



Quality Assurance Project Plan

Spokane River Toxics Reduction Strategy Study

Prepared for:
Spokane River Regional
Toxics Task Force

Draft

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LimnoTech 

Water | Scientists
Environment | Engineers



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Quality Assurance Project Plan
May 1, 2014

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Sampling Contractor – will be added when selected

Date:_____



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DISTRIBUTION LIST (A.3)

QUALITY ASSURANCE PROJECT PLAN

DRAFT

MAY 1, 2014

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

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LAB2-will be added				
Sampling Contractor —will be added				



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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. The Sampling Contractor (to be determined) will be responsible for sample collection. AXYS Analytical Services will perform laboratory analysis for PCB congeners for the project as a contractor to SRRTTF. The LAB2 (to be determined) 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	SRRTTF 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 Technical 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	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 Manager and SRRTTF-ACE
Cynthia Tomey – AXYS Analytical Services	Laboratory Project Manager Serves as main point of contact for laboratory Assist Laboratory Technical Director with management of laboratory QA systems Communicates with Project Manager
Dale Hoover-AXYS Analytical Services	Laboratory QA/QC Managers Manages Laboratory QA/QC activities



Name/Affiliation	Project Title/Responsibility
	Reviews and verifies field records, laboratory records and laboratory data Addresses nonconformance and carries out corrective actions at the laboratory.
Lab2 – to be determined	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
Lab2 – to be determined	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.
Sampling Contractor-to be determined	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.



