hazardous Waste Management Plan and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

The cold vapor atomic absorption technique is based on the absorption of radiation at 253.7 nm by mercury vapor. It relies on the volatility of elemental mercury at room temperature. During preparation, organic mercurials are oxidized and elemental mercury is ionized to Hg^{3+}. During instrumental analysis, mercuric ions are reduced to elemental mercury by the addition of stannous chloride. Elemental mercury is then aerated from solution and passes through a cell positioned in the path of a mercury spectrophotometer, where absorbance (peak height) is measured as a function of mercury concentration and recorded by the associated computer. The mercury vapor is then swept out of the instrument into an exhaust hood, where it is evacuated from the laboratory.

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3.0 INTERFERENCES

In addition to inorganic forms of mercury, organic mercurials may be present in environmental samples. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. The presence of undigested organo-mercurials in samples will result in a low bias for analytical results. Certain volatile organic materials will also non-specifically absorb radiation at the 253.7 nm analytical wavelength. The presence of such compounds may result in a high bias for analytical results. For these reasons, complete digestion using potassium permanganate and potassium persulfate is required for all environmental samples. Complete digestion is indicated by the persistence of the purple permanganate color (indicating the presence of excess permanganate) following digestion.

Sea waters, brines, and industrial effluents high in chlorides may require additional permanganate to maintain a persistent purple color following digestion. During the oxidation step, chlorides are converted to free chlorine which will absorb radiation at the 253.7 nm analytical wavelength. Any free chlorine thus generated will be present in the headspace of the digestion vessel following digestion. Because samples are poured into autosampler tubes prior to analysis by the mercury analyzer, any free chlorine present in the headspace of the digestion vessels is not sampled by the instrument and the analysis is free of chlorine interference.

4.0 APPARATUS AND MATERIALS

4.1 40 mL, 50 mL or 70 mL digestion tubes and appropriate watch glasses, for use as digestion vessels.

4.2 Heating block capable of maintaining a constant temperature of 95° C.

4.3 Adjustable volume automatic pipettes - 2 to 20 uL, 10 to 100 uL, 100 to 1000 uL. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.4 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, concentrated sulfuric acid, and other reagents

4.5 Battery powered Traceable Pocket-Size Thermometer from Fisher Scientific, NIST-traceable, covering the range from -50° to 750° C, for monitoring the temperature of the water bath. Mercury-filled thermometers are not acceptable for use in the metals laboratory, due to the possibility of breakage and consequent contamination.

4.6 Disposable graduated polystyrene sample cups, 200 mL capacity

4.7 CETAC M-6100 automated mercury analyzer and associated peripherals and parts

4.8 Disposable graduated dose cups, 30 mL capacity

Refer to Katahdin SOP CA-629, current revision, "Operation and Maintenance of the CETAC M-6100 Automated Mercury Analyzer" for additional required materials.

5.0 REAGENTS AND STANDARDS

5.1 Laboratory grade reagent water – mercury-free water meeting the specifications of ASTM Type II water

5.2 Concentrated sulfuric acid, trace metals grade

5.3 Concentrated nitric acid, trace metals grade

5.4 Concentrated hydrochloric acid, trace metal grade

5.5 Potassium permanganate solution, 5% w/v: Dissolve 50 g of potassium permanganate in 1 L laboratory grade reagent water. The source reagent should be labeled as suitable for use in mercury determination.

5.6 Potassium persulfate solution, 5% w/v: Dissolve 50g of potassium persulfate in 1L laboratory grade reagent water. The source reagent should be labeled as suitable for use in mercury determination.

5.7 Sodium chloride – hydroxylamine hydrochloride solution: Dissolve 120 g sodium chloride and 120 g hydroxylamine hydrochloride in laboratory grade reagent water and dilute to a final volume of 1 L.

5.8 Stannous chloride solution: Add 70 mL concentrated hydrochloric acid to 500 mL of laboratory grade reagent water. Add 100 g stannous chloride and bring to a final volume of 1 L. Mix to dissolve. Reagent should be labeled as suitable for use in mercury determination.

