



Quality Assurance Project Plan

Evaluation of PCBs in Spokane River Redband Trout

By

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Brandee Era-Miller

For the

Spokane River Regional Toxics Task Force

With support from the Washington State Departments of
Ecology and Fish & Wildlife

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Publication Information

This study is being conducted by the Spokane River Regional Toxics Task Force (Task Force) with support from the Washington State Department of Fish and Wildlife and the Washington State Department of Ecology and must have an approved Quality Assurance Project Plan (QAPP). The plan describes the objectives of the study and the procedures to be followed to achieve those objectives. After completing the study, the Task Force will post the final report of the study to the Internet.

This QAPP was approved to begin field work and finalized in October 2020.

The final QAPP is available on the Task Force website at: www.srrttf.org.

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Federal Clean Water Act 1996 303(d) Listings Addressed in this Study. See Section 3.1.

Data for this project will be available in both Ecology's [EIM Database](#) (Search Study ID: **SRRTTF-RedBT**) and in the SRRTTF Database (www.srrttf.org).

COVER PHOTO: A Redband Trout captured at Big Sheep Creek (tributary to the upper Columbia River). It was captured via hook and line in May 2011. PHOTO BY CHARLES LEE.

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October 2020

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Signature: Shea Hewage, General Manager, SGS AXYS Analytical Services, Ltd.	Date:
Signature: Arati Kaza, Ecology Quality Assurance Officer	Date:

Signatures are not available on the Internet version.

Ecology: Washington State Department of Ecology

WDFW: Washington Department of Fish and Wildlife

SRRTTF-ACE: Spokane River Regional Toxics Task Force – Contracting Entity

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2.0 Abstract

The Washington State Department of Fish and Wildlife (WDFW) in coordination with the Spokane River Regional Toxics Task Force (SRRTTF) will conduct a study to quantify concentrations of polychlorinated biphenyls (PCBs) in wild Redband Trout from the Spokane River. The results of this study will be used as a baseline for PCB concentrations in fish tissue and will be used as one measure of the effectiveness of PCB control actions aimed at the reduction of PCBs in the Spokane River. Fish collection will be conducted in the fall of 2020 and is intended to be repeated in two-year increments.

3.0 Background

3.1 Introduction and problem statement

Sections of the Spokane River have been placed on the 303(d) list of impaired waters for PCBs based on concentrations measured in fish tissue that exceed criteria for human consumption. The Spokane River Regional Toxics Task Force (SRRTTF) developed a “Comprehensive Plan” to identify sources of PCBs and implement control actions to reduce PCB levels in the Spokane River ([LimnoTech, 2016](#)). This study provides a standardized sampling framework and analyses to establish a baseline of PCB concentrations in fish tissue and can be used to help assess the control actions identified in the Comprehensive Plan to Reduce PCBs that have been implemented in the Spokane River.

The study utilizes index reaches that are comparable to past studies while including new reaches with similar hydrology for direct comparison across a geographic range. The study reduces variability by limiting the sampling to a single species with similar residence time in the river. Additionally, fish processing and analysis methods will be standardized to provide directly comparable results over time. The standardization allows the study to be repeated for use as a “yardstick” to monitor PCB concentrations in fish tissue over time. These analyses will provide a direct link to the efficacy of control actions on the bioaccumulation of PCBs in the tissue of Redband Trout in the Spokane River. This differs from the objectives of previous studies conducted by the Washington Department of Ecology.

In addition to the Redband Trout study, LimnoTech, technical consultant for SRRTTF, will direct a water column monitoring study in the Spokane River starting in summer of 2020 ([LimnoTech, 2020](#)). Both studies aim to establish a reference point for comparison to monitoring data in future years.

3.2 Study area and surroundings

The Spokane River originates at the outlet of Coeur d’Alene Lake in northern Idaho (river kilometer, rkm 178.8) and flows west 179 km through the City of Spokane to its confluence with Franklin D. Roosevelt Lake, an impoundment of the Columbia River in eastern Washington ([Figure 1](#)). This study is focused on an area of the Spokane River from the Washington/Idaho border to just downstream of the Riverside Water Reclamation Facility (rkm 154.5-108.0).