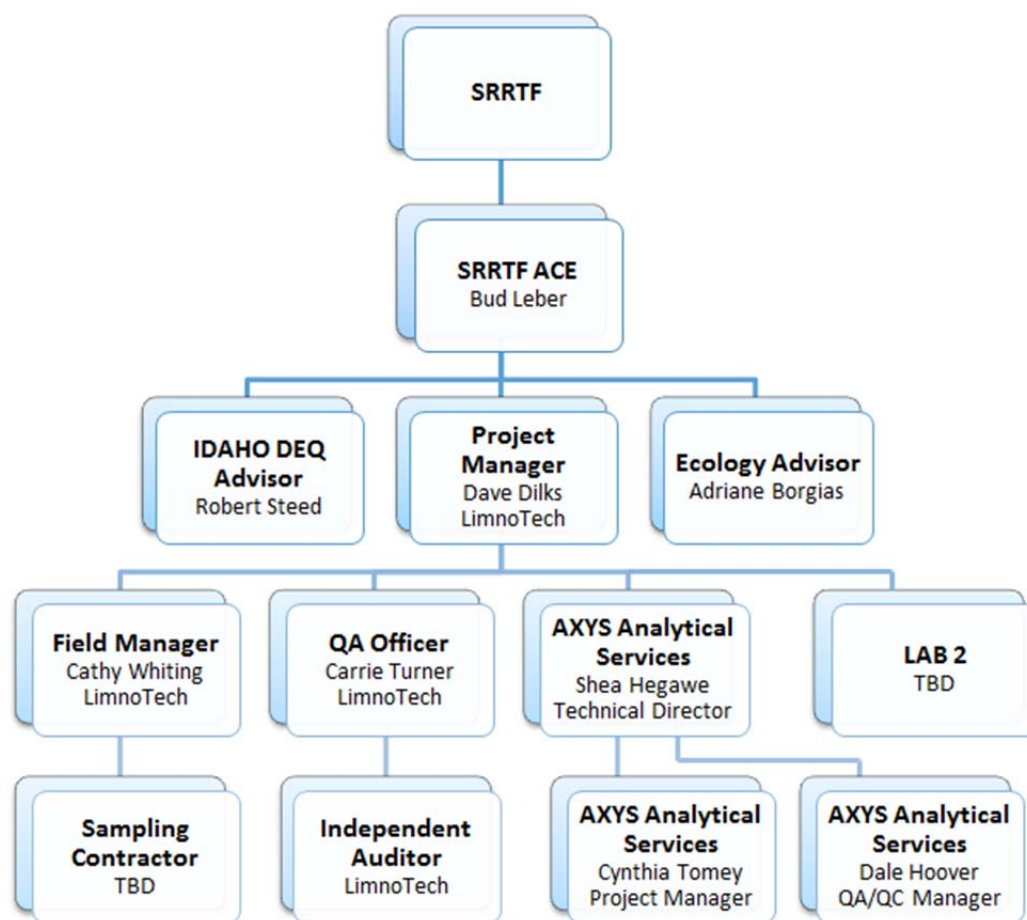


Figure 1. Project Team Organization

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 the Sampling CONTRACTOR (to be determined). 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 LAB2 (to be determined) 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) 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 (3.37 pg/L), based on a higher fish consumption rate than the general population (Serdar et al, 2013). 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 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.