5.9 Intermediate Mercury Standard A: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. This intermediate standard is used to prepare calibration standards, matrix spikes, CCVs, and laboratory control samples (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard A must be prepared fresh monthly ,and disposed of appropriately after use. (Note: the concentrations of all stock standards must be certified by the vendors as traceable to NIST reference materials).

5.10 Intermediate Mercury Standard B: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. The source of the stock standard used to prepare Intermediate Mercury Standard B must be distinct from that used to prepare Intermediate Mercury Standard A (i.e. obtained from a separate vendor). Intermediate Mercury Standard B is used to prepare the ICV (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate standard may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard B must be prepared fresh monthly, and disposed of appropriately after use.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Aqueous samples to be analyzed for mercury should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	28 days
Aqueous (dissolved)	P, G	250 mL	HNO_3 to pH < 2	28 days

[1] P = polyethylene or G = glass

7.0 PROCEDURES

BOTTLE PREPARATION

7.1 Mercury digestions are performed in two different types of vessels. Calibration standards, the Initial Calibration Verification (ICV) standard, and the Initial/Continuing Calibration Blank (ICB/CCB) are prepared in disposable graduated polystyrene sample cups. These are used to provide sufficient volumes of these standards to allow for multiple reanalyses when required. Field samples, Method Blanks, and Laboratory Control Samples are digested in 40 mL, 50 mL or 70 mL digestion tubes. These smaller vials provide enough digestate to allow one or two reanalyses when required, but reduce the amounts of samples consumed and waste generated.

The disposable graduated polystyrene sample cups in which standards are prepared are emptied, rinsed, and reused. Each of these bottles is permanently marked with the concentration of the standard it contains.

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PREPARATION OF STANDARDS, QC SAMPLES, AND BLANKS

7.2 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS Metals database and print out a copy of the sample prep bench sheet. All necessary details of sample preparation (standards preparation information, digestion times, initial and final volumes, pertinent observations, etc.) must be recorded on this spreadsheet, which will be bound in the Mercury Preparation Logbook. Refer to Figure 1 for an example page from the Mercury Preparation Logbook.

7.3 Using an industrial marker with super permanent ink, label clean sample containers with the appropriate sample numbers and standard identifications for each sample, preparation blank, laboratory control spike and matrix spike and standard to be digested.

7.4 Calibration Preparation –

- Using a bottle-top dispenser, add approximately 10 mL of laboratory grade reagent water to two standard digestion bottles (disposable graduated polystyrene sample cups).
- Using a repipettor, add 2.5 mL concentrated nitric acid and 5 mL concentrated sulfuric acid to each bottle.
- The blank calibration standard is prepared in the same manner as the high calibration standard except for the addition of mercury standard.
- Using a calibrated adjustable pipette, prepare the high calibration standard by adding 1000 uL of Intermediate Mercury Standard A to the appropriately labeled media bottle.
- Using a dose cup, add 15 mL potassium permanganate to each calibration standard.
- Using a repipettor, add 8 mL potassium persulfate solution to each calibration standard and swirl to mix.
- Add 6 mL Sodium chloride – hydroxylamine hydrochloride solution (sect. 5.7).
- Fill each bottle to 160 mL with laboratory grade reagent water. The mercury concentration of the high calibration standard is 10.0 ug/L.

Calibration levels 0.2 ug/L, 0.5 ug/L, 1.0 ug/L, 5.0 ug/L are made by diluting the 10.0 ug/L standard into calibration blank solution. See below for amounts. The 0.2 ug/L and 5 ug/L standards are analyzed after calibration as the PQL standard and the CCV (refer to Section 8.0), respectively, as well as being used in the creation of the calibration curve.