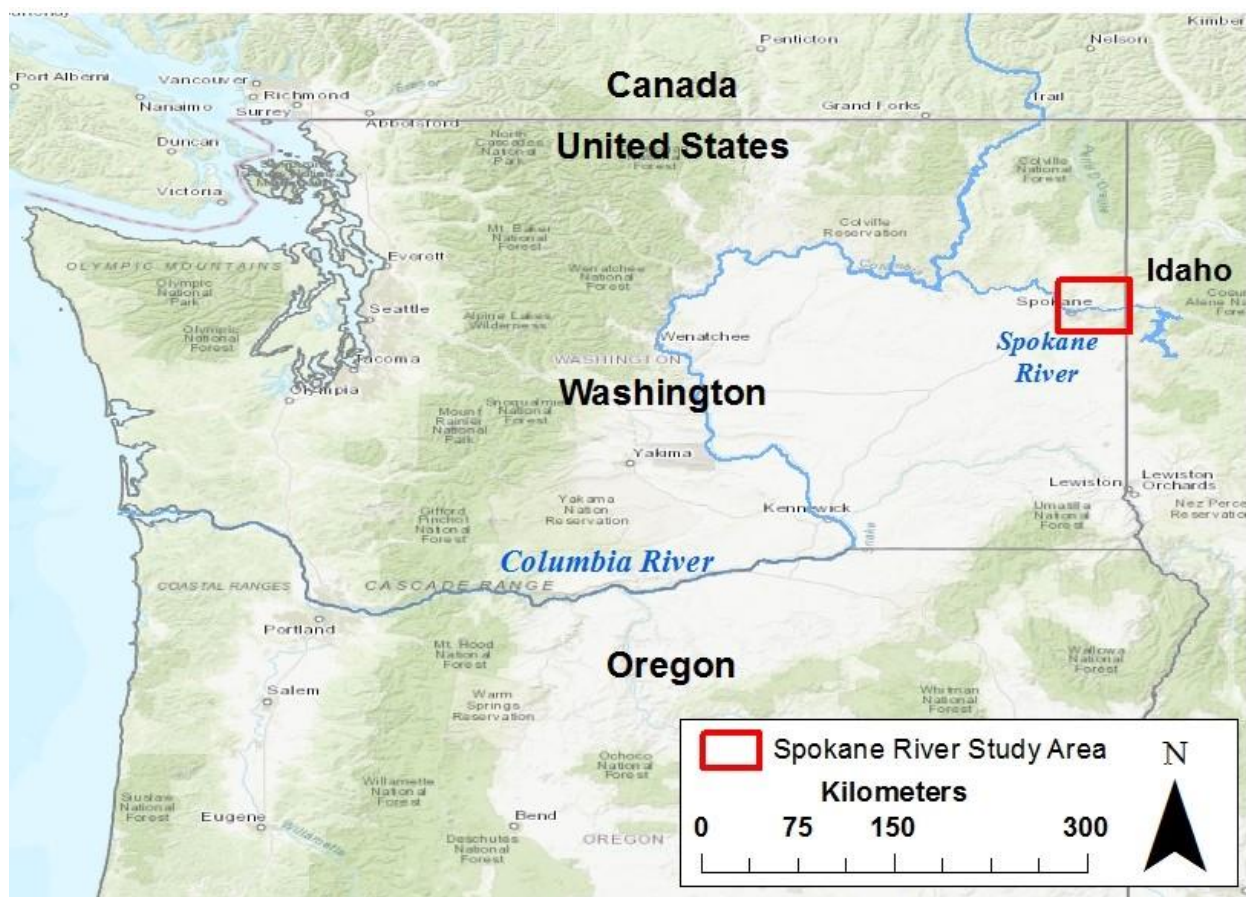


Figure 1. Map of Washington State and the Spokane River Redband Trout PCB study area.

3.2.1 History of study area

Prior to European settlement, the Spokane River flowed unpounded from its origin at the outlet of Coeur d'Alene Lake to the confluence with the Columbia River. The river supported anadromous salmon and steelhead runs, as well as a resident fish assemblage. The river also provided opportunity for urbanization and industry. The first hydroelectric development (HED) on the Spokane River was completed in 1890 and provided electrical power to the developing City of Spokane. Currently, seven HEDs are in operation on the Spokane River, including Post Falls (1906), Upriver (1933), Upper Falls (1922), Monroe Street (1890), Nine Mile (1908), Long Lake (1915), and Little Falls (1910). The construction and operation of the HEDs has changed the hydrodynamics of the river considerably, altering timing and volume of flows throughout its course. A thorough description of the study area was provided in the Comprehensive Plan (LimnoTech, 2016).

