


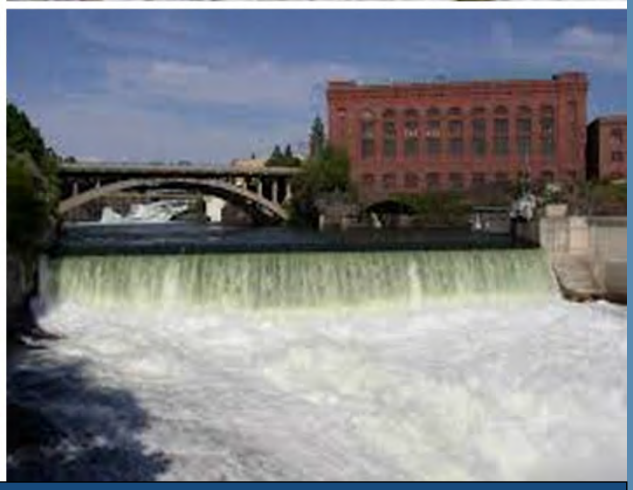

Quality Assurance Project Plan

Spokane River Toxics Reduction Strategy Study

Prepared for:
Spokane River Regional Toxics Task Force

FINAL

Approved by SRRTTF: July 23, 2014
Effective Date: July 23, 2014

LimnoTech
Water Environment | Scientists Engineers

APPROVALS (A.1)

Quality Assurance Project Plan

July 23, 2014

_____ Date:_____

Bud Leber, Kaiser Aluminum
SRRTTF ACE - President

_____ Date:_____

David Dilks, LimnoTech
Project Manager

_____ Date:_____

Jim Bellatty, Water Quality Section Manager
Eastern Region, Washington Department of Ecology

_____ Date:_____

Robert Steed, Surface Water Ecologist
Coeur d'Alene Regional Office
Idaho Department of Environmental Quality

_____ Date:_____

Cathy Whiting, LimnoTech
Field Manager

_____ Date:_____

Carrie Turner, LimnoTech
Project Quality Assurance Officer

_____ Date:_____

Shea Hewage, Operations Director
AXYS Analytical Services

_____ Date:_____

Dale Hoover, Laboratory QA Manager
AXYS Analytical Services

_____ Date:_____

John Kern, Laboratory Technical Director
SVL Analytical, Inc.

_____ Date:_____

Michael Desmarais, Laboratory QC Manager
SVL Analytical, Inc.

_____ Date:_____

Shawn Hinz, Project Manager
Gravity Environmental

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DISTRIBUTION LIST (A.3)

QUALITY ASSURANCE PROJECT PLAN

JULY 23, 2014

The approved Quality Assurance Project Plan, and any subsequent updates, will be distributed to the following list of project personnel:

Name	Project Role	Organization	Location	Phone Number
Bud Leber	SRRTF-ACE, President	Kaiser Aluminum Trentwood	Spokane Valley, WA	(509) 927-6554
David Dilks	Project Manager	LimnoTech	Ann Arbor, MI	(734) 332-1200
Adriane Borgias	Advisor	Washington State Department of Ecology	Spokane, WA	(509) 329-3515
Arianne Fernandez	Advisor	Washington State Department of Ecology	Spokane, WA	(509) 329-3498
Robert Steed	Advisor	Idaho DEQ	Coeur d'Alene, ID	(208) 769-1422
Cathy Whiting	Field Manager	LimnoTech	Ann Arbor, MI	(734) 332-1200
Carrie Turner	Project QA Officer	LimnoTech	Ann Arbor, MI	(734) 332-1200
Shea Hewage	Laboratory Operations Director	AXYS Analytical Services	Sidney, BC Canada	(250) 655-5800
Cynthia Tomey	Laboratory Project Manager	AXYS Analytical Services	Sidney, BC Canada	(250) 655-5800
Dale Hoover	Laboratory QA Manager	AXYS Analytical Services	Sidney, BC Canada	(250) 655-5800
John Kern	Laboratory Technical Director	SVL Analytical, Inc.	Coeur d'Alene, ID	(208) 784-1258
Michael Desmarais	Laboratory QC Manager	SVL Analytical, Inc.	Coeur d'Alene, ID	(208) 784-1258
Christine Meyer	Laboratory Project Manager	SVL Analytical, Inc.	Coeur d'Alene, ID	(208) 784-1258

Shawn Hinz	Project Manager	Gravity Environmental	Fall City, WA	(425) 281-1471

1. PROJECT MANAGEMENT (GROUP A)

The purpose of the Quality Assurance Project Plan (QAPP) is to document the necessary procedures required to assure that the project is executed in a manner consistent with applicable United States Environmental Protection Agency (U.S. EPA) guidance documents (EPA 1998, 2001), the Washington Department of Ecology (Ecology) guidance document (Washington Department of Ecology 2004) and with generally accepted and approved quality assurance objectives. This QAPP is organized in accordance with the basic groups and subgroup elements discussed in the U.S. EPA guidance for QAPPs. The four basic groups include project management (Group A); data generation and acquisition (Group B); assessment and oversight (Group C); and data validation and usability activities (Group D). The groups are subdivided into elements covering specific topics related to each group. The Section and Subsection headings of this QAPP include references to the U.S. EPA QAPP Guidance group letters and element numbers to facilitate cross-reference with the Guidance.

The QAPP integrates quality control policies and project-specific work tasks to successfully conduct water quality monitoring to support the toxics reduction strategy. The member organizations of the SRRTTF will actively participate and provide funds to the project. The SRRTTF-Administrative and Contracting Entity (SRRTTF-ACE) will serve as the contracting authority for the project and provide overall program management. The SRRTTF-ACE will coordinate communications to the SRRTTF regarding information and data that is generated as a result of this project. The SRRTTF has hired LimnoTech as a Technical Advisor. For the purposes of this project, LimnoTech serves as Project Manager, Field Manager, and Project Quality Assurance Officer (QAO). LimnoTech is responsible for the preparation of this Quality Assurance Plan and the associated Sampling and Analysis Plan (SAP) for the project. Gravity Environmental will be responsible for sample collection. AXYS Analytical Services will perform laboratory analysis for PCB congeners for the project as a contractor to SRRTTF. SVL Analytical, Inc. will perform laboratory analysis for all other parameters.

The QAPP has been prepared in compliance with U.S. EPA and Ecology requirements. It is the overall intent of the QAPP to provide professional guidelines for activities by all personnel on the project and to ensure that quality assurance/quality control (QA/QC) procedures are followed.

1.1 Project Organization (A.4)

Each of the organizations included in the project team has established an organizational structure for providing technical direction and administrative control to accomplish quality-related activities for the development of the project.

Key project personnel and their corresponding responsibilities are listed in Table 1 below and shown in Figure 1.

Table 1. Project Team Responsibilities

Name/Affiliation	Project Title/Responsibility
SRRTTF	**Oversight and direction** Secure funding for project activities Review and utilize project results Facilitate communications and provide public access to information Develop recommendations for controlling and reducing sources Develop comprehensive plan
Bud Leber – SRRTTF-ACE	**SRRTF ACE President** Manage contracts: review and approve project specifications Ensure project is completed in timely manner Receive deliverables and reports Manage data on behalf of SRRTTF Communicate with SRRTTF Communicate quality assurance issues with SRRTTF Ensure access to project information on the SRRTTF website Facilitate upload of data to EIM
David Dilks - LimnoTech	**Project Manager** General oversight Review/approval of all work products prior to delivery to SRRTTF-ACE Ensures that work is done in accordance with QAPP and SAP Reviews project with Laboratory Operations Directors prior to sampling Provides oversight of field activities (variances, documentation, QA/QC) Arranges for system audits
Jim Bellatty, Adriane Borgias – Department of Ecology	**Advisor** Reviews/approves QAPP
Robert Steed – Idaho DEQ	**Advisor** Reviews/approves QAPP
Cathy Whiting - LimnoTech	**Field Manager: Synoptic Survey and Quarterly sampling events** Direct all field activities, ensure samples handled in accordance with SAP Data screening, evaluation, validation, and usability determination Manage field variances, nonconformance, and corrective actions Manage reports, documentation, Project QA/QC file, and electronic data Communicates project specifics with Project Manager Conducts training of field sampling crew
Carrie Turner - LimnoTech	**Project Quality Assurance Officer** Performs systematic evaluation of data quality Receives notices, initiates investigation, and documents nonconformance with DQOs Manage the Project QA/QC file
LimnoTech	**Independent Auditor** Perform a critical, written evaluation of the work product Conducts audits at the direction of the Project Manager
Shea Hewage – AXYS Analytical Services	**Laboratory Operations Director** Sample analysis Serves as main point of contact for laboratory Manages laboratory Quality Assurance systems Final review and validation of data and field systems Initiates corrective actions for nonconformance Communicates with Project Manager and SRRTTF-ACE
Richard Grace – AXYS	**Laboratory Project Director** Oversight of all laboratory commercial and technical project specifications
Cynthia Tomey – AXYS Analytical Services	**Laboratory Project Manager** Serves as main point of contact for laboratory Assists Laboratory Operations Director with management of laboratory QA systems

Name/Affiliation	Project Title/Responsibility
	Communicates with Project Manager
Dale Hoover-AXYS Analytical Services	**Laboratory QA/QC Managers** Manages Laboratory QA/QC activities Reviews and verifies field records, laboratory records and laboratory data Addresses nonconformance and carries out corrective actions at the laboratory.
John Kern – SVL Analytical, Inc.	**Technical Director** Sample analysis Serves as main point of contact for laboratory Manages laboratory Quality Assurance systems Final review and validation of data and field systems Initiates corrective actions for nonconformance Communicates with Project Managers and SRRTTF-ACE
Michael Desmarais– SVL Analytical, Inc.	**Laboratory QA/QC Manager** Manages Laboratory QA/QC activities Reviews and verifies field records, laboratory records and laboratory data Addresses non-conformances and carries out corrective actions at the laboratory.
Christine Meyer – SVL Analytical, Inc.	**Laboratory Project Manager** Serves as main point of contact for laboratory Assists Laboratory Operations Director with management of laboratory QA systems
Shawn Hinz – Gravity Environmental	**Conducts Sample Collection** Collects samples in accordance with QAPP and SAP Prepares and follows the Invasive Species Plan Prepares and administers Health and Safety Plan for employees Maintains equipment logs, field records and data sheets Transfers field data to Field Manager Manages field equipment, conducts calibrations Addresses nonconformance findings and responds to corrective actions

All of the organizations in the project have the responsibility of ensuring that their employees receive the appropriate technical and administrative direction that is provided by this QAPP and the related SAP.

The lines of reporting for the organizations in the project are shown in the organization chart (Figure 1). Each team member has responsibility for performance of assigned quality control duties in the course of accomplishing identified activities. The quality control duties include:

- Completing the assigned task on or before schedule and in a quality manner in accordance with established procedures; and
- Ascertaining that the work performed is technically correct and meets all aspects of the QAPP.

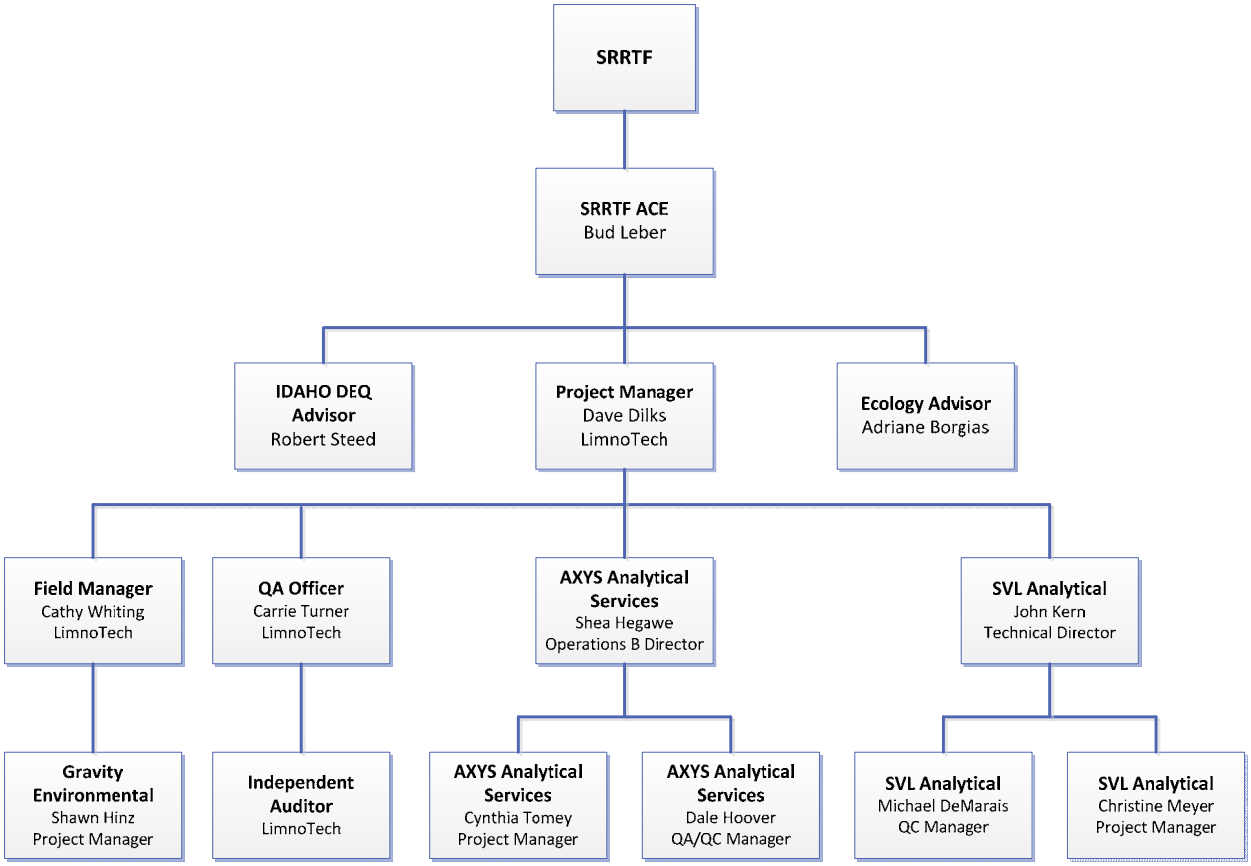

Figure 1. Project Team Organization

Project Team Responsibilities

LimnoTech's role is to ensure that the project is conducted in accordance with the requirements of the QAPP and SAP. LimnoTech is primarily responsible for preparation of the SAP and QAPP, field management, quality assurance and technical support. As Project Manager, David Dilks, LimnoTech, is responsible for general oversight of the project, including review and approval of all work products prior to delivery to SRRTTF-ACE.

Consultants to the project include LimnoTech of Ann Arbor, Michigan and Gravity Environmental of Fall City, Washington. The SRRTTF has oversight of the project and development of this QAPP. The SRRTTF-ACE is responsible for management and oversight of all consultants and deliverables. The SRRTTF-ACE oversees the development of the QAPP by LimnoTech, with the input of the SRRTTF. AXYS Analytical Services is responsible for laboratory analysis of PCBs and SVL Analytical, Inc. is responsible for testing associated with all other lab parameters.

1.2 Project Background (A.5)

The goal of the Spokane River Regional Toxics Task Force (SRRTTF) is to develop a comprehensive plan to reduce PCB inputs to the Spokane River and to bring into compliance with applicable water quality standards for PCB. PCBs are the pollutant of primary concern, however dioxins will be addressed as resources allow for inclusion in the comprehensive plan formulation (LimnoTech, 2014a).

The Spokane River and Lake Spokane exceed the water quality standard (170 pg/L – based on fish consumption rate of 6.5 g/day) for polychlorinated biphenyls (PCBs) in several segments. Fifteen waterbody segments of the Spokane River and Lake Spokane (also known as Long Lake, herein referred to as Lake Spokane) and one segment of the Little Spokane River are on the 2008 303 (d) list for exceeding human health water quality criteria for PCBs. The specific impairments are shown in Table 2. The Spokane Tribe of Indians have water quality standards for PCBs in the Spokane River below Lake Spokane (also known as the Spokane Arm of Lake Roosevelt) that are more than 95% lower than State standards (1.3 pg/L), based on a higher fish consumption rate (865 g/day) than the general population (Spokane Tribe of Indians, 2010). PCBs are not listed in Idaho.

In April 2011, the Department of Ecology published a PCB source assessment report based on data collected during the period of 2003 to 2007 (Publication No. 11-03-013). In Figure 19 of this report a schematic diagram summarized the state of knowledge with respect to identified sources and in-stream loads for Total PCB. This figure showed an identified source contribution to the river of 996.9 mg/day of PCB between the Idaho/Washington state line (RM 96.1) and Ninemile Dam (RM 58.1). In addition, the figure also showed an in-stream loading increase of 1,804 mg/day between these two locations. Thus, source contribution of 807.1 mg/day of Total PCB was not able to be accounted for – roughly 44.7% of the in-stream loading between those two points on the river.

To accomplish its goal, the SRRTTF is taking what has been referred to as a "Direct to Implementation" approach. In order to take this approach, the SRRTTF has determined that it needs to develop a sufficient clearer understanding of in-stream loadings and source contribution to the Spokane River between its headwaters at the outlet of Lake Coeur d'Alene (RM 111) and the Ninemile Dam (RM 58.1) (Figure 2). This 53 mile segment of the river has been chosen to be the focus of the SRRTTF's initial efforts for several reasons. In no particular order they are:

- Discharges from all of the major municipal and industrial sources in the watershed are located in this section
- Virtually all urban area storm runoff in the watershed (the largest identified source contribution from the 2003-2007 data) enters the river in this section
- This section of the river contains numerous river flow gauging stations, which will allow for the determination of in-stream loadings at multiple locations through mass balance calculations
- In this section of the river the vast majority of the aquifer/river interchange occurs, the impact of which has not been quantified by previous studies
- The likelihood of making near term source contribution reductions is greatest in this section of the river given the concentration of point source and storm runoff locations and the significant level of unidentified source contribution
- The ability to monitor and assess the effectiveness of PCB reductions is enhanced by the ability to track in-stream loadings with the infrastructure present (gauging stations) in this section of the river

To develop a sufficiently clear understanding of in-stream loadings and source contribution, data will need to be collected at various times of the year so that the seasonal variability of in-stream loading at the outlet of Lake Coeur d'Alene can be evaluated. In addition to potential seasonal loading variability, the contribution of groundwater as well as episodic storm runoff events to in-stream loading needs to be quantified and more clearly understood. Once a clearer understanding of in-stream loading and source contribution is obtained, the SRRTTF can then move forward with developing recommendations for controlling and reducing sources through such efforts as providing input on Toxic Management Plans, Source Management Plans, and Best Management Practices (BMPs).

This study uses the best technology available to assess current conditions of the river. The PCB concentrations in the water are expected to be very low, close to or below the limits of the analytic system to evaluate with statistical rigor.

As stated above, the data collection and analysis efforts of the SRRTTF are focused on supporting the "Direct to Implementation" approach. With this approach being the focus of the SRRTTF's efforts, data collection is not intended to satisfy the requirements of data collection needs for regulatory undertakings such as evaluating compliance with applicable water quality standards for PCB or developing information for Load or Wasteload Allocations. It is possible that the data collection on in-stream loadings and source contribution may be usable by some NPDES permit holders for fulfilling some permit monitoring requirements.

This QAPP was developed to address the first year of data collection and is designed to ensure that all monitoring activities undertaken result in representative water quality and quantity information necessary to support a low-flow mass balance assessment to the extent possible, given the limitations of the data, and assess the seasonal variability of upstream loads. Monitoring and sampling stations have been selected to provide appropriate coverage to meet the assessment needs of the task force.

Table 2. Spokane River 2012 303(d) listing for total PCB in fish tissue

Waterbody	Listing ID	Medium	Parameter	2012 Category*
Spokane River	8201	Fish tissue	PCB	5
Spokane River	8202	Fish tissue	PCB	5
Spokane River	8207	Fish tissue	PCB	5
Spokane Lake	9015	Fish tissue	PCB	5
Spokane Lake	9021	Fish tissue	PCB	5
Spokane River	9027	Fish tissue	PCB	5
Spokane River	9023	Fish tissue	PCB	5
Little Spokane River	9051	Fish tissue	PCB	5
Spokane River	14385	Fish tissue	PCB	5
Spokane River	14397	Fish tissue	PCB	5
Spokane River	14398	Fish tissue	PCB	5
Spokane River	14400	Fish tissue	PCB	5
Spokane River	14402	Fish tissue	PCB	5
Spokane Lake	36440	Fish tissue	PCB	5
Spokane Lake	36441	Fish tissue	PCB	5

* Category 5 means that Ecology has data showing that the water quality standards have been violated for one or more pollutants, and there is no TMDL or pollution control plan.

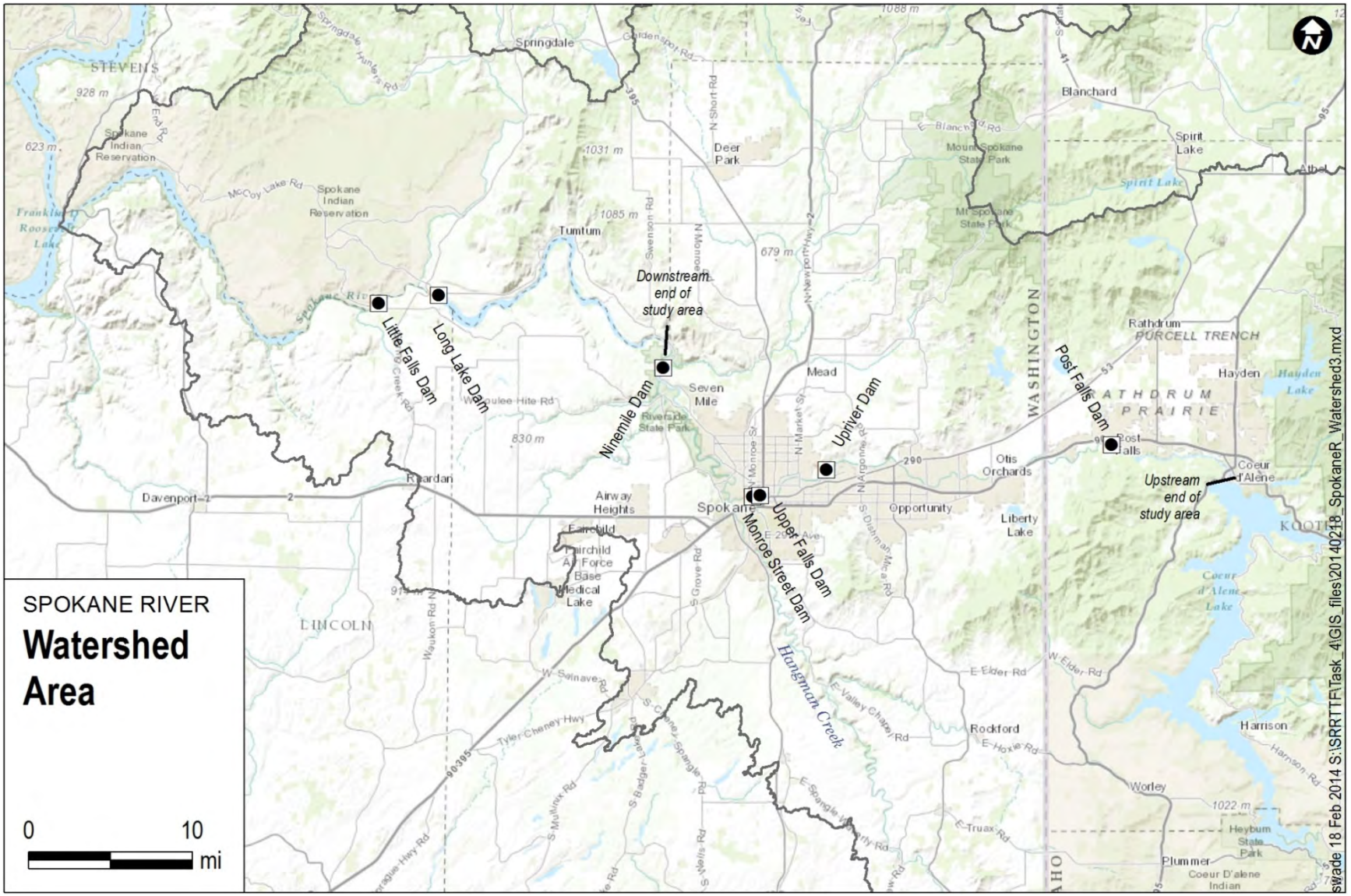

Figure 2. Spokane River Study Area

1.3 Project/Task Description (A.6) and Schedule

The Spokane River watershed has existing PCB monitoring data, which provide an estimate of the amount of PCBs entering the Spokane River from contributing source area categories (e.g. stormwater, WWTPs). Based on the Spokane River PCB Source Assessment 2004-2007 (Serdar et al, 2011), only 43% of the PCB source loading to the river between Stateline (RM 96.1) and Long Lake Dam (RM 33.9) could be identified. This is due in part to the uncertainty of the analyses and the high variability in the data. The existing data indicate that sources of PCBs are very diffuse throughout the watershed, such that more data will be needed to support development of a management plan with targeted control actions (LimnoTech, 2013a). Primary data gaps include:

- **The magnitude of true sources contributing to stormwater loads:** An existing dataset characterizes PCB concentration at numerous locations throughout the stormwater system, unfortunately these data indicate that PCB sources are very diffuse and difficult to trace back to their origin.
- **PCB sources upstream of the Idaho/Washington border:** PCBs entering from Idaho were estimated to represent 30% of the overall loading to the Spokane River in Washington.
- **The significance of loading from atmospheric and groundwater sources:** Insufficient data presently exist to define the magnitude of these source categories.

The objective of this project is to collect the necessary data to eliminate the data gaps in order to conduct a semi-quantitative PCB mass balance assessment of the Spokane River (LimnoTech, 2014a). Based on the results of the Confidence Interval Testing described below (LimnoTech, 2014c) the project objectives have been revised as follows:

a. The data shall be sufficient to support a semi-quantitative mass balance assessment, and be able to identify stream reaches where incremental loads lead to a significant increase in river concentrations.
b. The data shall be sufficient to support an adaptive management approach, where grab sample results can be directly compared to results from other sampling methodologies to allow determination of an improved monitoring approach for future phases of this work.