Calibration Level	Amount added	Amount calibration blank solution
0.2 ug/L	0.3 mL	14.7 mL
0.5 ug/L	0.5 mL	9.5 mL
1.0 ug/L	1 mL	9 mL
5.0 ug/L	10 mL	10 mL

7.5 Independent Calibration Verification Standard –

- Using a bottle-top dispenser add approximately 10 mL of laboratory grade reagent water to a digestion bottle.
- Using a repipettor, add 2.5 mL concentrated nitric acid and 5 mL concentrated sulfuric acid to the bottle.
- Using a calibrated adjustable pipette, add 600 uL of Intermediate Mercury Standard B to the bottle.
- Using a dose cup, add 15 mL potassium permanganate to each calibration standard.
- Using a repipettor, add 8 mL potassium persulfate solution and swirl to mix.
- Add 6 mL of Sodium chloride – hydroxylamine hydrochloride solution
- Fill to 160 mL with laboratory grade reagent water.
- The mercury concentration of the ICV standard is 6.0 ug/L.

7.6 Prepare an appropriate number of preparation blanks (PBW) by adding 25 mL of laboratory grade reagent water to labeled vials.

7.7 Prepare an appropriate number of laboratory control samples (LCSW) by adding 125 uL of Intermediate Mercury Standard A to labeled digestion vials containing 25 mL of laboratory grade reagent water. The mercury concentration of each LCSW is 5.0 ug/L.

7.8 Matrix spikes are prepared by adding 25 uL of Intermediate Mercury Std A to 25 mL aliquots of samples. The concentration of mercury added to each matrix spike is 1.0 ug/L.

7.9 Preparation blanks, laboratory control spikes and matrix spikes are digested in the same manner as client samples. Refer to Sample Preparation and Digestion, sections 7.10 through 7.13 of this SOP. The volumes of reagents added to the standards prepared in the media bottles are four times those listed in sections 7.10 through 7.13 but the standards are not heated.

SAMPLE PREPARATION AND DIGESTION

7.10 Using a graduated disposable dosecup or pour directly into graduated sample tube, transfer 25 mL of sample, or an aliquot diluted to 25 mL, to a digestion vial. Add

1.25 mL of concentrated sulfuric acid and 0.625 mL of concentrated nitric acid, swirling to mix after each addition. Add 3.75 mL of potassium permanganate solution, swirl to mix, and allow to stand for at least 15 minutes. Samples that contain large amounts of organic substances may require additional 3.75 mL aliquots of potassium permanganate solution. This is indicated by the failure of the purple permanganate color to persist for the entire 15 minute waiting period. Add additional 3.75 mL aliquots to samples as necessary until the purple color persists for 15 minutes. If any of the samples require these additional aliquots of potassium permanganate solution, record the additional volume used for each sample on the mercury preparation benchsheet.

Some samples may require dilution to 25 mL with potassium permanganate for digestion to be performed in the digestion vessel. Prepare method blank and LCS with equal amounts of potassium permanganate to check for potential mercury contamination.

7.11 Add 2 mL of potassium persulfate solution to each sample. Cap the vials, for 50 mL or 70 mL tubes, add ribbed watch glasses, and place them in a preheated heating block. Monitor the temperature of the bath with a thermometer throughout the digestion. The temperature of the water bath will fall below 90-95° C upon addition of the digestion vials. After the temperature of the bath has risen back to 90-95° C, continue heating the samples at 90-95° C for two hours. Record initial and final digestion times and temperatures in the mercury prepareation benchsheet.

7.12 Remove bottles from the heating black and allow to cool to room temperature. If the purple permanganate color has failed to persist after digestion in any of the samples, add additional 3.75 mL aliquots of potassium permanganate solution as required to the samples, and record these additions in the mercury preparation benchsheet. Heat the samples that required additional permanganate in the water bath at 90-95° C for an additional two hours. Remove the bottles from the water bath and allow to cool to room temperature. If the purple color fails to persist after the second heating step, consult the Department Manager for advice on how to proceed.

7.13 Add 1.5 mL of sodium chloride – hydroxylamine hydrochloride solution to each digestion vial and swirl to mix. This will reduce the excess permanganate, and the sample will change from purple to colorless. After color change, samples are filled to 40 mL with laboratory grade reagent water and mixed well. Wait at least 30 seconds before proceeding with analysis.