3.2.2 Summary of previous studies and existing data

Elevated levels of PCBs have been identified in the Spokane River by previous studies. [Maret and Dutton \(1999\)](#) reported PCB concentrations in Spokane River sediments which exceeded guidelines for Washington State freshwater sediment screening (0.021 ppm total PCBs). Initial

studies conducted prior to the implementation of the Comprehensive Plan detected concentrations of PCBs in sportfish fillets and whole fish which exceeded the human consumption criteria for edible fish (0.0053 ppm) and criterion for fish-eating wildlife (0.11 ppm) ([MacCoy, 2001](#); [USEPA, 1999](#); [Johnson, et. al., 1994](#); [Newall, et al. 1987](#)).

Subsequent reports by Ecology indicate that PCB concentrations in fish tissue decreased in some areas of the Spokane River, while results in other areas were variable but suggested no strong evidence of improving conditions ([Serdar and Johnson, 2006](#); [Seiders, et. al., 2014](#)).

The objectives of these previous Ecology fish tissue studies are different from the objective the current Redband Trout PCB study. The objectives of the previous studies were to provide high quality representative data to the Washington Department of Health for use in a human health assessment and in reviewing the fish consumption advisory stemming from data collected in 1999 and 2001. Secondary objectives were to examine contaminant trends within the river system.

The previous Ecology studies utilized composite samples of whole fish and fillets from a wide range of sizes and ages of multiple species. Not all species were collected at each location and therefore could not be compared throughout the geographical range of the study.

3.2.3 Parameters of interest and potential sources

[LimnoTech \(2020\)](#) describes PCBs and their properties as follows:

The contaminant of interest is total polychlorinated biphenyls (PCBs), which the sum of 209 individual congeners. PCBs are synthetic organochlorine compounds consisting of two benzene rings with one to ten chlorine atoms attached. PCBs have hydrophobic and lipophilic properties. They are persistent in the environment, bioaccumulative, and toxic. PCBs can affect the immune, reproductive, nervous, and endocrine system, and are known to be carcinogenic ([Davies, 2015](#)).

[LimnoTech \(2016\)](#) identified the primary delivery mechanisms of PCBs to the Spokane River as:

- industrial and municipal wastewater treatment plants,
- contaminated groundwater,
- PCB entering from the outlet of Lake Coeur d'Alene, and
- stormwater/combined sewer overflows.

3.2.3.1 Ancillary Parameters

Additional parameters will be analyzed in fish tissue samples and include weight, length, % lipids and moisture.

3.2.4 Regulatory criteria or standards

In this study, PCB concentrations are being used to support future temporal trend assessments. Results will not be compared to regulatory criteria or standards.

4.0 Project Description

Fish Sampling

This study is limited to a single species of a similar size and age: sub-adult Columbia River Redband Trout (*Oncorhynchus mykiss*, subspecies: *gairdneri*). The study will focus on the collection of more samples with similar life history strategies, habitat preferences, feeding preferences and residence times. Thus reducing the variability between samples and increasing the comparability throughout the geographic range of the study. Hatchery Rainbow Trout are excluded from the study because they are stocked as 1-year old fish and as such their PCB exposure is vastly different to that of wild Redband Trout.

Sub-adult fish are those that have not reached sexual maturity. Redband Trout primarily reach sexual maturity at age 2 (males) and age 3 (females). Based on 10 years of aging data, the vast majority of Redband Trout 200-300 mm total length were age 1. Redband Trout >300 mm TL exhibited substantial age overlap and age analysis using scales was less accurate. Thus, residence time was less predictable in larger fish. Additionally, it is unknown if or how the maturation process (both physiologically and behaviorally) affects PCB bioaccumulation. Therefore, using sub-adult Redband Trout (200-300 mm TL) reduces variability between samples and provides a “biological yardstick” to evaluate the efficacy of PCB abatement efforts in the Spokane River.