The first year of monitoring under this study includes the following tasks:

1. **Synoptic Study:** Conducted along the length of the river between Lake Coeur d'Alene and 9 Mile dam, during the summer low flow period.
2. **Seasonal Integrated Sampling:** Conducted at the Lake Coeur d'Alene outlet, during three different flow regimes.

1.3.1 Confidence Interval Testing

Confidence Interval Testing was performed in May 2014 to provide information for the sampling program. The Confidence Interval Testing was performed by Ecology as an initial task to confirm the appropriate sample volumes and frequencies. This initial sampling effort is described in the Confidence Interval Testing Memorandum (LimnoTech, 2014b) and is designed to generate information both on the temporal variability of PCB concentrations, as well as estimates of measurement uncertainty for the low PCB concentrations occurring in the Spokane River.

Five sampling events were conducted in May 2014 on the Spokane River at the State Park Parcel at River Mile 87, located between Mirabeau and Sullivan Parks (referred to as the Mirabeau Park site) and three sampling events at the Lake Coeur d'Alene outlet. Samples were collected for both discrete and composite analyses at

Mirabeau Park, while discrete samples were collected at the Lake Coeur d' Alene outlet. This information was used to satisfy three objectives:

1. Generate site-specific information on the sources of variability in PCB measurements (i.e. laboratory vs. variability in ambient concentrations)
2. Generate estimates of the confidence limits around the results to be obtained from the upcoming Synoptic Survey.
3. Determine if the proposed sampling methodology will provide data that can be distinguished from the lab blank.

As stated above, the draft QAPP (5/1/14) objectives have been revised based on the results of this sampling effort (LimnoTech, 2014c).

1.3.2 Synoptic Survey

The Synoptic Survey will consist of dry weather sampling at multiple locations in the Spokane River upstream of Lake Spokane, consisting of:

- River locations with flow gaging stations
- NPDES permitted sources
- Latah (Hangman) Creek Mouth

The Synoptic Surveys is designed to build upon the existing Ecology mass balance assessment (Serdar et al, 2011) and address data gaps related to groundwater and the nature of upstream sources of PCBs. Collection of data specifically at locations where flow gaging data are available will allow all concentration measurements to be converted to mass loads. While the results of the Confidence Interval Testing show that the planned monitoring may not distinguish differences in concentration between stations of up to 28 pg/l, the monitoring may be sufficient to identify incremental changes in concentration larger than that amount. Being able to identify the existence of larger increases in concentration will help support project objectives of identifying major PCB sources. In addition, the data shall be sufficient to support an adaptive management approach, where grab sample results can be directly compared to results from other sampling methodologies to allow determination of an improved monitoring approach for future phases of this work.

The Synoptic Survey sample locations are summarized in Table 3. River locations are identified as in-stream samples and NPDES permitted sources are identified as discharge samples. The point of discharge is determined to be the location identified in the dischargers NPDES permit or as determined in the field by the sampling team and approved by the project manager. The sample locations are shown in Figure 3.

Sampling will be conducted during the summer low flow period to minimize potential confounding effects of wet weather sources. Multiple river sampling events will be conducted (with some compositing to reduce analytical costs) over a two week sampling period to reduce the uncertainty in loading estimates caused by day to day variability in concentrations.

1.3.3 Seasonally Integrated Sampling

The Seasonally Integrated Sampling will consist of sampling at the outlet of Lake Coeur d'Alene. The intent of this monitoring is to provide information on the seasonal variability of upstream PCB loading to the Spokane River from Lake Coeur d'Alene, which will provide insight on the atmospheric contribution to the snow pack in the upstream watershed.

The sampling will be conducted on a seasonally integrated basis, with multiple samples taken and composited over each of three different flow regimes:

- Spring high flow
- Summer low flow (conducted as part of the Synoptic Survey)
- Winter moderate flow

The Seasonally Integrated Sampling locations are summarized in Table 3. The sample locations are shown in Figure 3.

1.3.4 Parameters

The study parameters include PCB congeners, total suspended solids (TSS), total dissolved solids (TDS), total organic carbon (TOC) and dissolved organic carbon (DOC). TSS, TOC and DOC will be used to provide information on the distribution of PCBs among various forms (i.e. purely dissolved, adsorbed to solids, sorbed to DOC), which will be needed if a fate and transport model is developed. TDS can be used as a tracer to provide information on groundwater contribution to the river. The parameters included in the Synoptic Survey and the Seasonally Integrated Sampling are listed in Table 4.

Sample collection details are provided in the Sampling and Analysis Plan (SAP).

1.3.5 Schedule

Key milestones associated with the project are described below along with their targeted completion dates:

QAPP and SAP approved by Task Force	May, 2014
Select laboratory	April 23, 2014
Sampling Contractor Request for Proposals sent out	May 7, 2014
Select Sampling Contractor	May, 2014
Confidence Interval Testing Sampling	May, 2014
Incorporate Confidence Interval Testing Results into QAPP/SAP	July 31, 2014
Contractor Training	August, 2014
Synoptic Survey	August, 2014
Seasonal Sampling – 1st Event	Fall, 2014
Seasonal Sampling – 2nd Event	Winter, 2015
Seasonal Sampling – 3rd Event	Spring, 2015
Draft Report	August 31, 2015
Final Report	October 31, 2015

1.3.6 Budget

The budget for this project includes the following:

Project Management	$90,000
Sampling	$145,000
Laboratory Analyses	$159,000
Confidence Interval Testing	$40,000
Total	$434,000

The funding provided by the Washington State Legislature ($350,000) must be expended by June 30, 2015.

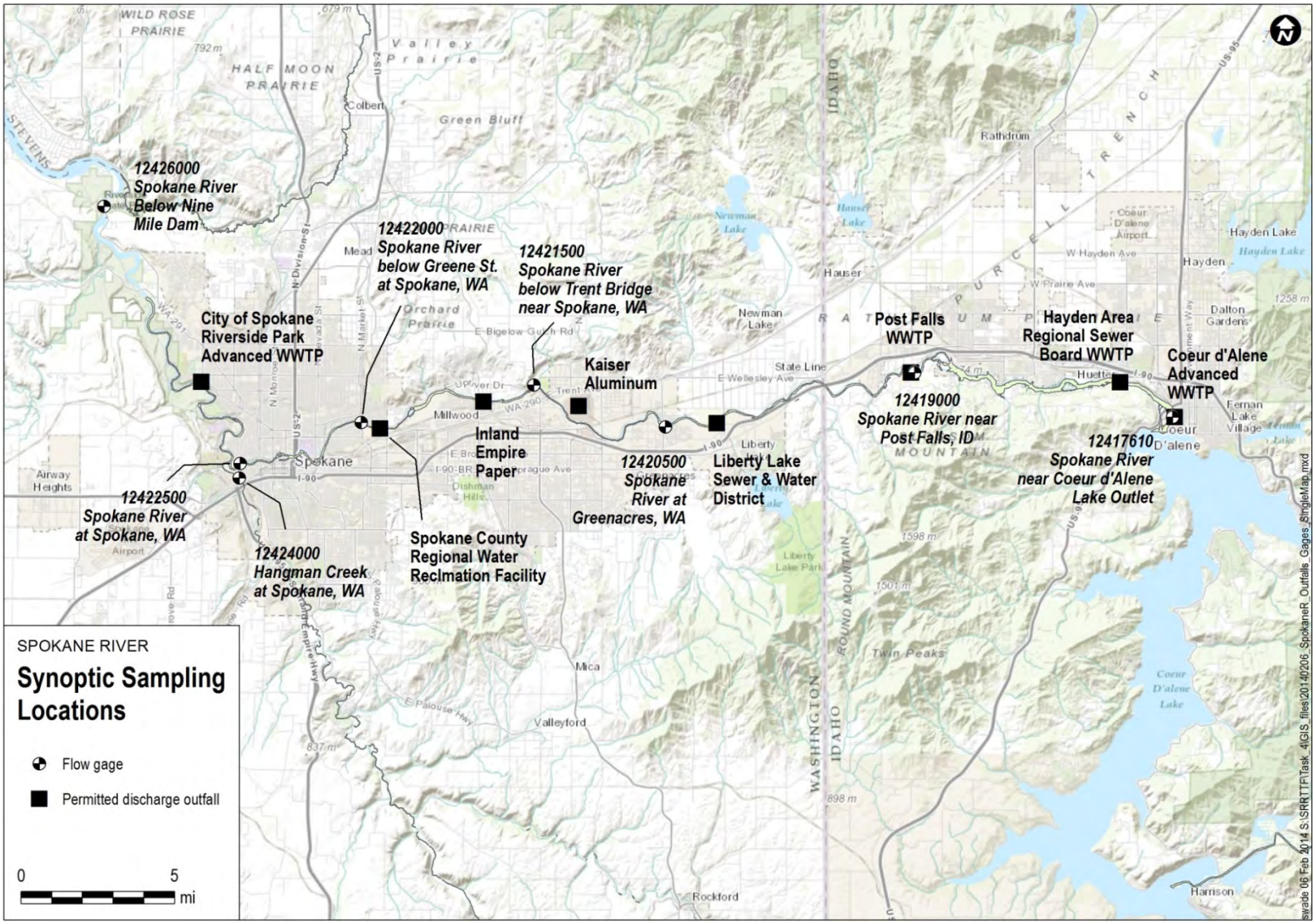

Figure 3. Spokane River Monitoring Locations Map

Table 3. Spokane River Monitoring Locations

Site	Location	Type of Sample	Low Flow Synoptic Survey	Seasonally Integrated Sampling
SR-1	Spokane River Below 9 Mile Dam	In-stream	X	
SR-2	City of Spokane Riverside Park Advanced WWTP	Discharge	X	
SR-3	Spokane River at Spokane	In-stream	X	
HC-1	Hangman Creek	In-stream	X	
SR-4	Spokane River at Greene Street Bridge	In-stream	X	
SR-5	Spokane County Regional Water Reclamation Facility	Discharge	X	
SR-6	Inland Empire Paper	Discharge	X	
SR-7	Spokane River at Below Trent Bridge	In-stream	X	
SR-8	Kaiser Aluminum	Discharge	X	
SR-9	Spokane River at Barker Road Bridge	In-stream	X	
SR-10	Liberty Lake Sewer & Water District Water Reclamation Facility	Discharge	X	
SR-11	Post Falls WWTP	Discharge	X	
SR-12	Spokane River at Post Falls	In-stream	X	
SR-13	Hayden Area Regional Sewer Board WWTP	Discharge	X	
SR-14	Coeur d'Alene Advanced WWTP	Discharge	X	
SR-15	Lake Coeur d'Alene Outlet	In-stream	X	X

Table 4. Spokane River Monitoring Parameters

Parameter	Type of Parameter
Polychlorinated Biphenyl (PCB)– 209 Congeners	Laboratory analytical
Dissolved Organic Carbon (DOC)	Laboratory analytical
Total Organic Carbon (TOC)	Laboratory analytical
Total Suspended Solids (TSS)	Laboratory analytical
Total Dissolved Solids (TDS)	Laboratory analytical
Temperature	In-situ measurement
Conductivity	In-situ measurement
pH	In-situ measurement
Dissolved Oxygen (DO)	In-situ measurement
Turbidity	In-situ measurement

1.4 Quality Objectives and Criteria (A.7)

The data quality objectives are intended to clarify the study's technical and quality objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of the data needed to support decisions. The data quality objectives for this study have been developed in order to ensure that the data collected are of acceptable quality and support the objectives of the project. It is anticipated that the PCB concentrations in the water will be very low, close to or below the limits of the analytic system to evaluate with statistical rigor. The sampling and analytical methods described in this QAPP are intended to provide a level of quality that allows the data to be suitable for a semi-quantitative low-flow mass balance assessment and to assess the seasonal variability of upstream loads. In addition, the data shall be sufficient to support an adaptive management approach, where

grab sample results can be directly compared to results from other sampling methodologies to allow determination of an improved monitoring approach for future phases of this work.

Confidence Interval Testing (LimnoTech, 2014b) was conducted prior to initiating the project to verify that the sampling and analytical protocols specified in this QAPP will be adequate. The results of the Confidence Interval Testing (LimnoTech, 2014c) were compared to the study's data quality objectives and data quality indicators. The draft QAPP (5/1/14) has been revised to address changes in project objectives and use of the data as described in Section 1.3.

The data that will be collected to support the Spokane River toxics reduction strategy will be evaluated relative to the data quality objectives outlined in this section. Data quality will be interpreted using the Data Quality Indicators (DQIs) which are the quantitative statistics and qualitative descriptors used to interpret the degree of acceptability of the data to the user. The DQIs include bias and precision, representativeness, completeness, comparability, and the required detection limits (sensitivity) for the analytical methods. These objectives also serve as a basis for developing the project's SAP.

The Data Quality Indicators and the measurement performance criteria for each are provided in Tables 5 and 6.

1.4.1 Accuracy

Accuracy is the degree of agreement between a measured value and the "true" or expected value. It represents an estimate of total error from a single measurement, including both systematic or matrix error (bias), and random error (precision) that may reflect variability due to sampling and analytical operations. Tables 5 and 6 provide a summary of the Data Quality Indicators.

Laboratory Bias

AXYS Analytical Services will do the PCB analyses using EPA Method 1668C/AXYS Method MLA-010 Revision 11 to perform low-level analysis for 209 PCB congeners using HRGS/HRMS instrumentation (Appendix A). Further information on the AXYS Analytical Services requirements is contained in the laboratory Request for Qualifications and Quote, which is included in Appendix B. SVL Analytical, Inc. will conduct the laboratory analyses for all other parameters. The laboratories will analyze field and laboratory QA/QC samples using the laboratory analytical procedures and the analytical method to assess data quality.

Laboratory bias will be assessed through daily calibration verification, the analysis of matrix spikes (if needed), and laboratory control samples (LCS) to determine if the percent recoveries (%R) meet the Data Quality Indicators. For PCB analyses the LCS samples are the Ongoing Precision and Recovery (OPR), internal standards and labeled compounds. Matrix spikes will not be analyzed for PCB analyses. Matrix spikes for other parameters will provide information concerning the effect of the sample matrix on the measurement methodology.

The percent recovery is calculated as follows:

$$\%R = [(C_s - C_u)/C_A] * 100$$

Where:

C_S = measured concentration of spiked sample, mg/L
C_U = measured concentration of unspiked sample, mg/L
C_A = actual concentration of spike added, mg/L

And:

$$C_A = \{[(V_u * C_u) + (V_{std} * C_{std})]/(V_u + V_{std})\} - C_U$$

Where:

V_u = Volume of unspiked sample, ml
V_{std} = Volume of known standard added as spike, ml
C_{std} = Concentration of known standard added as spike, mg/L

The percent recovery utilizing laboratory control samples is calculated as follows:

$$\%R = (C_M/C_A) * 100$$

Where:

C_M = measured concentration of control sample
C_A = actual concentration of control sample

1.4.2 Precision

Precision is a measure of reproducibility of analytical results. It can be defined as the measure of agreement among repeated measurements of the same property under identical, or substantially similar conditions. Total precision is a function of the variability associated with both sampling and analysis. Replicate analyses and the analysis of matrix spike replicates will be performed to verify analytical reproducibility. Field precision is assessed through the collection and measurement of field replicates, which are listed in Table 7. Relative Percent Difference (RPD) shall be calculated for each of the replicates collected for all the parameters analyzed.

Laboratory Precision

The precision of the laboratory analysis for PCB will be determined through a laboratory duplicate that will be generated in the laboratory and is spiked with all PCB natives and all surrogates. This sample is run with all batches.

The precision of the laboratory analysis of TOC and DOC is assessed by the comparison of matrix spikes (MS) and matrix spike duplicates (MSD). The RPD between the analyte levels measured in the MS sample and the MSD sample will be calculated as follows:

$$RPD = \frac{|C_{MS} - C_{MSD}|}{0.5(C_{MS} + C_{MSD})} \times 100$$

Where:

C_{MS} = measured concentration of the matrix spike
C_{MSD} = measured concentration of the matrix spike replicate

In situations where spiked samples are not practicable (such as TSS) to assess laboratory precision, a comparison of laboratory replicate analyses will be performed in order to calculate the RPD.

Field Precision

Field precision tests are conducted for grab samples and physical parameter readings. The precision of grab samples is assessed by the comparison of field replicates. The relative percent difference (RPD) between the analyte levels measured in the field replicates will be calculated as follows:

$$RPD = \frac{|C_A - C_B|}{0.5(C_A + C_B)} \times 100$$

Where:

C_A = measured concentration of the sample
C_B = measured concentration of the field replicate

1.4.3 Representativeness

Representativeness is the degree to which sample data accurately reflect the characteristics of a population of samples and appropriately reflect the environment or condition being measured. Surface water sampling will be conducted as specified in the SAP, so that the collected data appropriately reflect river conditions. All in stream water quality samples will be collected by wading into the main channel flow, if possible. Due to the heterogeneous nature of the river, it is not possible to establish a numeric Data Quality Indicator for representativeness.

The data review and validation process is intended to evaluate whether or not the measurements were made and the physical samples were collected in such a manner that the resulting data is representative of the river conditions at the time of sampling.

1.4.4 Completeness

Completeness is a measure of the amount of valid data obtained from the monitoring program compared to the amount of data that were expected. The completeness goal is 100%. However, events that may contribute to reduction in measurement completeness include sample container breakage, inaccessibility to proposed sampling locations, field equipment failure, and laboratory equipment failures.

The percent completeness (%C) is determined as follows:

$$\%C = \frac{(M_V)}{(M_P)} \times 100$$

Where:

M_V = number of valid measurements
M_P = number of planned measurements

If the completeness objectives are not achieved for any particular category of data, the Project Manager will provide documentation as to why the objective was not met and how the lower percentage impacted the overall study objectives. If the objectives of the study are compromised, re-sampling or re-measurement may be necessary.

Laboratory Completeness

Laboratory completeness is a measure of the amount of valid measurements obtained from all samples submitted for each sampling activity. The Laboratory validates the numbers of valid measurements based on

standard processes. The completeness criterion for all measurements is 95 percent. Qualified data are included as valid measurements and will be addressed in the data analysis. The completeness criterion will be evaluated by the Project Manager and QAO in accordance with the data analysis procedures. If the completeness goal is not met, re-sampling and/or re-analyzing may be necessary.

Field Completeness

Field completeness is determined by the number of measurements collected versus the number of measurements planned for collection. Due to a variety of circumstances, sometimes not all samples scheduled to be collected can be collected (e.g. a creek is dry, equipment malfunctions). The total number of samples to be collected is summarized in Table 7. The number of measurements collected is validated by the Field Manager. The completeness criterion for all measurements and sample collection is 95 percent, but will be influenced by environmental situations that may alter monitoring schedules. In order to meet this goal, replicate samples will be collected at each sample location. The replicate samples that are not analyzed as QA/QC replicate samples will be stored for use in the case of sample container breakage or other problems encountered that require additional sample volume. If the completeness goal is not met, re-sampling may be necessary.

1.4.5 Comparability

Comparability is the confidence with which one dataset can be compared to another. It is achieved by maintaining standard techniques and procedures for collecting and analyzing samples and reporting the analytical results in standard units. Results of performance evaluation samples and systems audits will provide additional information for assessing comparability of data among participating contract laboratories, if applicable.

The objective for data comparability is to generate data for each parameter that are comparable between sampling locations and comparable over time. Data comparability will be promoted by:

1. Using standard U.S. EPA approved methods, where possible.
2. Consistently following the sampling methods detailed in the SAP.
3. Consistently following the analytical methods detailed in the QAPP.
4. Achieving the required Estimated Detection Limits detailed in the QAPP.

All sample collection and analytical methods will be specified, and any deviations from the methods will be documented. All results will be reported in the standard units shown in Tables 5 and 6. All field and laboratory calibrations will be performed using standards traceable to National Institute of Science and Technology (NIST) or other U.S. EPA approved sources for TSS, TDS, TOC, and DOC. The standards used for PCB natives and labeled surrogate analyses will be procured from two qualified sources.

The data review and validation process is intended to evaluate whether or not the measurements were made and the physical samples were collected in such a manner that the resulting data is comparable with other datasets.

1.4.6 Sensitivity

Sensitivity is the capability of a method or instrument to discriminate between measurement responses representing different levels of the variable of interest. Sensitivity is determined by the minimum concentration or attribute that can be measured by a method (estimated detection limit), by an instrument (instrument detection limit), or by a laboratory (quantitation limit). The sensitivity requirements for PCB analysis are further described in the laboratory Request for Qualification and Quote (Appendix B).

Estimated Detection Limit (EDL) is defined as the concentration or amount of an analyte which can be determined to a specified level of certainty to be greater than zero. The Estimated Quantitation Limit (EQL) is the lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. EQLs are normally arbitrarily set rather than explicitly determined. The relationship between the EDL and EQL is shown in Figure 4.

The required detection limits are provided in Tables 5 and 6. Results will be reported down to the EDL, based on the signal-to-noise ratio of two ratioing peaks and two ratioing peaks from their corresponding surrogates. The EQL, which is based on the lowest validated standard in the calibration curve, will be provided for each analytic result. Detected values below the EQL will be qualified with a J flag. Results below the EDL will not be reported.

Refer to Table 8 for the specification limits of the field measurement instruments.

Table 5. PCB data quality Indicators

		BIAS	BIAS	BIAS		PRECISION	SENSITIVITY	COMPLETENESS
	Analytical Method	Daily Calibration Verification	Lab Control Sample Recovery*	Sample and Method Blank Surrogate Recovery	Method Blank	Duplicate Sample	Detection Limit (Level at which non-detects are reported)	Completeness Criteria
		% recovery limits	% recovery limits	% recovery limits	Concentration (pg/L)	RPD (valid for congeners > 10x EDL)	Concentration (pg/L)	%
PCB Congeners	EPA 1668C /AXYS Method MLA-010 Rev 11	50-145%	50-150%	25-150%*	Maximum = 127 pg/L (total) Laboratory will B-qualify congeners results < 3x the concentration in an associated blank	50%	1-20	95

*Per AXYS Method MLA-010 Revision 11 for OPR, internal standards and labeled compounds.

Table 6. Data quality indicators – DOC, TOC, TSS, TDS

DQI		BIAS	BIAS	BIAS	PRECISION	PRECISION	SENSITIVITY	COMPLETENESS
Parameter	Analytical Method	Lab Control Sample	Matrix Spikes	Lab Blanks	Replicate Samples	Matrix Spike Replicate	Detection Limit	Completeness Criteria
		% recovery limits	% recovery limits		RPD	RPD		%
DOC	EPA 415.3	80-120%	80-120%	< ½ EQL	30%	20%	1 mg/L	95
TOC	EPA 415.1	80-120%	80-120%	< ½ EQL	30%	20%	1 mg/L	95
TSS	EPA 160.2	80-120%	--	< ½ EQL	30%	--	1 mg/L	95
TDS	EPA 160.1	80-120%	--	< ½ EQL	30%	--	1 mg/L	95

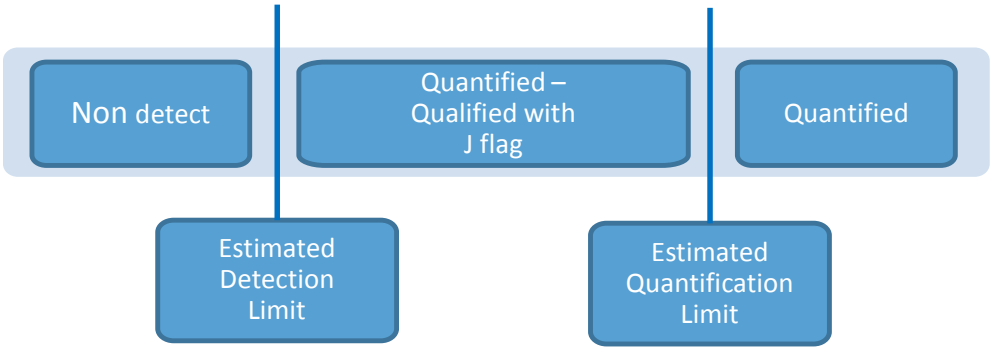

Figure 4. Schematic of detection limits

Table 7. Monitoring Program sample numbers

Parameter	Synoptic Survey Number of Samples Collected &Analyzed	Synoptic Survey Number of Replicate Samples Collected	Synoptic Survey Number of Replicate Samples Analyzed	Synoptic Survey Number of Composite Samples	Seasonally Integrated Sampling Number of Samples Collected & Analyzed	Seasonally Integrated Sampling Number of Replicate Samples Collected	Seasonally Integrated Sampling Number of Replicate Samples Analyzed	Seasonally Integrated Sampling Number of Composite Samples
PCB	80	80	10	16	10	10	10	2
Dissolved Organic Carbon	80	80	10	16	10	10	10	2
Total Organic Carbon	80	80	10	16	10	10	10	2
Total Suspended Solids	80	80	10	16	10	10	10	2
Total Dissolved Solids	80	80	10	16	10	10	10	2
Temperature	80	0	0	0	10	0	0	0
Conductivity	80	0	0	0	10	0	0	0
pH	80	0	0	0	10	0	0	0
Dissolved Oxygen	80	0	0	0	10	0	0	0
Turbidity	80	0	0	0	10	0	0	0

Table 8. Specification limits of field measurement instruments

Parameter	Instrument	Range	Accuracy	Resolution
Temperature	Hydrolab	-5 to 50°C	±0.10°C	0.01°C
	YSI	-5 to 45°C	±0.15°C	0.01°C
pH	Hydrolab	0 to 14 units	±0.2 units	0.01 units
	YSI	0 to 14 units	±0.2 units	0.01 units
Dissolved Oxygen	Hydrolab	0 to 20 mg/L	±0.2 mg/L	0.01 mg/L
	YSI	0 to 20 mg/L	±0.2 mg/L	0.01 mg/L
Conductivity	Hydrolab	0 to 100 mS/cm	±0.5% of range	1.0 uS/cm
	YSI	0 to 100 mS/cm	±1% of range	1.0 uS/cm
Turbidity	YSI	0-1000 NTU	±5% of range	0.1 units

1.5 Special Training/Certification (A.8)

Special training/certification needed for project personnel, including field, and laboratory staff in order to successfully complete project work is discussed in this section.

All laboratories will maintain the appropriate certifications and state approvals, which are included in Appendix C.