INSTRUMENTAL ANALYSIS

7.14 Digested mercury samples are analyzed using the CETAC M-6100 Automated Mercury Analyzer. Analysis is automated and is controlled by the QuickTrace Mercury Analyzer software running on a dedicated PC. Detailed instructions for

setting up the instrument and analyzing samples are given Katahdin SOP CA-629, "Operation and Maintenance of the CETAC M-6100 Automated Mercury Analyzer".

METHOD OF STANDARD ADDITIONS

7.15 The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. The method of standard additions shall be used for analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

7.15.1 The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_S of a standard analyte solution of concentration C_S. To the second aliquot (labeled B) is added the same volume V_S of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration C_x is calculated:

$$C_X = \frac{S_B V_S C_S}{(S_A - S_B) V_X}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_s and C_s should be chosen so that S_A is roughly twice S_B on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

7.15.2 Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is

shown in Figure 3. A linear regression program may be used to obtain the intercept concentration.

7.15.3 For the results of this MSA technique to be valid, the following limitations must be taken into consideration:

- The apparent concentrations from the calibration curve must be linear over the concentration range of concern. For the best results, the slope of the MSA plot should be nearly the same as the slope of the standard curve. If the slope is significantly different (greater than 20%), caution should be exercised.

- The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

- The determination must be free of spectral interference and corrected for nonspecific background interference.

DATA REDUCTION AND REPORTING

7.16 Results are obtained in concentration units (ug/L) from the instrument. Electronic instrument data files are imported into the Metals ACCESS database for data reduction. Sample preparation information (initial sample volumes and final digestate volumes) are entered directly into the Metals ACCESS database to allow calculation of final results for reporting. Results are calculated as follows:

$$\text{Mercury concentration (ug/L)} = \frac{MC \times DF \times IV}{FV}$$

where: MC = Measured mercury concentration (ug/L)
DF = Dilution factor at instrument
IV = Initial sample volume (mL)
FV = Final digestate volume (mL)

7.17 Results that exceed the calibration range of the instrument may not be reported - the sample must be appropriately diluted and reanalyzed. Results for diluted samples should be multiplied by the dilution factor prior to reporting. If additional aliquots of potassium permanganate were added during digestion, the resulting dilution must be corrected for before reporting.

7.18 Results are reported down to the laboratory's practical quantitation level (PQL), unless otherwise requested. Results below the PQL should be reported to the PQL and flagged with a “U” qualifier.

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8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 7470 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Preparation instructions and the resulting mercury concentrations for calibration standards, QC standards, and matrix spikes are detailed in Sections 7.4 through 7.8 of this SOP. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument by each method. This determination requires seven replicate analyses of a laboratory grade reagent water spiked at 3-5 times the anticipated detection limit for each analyte, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of laboratory grade reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain

the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 Limits of Detection (LOD) are used when evaluating data using DoD QSM. The LOD is established by spiking a quality system matrix at 2-3 times the detection limit for a single analyte standard and 1-4 times the detection limit for a multi-analyte standard. The LOD must be verified quarterly. For more information on performing LOD determinations, refer to the current revision of Katahdin SOP QA-806.

8.4 Instrument calibration - The instrument must be calibrated each time it is set up, and calibration standards must be prepared each day that samples are digested. Calibration includes analysis of a calibration blank and five calibration standards with graduated concentrations in the appropriate range. The concentration of one of the calibration standards must be at the Practical Quantitation Level (PQL). The intermediate standards used for preparing the calibration standards are prepared at least once per month in 2% nitric acid. Because mercury may be adsorbed onto the walls of glass and plastic containers, the calibration standards must be prepared fresh daily. The correlation coefficient for the calibration curve must be at least 0.995. If the calibration curve does not pass this test, analysis must be halted, the problem corrected, and the instrument recalibrated.

8.5 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared from a standard source different than that of the calibration standard and at a concentration within the working range of the instrument. The result of the ICV must fall within 90% to 110% of the expected value. If the ICV fails, results may not be reported from the run until the problem is corrected and a passing ICV has been analyzed.

8.6 The Continuing Calibration Verification (CCV) solution is analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standard used for calibration at a concentration near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected value. If a CCV fails, associated sample results may not be reported from the run until the problem is corrected and a passing CCV has been analyzed. Also, all samples analyzed after the last passing CCV must be reanalyzed.