WDFW will conduct boat electrofishing to capture Redband Trout in six sections from three reaches of the Spokane River in Washington state between the Washington/Idaho border to the Nine Mile HED. The three reaches from upstream to downstream are the Upper Spokane River (Post Falls HED to Upriver HED), Middle Spokane River (Upriver HED to Upper Falls HED), and Lower Spokane River (Monroe Street HED to Nine Mile HED). There are six survey sections or fish collection areas. The survey areas from upstream to downstream will be defined as:

- Section 1 (WA/ID State Line to McMillan Road; rkm 154.5-146.1),
- Section 2 (Flora Road to Donkey Island; rkm 143.1-134.8)
- Section 3 (Upriver Dam to Crestline Street; rkm 129.0-120.2),
- Section 4 (Crestline Street to Division Street; rkm 124.1-120.2),
- Section 5 (Water Street to and T.J. Meenach Bridge; rkm 117.9-112.3) and
- Section 6 (Riverside Water Reclamation Facility to the kayak takeout site approximately 650 m below the effluent pipe rkm 108.7-108.0).

Sampling will occur during the month of October when river discharge increases from summer low flows allowing safe navigation with a drift boat ($\geq 42.5 \text{ m}^3/\text{s}$).

A crew of 2-3 individuals, one boat captain/rower and 1-2 netters, will conduct the fishing surveys. A maximum of two sampling events will be conducted at each of the six survey sections. The WDFW will attempt to collect up to 25 sub-adult Redband Trout measuring 200-300 mm TL within each survey section. Data collection information will include species, total length (TL; mm), weight (WT; g), sample identification number and GPS coordinates of survey

sections where fish were collected. Redband Trout samples will be processed according to Ecology's Standard Operating Procedure (SOP) EAP009 ([Sandvik, 2018a](#)).

Analysis

Fish samples will be prepared for analysis and analyzed for PCB congeners (EPA Method 1668C) at the SGS-AXYS Laboratory in Sidney, B.C., Canada. Fish will be prepared as whole body composite samples of 5 fish per composite following Ecology's SOP EAP007 ([Sandvik, 2018b](#)) and SGS-AXYS's internal methods.

4.1 Project goals

The major goals of this project include:

- Develop a standardized sampling and analysis protocol for evaluating PCB concentrations in Redband Trout collected from six sections of the Spokane River.
- Determine PCB concentrations in sub-adult Redband Trout in six sections of the Spokane River from the Washington/Idaho border to Riverside Water Reclamation Facility to provide a baseline for future comparisons.

4.2 Project objectives

Project objectives include:

- Collect 25 sub-adult Redband Trout in each of six survey sections (n =150 fish total) of the Spokane River study area from the Washington/Idaho border to Riverside Water Reclamation Facility.
- Analyze PCB congeners in 30 composite samples; 5 composite samples, each consisting of 5 sub-adult Redband Trout collected from each of the six survey sections of the Spokane River.
- Establish a baseline of PCBs concentration in sub-adult Redband Trout within the Spokane River study area.

4.3 Information needed and sources

No further background data is necessary for this study. This study will collect new data on PCB congener concentrations in composite samples of whole sub-adult Redband Trout in the Spokane River.

4.4 Tasks required

WDFW Tasks:

1. Conduct a maximum of two sampling events at each of the six survey sections to collect a maximum of 25 sub-adult Redband Trout per Section.
2. Record electrofishing effort and biological data for each fish on standardized data sheet.

3. Preserve whole fish samples according to [Sandvik \(2018a\)](#) and maintain chain of custody.
4. Ship frozen fish to SGS-AXYS Laboratory in Sidney, B.C., Canada for sample processing and PCB congener analysis. Make sure to include the proper paper work for international shipping.
5. Enter boat electrofishing effort and biological data into a computer in an Excel file.
6. Provide a copy of datasheets and an electronic file of electrofishing effort and biological data to LimnoTech for inclusion into final data report.

SGS-AXYS

1. Process (homogenize and composite) whole body samples of Redband Trout following Ecology's fish processing SOP ([Sandvik, 2018b](#)) and appropriate internal processing methods.
2. Analyzed samples using EPA Method 1668 ([EPA, 2010](#)) and other internal methods as appropriate.
3. Report data in a level IV data package to LimnoTech for data validation.
4. Provide electronic data deliverables (EDDs) in the Equis-based format that is suited for upload into the SRRTTF database.