1.5.1 Project Staff

Professional staff (engineers, scientists and others) from LimnoTech and Gravity Environmental will be involved in this monitoring program. Personnel from AXYS Analytical Services and SVL Analytical, Inc. will conduct laboratory analysis of samples. Project staff will be assigned duties based on their qualifications to accomplish the task.

1.5.2 Field Staff

Field staff include the Field Manager (LimnoTech) and Gravity Environmental.

Training sessions will be conducted by the LimnoTech Field Manager for all field staff on proper sampling technique, sample handling and submission and general field procedures prior to conducting the first sampling event. Specific emphasis will be placed on QA/QC issues as well as on health and safety. Field staff will receive a safety briefing conducted by the LimnoTech field manager prior to the first sampling event. Emphasis will be on field hazards and materials handling. Gravity Environmental will develop the Health and Safety Plan.

Gravity Environmental will ensure that the field crews also receive training involving the operation, maintenance and calibration of field equipment including multi-parameter probes and all other on-site equipment used throughout the field program.

Standard Operating Procedures (SOPs) for program elements included in the SAP will be distributed to staff and available at all times.

1.5.3 Laboratory Staff

The Laboratory Project Managers will be the main points of contact for coordinating all sample receipt, etc. The Laboratory Project Manager will be assisted by the Laboratory Operations/Technical Director and Laboratory QA Manager in performing review and validation of all data generated to assure all data quality objectives have been met. The Laboratory Project Manager will contact the Project Manager immediately with any problems with samples noted during log in or with analysis. Prior to conducting the first sampling event, the Project Manager and Field Manager will meet with the Laboratory Project Manager to review details of the planned progression of sampling events.

AXYS Analytical Services will do the PCB analyses using EPA Method 1668C/AXYS Method MLA-010 Rev 11 to perform low-level analysis for 209 PCB congeners using HRGS/HRMS instrumentation (Appendix A). Further information on the PCB analysis requirements is contained in the laboratory Request for Qualifications and Quote, which is included in Appendix B. SVL Analytical, Inc. will conduct the laboratory analyses for all other parameters. The laboratories will analyze field and laboratory QA/QC samples using the procedures in the SAPs and the analytical method to assess data quality.

The Laboratory Technical Directors and QA Managers will ensure that all laboratory personnel have received training and have proven proficiency in their designated analytical procedures. Laboratory personnel will be provided copies of the appropriate Standard Analytical Procedures, which will be available at all times.

1.6 Documents and Records (A.9)

The approved QAPP and any approved updates will be distributed to the list of project personnel identified in the Distribution List at the beginning of this document. These personnel are responsible for distributing copies of the QAPP to relevant personnel within their organization.

The Project Manager is responsible for initiating project files and for overseeing maintenance of the files during the course of the project. All project files will be properly identified by client, project name, project number, file description, and file number for all appropriate correspondence, memoranda, calculations, technical work products, and other project-related data. In addition, a quality assurance file will be maintained by the Project QAO containing all QA/QC related information. A back up of all computer files containing important project information will also be maintained.

Documents to be generated by field activities include staff notes, field log sheets, equipment logs, field audit reports, sampling completion reports and chain of custody forms. Examples are included in the SAP. Documents to be generated by laboratory activities include QA/QC reports, laboratory bench sheets, laboratory results, and laboratory audit reports. These documents will be included in project reports.

At the conclusion of the project, all relevant information from the project files and electronic files will be turned over to SRRTTF-ACE who will manage the information on behalf of the SRRTTF. It is anticipated that the information will be uploaded into the Department of Ecology's Environmental Information Management (EIM) system and be available for public access.

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2. DATA GENERATION AND ACQUISITION (GROUP B)

This section of the QAPP addresses QA/QC elements related to the monitoring activities. The monitoring program QAPP was developed based on U.S. EPA requirements (EPA, 2001).

2.1 Sampling Process Design (B.1)

As described in the previous section, a Synoptic Survey will be conducted during the summer low flow period at numerous stations, and Seasonally Integrated Sampling will be conducted at the Lake Coeur d'Alene outlet during spring high flow, summer low flow and winter moderate flow. The sampling process design is discussed in the SAP.

2.2 Sampling Methods (B.2)

Standard operating procedures (SOPs) will be employed to provide consistency and reproducibility to the sampling methods used by field personnel. The SOPs are contained in the Sampling and Analysis Plan. The following sections present or reference the detailed methods for performing sampling activities including related support procedures for equipment cleaning, field measurements, and calibration and maintenance of field instruments. Sample custody procedures are presented in the Sample Handling and Custody Section of this QAPP. For all sampling related procedures, personnel will use personal protective equipment as required by the Health and Safety Plan (HASP), which will be prepared by Gravity Environmental.

2.2.1 Surface Water Sample Collection

Surface water sampling will be conducted as specified in the SAP, to minimize sample contamination. All in stream water quality samples will be collected by wading into the main channel flow. The best effort will be made without jeopardizing the safety of the sampling crew. If wading is not possible a boat will be used to access the main channel flow. The sample bottles will be filled by direct immersion into the sample bottle.

At NPDES permitted discharge locations the point of discharge is determined to be the location identified in the discharger's NPDES permit or as determined in the field by the sampling team and approved by the project manager. If an alternate sample collection method is required at discharger locations, such as using a sampling pole with a clean sample bottle attached, it will be documented on the field log sheet. In this situation a transfer blank will be required.

If a QC sample is to be collected at a given location, all containers designated for a particular analysis for both the sample and QC sample will be filled sequentially before containers for another analysis are filled. For field replicate samples, the sample and replicate will be filled one after the other. Once the samples have been collected they will be kept chilled and processed for transfer to the laboratory.

Care will also be taken to prevent the spread of non-native noxious weeds, pathogens and exotic flora and fauna among water bodies, by following the procedures specified in the SAP.

2.2.2 Field Water Quality Measurements and Monitoring

Instantaneous water quality measurements (temperature, conductivity, pH, dissolved oxygen and turbidity) using field instruments will be collected as specified in the SAP. Field measurements will be taken at each location prior to sample collection for laboratory analysis. All field instruments will be calibrated at the beginning of each day of sampling. Field instrument calibration and sample measurement data will be recorded on the field log sheet.

2.2.3 Field Variances

As conditions in the field vary, it may become necessary to implement minor modifications to the sampling procedures and protocols described in the QAPP. If this becomes necessary, Gravity Environmental will notify the Field Manager of the situation, who will discuss with the Project Manager. Gravity Environmental will obtain verbal approval prior to implementing any changes. The approval will be recorded in the field log sheet and included in the sampling completion report.

2.3 Sample Handling and Custody (B.3)

Sample handling will be the responsibility of Gravity Environmental and will be performed using methods as specified in the SAP, so that representative samples are collected, stored, and submitted to the laboratory for analysis. Sample containers, volumes, preservatives and holding times are summarized in Table 9. Proper sample handling and custody procedures will be employed as discussed in the following subsections of this QAPP.

Table 9. Guidelines for sample container preparation and preservation

Parameter	Container	Volume	Preservative	Holding Time
PCB	Amber glass	2.36 L	4° C	1 year
TSS	Polypropylene	1 L	4° C	7 days
TDS	Polypropylene	500 ml	4° C	7 days
TOC	Polypropylene	60 ml	4° C, H_2SO_4	28 days
DOC	Polypropylene	60 ml	4° C, H_2SO_4	28 days

2.3.1 Field Sample Custody

The objective of field sample custody is to assure that samples are traceable and are not tampered with between sample collection and receipt by the analytical laboratory. A person will have custody of a sample when:

- The person is one of the authorized personnel;
- The sample is in their physical possession;
- The sample is in their view after being in their possession;
- The sample is in their personal possession and secured to prevent tampering; and
- The sample is in a restricted area accessible only to authorized personnel.

Field custody documentation will consist of both field log sheet and chain of custody forms.

Chain-of-Custody Forms. Completed chain-of-custody forms will be required for all samples to be analyzed. Chain-of-custody forms will be filled-out by the field sampling crew during the sample collection events. The chain-of-custody form will contain the sample information:

- Unique identification number;
- Sample date and time;
- Sample description;
- Sample type;
- Sample preservation (if any);
- Analyses required.

The original chain-of-custody form will accompany the samples to the laboratory. Copies of the chain-of-custody form will be made prior to shipment for separate field documentation. A chain-of-custody form is included in the SAP. The chain-of-custody forms will remain with the samples at all times. The samples and signed chain-of-custody form will remain in the possession of the sampling crew until the samples are delivered to the express carrier (e.g., Federal Express or United Parcel Service) or to the laboratory.

Sample Packing and Shipping Requirements. Sample packaging and shipping procedures are designed to ensure that the samples and the chain-of-custody forms will arrive at the laboratory intact and together. Samples will be properly packaged for shipment according to the procedures presented in the SAP and submitted to the appropriate laboratory for analysis. Shipping containers will be secured with strapping tape and custody seals for shipment to the laboratory. The preferred procedure includes use of a custody seal attached to the front right and back left of the cooler. The custody seals are covered with clear plastic tape. The cooler is strapped shut with strapping tape in at least two locations.

All shipments will be accompanied by the chain-of-custody form identifying the contents. It is preferred that a separate chain-of-custody form be completed for and placed in each shipping container. The original form will accompany the shipment and copies will be retained by the sampler for the sampling records.

If sample containers are sent by common carrier (i.e., by Federal Express or United Parcel Service), the carrier need not sign the chain-of-custody form. In such cases, the chain-of-custody form should be sealed inside the sample container. The bill of lading (i.e., Federal Express label) serves as the custody documentation for the shipment so long as the container remains unopened until arrival at the laboratory. Copies of the bill of lading should be retained as part of the permanent documentation of the project.

2.3.2 Laboratory Sample Custody

Each laboratory will manage sample custody in accordance with the laboratory's procedures. Sample custody will also be consistent with the guidelines set forth in this section of the QAPP.

Each laboratory must have written standard operating procedures (SOPs) for sample custody including:

- Sample receipt and maintenance of custody;
- Sample storage; and
- Sample tracking.

In addition, each laboratory shall have written SOPs for laboratory safety, cleaning of analytical glass ware, and traceability of standards used in sample analysis QA/QC.

An SOP is defined as a written narrative step-wise description of laboratory operating procedures including examples of laboratory documentation. The SOPs must accurately describe the actual procedures used in the laboratory, and copies of the written SOPs shall be available to the appropriate laboratory personnel. These procedures are necessary to ensure that analytical data produced are acceptable for use. The laboratory SOPs shall provide mechanisms and documentation to meet the specification of the following sections.

Sample Receipt and Maintenance of Custody. Each laboratory shall have a designated sample custodian responsible for receipt of samples and have written SOPs describing duties and responsibilities.

Each laboratory shall have written SOPs for receiving and logging in of the samples. The procedures shall include but not be limited to documenting the following information:

- Presence or absence of chain-of-custody forms;
- Presence or absence of bills of lading;
- Presence or absence of custody seals on shipping and/or sample containers and their conditions;

- Presence or absence of sample labels;
- Sample label identification numbers if not recorded on the chain-of-custody record(s) or packing list(s);
- Condition of the shipping container;
- Condition of the sample bottles;
- Verification of agreement or non-agreement of information on receiving documents; and
- Resolution of problems or discrepancies.

Sample Storage. After samples are received, they are placed in secure storage where they are maintained at 4 degrees Celsius.

Each laboratory shall have written SOPs for maintenance of the security of samples after log-in and shall demonstrate security of the sample storage and laboratory areas. The SOPs shall specifically include descriptions of all storage areas for samples in the laboratory, and steps taken to prevent sample contamination. Only authorized personnel should have access or keys to secure storage areas.

Sample Tracking. Each laboratory shall have written SOPs for tracking the work performed on any particular sample. Documentation of sample receipt, sample storage, sample transfers, sample preparations, sample analyses, instrument calibration and other QA/QC activities shall be performed.

2.4 Analytical Methods (B.4)

The following section details the aspects of the analytical requirements, ensuring that appropriate analytical methods are employed. Tables 5 and 6 summarize the analytical methods to be used by the laboratory. Appendix D contains the relevant laboratory Standard Analytical Procedures for the project.

2.4.1 Parameter Specific Information

Table 9 displays the required container type, sample volume, preservation, and hold time for the study parameters according to the previously referenced methods. AXYS Analytical Services and SVL Analytical, Inc. will provide sample containers from a commercial supplier. All sample containers will be new and pre-cleaned by the supplier. In addition, the contract laboratories will provide sample labels for each bottle. The detection limits, expected concentrations, and analytical methods are included in Table 10 (Ecology, 2014).

Table 10. Parameters, Detection Limits, Expected Concentrations and Analytical Methods

Parameter	Detection Limit	Expected Concentrations	Analytical Method	Laboratory
PCB (pg/L)	1-20	10-10,000 total	EPA 1668C	AXYS Analytical Services
TSS (mg/L)	1	1-80	EPA 160.2	SVL Analytical, Inc.
DSS (mg/L)	1	1-80	EPA 160.1	SVL Analytical, Inc.
TOC (mg/L)	1	1-2	EPA 415.1	SVL Analytical, Inc.
DOC (mg/L)	1	1-2	EPA 415.3	SVL Analytical, Inc.

2.4.2 Laboratory Chain of Custody Procedures

Use of the chain-of-custody form will terminate when laboratory personnel receive the samples and sign the form. The laboratory custodian will open the sample coolers and carefully check the contents for evidence of leakage and to verify that samples were kept on ice. The laboratory will then verify that all information on the sample container label is correct and consistent with the chain-of-custody form. Any discrepancy between the sample bottle and the chain-of-custody form, any leaking sample containers, or any other abnormal situation will be reported to the Laboratory Operations/Technical Director and Laboratory Project Manager. The Laboratory Project Manager will inform the Project Manager of any such problem, and corrective actions will be discussed and implemented.

2.4.3 Analytical Records

The analytical data results, intra-laboratory QA/QC results, along with a case narrative will be submitted by the contract laboratory to the Project Manager in both an electronic format and also in hard copy within a specified time frame from the completion of each sampling event (synoptic and seasonal events) (standard turn around time 60 days). Also, at this time, the data sheets generated during the processing of these samples that include sample identification information will be submitted to the Project Manager for every sample analyzed. Copies of all bench sheets will be kept on file by the laboratory and made available for review upon request.

2.5 Quality Control (B.5)

Analytical quality control will be performed in accordance with the specified analytical methods and as discussed under the Quality Objectives and Criteria Section of this QAPP.

2.5.1 Field Sampling Quality Control

Field sampling QC consists of collecting field QC samples to help evaluate conditions resulting from field activities. Field QC is intended to support a number of data quality goals:

- Combined contamination from field sampling through sample receipt at the laboratory (to assess potential contamination from ambient conditions, sample containers, sample transport, and laboratory analysis) – assessed using trip blanks/transfer blanks.
- Combined sampling and analysis technique variability, as well as sample heterogeneity – assessed using field replicates.

Trip Blanks – Trip blanks will be used to evaluate whether contaminants have been introduced into the samples due to exposure to ambient conditions or from the sample containers themselves. A trip blank is a controlled water sample, with minimal concentrations of contaminants of concern, which is produced by the laboratory. The trip blank accompanies the sampling equipment into the field and is stored with the analytical samples. If transfer blanks are required, they will be obtained by pouring deionized water into the sample container in the field, preserved and shipped to the laboratory with the field samples. Trip/transfer blanks will be collected at a frequency of 10% or one blank per sampling round.

Trip/transfer blanks, as described above, will be preserved, packaged, and sealed in the same manner described for the surface water samples. A separate sample number and station number will be assigned to each blank. The samples will be submitted as "blind" samples to the laboratory for analysis. If target analytes are found in the blanks above the criteria, sampling and handling procedures will be reevaluated and corrective actions taken. These may consist of, but are not limited to, obtaining sampling containers from

new sources, training of personnel, discussions with the laboratory, invalidation of results, greater attention to detail during the next sampling event, or other procedures considered appropriate.

Field Replicate Samples – Field replicate samples will be collected to evaluate the precision of sample collection through analysis. Field replicates will be collected at designated sample locations by filling two distinct sample containers for each analysis. Field replicate samples will be preserved, packaged, and sealed in the same manner described for the surface water samples. A separate sample number and station number will be assigned to each replicate. The samples will be submitted as "blind" samples to the laboratory for analysis.

Field replicates will be collected for each analytical parameter at a frequency of 10% or one field replicate per sampling round, whichever is less. The replicate samples will be collected at random locations for each sampling event. If the acceptance criteria are exceeded, field sampling and handling procedures will be evaluated, and problems corrected through greater attention to detail, additional training, revised sampling techniques, or whatever appears to be appropriate to correct the problem.

2.5.2 Field Measurements Quality Control

Quality control requirements for field measurements are provided in Table 8.

Field instrumentation will be calibrated according to the manufacturer's requirements and will be calibrated daily. If a field instrument cannot be calibrated it should not be used.

2.5.3 Laboratory Analysis Quality Control

Laboratory QC is the responsibility of the laboratory personnel and QA/QC departments of AXYS Analytical Services and SVL Analytical, Inc. The laboratory's QA Manual details the QA/QC procedures it follows. The following elements are part of standard laboratory quality control practices:

- Analysis of method blanks
- Analysis of laboratory control samples
- Instrument calibration (including initial calibration, calibration blanks, and calibration verification)
- Analysis of matrix spikes (TOC/DOC)
- Analysis of duplicates

The data quality objectives for AXYS Analytical Services and SVL Analytical, Inc. (including frequency, QC acceptance limits, and corrective actions if the acceptance limits are exceeded) are detailed in this QAPP. Any excursions from these objectives must be documented by the laboratory and reported to the Project Manager/Project QAO.

Method Blanks – A method blank is an analyte-free matrix, analyzed as a normal sample by the laboratory using normal sample preparation and analytical procedures. A method blank is used for monitoring and documenting bias due to background contamination in the analytical environment. Method blanks can be used to estimate within- batch variability of the measurement system. Method blanks will be analyzed at a frequency of one per sample batch (or group of up to 20 samples analyzed in sequence using the same method). Corrective actions associated with exceeding acceptable method blank concentrations include isolating the source of contamination and re-digesting and/or re-analyzing the associated samples. Blank contamination will be noted in the laboratory reports, but sample results will not be corrected for blank contamination. Corrective actions will be documented in the laboratory report's narrative statement. Samples with results less than three times the level of the associated blank will be qualified by the laboratory with a B qualifier, as indicated in the laboratory Request for Qualifications and Quote and in Table 5. This

qualifier will be used to indicate samples at low concentrations where the blank contamination causes a significant bias.

Laboratory Control Samples – Laboratory control samples (LCS) are laboratory-generated samples analyzed as a normal sample by the laboratory using normal sample preparation and analytical procedures. An LCS is used to monitor the day-to-day performance (accuracy) of routine analytical methods. An LCS is an aliquot of clean water spiked with analytes of known concentrations corresponding to the analytical method. The LCS is used to verify that the laboratory can perform the analysis on a clean matrix within QC acceptance limits. Results are expressed as percent recovery of the known amount of the spiked analytical parameter.

One LCS is analyzed per sample batch. Acceptance criteria (control limits) for the LCS are defined by the laboratory and summarized in Tables 5 and 6. In general, the LCS acceptance criteria recovery range is 80 to 120 percent of the known amount of the spiked analytical parameter. Corrective action, consisting of a rerunning of all samples in the affected batch, will be performed if LCS recoveries fall outside of control limits. Such problems will be documented in the laboratory report's narrative statement.

Matrix Spikes – Matrix spikes (MS) are prepared by adding a known amount of the analyte of interest to a sample. MS are used as a similar function as the LCS, except that the sample matrix is a real time sample rather than a clean matrix. Results are expressed as percent recovery of the known amount of the spiked analytical parameter. Matrix spikes are used to verify that the laboratory can determine if the matrix is causing either a positive or negative influence on sample results.

One matrix spike is analyzed per sample batch or every 20 samples. Acceptance criteria for the MS are defined by the laboratory and summarized in Table 6. In general, the MS acceptance criteria recovery range is 80 to 120 percent of the known amount of the spiked analytical parameter. Generally, no corrective action is taken for matrix spike results exceeding the control limits, as long as the LCS recoveries are acceptable.

The PCB analysis does not include matrix spikes.

Laboratory Duplicates – A laboratory duplicate is a laboratory-generated split sample used to document the precision of the analytical method. Results are expressed as relative percent difference between the laboratory duplicate pair.

One laboratory duplicate will be run for each laboratory batch or every 20 samples, whichever is more frequent. Acceptance criteria for laboratory duplicates are specified in the laboratory QA Manual and SAPs and are summarized in Tables 5 and 6. If laboratory duplicates exceed criteria, the corrective action will be to repeat the analyses. If results remain unacceptable, the batch analyses will be rerun.

PCB: Labeled Compound, Cleanup, Internal and Injection Standards

Similar to surrogate spikes, these standards are 13C isotopes which are spiked into all field and laboratory samples prior to different points in the analytical process (extraction, cleanup and injection). 13C congener isotopes are added prior to extraction. These homologs are used for the purpose of quantifying target compounds. Cleanup 13C homologs are added prior to cleanup of samples for the purpose of monitoring their recoveries through the cleanup processes, for internal diagnostics only. The third 13C homologs (recovery standards) are added just prior to sample injection to monitor the recoveries of the pre-extraction homologs to insure they meet method criteria. Difficulties with the analytical method or sample matrix affect the recovery of these standards. If method criteria are not met the laboratory should take appropriate corrective action including re-extraction if necessary.

2.6 Instrument/Equipment Testing, Inspection, and Maintenance (B.6)

Field analytical equipment that may be used in this project includes instruments for measuring conductivity, pH, temperature, dissolved oxygen and turbidity. Testing, inspection and maintenance will be conducted in accordance with manufacturer instructions. Equipment logs will be maintained by Gravity Environmental, then submitted to and kept by the Field Manager. The log will document any maintenance and service of the equipment. A log entry will include the following information:

- Name of person maintaining the instrument/equipment,
- Date and description of the maintenance procedure,
- Date and description of any instrument/equipment problems,
- Date and description of action to correct problems,
- List of follow-up activities after maintenance, and
- Date the next maintenance will be needed.

Calibration frequency and preventative maintenance procedures are provided in SAP.

Laboratory instrumentation and equipment will follow manufacturer instructions and accepted procedures associated with the selected analytical methods, the laboratory's Standard Analytical Procedures.

2.7 Instrument/Equipment Calibration and Frequency (B.7)

Field analytical equipment that may be used in this project includes instruments for measuring conductivity, pH, temperature, dissolved oxygen and turbidity. Gravity Environmental will use the equipment manufacturer's calibration procedures for the equipment will follow manufacturer instructions. To maintain field precision and accuracy, the water quality instruments will be calibrated to known standards. Field analysis and operation procedures, including calibration and sample analysis, are provided in the SAP.

Laboratory instrument calibration will follow manufacturer instructions and accepted procedures associated with the selected analytical methods, each laboratory's Standard Analytical Procedures.

2.8 Inspection Acceptance of Supplies and Consumables (B.8)

All supplies and consumables for field and laboratory activities will be inspected for compliance with the acceptance criteria by the identified responsible party prior to use. Supplies or consumables not meeting the acceptance criteria upon inspection will not be used. Any equipment determined to be in an unacceptable condition will be replaced. Supplies and consumables will be stored in accordance with identified storage requirements.

2.9 Non-direct Measurements (B.9)

Non-direct measurements will not be used in implementation of the monitoring program.

2.10 Data Management (B.10)

Data generated through field and laboratory activities will be used for the mass balance assessment described in previous sections of this QAPP. The Project Manager will be responsible for organization and oversight of data generation, distribution, processing and storage so that the data will be documented, accessible and secure for the foreseeable time period of its use. The Laboratory Operations/Technical Director has the same responsibility for laboratory data and information.

Instrumentation used to generate, process and store data will be configured, maintained and operated in accordance with manufacturer recommendations and accepted industry standards. Generated raw data will be stored in formats compatible with the method or instrument of generation. Processed data will be stored in text files, Microsoft Excel spreadsheets or Access databases compatible with version 2007. Electronic data will be stored in project directories on a LimnoTech computer network server that is compatible with this software and that is backed up regularly. Data reported in paper format will be stored in the project files. The data will also be provided to the SRRTTF-ACE who is responsible for sharing the data with the SRRTTF. Following all data validation and verification procedures the data will be uploaded to the Washington State Department of Ecology EIM.

2.10.1 Field Data and Information Management

Field data reporting shall be conducted by Gravity Environmental principally through the transmission of field log sheets containing tabulated results of all measurements taken in the field, and documentation of all field calibration activities. Field log sheets and equipment logs will be turned over to the Field Manager following each monitored event. Following review by the Field Manager, the field log sheets will be transmitted to the Project Manager for review. Examples of standard field forms are provided in the SAP.

Field Logs

Field log sheets serve as a daily record of events, observations, and measurements during field activities. All information pertinent to sampling activities will be recorded in the log books. The logbooks may be bound with the pages sequentially numbered or include separate sheets for field notes and method specific data logs. Personal computers may also be used to record field data. Field log sheets will be maintained by field staff at all times documenting activities and conditions. Field log sheets will be turned in by field staff following each monitored event. Copies of all field log sheets will be made following each monitored event and maintained in the QA/QC project file.

Entries in the field log sheet will include:

- Name(s) of field crew
- Name(s) of site visitors
- Date and time of site entry
- Location of sampling activity
- Description of sample location
- Date and time of collection
- Sample identification numbers
- Sampling method
- Preservatives used
- Field measurements (pH, etc.)
- Field observations

Equipment Logs

Gravity Environmental will maintain equipment logs for all field equipment. As installation, calibration and maintenance functions are completed on equipment, equipment logs will be maintained and included in the QA/QC project file.

Field On-Site Measurements

Field measurement information recorded in the field log sheet will be compiled and the information transferred into electronic format by office staff. The Field Manager will review the source document and the electronic version to verify the accurate transfer of information. Following this review, electronic field data will be transferred to the Project Manager. The original field log sheets will be maintained in the Project QA/QC project file.

Labels

Gravity Environmental will label samples in a clear and precise way for proper identification in the field and for tracking in the laboratory. The samples will have pre-assigned, identifiable and unique numbers. At a minimum, the sample labels will contain the following information.

- Sampling location or name,
- Unique sample number,
- Sample description (e.g. grab, composite),
- Date and time of collection,
- Initials/signature of sampler,
- Analytical parameters, and
- Method of preservation.