8.7 A calibration blank is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If samples are being run using DoD QSM criteria, the absolute values of ICBs and CCBs must be less than the Limit of Detection (LOD). If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has

been analyzed. Also, all samples analyzed after the last passing CCB must be reanalyzed.

8.8 A standard with a mercury concentration that is at the Practical Quantitation Limit (PQL) is analyzed at the beginning of the run to determine calibration accuracy at the reporting limit. Result of the PQL standard should fall within 70% to 130% of the expected values. No corrective action has been established at this time.

PREPARATION BATCH QC SAMPLES

8.9 Preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) for each element. For DoD QSM acceptance criteria the results must be less than ½ the PQL except for common contaminants which must be less than the PQL. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL (greater than ½ PQL for DoD), associated sample results that are less than the PQL (less than ½ PQL for DoD) or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.10 A laboratory control sample (LCSW), consisting of spiked reagent carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless laboratory-generated statistical limits are available. If a laboratory control sample fails, results may not be reported from the digestion batch, and all associated samples must be redigested.

SAMPLE MATRIX QC SAMPLES

8.11 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. Matrix spike recoveries for these samples are calculated as follows:

$$\text{Recovery (\%)} = \frac{(P - S)}{A} \times 100\%$$

where: P = Spiked sample value
S = Original sample value
A = Spike amount

The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, a matrix interference should be suspected and the associated sample result should

be flagged on the report of analysis. If DoD QSM acceptance criteria are being used, recoveries must be the same as stated for laboratory control samples.

The relative percent difference between matrix spiked duplicate sample results is calculated as follows:

$$RPD\ (\%) = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = Spike sample result
D_2= Spike duplicate sample result

A control limit of 20% RPD is applied to matrix spike duplicate analysis. If the matrix spike duplicate analysis fails, the associated sample result should be flagged on the report of analysis.

8.12 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL or 50 x LOQ if using DoD QSM acceptance criteria), the measured concentration of a serial dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$Difference\ (\%) = \frac{|L-S|}{S} * 100\%$$

where: L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The associated sample result should be flagged on the report of analysis or the sample should be reanalyzed at dilution to eliminate the interference.

8.13 Post-digestion Spike (PDS) additions must be performed for DoD QSM samples if the serial dilution is not within acceptance criteria or if the analyte concentrations in all samples are less than 50x the LOD. The spike addition should produce a concentration that is between 10 and 100x the LOQ. The recovery of the PDS must be within 75-125%. If the PDS fails, all samples must be run by method of standard additions or appropriately flagged.

8.14 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the

contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of USEPA Method 7470 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Wastes, United States Environmental Protection Agency, USEPA SW 846, Third Edition, Final Update III (9/94), Method 7470A.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1.1, 2018.

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The 2009 TNI Standards

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.
Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications.

QuickTrace M6100 Mercury Analyzer Operator Manual Version 1.0.1, CETAC Technologies.

QuickTrace Mercury Analyzer Software Manual, CETAC Technologies.

List of Tables and Figures

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TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA 7470	Initial Calibration, 5 points plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient ≥ 0.995.	Correct problem and repeat calibration.
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	Correct problem and repeat calibration.
	Initial Calibration Blank (ICB)	Before beginning a sample run.	Less than PQL.	Correct problem and repeat calibration.
	Practical Quantitation Level Standard (PQL)	Before beginning a sample run.	Recovery within ± 30% of true value.	Correct problem and repeat calibration.
	Continuing Calibration Verification (CCV)	At beginning or run, after every 10 samples, and at end of the run	Recovery within ± 10% of true value	Repeat calibration and reanalyze all samples analyzed since the last successful CCV.
	Continuing Calibration Blank (CCB)	At beginning or run, after every 10 samples, and at end of the run	Less than PQL.	Repeat calibration and reanalyze all samples analyzed since the last successful CCB.
	Preparation Blank (PBW)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and < 10x the blank concentration.
	Laboratory Control Sample (LCSW)	One per digestion batch of 20 or fewer samples.	Recovery within ± 20% of true value.	Redigest all affected samples.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery ±25% of true value, if sample > 4x spike value.	Flag results.
	Matrix Spike Duplicate Sample (P)	One per digestion batch of 20 or fewer samples.	1) Recovery ± 25% of true value, if sample < 4x spike added. 2) RPD ≤20% for duplicate spikes.	Flag results
	Serial Dilution	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.
	Post-Digestion Spike	One per preparatory batch if MS or MSD fails.	Recovery within 80-120%	No specific CA, unless required by the project.