LimnoTech

1. Validate the data and determine usability.
2. Write up the results in a data report or other appropriate document for use by SRRTTF.

4.5 Systematic planning process

The project is funded in part by an Ecology contract with state funds. Therefore, any data collected requires an approved QAPP, Ecology as a signatory to the QAPP and entry of the final project data into Ecology's Environmental Information Management System (EIM).

This QAPP constitutes a suitable planning process for the project.

5.0 Organization and Schedule

5.1 Key individuals and their responsibilities

Table 1 shows the responsibilities of those who will be involved in this project.

Table 1. Organization of project staff and responsibilities.

Staff	Title	Responsibilities
Robert Lindsay President SRRTTF-ACE Phone: 509-477-7576	Task Force Client	Manage contracts: review and approve project specifications. Ensure project is completed in timely manner.
Charles Lee Biologist WDFW Phone: 509-892-1001	Field Lead	Lead author of the QAPP. Oversees field sampling and transportation of samples to the laboratory.
David Dilks LimnoTech Phone: 734-332-1200	Project Lead	Review/approve all work products prior to delivery to SRRTTF-ACE. Ensure that work is done in accordance with QAPP. Review project with Laboratory Operations Directors prior to sampling. Arrange for system audits. Writes data report for SRRTTF.
Adriane Borgias Water Quality Section Manager, Eastern Regional Office Phone: (509) 329-3515	Advisor	Review and approve QAPP.
Karl Rains Water Quality Planner, Eastern Regional Office Phone: (509) 329-3601	Contract Manager	Review and approve QAPP, manage SRRTTF contract.
Robert Betz LimnoTech Phone: 734-332-1200	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.
Shea Hewage SGS AXYS Analytical Services, Ltd. Phone: (250) 655-5800	Laboratory General Manager	Responsible for all aspects of the daily operation of the laboratory. Oversees laboratory operations including sample analysis and data reporting in accordance with defined procedures and client requirements. Oversees the completion of corrective actions to address any non-conformances.
Sean Campbell SGS AXYS Analytical Services, Ltd. Phone: (250) 655-5834	Laboratory Project Manager	Responsible for the execution of project-specific laboratory activities and interactions with the Task Force.
Richard Grace SGS AXYS Analytical Services, Ltd. Phone: 905-484-2314	Sales, Marketing, Service	Oversight of laboratory commercial terms. Serves as the main point of contact for laboratory for contract management or maintenance. Works closely with clients and laboratory management to develop project technical specifications
Dale Hoover SGS AXYS Analytical Services, Ltd. Phone: 250-655-5800	Laboratory Technical Manager	Oversees laboratory technical offerings. Monitors laboratory and method performance and analytical results for conformance with established quality
Arati Kaza Phone: 360-407-6964	Ecology Quality Assurance Officer	Reviews and approves the draft QAPP and the final QAPP.

5.2 Special training and certifications

Charles Lee is a biologist for the Washington Department of Fish and Wildlife and he will be overseeing all the activities surrounding fish collection, field processing, chain-of-custody and shipment of samples to the laboratory.

Mr. Lee has conducted research on fish populations in the Inland Northwest for over 20 years. He is currently a Biologist 3 for the Washington Department of Fish and Wildlife in Spokane, Washington, where he manages multiple projects conducting stock assessment of native salmonids in the upper Columbia River drainage and various other resident fisheries research and monitoring activities. Mr. Lee recently completed a 10 year study in coordination with Avista Utilities to monitor abundance and year class strength of Redband Trout in the Spokane River.

Education:

Eastern Washington University, Cheney, WA 99004 M.S. Biology-Fisheries 2005

Eastern Washington University, Cheney, WA 99004 B.S. Biology-Zoology 1999

5.3 Organization chart

The lines of reporting for the organizations in the project are shown in the organization chart below (Figure 2).