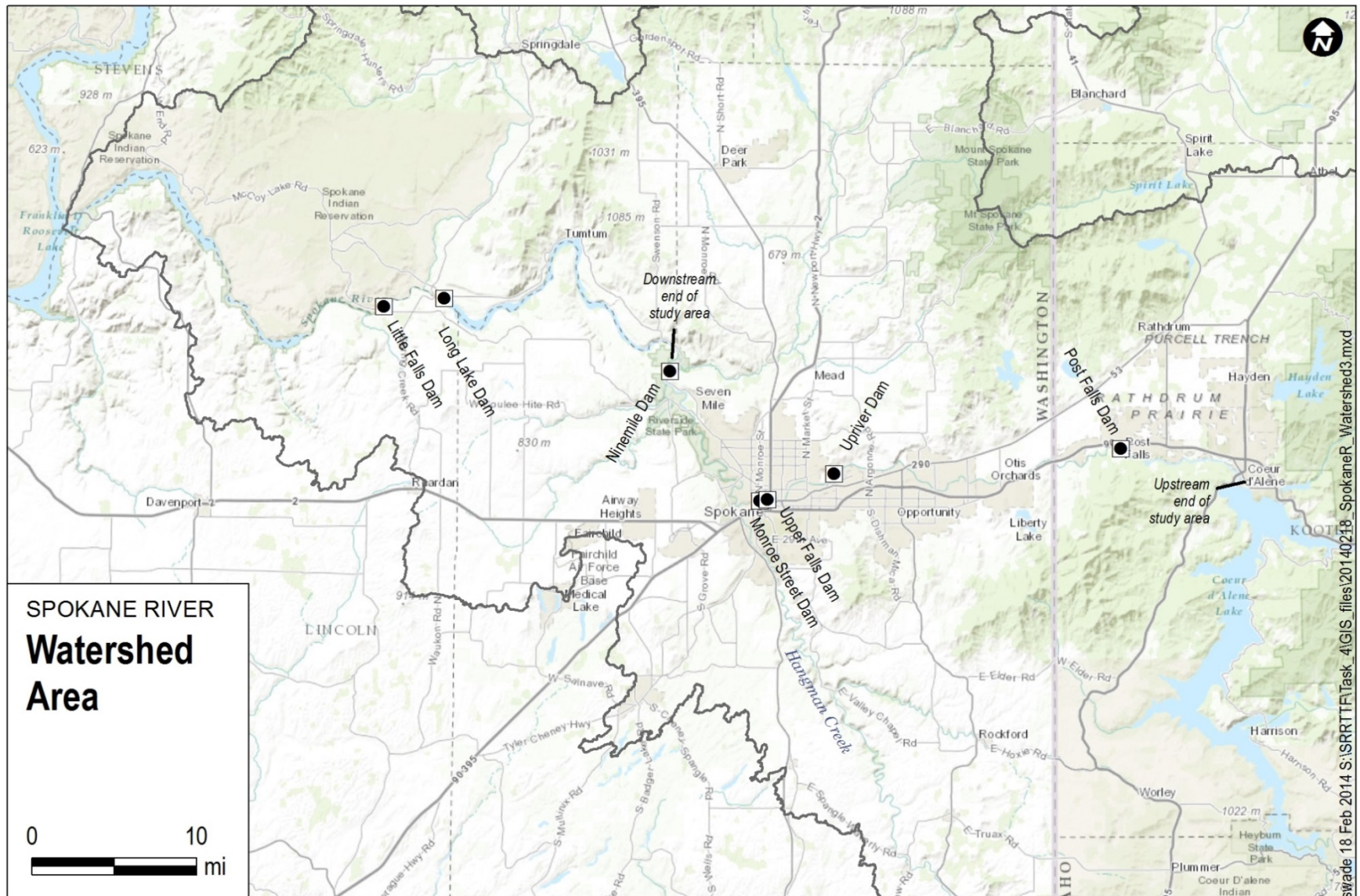


Figure 2. Spokane River Study Area

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

The Spokane River watershed has existing PCB monitoring data, which provide a good 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. 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 PCB mass balance assessment of the Spokane River (LimnoTech, 2014a). 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

The sampling program will be informed by the Confidence Interval Testing that will be conducted in May 2014. The Confidence Interval Testing will be 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 will be 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 will be collected for both discrete and composite analyses at Mirabeau Park, while discrete samples will be collected at the Lake Coeur d'Alene outlet. This information will be 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.

Based on the results of this sampling effort an addendum to the QAPP will be produced.



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. Knowledge of PCB mass loading at multiple river locations will allow the amount of PCB gained from and/or lost to groundwater between each station to be directly calculated. By extending the monitoring network to cover multiple locations in Idaho, this strategy will also provide necessary understanding of the relative contribution of the different Idaho sources (i.e. Lake Coeur d'Alene, point sources, groundwater).

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).

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
Seasonally Integrated Sampling – 1 st Event (done in conjunction with Synoptic Survey)	August, 2014
Seasonally Integrated Sampling – 2 nd Event	Winter, 2015
Seasonally Integrated Sampling – 3 rd Event	Spring, 2015
Draft Report	August 31, 2015
Final Report	October 31, 2015