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	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < PQL	1) Repeat IDL study. 2) Raise PQL.
	Limit of Detection (LOD) determination	Quarterly.	LOD = 2-3X MDL	Repeat LOD Determination.
	Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

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Table 2

DOD QSM REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analysis.	r2 = 0.99.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	FLAA and GFAA: minimum three standards and a calibration blank. CVAA/Mercury: minimum 5 standards and a calibration blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of the true value.	Correct problem. Rerun ICV. If that fails, Rerun ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reprepped or reanalyzed.
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.	Flagging is not appropriate.	Results may not be reported without a valid calibration blank. For CCB, failures due to carryover may not require an ICAL.

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QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to the source of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.
Dilution Test (Flame AA and GFAA only)	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations > 50 X LOQ (prior to dilution). Use along with MS/MSD or PDS data to confirm matrix effects.
Post-Digestion Spike (PDS) Addition (Flame AA and GFAA only)	One per preparatory batch if MS or MSD fails.	Recovery within 80-120%	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Criteria apply for samples with concentrations < 50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution or post digestion spike fails and if the required by project.	NA.	NA.	NA.	Document use of MSA in the case narrative.

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TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-615-12	USEPA METHOD 7470
Reagents	1) Stannous chloride dissolved in hydrochloric acid to prevent clogging of mercury analyzer, per instrument manufacturer's recommendation.	1) Stannous chloride dissolved/suspended in sulfuric acid.
Procedures	2) Sampling and gas stream switching performed automatically by mercury analyzer. 3) Working Mercury standard prepared monthly in 2% nitric; calibration standards prepared fresh daily.	2) Sampling and gas stream switching performed manually by analyst. 3) Working Mercury standard prepared fresh daily and acidity maintained at 0.15% nitric.
QC – Calibration Verification	4) Known reference sample (ICV) analyzed daily. 5) Calibration verified after every 10 samples with CCV.	4) Known reference sample analyzed quarterly. 5) Calibration verified after every 20 samples.
QC - Calibration Blanks	6) Acceptance criteria employed for 245.1: ± PQL	6) Acceptance criteria stated in 245.1: ± MDL

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FIGURE 1

EXAMPLE PAGE FROM MERCURY PREPARATION LOGBOOK

Katahdin Analytical Services, Inc. — **Metals Preparation Benchsheet**

Reagents and Consumables Information:

HNO3: MSR149 H2SO4: MSR103 Digestion Vessles: 180905 Method: 7470

KMNO4: MR2174 K2S2O8: MR2142 NH2OH-HCl: MR2168

REVIEWED JS 11.13.18 KATAHDIN ANALYTICAL METALS SECTION

Standards/Spiking Information:

1ppm A : MW18167
Pipet 1ppm B : MW18158
MH LCSW = 125uL of 1ppm A to 25mL
MH Spike(S/P) = 25uL of 1ppm A to 25mL
MP ICV = 600uL of 1ppm B to 100 mL
MP S10.0 = 1000uL of 1ppm A to 100 mL

Heat Source ID : B
Start Time: 1142 /Temp. 92 °C
End Time: 1342 /Temp. 90 °C
Thermometer ID/Pos: AL25/24

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Bottle
LCSWLK12HGW1	LK12HGW1	0.075	L	0.075	L	AQ	HG	AMJ	11/12/2018	
PBWLK12HGW1	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-002	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-003	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-004	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-005	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-006	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-006P	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	S
TL0847-006S	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	S
TL0847-007	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-008	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-009	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-010	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-011	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-012	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-013	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0912-001	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	C
TL0912-001P	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	C
TL0912-001S	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	C
TL0912-002	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	A
TL0912-003	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-004	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-005	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-006	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-007	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-008	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	