Field Quality Control Sample Records

Field QC samples (replicates and blanks) will be labeled as such in the field log sheet. They will be given unique sample identification numbers and will be submitted "blind" to the laboratory. The frequency of the QC sample collection will also be recorded in the field log sheet.

2.10.2 Laboratory Data and Information Management

The reporting of laboratory data will begin after the Laboratory Operations/Technical Director or designee has concluded the verification review. The contract laboratory will prepare and submit full analytical and QC reports to the LimnoTech Project Manager that will include the following, as appropriate.

- Case narrative, including a statement of the conditions that samples were received, description of any deviation from standard procedures, explanation of any data qualifiers used, and identification of any problems encountered during analysis.
- Computer generated report form containing all sample results
    - a hard copy version of the report
    - an electronic version of the report on CD
- Hard copy QC summary report for each parameter by batch including the results of replicates, matrix spikes, matrix spike duplicates, controls, dilution blanks, method blanks, verification tests, etc.
- Copies of all chain-of-custody forms.
- Copies of all laboratory bench sheets will be kept on file and made available for review, for a minimum of seven years.

Following receipt of laboratory data by the LimnoTech Project Manager, the data will be reviewed and validated by the Project Quality Assurance Officer (QAO) following the procedures outlined in Section 4.

2.10.3 Electronic Data Management

All data collected during the course of the study will be entered into a database by LimnoTech for use in the mass balance assessment. LimnoTech will manage and maintain the database.

All electronic files will be backed up on a regular basis. At the conclusion of the project all relevant information, project files and electronic data will be turned over to the SRRTTF –ACE, who will share with the SRRTTF. Validated and quality assured data will be made available for upload to the Washington State EIM.

3. ASSESSMENT AND OVERSIGHT (GROUP C)

The Group C Assessment and Oversight elements are addressed in this section.

3.1 Assessment and Response Actions (C.1)

Internal quality control checks are performed to ensure that the field and laboratory generated measurements meet the project quality assurance objectives. In addition, the quality control checks are intended to identify any need for corrective action.

3.1.1 Field Measurements

Field quality control checks will consist of QA/QC samples that will be collected or prepared by the field crews to be submitted for laboratory analysis. These samples will consist of replicates and trip blanks. Replicates will be collected at a 10% frequency (1 in 10 samples collected) and blanks will be submitted at a frequency of 10% (1 in 10 samples collected), or one replicate and blank per sampling round. The Field Manager will ensure that the correct number of QA/QC samples are collected during each event (Synoptic Survey or Seasonally Integrated Sampling event).

Quality control checks will be conducted in advance of using multi-parameter meters. The checks will involve the review of the previous calibration sheets. Any problems with sensors will be addressed immediately. Gravity Environmental will record the result of each review on the instrument's calibration sheet. At the conclusion of each event (Synoptic Survey or Seasonally Integrated Sampling event), all calibration sheets will be reviewed by the Field Manager to assess the adequacy of the quality control checks and to review the instruments' performance to identify any problems.

The Field Manager will inform the Project Manager in writing of any quality control check issues and to discuss corrective actions. All quality control documents will be contained in a file for each monitored event.

3.1.2 Laboratory Measurements

Each laboratory will perform quality control checks on all sample analyses, as specified in the laboratory Request for Qualifications and Quote (Appendix B). These will include replicates, matrix spikes, matrix spike duplicates, control samples, and method blanks as appropriate. Quality control procedures for analytical services will be conducted by the laboratories in accordance with their standard analytical procedures and the individual method requirements referenced by U.S. EPA or Standard Methods. The acceptable control limits are discussed in the laboratory Request for Qualifications and Quote and provided in Section 1.4. Each Laboratory Operations/Technical Director will inform the Laboratory QA Manager immediately of any quality control check issues and to discuss corrective actions.

At the conclusion of each event, the laboratories will provide a summary of all QA/QC results. The QA/QC summary will be reviewed by the Laboratory Technical Director and the QA Manager to assess the adequacy of the quality control checks and to identify any potential problems. Table 11 summarizes the laboratory quality control check frequencies.

Table 11. Laboratory quality control check frequencies

Parameter	Batch Size	QC Check	Frequency
TSS	20 Samples	Control	1 each per analytical batch
		Replicate	
		Method Blank	
TDS	20 Samples	Control	1 each per analytical batch
		Replicate	
		Method Blank	
TOC	20 Samples	Control	1 each per analytical batch
		MS/MSD	
		Method Blank	
DOC	20 Samples	Control	1 each per analytical batch
		MS/MSD	
		Method Blank	
PCB congeners	20 Samples	Control	1 each per analytical batch
		Replicate	
		Method Blank	

3.1.3 System Audits and Technical Reviews

All project team members are committed to providing quality services. The primary responsibility for the quality of work products rests with the individuals doing the work and with their immediate supervisors.

For certain project components an independent technical reviewer will audit or review the work products. LimnoTech Project Manager will coordinate the independent review process. The independent technical reviewer will perform a critical, written evaluation of the work product, and the independent technical audit or review will be incorporated in the project record.

The Project Manager is responsible for identifying the work products to be audited/reviewed and the scope of the audit/review, for scheduling independent technical audits/reviews, for assigning competent, qualified independent technical auditors/reviewers, and for making sure that appropriate follow-up actions are taken to correct reported deficiencies.

Field System Audits

Field system audits will be completed to ensure that the actual field procedures conform to those documented in the SAP and associated SOPs. The Project Manager will ensure that field system audits are performed. The audit will include a check of all field records and a review of all activities to document if procedures were conducted in compliance with the specified documentation.

Laboratory System Audits

Independent auditors will complete a lab audit of the contract laboratory at some point during the monitoring program. These auditors will be designated by the Project Manager. The audit will be scheduled if possible during analysis of project samples. The audits will include an assessment of all quality system documents as well as the laboratory Standard Analytical Procedures. In addition, the audit will include a laboratory site visit and discussions with the Laboratory Operations/Technical Director and Laboratory QA Manager. Also, spot checks will be performed to interview individual analysts with regard to methods used, knowledge of quality systems, training, and competency.

3.1.4 Corrective Action

Corrective actions will be implemented as required to rectify problems identified during the course of normal field and laboratory operations. Possible problems requiring corrective action include:

- Equipment malfunctions;
- Analytical methodology errors; or
- Non-compliance with quality control systems.

Equipment and analytical problems that require corrective action may occur during sampling and sample handling, sample preparation, and laboratory analysis.

For non-compliance problems, steps for corrective action will be developed and implemented at the time the problem is identified. The individual who identifies the problem is responsible for immediately notifying the Project Manager and the Project QAO.

Any non-conformance with the established quality control procedures outlined in the QAPP will be identified and corrected. The Project Manager will ensure that a Corrective Action Memorandum is issued for each non-conformance condition. All non-conformance memoranda will be discussed in the final report submitted to the SRRTTF-ACE.

Field Measurements and Sample Collection

Project staff will be responsible for reporting any suspected QA non-conformance or deficiencies to the Field Manager. The Field Manager will be responsible for assessing the suspected problems in consultation with the Project Manager to review the sampling protocols and provide additional training if necessary. If it is determined that the situation warrants a corrective action, then a Corrective Action Memorandum will be issued by the Field Manager.

The Field Manager will be responsible for ensuring that the corrective action for non-conformance takes place by:

- Evaluating all reported incidences of non-conformance;
- Controlling additional work on nonconforming items;
- Determining what corrective action is needed;
- Maintaining a log of non-conformance issues;
- Reviewing responses to corrective action memoranda;
- Ensuring that copies of corrective action memoranda and responses are included in the project files.

No additional work will be performed until appropriate corrective action has been implemented and documented in response to the corrective action memoranda.

Laboratory Analyses

Corrective actions are required whenever laboratory conditions, instrument malfunction or personnel situations have led or could potentially lead to errors in the analytical data. The corrective action taken will be dependent on the analysis and the event.

Laboratory personnel are alerted that corrective actions may be necessary if:

- QC data are outside the acceptable range for precision and accuracy as identified in Section 1.4;
- Blanks contain target analyses above acceptable levels;
- Undesirable trends are detected in spike recoveries or RPD between duplicates;

- Excessive interference is noted; or
- Deficiencies are detected by the Independent Auditor during laboratory system audits as described in Section 3.1.3.

Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, spike and calibration mixes, and instrument sensitivity, etc.

Corrective action taken within each laboratory is the responsibility of the Laboratory Operations/Technical Director. When a problem occurs, the Laboratory Technical Director informs the Project Manager about the problem and the steps taken to resolve it. Once the problem is resolved, full documentation of the corrective action procedure will be submitted to the Project Manager.

All non-conformance memoranda initiated by the contract laboratory will be discussed in the case narrative or included in the laboratory reports. The Project Manager will follow-up on all corrective actions that are taken to ensure that the memoranda are accurate.

3.2 Reports to Management (C.2)

The LimnoTech Project Manager and Laboratory Operations/Technical Directors will provide independent reporting to the SRRTTF-ACE and the SRRTTF on an as-needed basis. This communication is facilitated through the use of electronic mail, which provides ready access. In addition, the Project Manager will provide written reports to the SRRTTF-ACE on quality assurance issues as described in the QAPP. SRRTTF-ACE will ensure that the SRRTTF is informed of any quality assurance issues that could affect the ability to use the data for its intended purposes.

Field and laboratory system audits will be performed as described in Section 3.1.3 and the results will be provided to the SRRTTF-ACE who will ensure that the SRRTTF has access to the data. The results of all audits will be summarized in written reports, with copies retained in the Project Files. The audit reports will be completed for field and laboratory system audits according to the general outline described below.

All audit reports will include the following sections:

- Introduction – provides background of the project, laboratory, or program element, description of personnel and affiliation of all staff involved, the name of the auditor, the time and date of the audit, and a description of the activities audited.
- Audit Findings – describes the results of the audit including a deficiency report identifying all instances where the procedures in the SAP, QAPP, or laboratory QAP were not followed.
- Conclusions – summarizes the results of the audit and includes recommended actions to address any noted deficiencies.

Final Project Report

At the conclusion of the project a final report will be prepared, as stated in Section 1.3.5. The final report will contain the following elements:

- Information on the sampling locations, including geographic coordinates and maps
- Descriptions of field and laboratory methods
- Tables presenting all the data
- Discussion of project data quality
- Summary of significant findings

4. DATA VALIDATION AND USABILITY (GROUP D)

The Group D Data Validation and Usability elements are addressed in this section. The purpose of these elements is to determine if the data meet the project's Data Quality Objectives (validation) and to evaluate the data against the method, procedural and/or contractual requirements (verification). Data validation, verification, and usability assessment will be conducted as outlined in this QAPP.

The data generated from the sampling program will be subjected to a multi-tiered review process described below. This process includes:

- A review of the data at the bench and field levels;
- A secondary review of field records by the Field Manager and analytical results within the laboratory by the Laboratory QA Manager to verify the data against method and SAP requirements;
- A screening level review of the verified data by the LimnoTech QAO for reasonableness and to identify obvious data anomalies;
- A validation by an objective third party; and finally,
- An assessment of the data by project team members for its usability in the project as described in Section 4.1 of this QAPP.

4.1 Data Review, Verification and Validation (D.1)

All environmental measurement data collected by project staff will be subjected to quality control checks before being utilized in the interpretive reporting. A data generation system that incorporates reviews at several steps in the process is designed to protect the integrity of the data and reduce the number of data that do not meet the Data Quality Objectives or the project goals. This section describes the requirements of each review step that will be used in this project.

4.1.1 Data Verification Requirements

The definition of data verification, as described in the EPA's "Guidance on Environmental Data Verification and Data Validation" (EPA QA/G-8) is:

"…the process of evaluating the completeness, correctness, and conformance/compliance of a specific dataset against the method, procedural or contractual requirements."

Data verification will occur at the field and laboratory level as described in this section.

Field Activities Data Verification

The Field Manager will be responsible for ensuring that Gravity Environmental collects and handles samples in accordance with the procedures specified in the SAP. Sample collection verification will include confirming that the samples were collected with the proper equipment at the appropriate locations with the appropriate frequency. Sample handling verification will include confirming that the samples were stored in the appropriate containers (see Table 9) with the correct preservative, that the samples were stored at the proper temperature during transport from the field to the laboratory, and that all of the appropriate information is logged on the chain-of-custody records.

Lab Activities Data Verification

The Laboratory QA Manager will be responsible for verification of laboratory-generated data, although the laboratory Standard Analytical Procedures for each method require some components of the verification to

also be conducted at the bench level. Laboratory verification will include assessing that the procedures used to generate the data are consistent with the method requirements as specified in the laboratory's SOPs and that the QA/QC requirements for each method are met. Examples of method requirements include verifying the calibration and data reduction procedures. However, these requirements vary by analyte and are presented in more detail in the laboratory Standard Analytical Procedure. Once the data have been verified and approved by the laboratory, they will be released to SRRTTF-ACE.

4.1.2 Data Review Requirements

The Field Manager will perform data reviews that will consist of screening the field data sheets and laboratory data sheets according to established criteria listed in this section. If the established screening criteria are violated, an additional review of the quality control checks and any relevant laboratory bench sheets will be conducted. The investigation of the issue will be documented and the data will be discarded or flagged appropriately, identifying the limitations of the data. This is an additional step of review that is designed to provide an early assessment of the data's use in meeting the project goals by evaluating it within the context of well-understood constituent relationships.

Field Data Sheet Reviews

The following criteria will be used to screen the physical parameter measurements recorded by the field crews:

1. Temperature readings – do values seem reasonable
2. pH readings – do values seem reasonable
3. Dissolved oxygen readings – do concentrations compare to percent saturation
4. Conductivity readings – do concentrations seem reasonable

The values for these parameters measured by Ecology in 2012 and 2013 (Ecology, 2014) (Table 12) provide information on values expected to be measured in 2014 and 2015.

Table 12. In-Situ Parameter Measurements in 2012 and 2013.

Location	Stateline		Upriver Dam		Above Latah		Ninemile		Chamokane	
Date	10/24/12	10/25/12	10/24/12	10/25/12	10/24/12	10/25/12	10/24/12	10/25/12	10/24/12	10/25/12
Time	0950	0930	1701	1533	1805	1745	1500	1240	1135	1115
Sample No.	1210040-01		1210040-02		1210040-03		1210040-04		1210040-05	
Temperature (Deg. C)	10.46	10.36	9.84	10.04	9.84	9.68	9.46	9.83	12.65	12.58
Conductivity (uS/cm)	44.5	49.0	122.3	133.4	148.2	161.8	178.5	196.2	205	222
pH	7.50	7.47	7.90	7.87	8.18	8.24	7.83	8.00	8.14	8.18
Dissolved Oxygen (mg/L)	10.05	9.8	9.58	9.57	10.74	10.92	10.58	10.25	9.25	9.55
Dissolved Oxygen (% Sat.)	94.8	92.1	88.9	89.3	99.8	101.1	97.4	95.2	91.8	94.5
Date	5/23/13	5/24/13	5/23/13	5/24/13	5/23/13	5/24/13	5/23/13	5/24/13	5/23/13	5/24/13
Time	0935	0855	1031	0939	1145	1040	1323	1146	1440	1252
Sample No.	1305006-01		1305006-02		1305006-03		1305006-04		1305006-05	
Temperature (Deg. C)	12.98	13.27	12.72	12.59	12.88	12.47	13.14	12.71	14.84	14.64
Conductivity (uS/cm)	45.3	45.1	61.6	64.5	71.1	74.9	82.4	88.3	70.5	73.1
pH	7.55	6.98	7.35	7.25	7.55	7.47	7.48	7.55	7.53	7.42
Dissolved Oxygen (mg/L)	10.63	10.65	10.22	10.12	11.60	11.56	11.24	10.89	11.41	11.05
Dissolved Oxygen (% Sat.)	101.6	102.1	97.8	95.9	111.0	108.6	107.8	103.3	113.1	109.3

Laboratory Data Sheet Reviews

The following criteria, as specified in the laboratory Request for Qualification and Quote (Appendix B) will be used to screen the analytical measurements performed by the contract laboratory:

1. Trip blanks – are values less than detection limits?
2. Method blanks – are values less than detection limits?
3. Review of all values – do concentrations seem reasonable?

4.1.3 Data Validation Requirements

The purpose of data validation, as described in the EPA's "Guidance on Environmental Data Verification and Data Validation" (EPA QA/G-8) is:

> "…an analyte- and sample-specific process that extends the evaluation of data beyond method, procedural, or contractual compliance to determine the analytical quality of a specific data set."

According to U.S. EPA guidance, the data validation is typically performed by someone independent of the project activity and not associated with the organization responsible for producing the dataset. However, the data validator needs to be familiar with both the data validation requirements and the project objectives. LimnoTech's Project QAO will conduct the data validation since LimnoTech project staff are not directly involved in the field or laboratory operations.

The first requirement in this project's data validation is to inspect the data verification and review records to ensure that no oversights were made during that process. The second requirement of the data validation is to evaluate the data against the project's data quality objectives. The project-specific Data Quality Indicators are presented in Section 1.4. If data do not meet one or more of the DQIs, the data validation process will include an investigation into causes and an assessment of the impact of the noncompliant data on project objectives. The third requirement of the data validation is to evaluate the data in the context of the project's overall objectives, which are described in Section 1.3. The fourth requirement of the data validation is to communicate the data validation results to the rest of the project team.

4.2 Verification and Validation Methods (D.2)

All environmental measurement data and samples collected by project staff will be subjected to quality control prior to being entered into the project database. This is a multi-step process where the Laboratory QA/QC Manager will have primary responsibility for verifying the data and a third party, who is not involved in the data collection or analysis, conducts the data validation. These steps are described in more detail in the following sections.

4.2.1 Data Verification

This section describes the procedures that will be utilized in this project for verifying the data against method, procedural and/or contractual requirements.

Field Activities Data Verification

Individual crew leaders will verify the completion of their field data sheets and chain-of-custody forms. In addition, crew leaders will also verify the proper calibration and operation of their multi-parameter instruments. At the completion of each monitored event, the Field Managers will review all field data sheets, calibration sheets, and chain-of-custody forms for accuracy and completeness. The Field Managers will also

verify that monitoring QA objectives for all accuracy, precision, completeness, and adherence to the required collection techniques are being met.

Laboratory Analytical Results Verification

Individual analysts will verify the completion of the appropriate analytical test and required bench sheets. The Laboratory Operations/Technical Director or designee will review calculations and inspect laboratory bench sheets and log books daily to verify their accuracy, completeness, and adherence to the specified analytical method protocols. Calibration and QC data will be examined daily by the individual analyst. The Laboratory Technical Director or designee will verify that all instrument systems are under control and that QA objectives for accuracy, precision, completeness, and adherence to the required detection limits are being met.

A summary of all QA/QC results and any non-conformance issues will be included in the laboratory deliverable to the Project Manager.

4.2.2 Data Validation

This section describes the process that will be used to validate the data generated for this project. The first requirement in this project's data validation is to inspect the data, verification and review records to ensure that no oversights were made during that process. A complete set of field and laboratory information will be provided to the data validator for this task. The data management components described in Section 2.10 will be sufficient for this purpose.

The primary objective of the data validation in this project is to evaluate the data against the DQIs presented in Section 1.4. These DQIs include criteria for accuracy, precision, completeness, representativeness, comparability and compliance with required detection limits. The data management components described in Section 2.10 will provide the necessary information to make this evaluation. The following must be checked as part of the measurement data and analytical data validation activities.

1) field measurements data collection
2) field sample collection
3) sample custody
4) laboratory analytical results and case narrative
5) data reviews
6) quality control data

The Project QAO will conduct a systematic review of the data for compliance with the established quality control criteria based on replicate, spiked, control, and blank data results provided by the laboratory. In addition, quality assurance evaluations of data accuracy, precision, and completeness will be performed on the field measurement data and the laboratory analytical results for each monitored event. The data validation qualifiers listed in Table 13 will be used when validating the data:

Table 13. Data validation qualifiers

Qualifier	Definition
U	The analyte was not detected in the sample at the estimated detection limit.
J	The reported result is an estimate. The value is less than the minimum calibration level but greater than the estimated detection limit.
R	The data are unusable (note: analyte may or may not be present)
UJ	The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
B	Analyte found in sample at concentration less than 3 times the associate blank concentration.

All qualified data will be reported with validation qualifiers, however B flagged data will not be used in congener summations for total PCB.

If quality control checks or objectives were not met, an investigation of the non-conformance will be initiated by the Project QAO with the project team personnel, including the Field Manager, the Laboratory QA/QC Manager, and the Project Manager. The non-conformance will be documented and the affected data set will be flagged appropriately, identifying any limitations.

Another objective of the data validation is to evaluate the data within the context of the project goals, as discussed in Section 1.3. Suitable datasets for this project will be based on the data quality assessment described above as well as an assessment of the spatial and temporal extent of the sample collection. Comparability with other sources of data will be evaluated by comparing and, if necessary, plotting the data with previously collected data to identify outliers or anomalous values.

The data validation results will be communicated to the project team in the form of a summary table that lists the validation tasks performed and the associated results and conclusions. If the validated dataset includes non-compliant data, this data will be addressed in a memo that accompanies the summary table. Data qualifiers assigned to the data during validation will be maintained in the project database to ensure communication of validation results with current and future data users.

4.3 Reconciliation with User Requirements (D.3)

Once all field measurements and analytical data have been reviewed, quality control measures assessed, and any problems addressed, the measurement and analytical data will be assessed.

The assessment of the information generated from the monitoring program will be initiated by entering all analytical data and field measurement data into the project database. In addition flow data, stage data, field notes, and information on any sampling anomalies will be appended. All of these data will be evaluated and any relationships or correlations will be noted. The compilation of all information surrounding a sampling and/or monitoring event will be available to facilitate reconciliation with user requirements. Ultimately these data will be used to support a semi-quantitative low-flow mass balance assessment and assess the seasonal variability of upstream loads to the Spokane River. In addition, the data shall be sufficient to support an adaptive management approach, where grab sample results can be directly compared to results from other sampling methodologies to allow determination of an improved monitoring approach for future phases of this work.

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5. REFERENCES

Ecology, 2014. Spokane River Toxics Sampling 2012-2013 – Surface Water, CLAM and Sediment Trap Results. Technical Memorandum from Brandi Era-Miller to Dale Norton.

LimnoTech, 2013a. Identification of Data Gaps-Final. Memorandum from Dave Dilks, Tim Towey and Kat Ridolfi to Spokane River Regional Toxics Task Force. November 14, 2013.

LimnoTech, 2013b. Initial Conceptual Models of PCBs and Dioxins in the Spokane River Watershed - Final. Memorandum from Dave Dilks, Tim Towey and Kat Ridolfi to Spokane River Regional Toxics Task Force. November 14, 2013.

LimnoTech, 2014a. Data Collection Strategy for PCB Comprehensive Plan - Draft. Memorandum from Dave Dilks to Spokane River Regional Toxics Task Force. February 4, 2014.

LimnoTech, 2014b. Sampling Recommendations for Spokane River PCB Confidence Testing – Draft. Memorandum from Dave Dilks to the Spokane River Regional Toxics Task Force. April 7, 2014.

LimnoTech, 2014c. Confidence Testing Results from Spokane River PCB Sampling – Draft. Memorandum from Dave Dilks to the Spokane River Regional Toxics Task Force. July 15, 2014.

Serdar, D., B. Lubliner, A. Johnson, D. Norton, 2011. Spokane River PCB Source Assessment 2003-2007. Publication No. 11-03-013.

Spokane Tribe of Indians, 2010. Surface Water Quality Standards, Resolution 2010-173. February 25, 2010.

United States Environmental Protection Agency (EPA), 1998. EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5. Washington , DC.

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United States Environmental Protection Agency (EPA), 2002. Guidance on Environmental Verification and Data Validation. EPA QA/G-8. Washington, DC.

Washington Department of Ecology, 2004. Guidelines for Preparing Quality Assurance Project Plans for Environmental Studies. Publication No. 04-03-030, Revision Publication No. 01-03-003.

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APPENDIX A

EPA METHOD 1668C

This document is located at:

http://srrttf.org/wp-content/uploads/2014/05/M1668C_11June10-PCB_Congeners.pdf

APPENDIX B

LABORATORY REQUEST FOR QUALIFICATIONS AND QUOTE/ECOLOGY CERTIFICATION

(AXYS ANALYTICAL SERVICES AND SVL ANALYTICAL, INC.)

SPOKANE RIVER
REGIONAL TOXICS TASK FORCE

COLLABORATION INNOVATION PROGRESS

April 3, 2014

ALS Environmental
1317 South 13th Avenue
Kelso, WA 98626
Attn: Ron McLeod

AXYS Analytical Services, Ltd
2045 Mills Road W
Sidney, BC
Canada V8L 5X2
Attn: Richard Grace

Pacific Rim Laboratories
#103, 19575 – 55A Avenue
Surrey, BC
Canada V3S 8P8
Attn: Dave Hope

Vista Analytical Laboratory
1104 Windfield Way
El Dorado Hills, CA 95762
Attn: Jennifer Miller

Dear Potential Supplier:

Attached is an updated Specification No. 1 (Revision 1) date April 3, 2014. This version of the specification attempts to address all the question and clarifications that were raised or requested by various parties. In addition a Response to Questions sheet is also enclosed that provides additional clarifications.

Submittals for bidding on this project are to be sent electronically to the SRRTTF facilitator and project associate. From there the submittal will be forwarded to the Technical Work Group of the SRRTTF for review. Following the review of the bids received, the Technical Work Group will make a recommendation to the full Task Force for their decision.

The contact information for the SRRTTF facilitator and project associate is as follows:

Chris Page
William D. Ruckelshaus Center
901 Fifth Avenue, Suite 2900
Seattle, WA 98164
(206) 770-6060
c.page@wsu.edu

Aubri Denevan
William D. Ruckelshaus Center
901 Fifth Avenue, Suite 2900
Seattle, WA 98164
(206) 219-2432
aubri.denevan@wsu.edu

For technical questions on the Specification or the project bid submittal, please contact me. My contact information is as follows:

Bud Leber
Kaiser Aluminum
PO Box 15108, Mail Stop #32
Spokane Valley, WA 99215
(509) 927-6554
bud.leber@kaisertwd.com

Exhibit "B" and the requested information are to be provided to Chris and Aubri at the e-mail addresses provided above by the close of business on April 11, 2014.