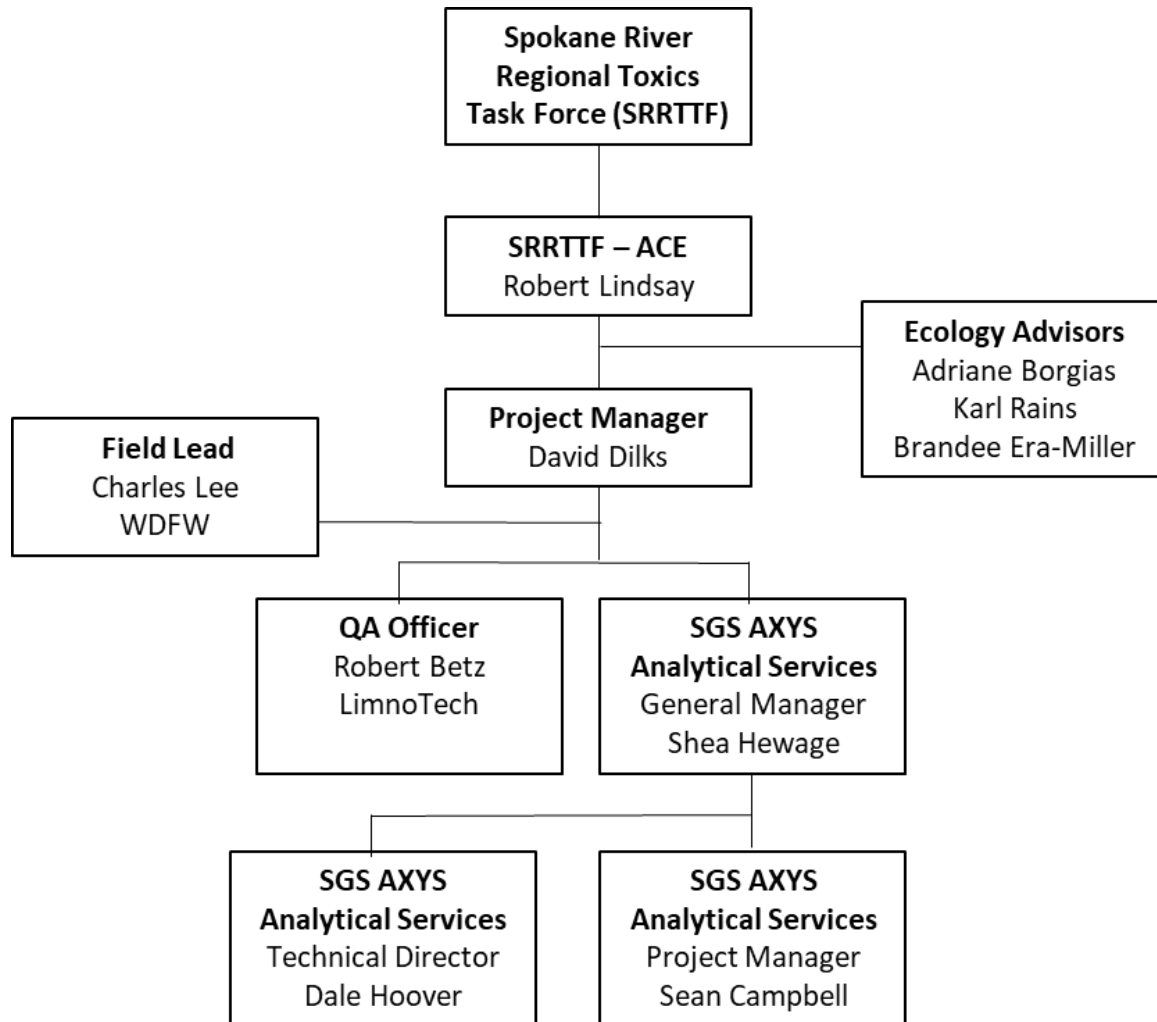


Figure 2. Project organization chart.

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

5.4 Proposed project schedule

Table 2 lists key activities, due dates, and lead staff for this project.

Table 2. Proposed schedule for completing field and laboratory work, data entry into EIM and SRRTTF Databases and reports.

Work type	Due date	Lead staff	Organization
Field and laboratory work			
Field work completed	November 2020	Charles Lee	WDFW
Laboratory analyses completed	February 2021	Sean Campbell	SGS-AXYS
Laboratory data validation	April 2021	Robert Betz	SGS-AXYS
Database			
EIM database entry and review	August 2021	TBD	SRRTTF
SRRTTF database entry and review	August 2021	Amy Sumner	Spokane County
Final report			
Draft report to Task Force	December 2021	Dave Dilks	Limnotech
Final report on web	March 2022	Dave Dilks	Limnotech

TBD = to be determined

5.5 Budget and funding

SRRTTF will pay for the project with funding in part by an Ecology contract and state funds. Redband Trout collection by WDFW and associated costs are itemized in Table 3. Table 4 includes the overall project budget.