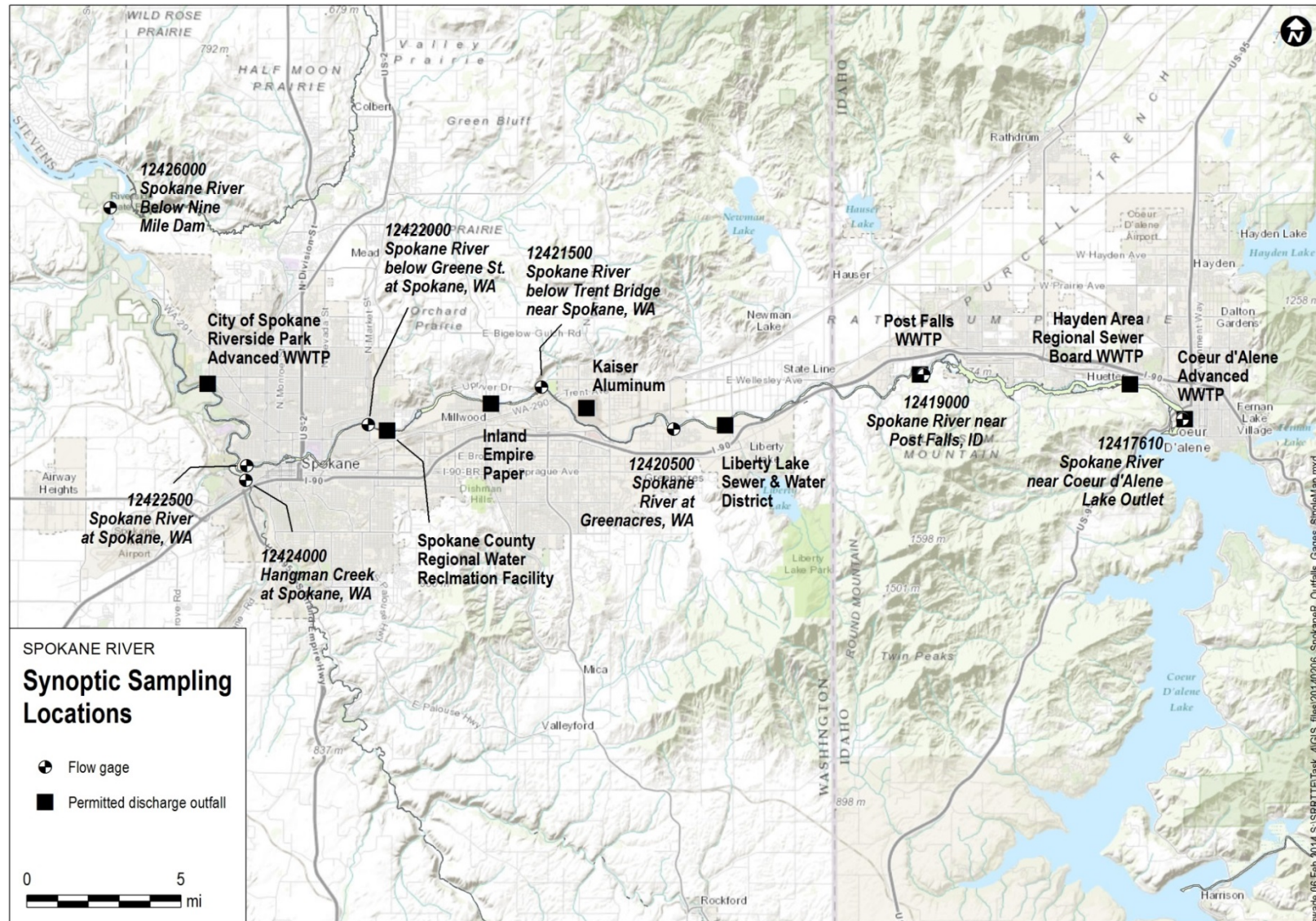


Figure 3. Spokane River Monitoring Locations Map

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 low-flow mass balance assessment and to assess the seasonal variability of upstream loads.



Confidence Interval Testing (LimnoTech, 2014b) will be 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 will be compared to the study's data quality objectives and data quality indicators. If necessary, adjustments will be made to the QAPP to accommodate changes in sampling and analytical protocols. Alternative sampling methods to be considered include a larger sample size, the Continuous Low-level Aquatic Monitoring (CLAM) device, or the XAD Resin. Of these options, recent tests by the Department of Ecology indicate that the CLAM would be reliable in river conditions. However other tests conducted by Kaiser Aluminum on wastewater effluent, indicate concerns over the ability to accurately assess flow rates through the sampler.

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 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. LAB2 (to be determined) 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, internal standards and labeled compounds. Matrix spikes 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 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 Technical Director validates the numbers of valid measurements. 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 analyses will be procured from Wellington Laboratories and Cambridge Isotope Laboratories.

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	50-145%	50-150%	25-150%*	Maximum = 50 pg/L Laboratory will B-qualify congeners results < 3x the concentration in an associated blank	50%	1-20	95

*Per Method for Ongoing Precision and Recovery (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



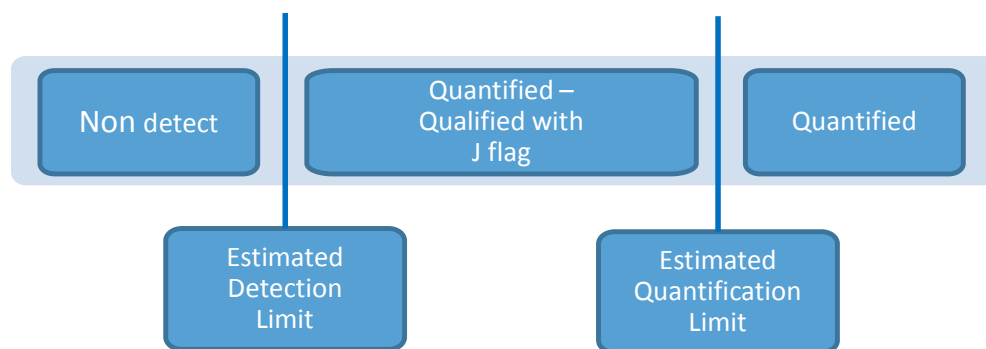


Figure 4. Schematic of detection limits

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 Sampling Contractor (to be determined) will be involved in this monitoring program. Personnel from AXYS Analytical Services and LAB2 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 Sampling Contractor (to be determined).