JS 11.13.18

QA-066-Revision 4 - 09/28/2018 Digestion performed by: AMJ On: 11/12/18 Page: LK061 Revision: 00

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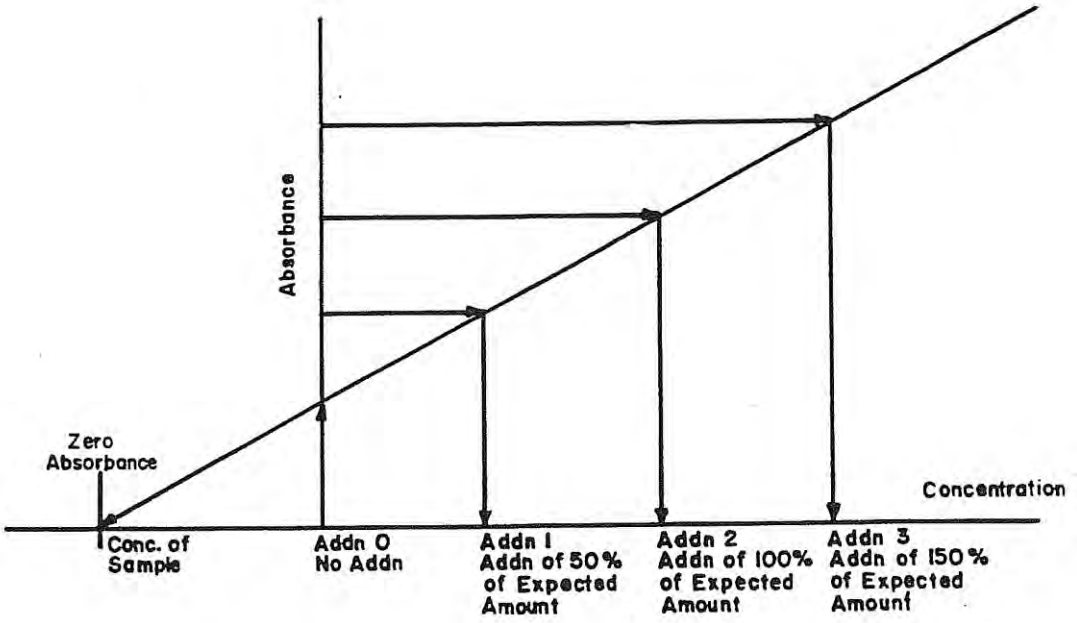
FIGURE 2

STANDARD ADDITIONS PLOT



Attachment 3

Field Modification Form (Template)

FIELD MODIFICATION FORM
FOR
GOWANUS CANAL SUPERFUND SITE
WSP

Date:

Document:

Activity:

Requested Modification:

Rationale:

Attachments:

Investigative Organization Project Manager: _____

Investigative Organization Technical Lead: _____

Investigative Organization Site Quality Control Officer: _____

Attachment 4

Non-Conformance Form (Template)

GOWANUS CANAL SUPERFUND SITE

NON-CONFORMANCE/QUALITY CONTROL

REPORT

Date: _____

Organization Name: _____

Initiator's Name & Title: _____

Problem Description:

_____

_____

_____

_____

_____

_____

_____

Reported To: _____

Corrective Action:

_____

_____

_____

_____

_____

_____

_____

Reviewed and Implemented by: _____

Attachment 5

Example Chain of Custody (COC) for shipment of samples to laboratory

Gowanus Canal Superfund Site
Date Shipped:
Carrier Name:
Airbill No:

CHAIN OF CUSTODY RECORD
Gowanus Superfund Site
Contact #:
Contact Phone:

No: X-XXXXX-XXXXXX-XXXX
Cooler #
Lab:
Lab Phone:

Lab #	Sample #	Location	Analyses	Matrix	Sample Date	Sample Time	Numb Cont	Container	Preservative	Lab QC