If you should have any questions or need any clarifications, please contact me.

Sincerely,

Bernard P. Leber Jr.

Bernard P. (Bud) Leber, Jr.
SRRTTF Technical Work Group Chair

Response to Specification Questions

Idaho Department of Environmental Quality (IDEQ) Laboratory Accreditation

IDEQ does not have a laboratory accreditation program that applies to this Specification. The Specification has been revised to remove this as a qualification requirement.

Exhibit "B" Revision – Sample Types

The table in Exhibit "B" with respect to the column labeled "Sample Volume or Type" has been revised. The reference to samples collected by XAD2 resin has been removed. This method of collection is no longer being considered at this time. Please note that other revisions have been made to this table and a section has been added where any explanations or additional information related to the proposal can be provided.

Sample Details

A table has been added to Exhibit "A" (Scope of Work). This table provides additional details on sources being sampled and other details such as sample compositing for each event. Where available, TSS information specific to the samples has been provided.

The final sample collection method will be decided upon by SRTTF-ACE based on a combination of lowest cost and lowest method blank contamination level.

CLAM Details

The following information is provided relative to the potential use of CLAMs for sample collection:

- *With respect to the sourcing of the CLAM, SRRTTF-ACE would purchase and supply the CLAM media to the laboratory for preparation (conditioning and pre-deployment spiking using labeled compounds used for cleanup standards by the laboratory). The cost for this preparation should be included in the per sample cost in the table in Exhibit "B". All field sample collection work will be performed by a separate contractor. No quotes related to field equipment are required.*
- *Each CLAM is expected to have processed between 55 L and 90 L of water with an average of 60 L.*
- *No pre-filter would be used for any samples collected by a CLAM. The laboratory is to report the total amount extracted from the CLAM.*
- *With respect to "blank proofing", (Proof of Clean Certification), one conditioned and spiked CLAM for the CLAM Method Blank for each batch of 20 or fewer samples and one for the CLAM ORP*
- *Target Reporting Limits are provided in the table in the Reporting of Results Section, Paragraph 6.A. of Exhibit "A". Please note: Data reported below the lab's QL will not be within the calibration range, whether diluted or not, and must therefore be qualified as estimated.*

General Questions

For Proof of Clean Certification, no additional "blank proofing" by the lab is needed, as long as the sample containers used are certified as clean by the manufacturer.

With respect to the table in Exhibit "B", pricing should be provided for each Method Blank column for each sample volume/type identified. If the Method Blank level cannot be achieved, enter "NB" in the column.

For reporting of blank levels requested in the Specification, please provide the mean and 2 sigma of the mean, as well as actual concentrations for each individual blank, reported to the EDL.

A section has been added to Exhibit "B" after the pricing table so that any additional information or qualifications can be provided.

Bookmarking of pdf documents is not required, but is preferred.

With respect to the requirement for labeled standard recovery in sample and Method Blanks, at a minimum the limits from the revised 1668A (2003) (15% - 150% for the monochlorobiphenyls) should be observed.

With respect to Exhibit "A", Reporting of Results Section, Paragraph 5G, the redrawn baseline must be visible to the data reviewer.

The additional CS-0.2 calibration standard must meet all method criteria.

Any GC column allowed for in the method may be used, regardless of co-elutions.

SRRTTF-ACE
Specification No. 1
(Revision 1)

April 3, 2014

PCB Analytical Services by EPA Method 1668C

The Spokane River Regional Toxics Task Force (SRRTTF) through its Administrative and Contracting Entity (SRRTTF-ACE) will be conducting PCB source identification studies on the Spokane River for PCB. These studies will require analytical services for PCB by EPA Method 1668C.

Scope of Qualifications

1.0 Provide Analytical Services

Project details are provided in the attached Exhibit "A", Scope of Work (SOW). To be considered for this project, the Contractor must electronically provide the following documentation/information:

1.1 Provide documentation that the Contractor's laboratory is currently accredited by the Department of Ecology's Laboratory Accreditation Unit for all analyses described in the attached SOW.

1.2 Provide documentation that the Contractor has a minimum of 5 years of experience with the method.

1.3 Provide documentation that the Contractor has participated in an International Round Robin Intercalibration Study (and provide the most recent results) for the relevant analyses described in the attached SOW.

1.4 Provide documentation that the Contractor can provide the analysis as requested, including but not limited to a Method Detection Limit (MDL) supporting the requested reporting limits including documentation of a standard analyzed at the reporting limit requested for this SOW.

1.5 Submit Method Blank demonstrating that the Contractor can meet the required Method Blank contamination limits described in the SOW.

1.6 Provide documentation of the quantitation limits (based on the lowest calibration standard) that the instrument can achieve.

1.7 Provide quality control limits for laboratory control samples, duplicates, matrix spikes, etc., for all analyses in this SOW.

1.8 Provide a contact name, company name, address, and phone number for three Contractor client references who have had the requested analyses performed on the matrices specified in the SOW.
1.9 Demonstrate that the Contractor can provide the analytical reports as requested in the attached SOW.
1.10 Demonstrate that the Contractor has the ability to process multiple aliquots of larger volume samples (i.e. – 1 gallon) and combine the extracts as well as the ability to composite samples in-house.

2.0 Other Factors

In addition to the analytical qualifications described in Section 1.0 above, the Contractor must electronically provide the following documentation/information:

2.1 Provide a maximum three-page length description of their qualifications specific to the SOW and their intended approach to performing the analysis, electronically. This should also include information on capabilities for performing this method in various matrices: water, sediment/soil, animal tissue, and other materials. Include details of preparation method to be used on these samples.
2.2 Submit an example work product in the form of one fully bookmarked and searchable PDF file. This work product must include all raw data that would be needed to perform an independent review of the results: calibration reports, chromatograms, spectra, bench sheets, etc.
2.3 Submit the 20 most recent Method Blanks for the matrix/matrices of interest in the SOW.
2.4 Submit the 20 most recent Ongoing Precision and Recovery Standards - OPRs (LCS) for the matrix/matrices of interest in the SOW.

Contractor Selection Process

3.0 Selection Criteria

The selection process will be based on cost, relevant experience, and ability to provide the specified deliverables according to schedule. The following criteria will be used:

3.1 Submittal was received by the date and time specified.
3.2 Submittal contained all required documentation/information.
3.3 Submittal shows a good understanding of project goals and needs.
3.4 Submittal demonstrates relevant experience with similar environmental samples.
3.5 Submittal demonstrates capability to meet all technical specifications. This includes evaluation of 20 blanks and 20 OPRs for conformance to criteria in this

SOW (1668C criteria for the OPRS and Paragraph 9D in the SOW under Reporting of Results).

3.6 Submittal demonstrates the ability to meet the specified schedule for sample analysis and reporting.
3.7 Submittal provided complete and clear cost information.

Additional Information

4.0 Errors in Submittal

Contractor is liable for all errors or omissions contained in their submittals. Contractor will not be allowed to alter submittals after the submission deadline. SRRTTF-ACE is not liable for any errors in submittals. SRRTTF-ACE reserves the right to contact Contractor for clarification of submittal contents. If clarification questions result in a required revision by the Contractor, only revisions addressing the clarification will be allowed.

5.0 Vendor Questions and Exceptions

Any Contractor questions must be transmitted by electronic mail. Only written questions will receive official written responses. Should a Contractor question result in a revision to this specification, all potential Contractors will be advised and the submittal date will be revised if appropriate.

With respect to any exceptions that the Contractor may have with respect to this specification, these shall be noted on Exhibit "B".

6.0 Proprietary or Confidential Information

Any proprietary or confidential contained in the Contractor's submittal must be clearly identified. Marking of the entire or entire sections of the submittal as proprietary or confidential will not be accepted nor honored. SRRTTF-ACE will not accept submittals where pricing is identified as proprietary or confidential.

7.0 Submittal

The submittal by the Contractor shall include the documentation/information described above as well as Exhibit "B".

Exhibit "A"
Specification No. 1
Scope of Work
(SOW)

This SOW does not include the collection of any samples.

SRRTTF-ACE will send approximately 161 water samples over approximately 4 events for PCB congeners by High Resolution Mass Spectrometer (HRMS) analysis, EPA Method 1668C. The successful laboratory must follow the quality control criteria in EPA Method 1668C with the following exception. The labeled compound percent recovery for Sample and Method Blank Standard Recovery must be within the range of 25% to 150% (15% - 150% for the monochlorobiphenyls should be observed). Samples may be collected in various volumes or types such as 1 liter, 2.36 liters, 4.0 liters or CLAM Cartridges. A lab duplicate, matrix spike, and matrix spike duplicate will be requested for each sample event. The following tables provide sampling time frames and sample count details:

May 2014 Sampling Event	
Total Sample Count	Sample Details
8	Riverine samples from 2 locations (3 from one and 5 from the other)
5	Trip Blanks (1 per sampling day)
5	Replicates (1 per sampling day)
1	3 samples to be composited into 1
1	5 samples to be composited into 1

August 2014 Sampling Event	
Total Sample Count	Sample Details
56	7 riverine samples from 8 locations
24	3 point source samples from 8 locations
8	7 samples to be composited from each of 8 riverine locations
8	3 samples to be composited from each of 8 point source locations
7	Trip Blanks (1 per sampling day)
7	Riverine Replicates (1 per sampling day)
3	Point Source Replicates (1 per sampling day)

December 2014 / February 2015 Sampling Event	
5	Riverine samples from 1 location
1	5 samples to be composited into 1
5	Replicate (1 per sampling day)
5	Trip Blanks (1 per sampling day)

May 2015 Sampling Event	
5	Riverine samples from 1 location
1	5 samples to be composited into 1
5	Replicate (1 per sampling day)
5	Trip Blank (1 per sampling day)

(TSS levels in riverine samples are expected to be as follows: minimum – 1 mg/L; maximum – 79 mg/L; median – 2 mg/L; mean – 3 mg/l)

Laboratories must analyze and provide data for an independent source standard (different vendor than the calibration standards).

The estimated cost of ground shipping sample containers, field blank water, coolers, and blue ice are to be included in the price quote.

The laboratory must document which preparation and extraction procedures are performed – and how - for the samples from this project. The laboratory must also document in a logbook, and in a case narrative, any deviations from their Standard Operating Procedures (SOP) performed for this project.

The final data package is to include:

a) All raw data (EPA "Tier IV" or "Level 4" deliverables) in a fully bookmarked PDF file; and
b) All results in an electronic data deliverable (EDD) format as shown in Section 13 of **Reporting of Results** below. The EDD format is needed for loading results to Ecology's Information Management (EIM) database.

Other items may be included as needed to help understand the data package.

Data Turnaround Time

45 days from sample receipt for May 2014 samples.
60 days from sample receipt for all other sample events.

Analytical Details

1. Section 9.5.1 in all versions of EPA Method 1668 state: "Analyze the blank immediately after analysis of the ongoing precision and recovery standards (OPR) (Section 15.5) to demonstrate freedom from contamination." However, as mentioned in EPA Method 1668, Revision C, if congeners will be carried from the OPR into the Method Blank, analyze one or more aliquots of solvent between the OPR and the Method Blank.
2. Perform all result calculations using the initial calibration as per the method. In other words, do not use a single point calibration standard. Also, do not average in additional standards analyzed on a different day, or analyzed after the samples have been analyzed.
3. PCB congeners: Use the combined 209 congener standard solution for calibration verification. (Including the labeled and native toxics/Level of Chlorination (LOC)/window-defining congeners in the calibration verification allows a check against the Initial Calibration (ICAL) for those congeners.)

   Alternatively, a separate solution may be analyzed for each, but both solutions must be analyzed on the method schedule for calibration verification. SRRTTF-ACE must be able to evaluate the daily 209 standard against the initial analysis of this standard.
4. All congeners and labeled compounds in the low level Calibration Standard (CS-0.2 standard) must be within the method QC limits for their respective ion abundance ratios; otherwise, the mass spectrometer must be adjusted and this test repeated until the m/z ratios fall within the limits specified. (If the adjustment alters the resolution of the mass spectrometer, resolution must be verified prior to repeat of the test.)
5. Because of the low reporting limits requested, it is recommended the lab add in an extra standard to the initial calibration curve. This will account for increased sensitivity potentially causing analyte saturation at the high end of the curve, and allow a minimum of 5 points to be used in calculating analyte concentration. This will be accomplished by use of the CS-0.2 standard specified in the method.
6. HRMS instrument resolution must be 10,000 or better. Proof (in the form of an instrument printout) must be submitted with the data.

Reporting of Results

1. Report all results in pg/L for water.
2. Include a copy of the "Request for Laboratory Services" with signed and dated Chain of Custody section; this form will be provided by the SRRTTF-ACE Sampling Contractor. Proof of Clean Certification must be provided for project sample containers.
3. Include Case Narratives and corrective action reports.
4. Provide description of: analytical method used; any modifications to the method, Quality Assurance/Quality Control (QA/QC) performed and results; definitions of all data flags and qualifiers used; and any other information that helps client understand the data package.
5. Provide fully validatable deliverables package: Deliverables shall include copies of all raw

data necessary to perform an independent evaluation of the results, including, but not limited to initial calibration and verification standards, sample and QC chromatograms and spectra, analytical sequence (run) logs, bench sheets, standard logs and Certificates of Analysis for standards, etc.

A. Include a fully paginated and bookmarked Adobe Acrobat (PDF) file on compact disk (CD).

B. Bookmark *each individual sample and each standard chromatogram* for ease of review.

C. Rotate landscape pages as needed so that all information is viewable left to right in the electronic file.

D. Clearly identify all field and QC samples with the sample number or QC name in the raw data and report.

E. All initial calibration (ICAL) standards and Calibration Verification Standard (VER), and the single point 209 PCB standard, shall be clearly identified in the raw data and separately bookmarked in the electronic file. (For example: CS-0.2, CS-1, etc., for the ICAL.)

F. An Independent Calibration Verification (ICV) standard must be analyzed from a separate source in order to verify the initial calibration standards. The ICV must be analyzed each time a new standard curve is prepared. Provide the results of the most recent ICV with the data. This is equivalent to the Quality Control Check Sample in the method.

G. Provide before and after printouts of any and all manual integrations.

H. Provide analytical sequence logs that include the date, time, and filename for the initial and continuing calibrations, all field and QC samples, check standards, etc., associated with the project.

6. Reporting Limits (RL), Quantitation Limit (QL), Method Detection Limit (MDL), Estimated Detection Limit (EDL).

A. Maximum RLs are defined in the table below.

Analytical Methods and Reporting Limits			
Analysis	**Analyte**	**Water**	**Sediment**
EPA 1668C	PCB congeners	1-20 pg/L (depending on congener)	NA

B. If any of these limits cannot be met for individual samples due to interference or other issues, contact the client to discuss action to take.

C. Provide the QL for each result in the electronic results file. (The QL is based on the lowest validated standard in calibration curve; and equivalent to "Minimum Level or ML"

in 1668C).

D. Provide the most recent MDL results for each analyte and include the date performed.

E. Report down to the (EDL) - aka Instrument Detection Limits (IDL) or Sample Detection Limits (SDL) - based on 2.5 times the signal-to-noise ratio. Provide this value for each target analyte in the electronic results file.

F. Dilutions

   a. Any results above the range of the calibration curve must be diluted to be within the range of the calibration curve.

   b. All results reported from dilution analyses must be within the range of the calibration curve.

G. For non-detect values, record the EDL in the "Result Reported Value" column and a "UJ" the "Result Data Qualifier" column.

H. Qualify detected values that are below the QL as estimates ("J").

I. Do not report below the EDL. Where the EDL is above the QL due to interference, raise any values below the EDL to the value of the EDL and qualify "UJ".

J. Report total homologs when not detected as "U" without a value.

K. Calculate and report the Estimated Maximum Possible Concentration (EMPC) value for results that do not meet ion abundance ratio criteria. Qualify these results with "NJ". Provide an example calculation if the result value is adjusted.

7. The qualifiers used above are defined as:

   A. "J" – The analyte was positively identified. The associated numerical result is an estimate.

   B. "U" – The analyte was not detected above the reporting limit. (This qualifier will likely be used only for total homologs, since all analytes are to be reported down to the level of the EDL.)

   C. "UJ" – The analyte was not detected at or above the estimated reporting limit.

   D. "NJ" – The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration. (See 6. K., above.)

8. Perform all QC samples as specified in the method.

   A. Report results of Laboratory Control Samples (On-going Precision and Recovery standards), labeled compounds, (including cleanup standards and extraction internal standard/surrogates) as % recoveries in the EDD.

9. Method Blanks.

   A. Clearly identify samples associated with each laboratory Method Blank.

B. If sample results are less than three times the concentration in the associated method blank, flag sample results with "B" – even if the sample result has already been qualified "NJ"; but not when the blank result is qualified "NJ". Discuss in the Case Narrative whether these qualified results are included in the summing of total homolog results and Total PCBs; where applicable.

C. Total PCBs in the Method Blank, at a maximum, must not exceed 50 pg/L. Method Blanks for Total PCBs in the range of 10pg/L to 1 pg/L are desired. If the 50 pg/L or other established limit is exceeded, contact SRRTTF-ACE to discuss actions to take. Most likely, any blanks with individual results greater than half the EQL should be re-extracted along with any associated samples.

D. Concentrations of congeners in a minimum of 10 blanks must be significantly below the ML {QL}. "Significant" means that the ML for the congener is no less than 2 standard deviations above the mean (average) level in the minimum of 10 blanks. The blanks must be analyzed during the same period that samples are analyzed, ideally over an approximately 1-month period.

10. Treatment of result qualifiers for and summing of homologs.

A. Describe in the case narrative how totals were derived for PCB homolog groups and Total PCBs (e.g. what rules are used for rounding values, dealing with non-detects, blank detects, qualifier definitions, etc.).

B. Report Total PCB results for each homolog group in the EDD. However, do not report a QL or an EDL (leave these columns blank for summed values).

C. Do not include EMPC results in the calculations of the total homologs.

11. Sample identification.

A. Provide the client sample ID (field ID) associated with all sample results.

B. Provide the lab's internal sample ID associated with all results OR a table that cross-references field ID with the lab's internal sample ID.

C. Clearly identify QA/QC samples and results: blanks, matrix spikes, Standard Reference Materials (SRM), lab duplicates. If samples are reanalyzed, these results need be clearly identified as such.

D. Label all analyte peaks on chromatograms with either the congener name or the retention time and scale chromatograms such that peaks are visible above the baseline.

12. Analyte identification.

A. Provide the Chemistry Abstract Service Registry Number (CAS RN) for individual congeners/each analyte.

B. PCB Congener Numbering.

a. Name PCB congeners using the naming convention given by Guitart, et al.

(Guitart R., Puig P., Gomez-Catalan J., Chemosphere 27 1451-1459, 1993).

See http://www.epa.gov/osw/hazard/tsd/pcbs/pubs/congeners.htm

b. Modify to a 7-character format that uses leading zeroes for congener numbers below 100 (e.g. PCB-008). (Conversely, the value "PCB-001" appears to have 7 characters yet actually has 11 since there are 4 spaces after the 001. This complicates export into databases and statistical packages.)

c. Records for co-eluting congeners must have no CAS number.

C. Co-eluting congeners for PCBs should be numbered in ascending order (e.g.: PCB-040/041/071), and records for co-eluting congeners must have no CAS number.

13. Electronic results must be in Excel-compatible format as in table below:

Required Fields for Electronic Data Deliverables		
Preferred Order	**Field Name**	**Example**
1	MEL (Client) Sample ID	1311021-03
2	Field ID (sample name on tag)	COLRIV034
3	Result Congener Name	2,3'-DiCB
4	Result Parameter Name	PCB-006
5	Result Parameter CAS Number	25569-80-6
6	Sample Extraction Date	11/14/2013**(format as numerical date)**
7	Sample Analysis Date	11/15/2013 **(format as numerical date)**
8	Lab Duplicate Flag	"Y" if lab duplicate, leave blank or "N" if not
9	Re-analysis Flag	"Y" if a re-analysis, leave blank or "N" if not
10	Result Reported Value	7.9 (format as number)
11	Result Data Qualifier	J
12	Result Value Units of Measure	pg/L
13	Result Value QL *	10 (format as number)
14	Result Value EDL**	3.42 (format as number)
15	Result Method Code	EPA 1668C
16	Result Lab Name	Laboratory Name
17	Contract Lab Sample ID	PR137954
18	Others as needed by contract lab or MEL.	If used, clearly identify field and content
	* = Estimated Quantitation Limit (Based on the lowest validated standard in the calibration curve and adjusted for weight, volume, % solids, etc., as applicable).	
	** = Estimated Sample Detection Limit; calculated from signal for each sample)	

Exhibit "B"
Specification No. 1

The Request for Proposal, Specification No. 1, sets forth the requirements for providing PCB analytical services utilizing EPA Method 1668C. This Exhibit and the requested documentation/information is to be provided to SRRTTF-ACE as identified in the bid package cover letter.

Contact Information

Please provide the following information:

Laboratory Name:	
Laboratory Address:	
Project Contact Name:	
Project Contact Phone:	
Project Contact E-mail:	

Is the Contractor a Minority or Women's Business Enterprise ☐ Yes ☐ No

It is the Owner's intention to select a Contractor on the basis of both laboratory performance and the competitiveness of Contractor's commercial proposal.

To assist the Owner in evaluating the various proposals, Contractor shall furnish the following information.

1. Laboratory Performance

As described in Specification No. 1, please provide the following:

Qualifications

Provide the documentation and/or information requested as described Section 1.0 of the Specification.

Other Factors

Provide the documentation and/or information requested as described in Section 2.0 of the Specification with respect to the Scope of Work described in Exhibit A.

All responses to these document/information requests are to be provided electronically.

2. Commercial

Price Breakdown

It is the Owner's intent to award all work covered under Specification No. 1 to a single Contractor. In order to assist the Owner in evaluating bids and to eliminate any obvious errors in bid pricing, the following price breakdown is requested. With respect to the multiple Method Blanks listed, if a Method Blank cannot be achieved for the sample volume or type listed, enter "NB" in the appropriate column.

Unit Price per Sample (US$ per Sample)			
Sample Volume or Type	Method Blank Level		
	Total PCB <50 pg/L	Total PCB <10 pg/L	Total PCB < 1 pg/L
2.36 Liter			
4.0 Liter			
CLAM			

Note: for each of the 2.36 Liter and 4.0 Liter sample sizes above, a Method Blank of the same volume is to be analyzed.

Provide pricing for water sample compositing:

Unit Price per Composite (US$ per Composite)			
	3 Sample Composite	5 Sample Composite	7 Sample Composite
2.36 Liter			
4.0 Liter			

Provide pricing estimate and assumptions for sample containers and shipping for the sample volumes/types listed above:

Provide pricing for the EDD per sampling event (two copies per event to be provided):

3. Conditions of Contract

List any exceptions, if any, taken to Specification No. that need to be addressed with respect to a contract to perform this work.

4. Additional Information

Provide any additional explanatory information related to this quotation.

Potential Contractors are advised that any or all of the information furnished in response to this Exhibit "B" may, as mutually agreed upon, become part of the contract.

SRRTTF-ACE
Specification No. 3
(Revision 0)

May 13, 2014

Analytical Services for Conventional Parameters

The Spokane River Regional Toxics Task Force (SRRTTF) through its Administrative and Contracting Entity (SRRTTF-ACE) will be conducting PCB source identification studies on the Spokane River for PCB. In addition to PCB, these studies will require that field samples be collected and analyzed for conventional parameters as well. To perform this work, the laboratory must be a Washington Department of Ecology Certified Laboratory for the parameters of interest.

1.0 Scope

1.1 Work Included

Supplier shall provide all necessary materials and staff to conduct laboratory chemical analyses for conventional parameters as contained in the Sampling and Analysis Plan and the Quality Assurance Project Plan.

1.2 Work Not Included

Supplier is not responsible for the return of field collected samples to the laboratory for analysis.

1.3 Owner Furnished Items

The Owner shall provide the Supplier with the Sampling and Analysis Plan (SAP) and the Quality Assurance Project Plan (QAPP), which are located in Attachment A.

2.0 Detailed Scope of Work

2.1 Conventional Parameters for Analysis

Analytical services for each sample for the following parameters are to be provided on a standard turnaround basis:

- Total Organic Carbon (TOC) – EPA Method 415.1
- Dissolved Organic Carbon (DOC) – EPA Method 415.3
- Nonfilterable Residue (TSS) – EPA Method 160.2
- Filterable Residue – (TDS) – EPA Method 160.1

2.2 Sample Count

The work will take place during three sampling events: August 2014, December 2014/February 2015, and May 2015. The following tables provide a summary of the sample collection and analysis effort.

August 2014 Sampling Event	
Total Sample Count	Sample Details
56	7 riverine samples from 8 locations
24	3 point source samples from 8 locations
7	Trip Blanks (1 per sampling day)
7	Riverine Replicates (1 per sampling day)
3	Point Source Replicates (1 per sampling day)
December 2014 / February 2015 Sampling Event	
5	Riverine samples from 1 location
5	Replicate (1 per sampling day)
5	Trip Blanks (1 per sampling day)
May 2015 Sampling Event	
5	Riverine samples from 1 location
5	Replicate (1 per sampling day)
5	Trip Blank (1 per sampling day)

2.3 Sample Containers

Supplier shall provide all sample containers, Chain of Custody forms, field blank water, coolers and blue ice. The Supplier shall ship required sample containers via ground transportation.

2.4 Data Packages

Supplier shall provide all laboratory data packages in electronic format.

The State of Washington Department of Ecology

Axys Analytical Services Ltd
Sidney, BC

has complied with provisions set forth in Chapter 173-50 WAC and is hereby recognized by the Department of Ecology as an ACCREDITED LABORATORY for the analytical parameters listed on the accompanying Scope of Accreditation. This certificate is effective May 29, 2014 and shall expire May 28, 2015.

Witnessed under my hand on May 16, 2014

Alan D. Rue
Lab Accreditation Unit Supervisor

Laboratory ID
C404

WASHINGTON STATE DEPARTMENT OF ECOLOGY

ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

SCOPE OF ACCREDITATION

Axys Analytical Services Ltd

Sidney, BC

is accredited for the analytes listed below using the methods indicated. Full accreditation is granted unless stated otherwise in a note. Accreditation for U.S. Environmental Protection Agency (EPA) "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods" (SW-846) is for the latest version of the method. SM refers to EPA approved editions of "Standard Methods for the Examination of Water and Wastewater." ASTM is the American Society for Testing and Materials. Other references are described in notes.