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. The Sampling Contractor will develop the Health and Safety Plan.

The Sampling Contractor 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 Technical Directors will be the main points of contact for coordinating all sample receipt, etc. The Laboratory Technical Directors will be assisted by the Laboratory Project Managers and QA/QC Managers in performing review and validation of all data generated to assure all data quality objectives have been met. The Laboratory Technical Directors or Laboratory QA/QC Managers 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 Technical Directors to review details of the planned progression of sampling events.

AXYS Analytical Services will do the PCB analyses using EPA Method 1668C 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. LAB2 (to be determined) 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 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 LimnoTech 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.



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 the Sampling Contractor.

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, if possible. The best effort will be made without jeopardizing the safety of the sampling crew. 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, the sampling contractor will notify the Field Manager of the situation, who will discuss with the Project Manager. The Sampling Contractor 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 the Sampling Contractor (to be determined) 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 ₂ SO ₄	28 days
DOC	Polypropylene	60 ml	4° C, H ₂ SO 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 all 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 LAB2 (to be determined) 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	LAB2
DSS (mg/L)	1	1-80	EPA 160.1	LAB2
TOC (mg/L)	1	1-2	EPA 415.1	LAB2
DOC (mg/L)	1	1-2	EPA 415.3	LAB2



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 Technical Director. The Laboratory Technical Director 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 LAB2 (to be determined). 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
- Analysis of duplicates

The data quality objectives for AXYS Analytical Services and LAB2 (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.

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 homologs 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. The third ¹³C homologs 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 the sampling contractor, 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. The sampling contractor 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 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 the Sampling Contractor 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 and title of author
- Name(s) of field crew
- Name(s) of site visitors
- Date and time of site entry
- Location of sampling activity
- Description of sample location
- Number and volume of samples taken
- Date and time of collection
- Sample identification numbers
- Sampling method
- Preservatives used
- Field measurements (pH, etc.)
- Date and time of shipment
- Shipment method
- Field observations

Equipment Logs

The sampling contractor 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

The sampling contractor 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 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. The sampling contractor 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 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 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 SRR-TTF-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 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 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 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.



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 the sampling contractor 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 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 described in Section 1, these goals include providing datasets for mass balance assessment. 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 low-flow mass balance assessment and assess the seasonal variability of upstream loads to the Spokane River.



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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.
- 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.
- LimnoTech, 2014a. Data Collection Strategy for PCB Comprehensive Plan - Draft. Memorandum from Dave Dilks to Spokane River Regional Toxics Task Force.
- LimnoTech, 2014b. Sampling Recommendations for Spokane River PCB Confidence Testing – Draft. Memorandum from Dave Dilks to the Spokane River Regional Toxics Task Force.
- Serdar, D., B. Lubliner, A. Johnson, D. Norton, 2011. Spokane River PCB Source Assessment 2003-2007. Publication No. 11-03-013.
- United States Environmental Protection Agency (EPA), 1998. EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5. Washington , DC.
- United States Environmental Protection Agency (EPA), 2001. EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5. Washington, DC.
- 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://srtrtf.org/wp-content/uploads/2014/05/M1668C_11June10-PCB_Congeners.pdf





APPENDIX B

LABORATORY REQUEST FOR QUALIFICATIONS AND QUOTE





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. (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, SRTTF-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.

Specification No. 1

April 3, 2014

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- 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:

- All raw data (EPA “Tier IV” or “Level 4” deliverables) in a fully bookmarked PDF file; and
- 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.

Specification No. 1

April 3, 2014

Page 1 of 3



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.

APPENDIX C

LABORATORY CERTIFICATIONS





APPENDIX D
LABORATORY
STANDARD ANALYTICAL PROCEDURES





APPENDIX E

GLOSSARY



Blank Page



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 priori” 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.