Special Instructions:	**Samples Transferred From**
	Chain of Custody #

Items / Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt

Attachment 6

Field Mobilization Protocol

NYCDEP Proposed Mobilization Protocol for Sampling
Gowanus Canal CSOs

1. NYCDEP Mobilization Procedure Overview:

   a) NYCDEP will track forecast daily to look for approaching storms that meet criteria for successful CSO sampling

   b) NYCDEP will provide a series of alerts as storms meeting criteria approach

   c) Criteria will be adaptively managed

2. Rainfall Thresholds for mobilization:

   a) 48-hour antecedent dry (<0.1" rain) period: per EPA, wet-weather sampling should be performed after a sufficient period with no runoff. Generally, up to about 0.1 inches of rain is necessary to wet the ground before runoff may occur.

   b) CSO discharges: combined sewers overflow under certain hydraulic conditions. The primary factor is the amount of stormwater in the system. Another factor is tidal water elevation. Each outfall has a unique hydraulic condition at which it overflows. See Table 1 below

Table 1. Rainfall Thresholds for Discharge from CSOs

CSO Outfall	MinimumHourly Intensity [2](in/hr)	MinimumTotal Rain[3] (in)
OH-007	0.20	0.55
RH-034	0.20	0.20

Notes:

1. Assumes 72-hr antecedent dry-weather (≤ 0.1 inch rain) conditions.
2. Minimum hourly rainfall intensity over a 3-hour period required to generate a discharge, given the typical daily range of tidal elevations.
3. Minimum total rainfall over a 3-hour period required to generate a discharge, given the minimum hourly intensity shown and the typical daily range of tidal elevations.

3. Alerts:

   a) NYCDEP will issue "Storm Watch" >48 hours before forecasted rain meeting criteria for triggering a CSO/MS4 event:

      i. Criteria:

- >50% chance of targeted rain (may vary depending on status of program)
- >48 hrs dry (less than 0.1" rain)
- Lab turn-around time (minimum 10 business days between sampling events)

- No "black-out periods" (such as holidays, other reasons)

ii. Action:

- NYCDEP designated mobilization leader issues "Storm Watch" with timing to field crews

iii. Response:

- Potential field personnel are identified and asked to reserve the time

b) NYCDEP will issue "Storm Warning" >24 hours before rain for triggering a CSO/MS4 event:

i. Criteria:

- >80-85% chance of targeted rain (may vary depending on status of program
- Likely favorable tidal conditions at onset of storm (receding/low tides at onset of storm will avoid sampling complications associated with backwater)
- >48 hrs dry (less than 0.1" rain)
- Lab turn-around time (minimum 10 business days between sampling events)
- No "black-out periods" (such as holidays, other reasons)

ii. Action:

- NYCDEP mobilization leader issues "Storm Warning" with timing to field crews.

iii. Response:

- Field crews on stand-by to deploy for mobilization

c) NYCDEP will issue "Mobilization Notice" issued 4-6 hours before rain and/or anticipated CS)/MS4 discharge period:

i. Criteria:

- >90% chance of targeted rain (may vary depending on status of program)
- Favorable tidal conditions at onset of storm (receding/low tides at onset of storm will avoid sampling complications associated with backwater)
- >48 hrs dry (less than 0.1" rain)
- Lab turn-around time (minimum 10 business days between sampling events)
- No "black-out periods" (such as holidays, other reasons)

ii. Action:

- NYCDEP mobilization leader issues "Mobilization Notice" with timing and muster location to field crews
- NYCDEP mobilization leader provides required notices to DEP and EPA as necessary.

iii. Response:

- Field crews deploy to muster point

d) NYCDEP will issue "Cancellation Notices" after deployment:

i. Criteria:

- Determined by NYCDEP Mobilization Leader or Field Leader
- May be based on change in forecast or development of unsafe conditions (such as lightening, excessive winds, etc.)

ii. Action:

- NYCDEP Mobilization leader or Field Leader issues "Cancellation Notice" to field crews, DEP, EPA, NCG, etc. as necessary

iii. Response:

- Field crews return to muster point and demobilize.