Matrix/Analyte	Method	Notes
Non-Potable Water		
2,4'-DDD	AXYS MLA-028	1
2,4'-DDE	AXYS MLA-028	1
2,4'-DDT	AXYS MLA-028	1
4,4'-DDD	AXYS MLA-028	1
4,4'-DDE	AXYS MLA-028	1
4,4'-DDT	AXYS MLA-028	1
Aldrin	AXYS MLA-028	1
alpha-BHC (alpha-Hexachlorocyclohexane)	AXYS MLA-028	1
alpha-Chlordane	AXYS MLA-028	1
beta-BHC (beta-Hexachlorocyclohexane)	AXYS MLA-028	1
cis-Nonachlor	AXYS MLA-028	1
delta-BHC	AXYS MLA-028	1
Dieldrin	AXYS MLA-028	1
Endosulfan I	AXYS MLA-028	1
Endosulfan II	AXYS MLA-028	1
Endosulfan sulfate	AXYS MLA-028	1
Endrin	AXYS MLA-028	1
Endrin aldehyde	AXYS MLA-028	1
Endrin ketone	AXYS MLA-028	1
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	AXYS MLA-028	1
gamma-Chlordane	AXYS MLA-028	1
Heptachlor	AXYS MLA-028	1

Matrix/Analyte	Method	Notes
1,2,3,6,7,8-Hxcdd	EPA 1613_1994	1
1,2,3,6,7,8-Hxcdf	EPA 1613_1994	1
1,2,3,7,8,9-Hxcdd	EPA 1613_1994	1
1,2,3,7,8,9-Hxcdf	EPA 1613_1994	1
1,2,3,7,8-Pecdd	EPA 1613_1994	1
1,2,3,7,8-Pecdf	EPA 1613_1994	1
2,3,4,6,7,8-Hxcdf	EPA 1613_1994	1
2,3,4,7,8-Pecdf	EPA 1613_1994	1
2,3,7,8-TCDD	EPA 1613_1994	1
2,3,7,8-TCDF	EPA 1613_1994	1
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ-206)	EPA 1668C_2010	1
2,2',3,3',4,4',5,5'-Octachlorobiphenyl (BZ-194)	EPA 1668C_2010	1
2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl (BZ-207)	EPA 1668C_2010	1
2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ-195)	EPA 1668C_2010	1
2,2',3,3',4,4',5,6'-Octachlorobiphenyl (BZ-196)	EPA 1668C_2010	1
2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ-170)	EPA 1668C_2010	1
2,2',3,3',4,4',6,6'-Octachlorobiphenyl (BZ-197)	EPA 1668C_2010	1
2,2',3,3',4,4',6-Heptachlorobiphenyl (BZ-171)	EPA 1668C_2010	1
2,2',3,3',4,4'-Hexachlorobiphenyl (BZ-128)	EPA 1668C_2010	1
2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl (BZ-208)	EPA 1668C_2010	1
2,2',3,3',4,5,5',6-Octachlorobiphenyl (BZ-198)	EPA 1668C_2010	1
2,2',3,3',4,5,5',6'-Octachlorobiphenyl (BZ-199)	EPA 1668C_2010	1
2,2',3,3',4,5,5'-Heptachlorobiphenyl (BZ-172)	EPA 1668C_2010	1
2,2',3,3',4,5,6,6'-Octachlorobiphenyl (BZ-200)	EPA 1668C_2010	1
2,2',3,3',4,5',6,6'-Octachlorobiphenyl (BZ-201)	EPA 1668C_2010	1
2,2',3,3',4,5,6-Heptachlorobiphenyl (BZ-173)	EPA 1668C_2010	1
2,2',3,3',4,5,6'-Heptachlorobiphenyl (BZ-174)	EPA 1668C_2010	1
2,2',3,3',4,5',6-Heptachlorobiphenyl (BZ-175)	EPA 1668C_2010	1
2,2',3,3',4,5',6'-Heptachlorobiphenyl (BZ-177)	EPA 1668C_2010	1
2,2',3,3',4,5-Hexachlorobiphenyl (BZ-129)	EPA 1668C_2010	1
2,2',3,3',4,5'-Hexachlorobiphenyl (BZ-130)	EPA 1668C_2010	1
2,2',3,3',4,6,6'-Heptachlorobiphenyl (BZ-176)	EPA 1668C_2010	1
2,2',3,3',4,6-Hexachlorobiphenyl (BZ-131)	EPA 1668C_2010	1
2,2',3,3',4,6'-Hexachlorobiphenyl (BZ-132)	EPA 1668C_2010	1
2,2',3,3',4-Pentachlorobiphenyl (BZ-82)	EPA 1668C_2010	1
2,2',3,3',5,5',6,6'-Octachlorobiphenyl (BZ-202)	EPA 1668C_2010	1

Matrix/Analyte	Method	Notes
2,2',3,3',5,5',6-Heptachlorobiphenyl (BZ-178)	EPA 1668C_2010	1
2,2',3,3',5,5'-Hexachlorobiphenyl (BZ-133)	EPA 1668C_2010	1
2,2',3,3',5,6,6'-Heptachlorobiphenyl (BZ-179)	EPA 1668C_2010	1
2,2',3,3',5,6-Hexachlorobiphenyl (BZ-134)	EPA 1668C_2010	1
2,2',3,3',5,6'-Hexachlorobiphenyl (BZ-135)	EPA 1668C_2010	1
2,2',3,3',5-Pentachlorobiphenyl (BZ-83)	EPA 1668C_2010	1
2,2',3,3',6,6'-Hexachlorobiphenyl (BZ-136)	EPA 1668C_2010	1
2,2',3,3',6-Pentachlorobiphenyl (BZ-84)	EPA 1668C_2010	1
2,2',3,3'-Tetrachlorobiphenyl (BZ-40)	EPA 1668C_2010	1
2,2',3,4,4',5,5',6-Octachlorobiphenyl (BZ-203)	EPA 1668C_2010	1
2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ-180)	EPA 1668C_2010	1
2,2',3,4,4',5,6,6'-Octachlorobiphenyl (BZ-204)	EPA 1668C_2010	1
2,2',3,4,4',5,6-Heptachlorobiphenyl (BZ-181)	EPA 1668C_2010	1
2,2',3,4,4',5,6'-Heptachlorobiphenyl (BZ-182)	EPA 1668C_2010	1
2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ-183)	EPA 1668C_2010	1
2,2',3,4,4',5-Hexachlorobiphenyl (BZ-137)	EPA 1668C_2010	1
2,2',3,4,4',5'-Hexachlorobiphenyl (BZ-138)	EPA 1668C_2010	1
2,2',3,4,4',6,6'-Heptachlorobiphenyl (BZ-184)	EPA 1668C_2010	1
2,2',3,4,4',6-Hexachlorobiphenyl (BZ-139)	EPA 1668C_2010	1
2,2',3,4,4',6'-Hexachlorobiphenyl (BZ-140)	EPA 1668C_2010	1
2,2',3,4,4'-Pentachlorobiphenyl (BZ-85)	EPA 1668C_2010	1
2,2',3,4,5,5',6-Heptachlorobiphenyl (BZ-185)	EPA 1668C_2010	1
2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ-187)	EPA 1668C_2010	1
2,2',3,4,5,5'-Hexachlorobiphenyl (BZ-141)	EPA 1668C_2010	1
2,2',3,4',5,5'-Hexachlorobiphenyl (BZ-146)	EPA 1668C_2010	1
2,2',3,4,5,6,6'-Heptachlorobiphenyl (BZ-186)	EPA 1668C_2010	1
2,2',3,4',5,6,6'-Heptachlorobiphenyl (BZ-188)	EPA 1668C_2010	1
2,2',3,4,5,6-Hexachlorobiphenyl (BZ-142)	EPA 1668C_2010	1
2,2',3,4,5,6'-Hexachlorobiphenyl (BZ-143)	EPA 1668C_2010	1
2,2',3,4,5',6-Hexachlorobiphenyl (BZ-144)	EPA 1668C_2010	1
2,2',3,4',5,6-Hexachlorobiphenyl (BZ-147)	EPA 1668C_2010	1
2,2',3,4',5,6'-Hexachlorobiphenyl (BZ-148)	EPA 1668C_2010	1
2,2',3,4',5',6-Hexachlorobiphenyl (BZ-149)	EPA 1668C_2010	1
2,2',3,4,5-Pentachlorobiphenyl (BZ-86)	EPA 1668C_2010	1
2,2',3,4,5'-Pentachlorobiphenyl (BZ-87)	EPA 1668C_2010	1
2,2',3,4',5-Pentachlorobiphenyl (BZ-90)	EPA 1668C_2010	1

Matrix/Analyte	Method	Notes
2,2',3,4',5'-Pentachlorobiphenyl (BZ-97)	EPA 1668C_2010	1
2,2',3,4,6,6'-Hexachlorobiphenyl (BZ-145)	EPA 1668C_2010	1
2,2',3,4',6,6'-Hexachlorobiphenyl (BZ-150)	EPA 1668C_2010	1
2,2',3,4,6-Pentachlorobiphenyl (BZ-88)	EPA 1668C_2010	1
2,2',3,4,6'-Pentachlorobiphenyl (BZ-89)	EPA 1668C_2010	1
2,2',3,4',6-Pentachlorobiphenyl (BZ-91)	EPA 1668C_2010	1
2,2',3,4',6'-Pentachlorobiphenyl (BZ-98)	EPA 1668C_2010	1
2,2',3,4-Tetrachlorobiphenyl (BZ-41)	EPA 1668C_2010	1
2,2',3,4'-Tetrachlorobiphenyl (BZ-42)	EPA 1668C_2010	1
2,2',3,5,5',6-Hexachlorobiphenyl (BZ-151)	EPA 1668C_2010	1
2,2',3,5,5'-Pentachlorobiphenyl (BZ-92)	EPA 1668C_2010	1
2,2',3,5,6,6'-Hexachlorobiphenyl (BZ-152)	EPA 1668C_2010	1
2,2',3,5,6-Pentachlorobiphenyl (BZ-93)	EPA 1668C_2010	1
2,2',3,5,6'-Pentachlorobiphenyl (BZ-94)	EPA 1668C_2010	1
2,2',3,5',6-Pentachlorobiphenyl (BZ-95)	EPA 1668C_2010	1
2,2',3,5-Tetrachlorobiphenyl (BZ-43)	EPA 1668C_2010	1
2,2',3,5'-Tetrachlorobiphenyl (BZ-44)	EPA 1668C_2010	1
2,2',3,6,6'-Pentachlorobiphenyl (BZ-96)	EPA 1668C_2010	1
2,2',3,6-Tetrachlorobiphenyl (BZ-45)	EPA 1668C_2010	1
2,2',3,6'-Tetrachlorobiphenyl (BZ-46)	EPA 1668C_2010	1
2,2',3-Trichlorobiphenyl (BZ-16)	EPA 1668C_2010	1
2,2',4,4',5,5'-Hexachlorobiphenyl (BZ-153)	EPA 1668C_2010	1
2,2',4,4',5,6'-Hexachlorobiphenyl (BZ-154)	EPA 1668C_2010	1
2,2',4,4',5-Pentachlorobiphenyl (BZ-99)	EPA 1668C_2010	1
2,2',4,4',6,6'-Hexachlorobiphenyl (BZ-155)	EPA 1668C_2010	1
2,2',4,4',6-Pentachlorobiphenyl (BZ-100)	EPA 1668C_2010	1
2,2',4,4'-Tetrachlorobiphenyl (BZ-47)	EPA 1668C_2010	1
2,2',4,5,5'-Pentachlorobiphenyl (BZ-101)	EPA 1668C_2010	1
2,2',4,5,6'-Pentachlorobiphenyl (BZ-102)	EPA 1668C_2010	1
2,2',4,5',6-Pentachlorobiphenyl (BZ-103)	EPA 1668C_2010	1
2,2',4,5-Tetrachlorobiphenyl (BZ-48)	EPA 1668C_2010	1
2,2',4,5'-Tetrachlorobiphenyl (BZ-49)	EPA 1668C_2010	1
2,2',4,6,6'-Pentachlorobiphenyl (BZ-104)	EPA 1668C_2010	1
2,2',4,6-Tetrachlorobiphenyl (BZ-50)	EPA 1668C_2010	1
2,2',4,6'-Tetrachlorobiphenyl (BZ-51)	EPA 1668C_2010	1
2,2',4-Trichlorobiphenyl (BZ-17)	EPA 1668C_2010	1

Matrix/Analyte	Method	Notes
2,2',5,5'-Tetrachlorobiphenyl (BZ-52)	EPA 1668C_2010	1
2,2',5,6'-Tetrachlorobiphenyl (BZ-53)	EPA 1668C_2010	1
2,2',5-Trichlorobiphenyl (BZ-18)	EPA 1668C_2010	1
2,2',6,6'-Tetrachlorobiphenyl (BZ-54)	EPA 1668C_2010	1
2,2',6-Trichlorobiphenyl (BZ-19)	EPA 1668C_2010	1
2,2'-Dichlorobiphenyl (BZ-4)	EPA 1668C_2010	1
2,3,3',4,4',5,5',6-Octachlorobiphenyl (BZ-205)	EPA 1668C_2010	1
2,3,3',4,4',5,5'-Heptachlorobiphenyl (BZ-189)	EPA 1668C_2010	1
2,3,3',4,4',5,6-Heptachlorobiphenyl (BZ-190)	EPA 1668C_2010	1
2,3,3',4,4',5',6-Heptachlorobiphenyl (BZ-191)	EPA 1668C_2010	1
2,3,3',4,4',5-Hexachlorobiphenyl (BZ-156)	EPA 1668C_2010	1
2,3,3',4,4',5'-Hexachlorobiphenyl (BZ-157)	EPA 1668C_2010	1
2,3,3',4,4',6-Hexachlorobiphenyl (BZ-158)	EPA 1668C_2010	1
2,3,3',4,4'-Pentachlorobiphenyl (BZ-105)	EPA 1668C_2010	1
2,3,3',4,5,5',6-Heptachlorobiphenyl (BZ-192)	EPA 1668C_2010	1
2,3,3',4',5,5',6-Heptachlorobiphenyl (BZ-193)	EPA 1668C_2010	1
2,3,3',4,5,5'-Hexachlorobiphenyl (BZ-159)	EPA 1668C_2010	1
2,3,3',4',5,5'-Hexachlorobiphenyl (BZ-162)	EPA 1668C_2010	1
2,3,3',4,5,6-Hexachlorobiphenyl (BZ-160)	EPA 1668C_2010	1
2,3,3',4',5,6-Hexachlorobiphenyl (BZ-163)	EPA 1668C_2010	1
2,3,3',4',5',6-Hexachlorobiphenyl (BZ-164)	EPA 1668C_2010	1
2,3,3',4,5',6-Hexachlorobiphenyl (BZ-161)	EPA 1668C_2010	1
2,3,3',4,5-Pentachlorobiphenyl (BZ-106)	EPA 1668C_2010	1
2,3,3',4',5-Pentachlorobiphenyl (BZ-107)	EPA 1668C_2010	1
2,3,3',4,5'-Pentachlorobiphenyl (BZ-108)	EPA 1668C_2010	1
2,3,3',4',5'-Pentachlorobiphenyl (BZ-122)	EPA 1668C_2010	1
2,3,3',4,6-Pentachlorobiphenyl (BZ-109)	EPA 1668C_2010	1
2,3,3',4',6-Pentachlorobiphenyl (BZ-110)	EPA 1668C_2010	1
2,3,3',4-Tetrachlorobiphenyl (BZ-55)	EPA 1668C_2010	1
2,3,3',4'-Tetrachlorobiphenyl (BZ-56)	EPA 1668C_2010	1
2,3,3',5,5',6-Hexachlorobiphenyl (BZ-165)	EPA 1668C_2010	1
2,3,3',5,5'-Pentachlorobiphenyl (BZ-111)	EPA 1668C_2010	1
2,3,3',5,6-Pentachlorobiphenyl (BZ-112)	EPA 1668C_2010	1
2,3,3',5',6-Pentachlorobiphenyl (BZ-113)	EPA 1668C_2010	1
2,3,3',5-Tetrachlorobiphenyl (BZ-57)	EPA 1668C_2010	1
2,3,3',5'-Tetrachlorobiphenyl (BZ-58)	EPA 1668C_2010	1

Matrix/Analyte	Method	Notes
2,3,3',6-Tetrachlorobiphenyl (BZ-59)	EPA 1668C_2010	1
2,3,3'-Trichlorobiphenyl (BZ-20)	EPA 1668C_2010	1
2,3',4,4',5,5'-Hexachlorobiphenyl (BZ-167)	EPA 1668C_2010	1
2,3,4,4',5,6-Hexachlorobiphenyl (BZ-166)	EPA 1668C_2010	1
2,3',4,4',5',6-Hexachlorobiphenyl (BZ-168)	EPA 1668C_2010	1
2,3,4,4',5-Pentachlorobiphenyl (BZ-114)	EPA 1668C_2010	1
2,3',4,4',5-Pentachlorobiphenyl (BZ-118)	EPA 1668C_2010	1
2,3',4,4',5'-Pentachlorobiphenyl (BZ-123)	EPA 1668C_2010	1
2,3,4,4',6-Pentachlorobiphenyl (BZ-115)	EPA 1668C_2010	1
2,3',4,4',6-Pentachlorobiphenyl (BZ-119)	EPA 1668C_2010	1
2,3,4,4'-Tetrachlorobiphenyl (BZ-60)	EPA 1668C_2010	1
2,3',4,4'-Tetrachlorobiphenyl (BZ-66)	EPA 1668C_2010	1
2,3',4,5,5'-Pentachlorobiphenyl (BZ-120)	EPA 1668C_2010	1
2,3',4',5,5'-Pentachlorobiphenyl (BZ-124)	EPA 1668C_2010	1
2,3,4,5,6-Pentachlorobiphenyl (BZ-116)	EPA 1668C_2010	1
2,3,4',5,6-Pentachlorobiphenyl (BZ-117)	EPA 1668C_2010	1
2,3',4,5',6-Pentachlorobiphenyl (BZ-121)	EPA 1668C_2010	1
2,3',4',5',6-Pentachlorobiphenyl (BZ-125)	EPA 1668C_2010	1
2,3,4,5-Tetrachlorobiphenyl (BZ-61)	EPA 1668C_2010	1
2,3,4',5-Tetrachlorobiphenyl (BZ-63)	EPA 1668C_2010	1
2,3',4,5'-Tetrachlorobiphenyl (BZ-68)	EPA 1668C_2010	1
2,3',4',5-Tetrachlorobiphenyl (BZ-70)	EPA 1668C_2010	1
2,3',4',5'-Tetrachlorobiphenyl (BZ-76)	EPA 1668C_2010	1
2,3',4,5-Tetrachlorobiphenyl (BZ-67)	EPA 1668C_2010	1
2,3,4,6-Tetrachlorobiphenyl (BZ-62)	EPA 1668C_2010	1
2,3,4',6-Tetrachlorobiphenyl (BZ-64)	EPA 1668C_2010	1
2,3',4,6-Tetrachlorobiphenyl (BZ-69)	EPA 1668C_2010	1
2,3',4',6-Tetrachlorobiphenyl (BZ-71)	EPA 1668C_2010	1
2,3,4-Trichlorobiphenyl (BZ-21)	EPA 1668C_2010	1
2,3,4'-Trichlorobiphenyl (BZ-22)	EPA 1668C_2010	1
2,3',4-Trichlorobiphenyl (BZ-25)	EPA 1668C_2010	1
2,3',4'-Trichlorobiphenyl (BZ-33)	EPA 1668C_2010	1
2,3',5,5'-Tetrachlorobiphenyl (BZ-72)	EPA 1668C_2010	1
2,3,5,6-Tetrachlorobiphenyl (BZ-65)	EPA 1668C_2010	1
2,3',5',6-Tetrachlorobiphenyl (BZ-73)	EPA 1668C_2010	1
2,3,5-Trichlorobiphenyl (BZ-23)	EPA 1668C_2010	1

Matrix/Analyte	Method	Notes
2,3',5-Trichlorobiphenyl (BZ-26)	EPA 1668C_2010	1
2,3',5'-Trichlorobiphenyl (BZ-34)	EPA 1668C_2010	1
2,3,6-Trichlorobiphenyl (BZ-24)	EPA 1668C_2010	1
2,3',6-Trichlorobiphenyl (BZ-27)	EPA 1668C_2010	1
2,3-Dichlorobiphenyl (BZ-5)	EPA 1668C_2010	1
2,3'-Dichlorobiphenyl (BZ-6)	EPA 1668C_2010	1
2,4,4',5-Tetrachlorobiphenyl (BZ-74)	EPA 1668C_2010	1
2,4,4',6-Tetrachlorobiphenyl (BZ-75)	EPA 1668C_2010	1
2,4,4'-Trichlorobiphenyl (BZ-28)	EPA 1668C_2010	1
2,4,5-Trichlorobiphenyl (BZ-29)	EPA 1668C_2010	1
2,4',5-Trichlorobiphenyl (BZ-31)	EPA 1668C_2010	1
2,4,6-Trichlorobiphenyl (BZ-30)	EPA 1668C_2010	1
2,4',6-Trichlorobiphenyl (BZ-32)	EPA 1668C_2010	1
2,4-Dichlorobiphenyl (BZ-7)	EPA 1668C_2010	1
2,4'-Dichlorobiphenyl (BZ-8)	EPA 1668C_2010	1
2,5-Dichlorobiphenyl (BZ-9)	EPA 1668C_2010	1
2,6-Dichlorobiphenyl (BZ-10)	EPA 1668C_2010	1
2-Chlorobiphenyl (BZ-1)	EPA 1668C_2010	1
3,3',4,4',5,5'-Hexachlorobiphenyl (BZ-169)	EPA 1668C_2010	1
3,3',4,4',5-Pentachlorobiphenyl (BZ-126)	EPA 1668C_2010	1
3,3',4,4'-Tetrachlorobiphenyl (BZ-77)	EPA 1668C_2010	1
3,3',4,5,5'-Pentachlorobiphenyl (BZ-127)	EPA 1668C_2010	1
3,3',4,5-Tetrachlorobiphenyl (BZ-78)	EPA 1668C_2010	1
3,3',4,5'-Tetrachlorobiphenyl (BZ-79)	EPA 1668C_2010	1
3,3',4-Trichlorobiphenyl (BZ-35)	EPA 1668C_2010	1
3,3',5,5'-Tetrachlorobiphenyl (BZ-80)	EPA 1668C_2010	1
3,3',5-Trichlorobiphenyl (BZ-36)	EPA 1668C_2010	1
3,3'-Dichlorobiphenyl (BZ-11)	EPA 1668C_2010	1
3,4,4',5-Tetrachlorobiphenyl (BZ-81)	EPA 1668C_2010	1
3,4,4'-Trichlorobiphenyl (BZ-37)	EPA 1668C_2010	1
3,4,5-Trichlorobiphenyl (BZ-38)	EPA 1668C_2010	1
3,4',5-Trichlorobiphenyl (BZ-39)	EPA 1668C_2010	1
3,4-Dichlorobiphenyl (BZ-12)	EPA 1668C_2010	1
3,4'-Dichlorobiphenyl (BZ-13)	EPA 1668C_2010	1
3,5-Dichlorobiphenyl (BZ-14)	EPA 1668C_2010	1
3-Chlorobiphenyl (BZ-2)	EPA 1668C_2010	1

Matrix/Analyte	Method	Notes
4,4'-Dichlorobiphenyl (BZ-15)	EPA 1668C_2010	1
4-Chlorobiphenyl (BZ-3)	EPA 1668C_2010	1
Decachlorobiphenyl (BZ-209)	EPA 1668C_2010	1
Total Dichlorobiphenyls	EPA 1668C_2010	1
Total Heptachlorobiphenyls	EPA 1668C_2010	1
Total Hexachlorobiphenyls	EPA 1668C_2010	1
Total Monochlorobiphenyls	EPA 1668C_2010	1
Total Nonachlorobiphenyls	EPA 1668C_2010	1
Total Octachlorobiphenyls	EPA 1668C_2010	1
Total Pentachlorobiphenyls	EPA 1668C_2010	1
Total Tetrachlorobiphenyls	EPA 1668C_2010	1
Total Trichlorobiphenyls	EPA 1668C_2010	1

Accredited Parameter Note Detail

(1) Accreditation based in part on recognition of CALA accreditation.

05/16/2014

Authentication Signature
Alan D. Rue, Lab Accreditation Unit Supervisor

Date

Matrix/Analyte	Method	Notes
Heptachlor epoxide	AXYS MLA-028	1
Hexachlorobenzene	AXYS MLA-028	1
Methoxychlor	AXYS MLA-028	1
Mirex	AXYS MLA-028	1
Oxychlordane	AXYS MLA-028	1
trans-Nonachlor	AXYS MLA-028	1
1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	EPA 1613_1994	1
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	EPA 1613_1994	1
1,2,3,4,6,7,8-Heptachlorodibenzofuran	EPA 1613_1994	1
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	EPA 1613_1994	1
1,2,3,4,7,8,9-Heptachlorodibenzofuran	EPA 1613_1994	1
1,2,3,4,7,8-Hxcdd	EPA 1613_1994	1
1,2,3,4,7,8-Hxcdf	EPA 1613_1994	1
1,2,3,6,7,8-Hxcdd	EPA 1613_1994	1
1,2,3,6,7,8-Hxcdf	EPA 1613_1994	1
1,2,3,7,8,9-Hxcdd	EPA 1613_1994	1
1,2,3,7,8,9-Hxcdf	EPA 1613_1994	1
1,2,3,7,8-Pecdd	EPA 1613_1994	1
1,2,3,7,8-Pecdf	EPA 1613_1994	1
2,3,4,6,7,8-Hxcdf	EPA 1613_1994	1
2,3,4,7,8-Pecdf	EPA 1613_1994	1
2,3,7,8-TCDD	EPA 1613_1994	1
2,3,7,8-TCDF	EPA 1613_1994	1
2,2',3,3',4,4',5,5',6-Nonabromodiphenylether (BDE-206)	EPA 1614A_2010	1
2,2',3,3',4,4',5,6,6'-Nonabromodiphenylether (BDE-207)	EPA 1614A_2010	1
2,2',3,3',4,5,5',6,6'-Nonabromodiphenylether (BDE-208)	EPA 1614A_2010	1
2,2',3,4,4',5,6-Heptabromodiphenylether (BDE-181)	EPA 1614A_2010	1
2,2',3,4,4',5',6-Heptabromodiphenylether (BDE-183)	EPA 1614A_2010	1
2,2',3,4,4',6'-Hexabromodiphenylether (BDE-140)	EPA 1614A_2010	1
2,2',3,4,4'-Pentabromodiphenylether (BDE-85)	EPA 1614A_2010	1
2,2',4,4',5,5'-Hexabromodiphenyl ether (BDE-153)	EPA 1614A_2010	1
2,2',4,4',5',6-Hexabromodiphenylether (BDE-154)	EPA 1614A_2010	1
2,2',4,4',5-Pentabromodiphenyl ether (BDE-99)	EPA 1614A_2010	1
2,2',4,4',6,6'-Hexabromodiphenylether (BDE-155)	EPA 1614A_2010	1
2,2',4,4',6-Pentabromodiphenyl ether (BDE-100)	EPA 1614A_2010	1
2,2',4,4'-Tetrabromodiphenyl ether (BDE-47)	EPA 1614A_2010	1

Matrix/Analyte	Method	Notes
2,2',4,5'-Tetrabromodiphenylether (BDE-49)	EPA 1614A_2010	1
2,2',4-Tribromodiphenylether (BDE-17)	EPA 1614A_2010	1
2,3,3',4,4',5,6-Heptabromodiphenylether (BDE-190)	EPA 1614A_2010	1
2,3,3',4,4'-Pentabromodiphenylether (BDE-105)	EPA 1614A_2010	1
2,3,4,4',5,6-Hexabromodiphenylether (BDE-166)	EPA 1614A_2010	1
2,3',4,4',6-Pentabromodiphenylether (BDE-119)	EPA 1614A_2010	1
2,3',4,4'-Tetrabromodiphenylether (BDE-66)	EPA 1614A_2010	1
2,3,4,5,6-Pentabromodiphenylether (BDE-116)	EPA 1614A_2010	1
2',3,4,-Tribromodiphenylether (BDE-33)	EPA 1614A_2010	1
2,3',4-Tribromodiphenylether (BDE-25)	EPA 1614A_2010	1
2,4,4',6-Tetrabromodiphenylether (BDE-75)	EPA 1614A_2010	1
2,4,4'-Tribromodiphenylether (BDE-28)	EPA 1614A_2010	1
2,4,6-Tribromodiphenylether (BDE-30)	EPA 1614A_2010	1
2,4-Dibromodiphenylether (BDE-7)	EPA 1614A_2010	1
2,4'-Dibromodiphenylether (BDE-8)	EPA 1614A_2010	1
2,6-Dibromodiphenylether (BDE-10)	EPA 1614A_2010	1
3,3',4,4',5-Pentabromodiphenylether (BDE-126)	EPA 1614A_2010	1
3,3',4,4'-Tetrabromodiphenylether (BDE-77)	EPA 1614A_2010	1
3,3',4-Tribromodiphenylether (BDE-35)	EPA 1614A_2010	1
3,3'-Dibromodiphenylether (BDE-11)	EPA 1614A_2010	1
3,4,4'-Tribromodiphenylether (BDE-37)	EPA 1614A_2010	1
3,4-Dibromodiphenylether (BDE-12)	EPA 1614A_2010	1
3,4'-Dibromodiphenylether (BDE-13)	EPA 1614A_2010	1
4,4'-Dibromodiphenylether (BDE-15)	EPA 1614A_2010	1
Decabromodiphenylether (BDE-209)	EPA 1614A_2010	1
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ-206)	EPA 1668C_2010	1
2,2',3,3',4,4',5,5'-Octachlorobiphenyl (BZ-194)	EPA 1668C_2010	1
2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl (BZ-207	EPA 1668C_2010	1
2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ-195)	EPA 1668C_2010	1
2,2',3,3',4,4',5,6'-Octachlorobiphenyl (BZ-196)	EPA 1668C_2010	1
2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ-170)	EPA 1668C_2010	1
2,2',3,3',4,4',6,6'-Octachlorobiphenyl (BZ-197)	EPA 1668C_2010	1
2,2',3,3',4,4',6-Heptachlorobiphenyl (BZ-171)	EPA 1668C_2010	1
2,2',3,3',4,4'-Hexachlorobiphenyl (BZ-128)	EPA 1668C_2010	1
2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl (BZ-208	EPA 1668C_2010	1
2,2',3,3',4,5,5',6-Octachlorobiphenyl (BZ-198)	EPA 1668C_2010	1

Matrix/Analyte	Method	Notes
2,2',3,3',4,5,5',6'-Octachlorobiphenyl (BZ-199)	EPA 1668C_2010	1
2,2',3,3',4,5,5'-Heptachlorobiphenyl (BZ-172)	EPA 1668C_2010	1
2,2',3,3',4,5,6,6'-Octachlorobiphenyl (BZ-200)	EPA 1668C_2010	1
2,2',3,3',4,5',6,6'-Octachlorobiphenyl (BZ-201)	EPA 1668C_2010	1
2,2',3,3',4,5,6-Heptachlorobiphenyl (BZ-173)	EPA 1668C_2010	1
2,2',3,3',4,5,6'-Heptachlorobiphenyl (BZ-174)	EPA 1668C_2010	1
2,2',3,3',4,5',6-Heptachlorobiphenyl (BZ-175)	EPA 1668C_2010	1
2,2',3,3',4,5',6'-Heptachlorobiphenyl (BZ-177)	EPA 1668C_2010	1
2,2',3,3',4,5-Hexachlorobiphenyl (BZ-129)	EPA 1668C_2010	1
2,2',3,3',4,5'-Hexachlorobiphenyl (BZ-130)	EPA 1668C_2010	1
2,2',3,3',4,6,6'-Heptachlorobiphenyl (BZ-176)	EPA 1668C_2010	1
2,2',3,3',4,6-Hexachlorobiphenyl (BZ-131)	EPA 1668C_2010	1
2,2',3,3',4,6'-Hexachlorobiphenyl (BZ-132)	EPA 1668C_2010	1
2,2',3,3',4-Pentachlorobiphenyl (BZ-82)	EPA 1668C_2010	1
2,2',3,3',5,5',6,6'-Octachlorobiphenyl (BZ-202)	EPA 1668C_2010	1
2,2',3,3',5,5',6-Heptachlorobiphenyl (BZ-178)	EPA 1668C_2010	1
2,2',3,3',5,5'-Hexachlorobiphenyl (BZ-133)	EPA 1668C_2010	1
2,2',3,3',5,6,6'-Heptachlorobiphenyl (BZ-179)	EPA 1668C_2010	1
2,2',3,3',5,6-Hexachlorobiphenyl (BZ-134)	EPA 1668C_2010	1
2,2',3,3',5,6'-Hexachlorobiphenyl (BZ-135)	EPA 1668C_2010	1
2,2',3,3',5-Pentachlorobiphenyl (BZ-83)	EPA 1668C_2010	1
2,2',3,3',6,6'-Hexachlorobiphenyl (BZ-136)	EPA 1668C_2010	1
2,2',3,3',6-Pentachlorobiphenyl (BZ-84)	EPA 1668C_2010	1
2,2',3,3'-Tetrachlorobiphenyl (BZ-40)	EPA 1668C_2010	1
2,2',3,4,4',5,5',6-Octachlorobiphenyl (BZ-203)	EPA 1668C_2010	1
2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ-180)	EPA 1668C_2010	1
2,2',3,4,4',5,6,6'-Octachlorobiphenyl (BZ-204)	EPA 1668C_2010	1
2,2',3,4,4',5,6-Heptachlorobiphenyl (BZ-181)	EPA 1668C_2010	1
2,2',3,4,4',5,6'-Heptachlorobiphenyl (BZ-182)	EPA 1668C_2010	1
2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ-183)	EPA 1668C_2010	1
2,2',3,4,4',5-Hexachlorobiphenyl (BZ-137)	EPA 1668C_2010	1
2,2',3,4,4',5'-Hexachlorobiphenyl (BZ-138)	EPA 1668C_2010	1
2,2',3,4,4',6,6'-Heptachlorobiphenyl (BZ-184)	EPA 1668C_2010	1
2,2',3,4,4',6-Hexachlorobiphenyl (BZ-139)	EPA 1668C_2010	1
2,2',3,4,4',6'-Hexachlorobiphenyl (BZ-140)	EPA 1668C_2010	1
2,2',3,4,4'-Pentachlorobiphenyl (BZ-85)	EPA 1668C_2010	1

Matrix/Analyte	Method	Notes
2,2',3,4,5,5',6-Heptachlorobiphenyl (BZ-185)	EPA 1668C_2010	1
2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ-187)	EPA 1668C_2010	1
2,2',3,4,5,5'-Hexachlorobiphenyl (BZ-141)	EPA 1668C_2010	1
2,2',3,4',5,5'-Hexachlorobiphenyl (BZ-146)	EPA 1668C_2010	1
2,2',3,4,5,6,6'-Heptachlorobiphenyl (BZ-186)	EPA 1668C_2010	1
2,2',3,4',5,6,6'-Heptachlorobiphenyl (BZ-188)	EPA 1668C_2010	1
2,2',3,4,5,6-Hexachlorobiphenyl (BZ-142)	EPA 1668C_2010	1
2,2',3,4,5,6'-Hexachlorobiphenyl (BZ-143)	EPA 1668C_2010	1
2,2',3,4,5',6-Hexachlorobiphenyl (BZ-144)	EPA 1668C_2010	1
2,2',3,4',5,6-Hexachlorobiphenyl (BZ-147)	EPA 1668C_2010	1
2,2',3,4',5,6'-Hexachlorobiphenyl (BZ-148)	EPA 1668C_2010	1
2,2',3,4',5',6-Hexachlorobiphenyl (BZ-149)	EPA 1668C_2010	1
2,2',3,4,5-Pentachlorobiphenyl (BZ-86)	EPA 1668C_2010	1
2,2',3,4,5'-Pentachlorobiphenyl (BZ-87)	EPA 1668C_2010	1
2,2',3,4',5-Pentachlorobiphenyl (BZ-90)	EPA 1668C_2010	1
2,2',3,4',5'-Pentachlorobiphenyl (BZ-97)	EPA 1668C_2010	1
2,2',3,4,6,6'-Hexachlorobiphenyl (BZ-145)	EPA 1668C_2010	1
2,2',3,4',6,6'-Hexachlorobiphenyl (BZ-150)	EPA 1668C_2010	1
2,2',3,4,6-Pentachlorobiphenyl (BZ-88)	EPA 1668C_2010	1
2,2',3,4,6'-Pentachlorobiphenyl (BZ-89)	EPA 1668C_2010	1
2,2',3,4',6-Pentachlorobiphenyl (BZ-91)	EPA 1668C_2010	1
2,2',3,4',6'-Pentachlorobiphenyl (BZ-98)	EPA 1668C_2010	1
2,2',3,4-Tetrachlorobiphenyl (BZ-41)	EPA 1668C_2010	1
2,2',3,4'-Tetrachlorobiphenyl (BZ-42)	EPA 1668C_2010	1
2,2',3,5,5',6-Hexachlorobiphenyl (BZ-151)	EPA 1668C_2010	1
2,2',3,5,5'-Pentachlorobiphenyl (BZ-92)	EPA 1668C_2010	1
2,2',3,5,6,6'-Hexachlorobiphenyl (BZ-152)	EPA 1668C_2010	1
2,2',3,5,6-Pentachlorobiphenyl (BZ-93)	EPA 1668C_2010	1
2,2',3,5,6'-Pentachlorobiphenyl (BZ-94)	EPA 1668C_2010	1
2,2',3,5',6-Pentachlorobiphenyl (BZ-95)	EPA 1668C_2010	1
2,2',3,5-Tetrachlorobiphenyl (BZ-43)	EPA 1668C_2010	1
2,2',3,5'-Tetrachlorobiphenyl (BZ-44)	EPA 1668C_2010	1
2,2',3,6,6'-Pentachlorobiphenyl (BZ-96)	EPA 1668C_2010	1
2,2',3,6-Tetrachlorobiphenyl (BZ-45)	EPA 1668C_2010	1
2,2',3,6'-Tetrachlorobiphenyl (BZ-46)	EPA 1668C_2010	1
2,2',3-Trichlorobiphenyl (BZ-16)	EPA 1668C_2010	1

Matrix/Analyte	Method	Notes
2,2',4,4',5,5'-Hexachlorobiphenyl (BZ-153)	EPA 1668C_2010	1
2,2',4,4',5,6'-Hexachlorobiphenyl (BZ-154)	EPA 1668C_2010	1
2,2',4,4',5-Pentachlorobiphenyl (BZ-99)	EPA 1668C_2010	1
2,2',4,4',6,6'-Hexachlorobiphenyl (BZ-155)	EPA 1668C_2010	1
2,2',4,4',6-Pentachlorobiphenyl (BZ-100)	EPA 1668C_2010	1
2,2',4,4'-Tetrachlorobiphenyl (BZ-47)	EPA 1668C_2010	1
2,2',4,5,5'-Pentachlorobiphenyl (BZ-101)	EPA 1668C_2010	1
2,2',4,5,6'-Pentachlorobiphenyl (BZ-102)	EPA 1668C_2010	1
2,2',4,5',6-Pentachlorobiphenyl (BZ-103)	EPA 1668C_2010	1
2,2',4,5-Tetrachlorobiphenyl (BZ-48)	EPA 1668C_2010	1
2,2',4,5'-Tetrachlorobiphenyl (BZ-49)	EPA 1668C_2010	1
2,2',4,6,6'-Pentachlorobiphenyl (BZ-104)	EPA 1668C_2010	1
2,2',4,6-Tetrachlorobiphenyl (BZ-50)	EPA 1668C_2010	1
2,2',4,6'-Tetrachlorobiphenyl (BZ-51)	EPA 1668C_2010	1
2,2',4-Trichlorobiphenyl (BZ-17)	EPA 1668C_2010	1
2,2',5,5'-Tetrachlorobiphenyl (BZ-52)	EPA 1668C_2010	1
2,2',5,6'-Tetrachlorobiphenyl (BZ-53)	EPA 1668C_2010	1
2,2',5-Trichlorobiphenyl (BZ-18)	EPA 1668C_2010	1
2,2',6,6'-Tetrachlorobiphenyl (BZ-54)	EPA 1668C_2010	1
2,2',6-Trichlorobiphenyl (BZ-19)	EPA 1668C_2010	1
2,2'-Dichlorobiphenyl (BZ-4)	EPA 1668C_2010	1
2,3,3',4,4',5,5',6-Octachlorobiphenyl (BZ-205)	EPA 1668C_2010	1
2,3,3',4,4',5,5'-Heptachlorobiphenyl (BZ-189)	EPA 1668C_2010	1
2,3,3',4,4',5,6-Heptachlorobiphenyl (BZ-190)	EPA 1668C_2010	1
2,3,3',4,4',5',6-Heptachlorobiphenyl (BZ-191)	EPA 1668C_2010	1
2,3,3',4,4',5-Hexachlorobiphenyl (BZ-156)	EPA 1668C_2010	1
2,3,3',4,4',5'-Hexachlorobiphenyl (BZ-157)	EPA 1668C_2010	1
2,3,3',4,4',6-Hexachlorobiphenyl (BZ-158)	EPA 1668C_2010	1
2,3,3',4,4'-Pentachlorobiphenyl (BZ-105)	EPA 1668C_2010	1
2,3,3',4,5,5',6-Heptachlorobiphenyl (BZ-192)	EPA 1668C_2010	1
2,3,3',4',5,5',6-Heptachlorobiphenyl (BZ-193)	EPA 1668C_2010	1
2,3,3',4,5,5'-Hexachlorobiphenyl (BZ-159)	EPA 1668C_2010	1
2,3,3',4',5,5'-Hexachlorobiphenyl (BZ-162)	EPA 1668C_2010	1
2,3,3',4,5,6-Hexachlorobiphenyl (BZ-160)	EPA 1668C_2010	1
2,3,3',4',5,6-Hexachlorobiphenyl (BZ-163)	EPA 1668C_2010	1
2,3,3',4',5',6-Hexachlorobiphenyl (BZ-164)	EPA 1668C_2010	1

Matrix/Analyte	Method	Notes
2,3,3',4,5',6-Hexachlorobiphenyl (BZ-161)	EPA 1668C_2010	1
2,3,3',4,5-Pentachlorobiphenyl (BZ-106)	EPA 1668C_2010	1
2,3,3',4',5-Pentachlorobiphenyl (BZ-107)	EPA 1668C_2010	1
2,3,3',4,5'-Pentachlorobiphenyl (BZ-108)	EPA 1668C_2010	1
2,3,3',4',5'-Pentachlorobiphenyl (BZ-122)	EPA 1668C_2010	1
2,3,3',4,6-Pentachlorobiphenyl (BZ-109)	EPA 1668C_2010	1
2,3,3',4',6-Pentachlorobiphenyl (BZ-110)	EPA 1668C_2010	1
2,3,3',4-Tetrachlorobiphenyl (BZ-55)	EPA 1668C_2010	1
2,3,3',4'-Tetrachlorobiphenyl (BZ-56)	EPA 1668C_2010	1
2,3,3',5,5',6-Hexachlorobiphenyl (BZ-165)	EPA 1668C_2010	1
2,3,3',5,5'-Pentachlorobiphenyl (BZ-111)	EPA 1668C_2010	1
2,3,3',5,6-Pentachlorobiphenyl (BZ-112)	EPA 1668C_2010	1
2,3,3',5',6-Pentachlorobiphenyl (BZ-113)	EPA 1668C_2010	1
2,3,3',5-Tetrachlorobiphenyl (BZ-57)	EPA 1668C_2010	1
2,3,3',5'-Tetrachlorobiphenyl (BZ-58)	EPA 1668C_2010	1
2,3,3',6-Tetrachlorobiphenyl (BZ-59)	EPA 1668C_2010	1
2,3,3'-Trichlorobiphenyl (BZ-20)	EPA 1668C_2010	1
2,3',4,4',5,5'-Hexachlorobiphenyl (BZ-167)	EPA 1668C_2010	1
2,3,4,4',5,6-Hexachlorobiphenyl (BZ-166)	EPA 1668C_2010	1
2,3',4,4',5',6-Hexachlorobiphenyl (BZ-168)	EPA 1668C_2010	1
2,3,4,4',5-Pentachlorobiphenyl (BZ-114)	EPA 1668C_2010	1
2,3',4,4',5-Pentachlorobiphenyl (BZ-118)	EPA 1668C_2010	1
2,3',4,4',5'-Pentachlorobiphenyl (BZ-123)	EPA 1668C_2010	1
2,3,4,4',6-Pentachlorobiphenyl (BZ-115)	EPA 1668C_2010	1
2,3',4,4',6-Pentachlorobiphenyl (BZ-119)	EPA 1668C_2010	1
2,3,4,4'-Tetrachlorobiphenyl (BZ-60)	EPA 1668C_2010	1
2,3',4,4'-Tetrachlorobiphenyl (BZ-66)	EPA 1668C_2010	1
2,3',4,5,5'-Pentachlorobiphenyl (BZ-120)	EPA 1668C_2010	1
2,3',4',5,5'-Pentachlorobiphenyl (BZ-124)	EPA 1668C_2010	1
2,3,4,5,6-Pentachlorobiphenyl (BZ-116)	EPA 1668C_2010	1
2,3,4',5,6-Pentachlorobiphenyl (BZ-117)	EPA 1668C_2010	1
2,3',4,5',6-Pentachlorobiphenyl (BZ-121)	EPA 1668C_2010	1
2,3',4',5',6-Pentachlorobiphenyl (BZ-125)	EPA 1668C_2010	1
2,3,4,5-Tetrachlorobiphenyl (BZ-61)	EPA 1668C_2010	1
2,3,4',5-Tetrachlorobiphenyl (BZ-63)	EPA 1668C_2010	1
2,3',4,5'-Tetrachlorobiphenyl (BZ-68)	EPA 1668C_2010	1

Matrix/Analyte	Method	Notes
2,3',4',5-Tetrachlorobiphenyl (BZ-70)	EPA 1668C_2010	1
2,3',4',5'-Tetrachlorobiphenyl (BZ-76)	EPA 1668C_2010	1
2,3',4,5-Tetrachlorobiphenyl (BZ-67)	EPA 1668C_2010	1
2,3,4,6-Tetrachlorobiphenyl (BZ-62)	EPA 1668C_2010	1
2,3,4',6-Tetrachlorobiphenyl (BZ-64)	EPA 1668C_2010	1
2,3',4,6-Tetrachlorobiphenyl (BZ-69)	EPA 1668C_2010	1
2,3',4',6-Tetrachlorobiphenyl (BZ-71)	EPA 1668C_2010	1
2,3,4-Trichlorobiphenyl (BZ-21)	EPA 1668C_2010	1
2,3,4'-Trichlorobiphenyl (BZ-22)	EPA 1668C_2010	1
2,3',4-Trichlorobiphenyl (BZ-25)	EPA 1668C_2010	1
2,3',4'-Trichlorobiphenyl (BZ-33)	EPA 1668C_2010	1
2,3',5,5'-Tetrachlorobiphenyl (BZ-72)	EPA 1668C_2010	1
2,3,5,6-Tetrachlorobiphenyl (BZ-65)	EPA 1668C_2010	1
2,3',5',6-Tetrachlorobiphenyl (BZ-73)	EPA 1668C_2010	1
2,3,5-Trichlorobiphenyl (BZ-23)	EPA 1668C_2010	1
2,3',5-Trichlorobiphenyl (BZ-26)	EPA 1668C_2010	1
2,3',5'-Trichlorobiphenyl (BZ-34)	EPA 1668C_2010	1
2,3,6-Trichlorobiphenyl (BZ-24)	EPA 1668C_2010	1
2,3',6-Trichlorobiphenyl (BZ-27)	EPA 1668C_2010	1
2,3-Dichlorobiphenyl (BZ-5)	EPA 1668C_2010	1
2,3'-Dichlorobiphenyl (BZ-6)	EPA 1668C_2010	1
2,4,4',5-Tetrachlorobiphenyl (BZ-74)	EPA 1668C_2010	1
2,4,4',6-Tetrachlorobiphenyl (BZ-75)	EPA 1668C_2010	1
2,4,4'-Trichlorobiphenyl (BZ-28)	EPA 1668C_2010	1
2,4,5-Trichlorobiphenyl (BZ-29)	EPA 1668C_2010	1
2,4',5-Trichlorobiphenyl (BZ-31)	EPA 1668C_2010	1
2,4,6-Trichlorobiphenyl (BZ-30)	EPA 1668C_2010	1
2,4',6-Trichlorobiphenyl (BZ-32)	EPA 1668C_2010	1
2,4-Dichlorobiphenyl (BZ-7)	EPA 1668C_2010	1
2,4'-Dichlorobiphenyl (BZ-8)	EPA 1668C_2010	1
2,5-Dichlorobiphenyl (BZ-9)	EPA 1668C_2010	1
2,6-Dichlorobiphenyl (BZ-10)	EPA 1668C_2010	1
2-Chlorobiphenyl (BZ-1)	EPA 1668C_2010	1
3,3',4,4',5,5'-Hexachlorobiphenyl (BZ-169)	EPA 1668C_2010	1
3,3',4,4',5-Pentachlorobiphenyl (BZ-126)	EPA 1668C_2010	1
3,3',4,4'-Tetrachlorobiphenyl (BZ-77)	EPA 1668C_2010	1

Matrix/Analyte	Method	Notes
3,3',4,5,5'-Pentachlorobiphenyl (BZ-127)	EPA 1668C_2010	1
3,3',4,5-Tetrachlorobiphenyl (BZ-78)	EPA 1668C_2010	1
3,3',4,5'-Tetrachlorobiphenyl (BZ-79)	EPA 1668C_2010	1
3,3',4-Trichlorobiphenyl (BZ-35)	EPA 1668C_2010	1
3,3',5,5'-Tetrachlorobiphenyl (BZ-80)	EPA 1668C_2010	1
3,3',5-Trichlorobiphenyl (BZ-36)	EPA 1668C_2010	1
3,3'-Dichlorobiphenyl (BZ-11)	EPA 1668C_2010	1
3,4,4',5-Tetrachlorobiphenyl (BZ-81)	EPA 1668C_2010	1
3,4,4'-Trichlorobiphenyl (BZ-37)	EPA 1668C_2010	1
3,4,5-Trichlorobiphenyl (BZ-38)	EPA 1668C_2010	1
3,4',5-Trichlorobiphenyl (BZ-39)	EPA 1668C_2010	1
3,4-Dichlorobiphenyl (BZ-12)	EPA 1668C_2010	1
3,4'-Dichlorobiphenyl (BZ-13)	EPA 1668C_2010	1
3,5-Dichlorobiphenyl (BZ-14)	EPA 1668C_2010	1
3-Chlorobiphenyl (BZ-2)	EPA 1668C_2010	1
4,4'-Dichlorobiphenyl (BZ-15)	EPA 1668C_2010	1
4-Chlorobiphenyl (BZ-3)	EPA 1668C_2010	1
Decachlorobiphenyl (BZ-209)	EPA 1668C_2010	1
Total Dichlorobiphenyls	EPA 1668C_2010	1
Total Heptachlorobiphenyls	EPA 1668C_2010	1
Total Hexachlorobiphenyls	EPA 1668C_2010	1
Total Monochlorobiphenyls	EPA 1668C_2010	1
Total Nonachlorobiphenyls	EPA 1668C_2010	1
Total Octachlorobiphenyls	EPA 1668C_2010	1
Total Pentachlorobiphenyls	EPA 1668C_2010	1
Total Tetrachlorobiphenyls	EPA 1668C_2010	1
Total Trichlorobiphenyls	EPA 1668C_2010	1
Solid and Chemical Materials		
1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	EPA 1613_1994	1
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	EPA 1613_1994	1
1,2,3,4,6,7,8-Heptachlorodibenzofuran	EPA 1613_1994	1
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	EPA 1613_1994	1
1,2,3,4,7,8,9-Heptachlorodibenzofuran	EPA 1613_1994	1
1,2,3,4,7,8-Hxcdd	EPA 1613_1994	1
1,2,3,4,7,8-Hxcdf	EPA 1613_1994	1

The State of Washington
Department of Ecology

SVL Analytical, Incorporated
Kellogg, ID

has complied with provisions set forth in Chapter 173-50 WAC and is hereby recognized by the Department of Ecology as an ACCREDITED LABORATORY for the analytical parameters listed on the accompanying Scope of Accreditation. This certificate is effective May 17, 2014 and shall expire May 16, 2015.

Witnessed under my hand on May 13, 2014

Alan D. Rue
Lab Accreditation Unit Supervisor

Laboratory ID
C573

APPENDIX C

AXYS

STANDARD ANALYTICAL PROCEDURE

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.

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

The analysis of chlorinated pesticides, polybrominated diphenylethers, polychlorinated naphthalenes, and/or polybrominated biphenyls 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 AXYS method documents MLA-007[4], MLA-028[5], MLA-033[6,7] MLA-030[8] and MLA-026[9]. 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 by: _____ Date: _____
John Cosgrove, President and Senior Technical Director

Approved by: _____ Date: _____
Shea Hewage, Director of Operations

Approved by: _____ Date: _____
Dale Hoover, QA Manager

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. AXYS Method Document MLA-030 *Analytical Method for the Determination of Polychlorinated Naphthalenes (PCN) by High Resolution GC/MS.*

9. AXYS Method Document MLA-026. *Analytical Method for the Determination of Polybrominated Biphenyls (PBBs) by High Resolution GC/MS.*

10. 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)

CHANGES FROM PREVIOUS REVISIONS or VERSIONS

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.
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"

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.

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.

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.*

The analysis of polybrominated biphenyls is carried out using the extraction and cleanup procedures described in this method, as documented in AXYS Method Document MLA-026, *Analytical Method for the Determination of Polybrominated Biphenyls (PBBs) by High Resolution GC/MS.*

The analysis of chloronaphthalenes is carried out using the extraction and cleanup procedures described in this method, as documented in AXYS Method Document MLA-030, *Analytical Method for the Determination of Polychlorinated Naphthalenes (PCN) by High Resolution GC/MS*.

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) 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.

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.

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 (Teflon 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.

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.

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.

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
Magnetic stirring plate, with pre-cleaned PTFE coated magnetic stirring bars

Millipore Filtration Apparatus
Soxhlet apparatus (pre-cleaned) with heating mantle
Soxhlet/Dean-Stark apparatus (pre-cleaned)
Water bath capable of maintaining up to 50ºC
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).

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 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.

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.

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

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

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

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.

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).

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.

Hydrochloric Acid (1 M) is prepared by adding HCL (100 mL, conc,) to ultra pure water (1 L).

Potassium Hydroxide (KOH, 1M) is prepared by dissolving potassium hydroxide pellets (56 g, Fisher, certified) in ethanol (1000 mL).

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.

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 soaked in Contrad 70 cleaning solution, rinsed well with water and are baked overnight at a minimum of 300°C.

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

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 (Mallinckrodt, SilicAR 100-200 mesh) 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.

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, 2.1% 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).

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.

- 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.

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".

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	$\overline{X}$	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	-	-

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
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

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	$\bar{X}$	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	-	-

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
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

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.

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

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-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.

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 my 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. 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.

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. 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.

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).

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. 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 to 1 mL by rotary evaporation (water bath <30°C) and add hexane (1 mL) to the extract.

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).

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.

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).

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.

5.5.2 Extraction of Samples with ≤1% Solids

5.5.2.1 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 and dry over anhydrous sodium sulphate for at least 30 minutes. Transfer the extract to a round bottom flask with dichloromethane rinses. Add an aliquot of cleanup standard solution. Add hexane (2 mL) and concentrate the extract to 1-2 mL. 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 60°C). The extract is ready for chromatographic cleanup procedures (Section 6).

5.5.2.2 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 over anhydrous sodium sulphate for at least 30 minutes. Transfer the extract to a round bottom flask with dichloromethane rinses. Add an aliquot of cleanup standard solution. Add hexane (2 mL) and concentrate the extract to 1-2 mL (water bath 60°C). 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 60°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:

5.5.3.1 Default Centrifugation Procedure for Separation of Particulate

Note: the following procedure cannot be used if particulate weight is required.

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 over anhydrous sodium sulphate for at least 30 minutes.

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 and dry over anhydrous sodium sulphate for at least 30 minutes.

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). 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 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 60°). 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..

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

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

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.

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.

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.

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.

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).

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 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 (water bath <30°C). 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).

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. If brominated analytes (PBB) are also being determined the apparatus must be extracted with dichloromethane for a minimum of 3 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 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).

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.**

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.

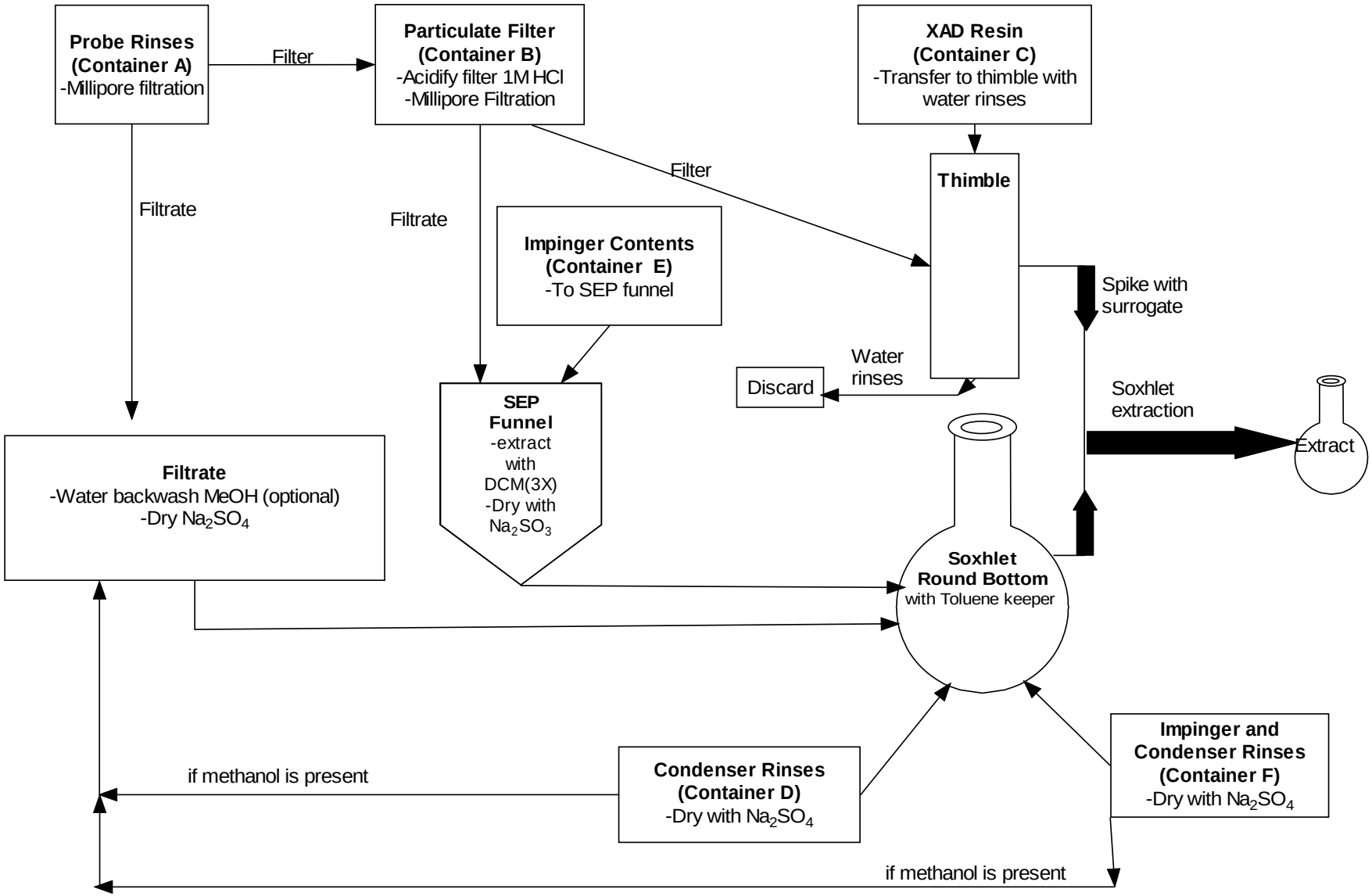
2. Remove water from the extract as follows:
   - If less than 3 mL of water is visible, quantitatively transfer the entire extract to an Erlenmeyer flask with toluene rinses.

- 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.

Figure 1: Processing of Sample Trains



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.

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 and/or polychlorinated naphthalenes analytes 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-033	Analytical Method for the Determination of Brominated Diphenylethers (PBDE) by EPA Method 1614
MLA-030	Analytical Method for the Determination of Polychlorinated Naphthalenes (PCN)

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 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.

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. Load the extract (in hexane) and rinse 3 times with the E1 elution solvent (15:85 dichloromethane:hexane) on a Florisil column (8 g, 2.1% deactivated) prepared as described in Section 3.6.4. Elute the column with 15:85 dichloromethane:hexane (E1) using the volume of solvent determined from the most recent Florisil cutpoint determination. 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.

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.

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:

SLA-008	Preparing Extracts for Instrumental Analysis
SLA-072	Computer Preparation of Labels

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

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.

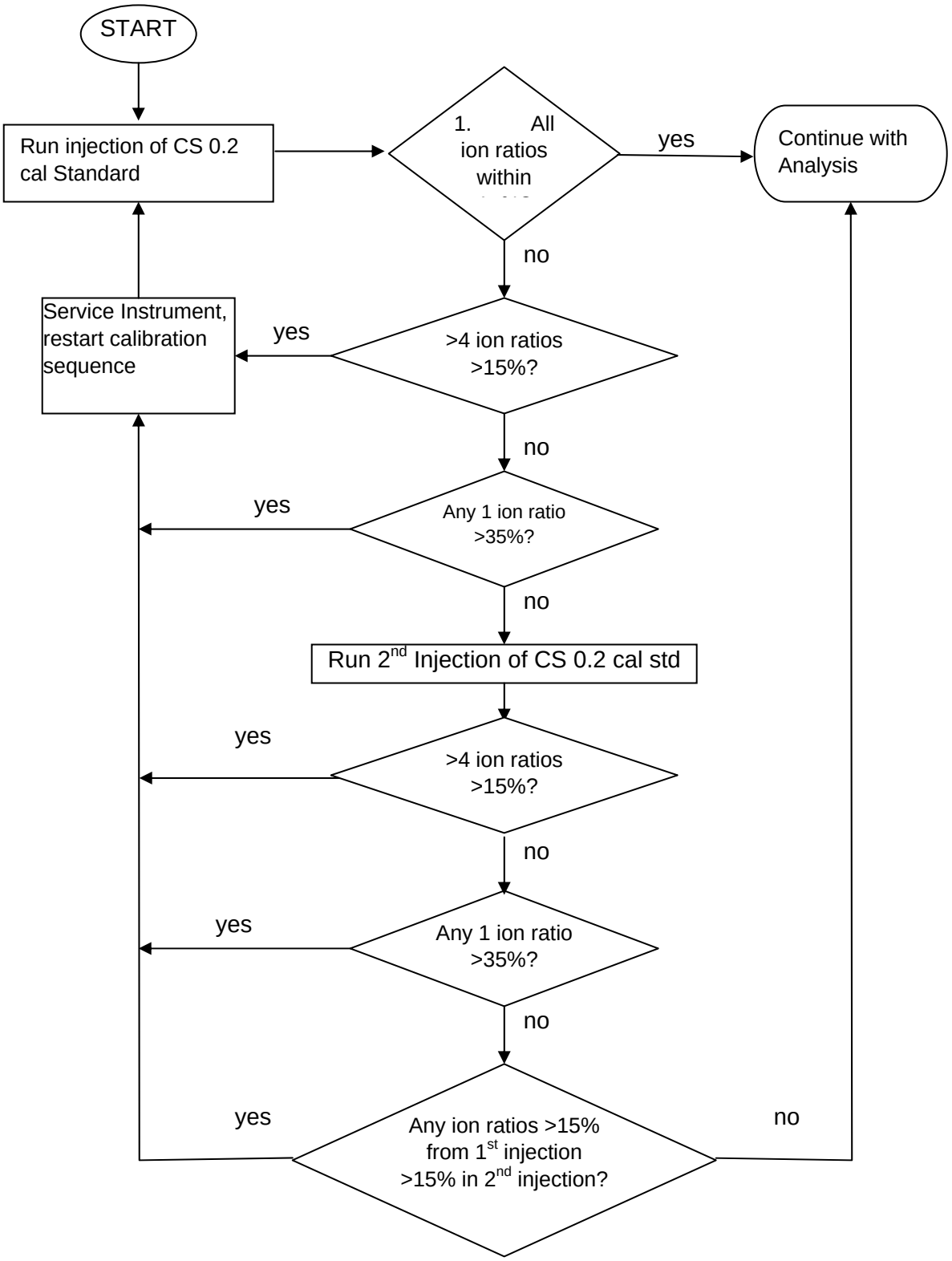
If all ion peak ratios are not within 15% of theoretical for the first injection of the CS-0.2 standard but the ion peak ratios for a maximum of four congeners differ from theoretical by no more than 35% and the ion ratios for all remaining congeners are within 15% of theoretical, then further evaluation 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.

Figure 2. Procedure for Ion Ratio Criteria for CS-0.2 Initial Calibration Standard



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.

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

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, 99 concentrations multiplied by 8.0 (for EPA 1668A); OR
The sum of PCBs 86/87/97/**109**/119/125, 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

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

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

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							

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

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							

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

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

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
22'344'5'6 - 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

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

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)

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

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

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

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 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 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 lower reporting limit for blood/serum/plasma samples is the SDL, for other matrices the lower reporting limit is 0.5 pg/sample (or as specified by client).

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 if is determined for the calculation of a dry weight.

Percent lipid is reported if determined on a sample.

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 2.1% 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

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.

$$Conc_i = \frac{A_i}{A_{si}} \times \frac{M_{si}}{RRF_{i,si}} \times \frac{1}{M_x}$$

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:

$$\cdot \frac{A_{si}}{A_{rs}} \times \frac{M_{rs}}{M_{si}}$$

APPENDIX A

OPTIONAL ANALYSES

Chlorinated Pesticides

Polybrominated Diphenylethers

Polybrominated Biphenyls

Polychlorinated Naphthalenes

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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, 2.1% 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

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).

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.

Polybrominated Biphenyls (PBB)

The analysis of polybrominated biphenyls may be carried out using the extraction procedure as described in Section 5 and the cleanup procedures described in Section 6. Sample matrices are spiked with the PCB surrogate, which is used for the quantification of PBBs. The analyst is referred to the most recent version of AXYS Method Document MLA-026. *Analytical Method for the Determination of Polybrominated Biphenyls (PBBs) by High Resolution GC/MS* for details regarding instrumental analysis, analyte identification and quantification procedures and QA/QC criteria.

Polychlorinated Naphthalenes (PCN)

The analysis of polychlorinated naphthalenes may be carried out using the extraction procedure as described in Section 5 of this document. 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-030, *Analysis Method for the Determination of Polychlorinated Naphthalenes by High Resolution GC/MS* for details regarding 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.

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

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

Figure B-1: Typical Chromatogram of a Well-defined Shoulder of PCB-126 on SPB-Octyl Column

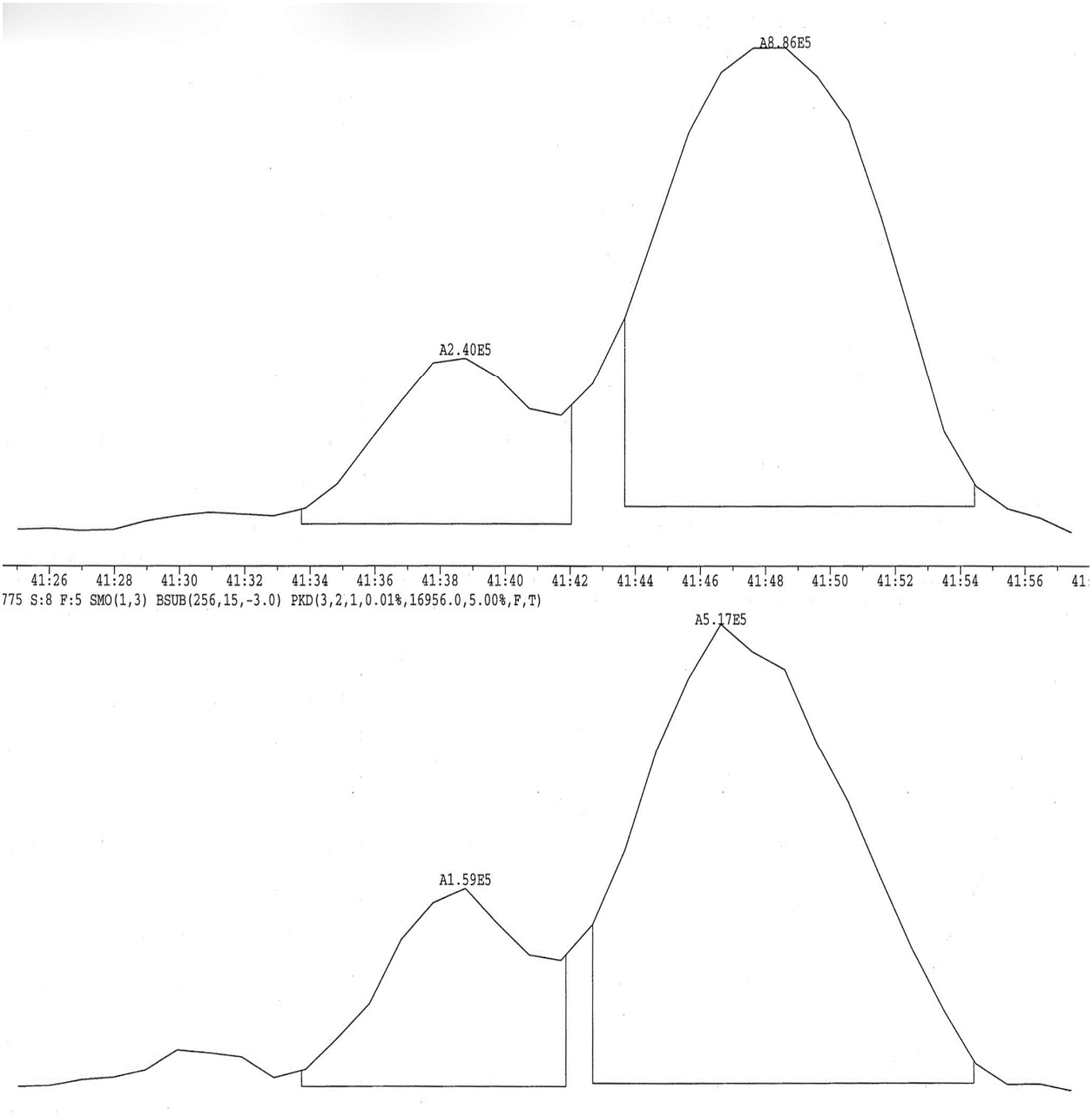
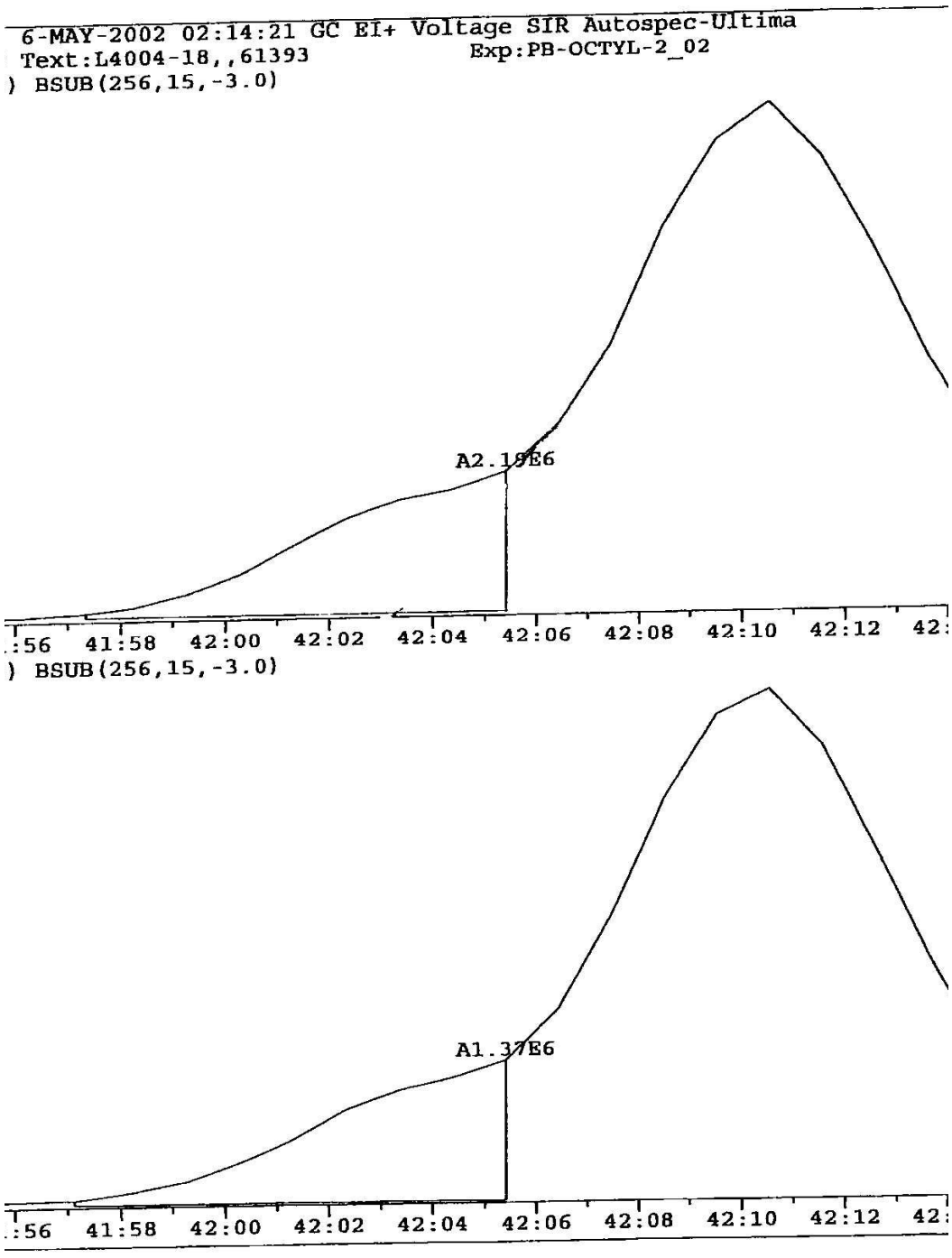


Figure B-2: Typical Chromatogram of a Less-defined Shoulder of PCB-126 on SPB-Octyl Column



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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. Tissue samples are extracted by Soxhlet extraction for 18-24 hours, rather than 16-20 hours.
3. Aqueous samples are extracted by liquid-liquid extraction using separatory funnels, rather that by magnetic stirring extraction.
4. Carbon column isolation of PCBs 77, 126, and 169 is required if detected. This requirement may be waived by contract.
5. Hepta-PCBs are quantified against ^{13}C-PCB 188 and ^{13}C-PCB 189 only.
6. Quantification reports include the signal:noise (S:N) ratio for all analytes and standards.
7. 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.

APPENDIX D

GLOSSARY

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Accuracy – An estimate of closeness of a measurement result to the true value.

Bias – The difference between the population mean and the true value.

Blank – A sample prepared to contain none of the analyte of interest.

Calibration – The process of establishing the relationship between the response of a measurement system and the value of the parameter being measured.

Check standard – A QC sample prepared independently of calibration standards and analyzed along with the samples to check the precision of the measurement system. A check standard can also be used to check the bias due to the way calibration is done. It is also called a lab control sample.

Data Quality Objectives Process – EPA's recommended systematic planning process when environmental data are used to decide between two opposing conditions (e.g., compliance or non-compliance with a standard).

Data validation – An analyte-specific and sample-specific process that extends the evaluation of data beyond data verification to determine the analytical quality of a specific data set. It involves a detailed examination of the data package using professional judgment to determine whether the MQOs for precision, bias, and sensitivity have been met.

Data verification – Examination of the data for errors or omissions and the QC results for compliance with acceptance criteria.

Duplicates – Two samples collected or measurements made at the same time and location, or two aliquots of the same sample prepared and analyzed in the same batch.

Estimated Detection Limit (limit of detection) – The concentration or amount of an analyte which, on an "a prior" basis, can be determined to a specified level of certainty to be greater than zero.

Estimated Quantitation Limit – Lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. EQLs are normally arbitrarily set rather than explicitly determined.

Field blank – A blank used to obtain information on contamination introduced during sample collection, storage, and transport.

Laboratory Control Sample (LCS) – See "Check Standard".

Matrix spike – A QC sample prepared by adding a known amount of the target analyte to an aliquot of a sample to check for bias due to interference or matrix effects.

Measurement Quality Objectives (MQOs) – The performance or acceptance criteria for individual data quality indicators, including precision, bias and sensitivity.

Measurement result – A value obtained by carrying out the procedure described in the method.

Method – A set of written instructions completely defining the procedure to be used.

Method blank – A blank prepared to represent the sample matrix and analyzed in a batch of samples.

Parameter – A specified characteristic of a population or sample.

Population – The hypothetical set of all possible observations of the type which is being investigated.

Precision – A measure of the variability in the results of replicate measurements due to random error.

Quality Assurance (QA) – Adherence to a system for assuring the reliability of measurement data.

Quality Assurance Project Plan (QAPP) – A document that describes the objectives of a project and the procedures necessary to acquire data that will serve those objectives.

Quality Control (QC) – The routine application of statistical procedures to evaluate and control the accuracy of measurement data.

Relative percent difference (RPD) – The difference between two values divided by their mean and multiplied by 100.

Replicates – Two or more samples collected or measurements made at the same time and place.

Reporting Limit -

Sensitivity – In general, denotes the rate at which the analytical response (e.g., absorbance, volume, meter reading) varies with the concentration of the parameter being determined. In a specialized sense, it has the same meaning as the detection limit.

Standard Operating Procedure (SOP) – A document that describes in detail the approved way for performing a routine procedure.