Table 3. Budget for Redband Trout sample collection.

Description	Cost
WDFW Personnel Subtotal (Salaries+Fringe)	\$12,976
Vehicle/Vessel Operation & Maintenance	\$1,318
Field Equipment/Supplies	\$350
Administrative Expenses	\$4,436
Fish Sampling Total	\$19,080

Table 4. Project budget and funding.

Description					Cost
LimnoTech (Data Validation and Report)					10,000
Fish Sampling					19,080
Parameter	Number of Samples	Number of QA Samples	Total Number of Samples	Cost Per Sample*	Lab Subtotal
PCB Congeners, lipids and % moisture	30	3**	33	1100	36,300
Project Grand Total					\$65,380

*Cost per sample includes processing of fish tissue, chemical analysis and providing a level IV data package and an electronic data deliverable (EDD) suited for upload into the SRRTTF database.

**Three duplicate samples will be analyzed as separate aliquots from the same composite samples and will be given separate sample numbers. These duplicates are sometimes referred to as “split samples”. In addition, the lab will perform two laboratory duplicates at no extra charge.

6.0 Quality Objectives

6.1 Data quality objectives

The main data quality of objective (DQO) for this project is to collect up to 25 sub-adult Redband Trout from six sections of the Spokane River, preserve the samples according to [Sandvik \(2018a\)](#) and ship samples under chain of custody to SGS-AXYS laboratory for PCB congener analysis. The analysis will use EPA methods with high-resolution gas chromatography-mass spectrometry to resolve the congener distribution present. Measurement quality objectives (MQOs) described in the subsequent section detail the targets for analytical precision, bias, and sensitivity.

6.2 Measurement quality objectives

The MQOs for this study are detailed in [Tables 5 and 6](#).

6.2.1 Targets for precision, bias, and sensitivity

The MQOs for project results, expressed in terms of acceptable precision, bias, and sensitivity, are described in this section and summarized in [Tables 5 and 6](#) below. Method detection limits, calibration limits and reporting limits for SGS-AXYS's Method MLA-010, their modified version of EPA Method 1668C, are detailed in [Appendix A, Table A-1](#), on a per congener basis.

Table 5. Measurement quality objectives for laboratory analyses of tissue samples.

MQO →	Precision	Bias		Sensitivity
Parameter	Duplicate Samples	Verification Standards (LCS,CRM,CCV)	Surrogate Standards*	Lowest Concentration of Interest
	Relative Percent Difference (% RPD)	Recovery Limits (%)		Concentration Units
PCB Congeners	≤50%	Compound Specific	25-150	0.05 pg/g per congener
Lipids	≤20%	NA	NA	0.1%
% Moisture	≤20%	NA	NA	0.1%

* Surrogate recoveries are compound-specific (see [Table 6](#)).

Table 6. Detailed Measurement quality objectives for PCB congener analyses.

Congener	Cong. No.	Test conc. ng/mL	CAL/VER (%)		IPR ²		OPR ²		Labelled compound ²	
					(%)		(%)		% recovery in samples	
			Warning limits	Accept. limits	RSD	×	Warning limits	Accept. limits	Warning limits	Accept. limits
2-MoCB	1	50	75-125	75-125	25	70-130	70-130	60-135	-	-
4-MoCB	3	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2'-DiCB	4	50	75-125	75-125	25	70-130	70-130	60-135	-	-
4,4'-DiCB	15	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2'6-TrCB	19	50	75-125	75-125	25	70-130	70-130	60-135	-	-
3,4,4'-TrCB	37	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2'6,6'-TeCB	54	50	75-125	75-125	25	70-130	70-130	60-135	-	-
3,3',4,4'-TeCB	77	50	75-125	75-125	25	70-130	70-130	60-135	-	-
3,4,4',5'-TeCB	81	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',4,6,6'-PeCB	104	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,3',4,4'-PeCB	105	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,4,4',5'-PeCB	114	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3',4,4',5'-PeCB	118	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2',3,4,4',5'-PeCB	123	50	75-125	75-125	25	70-130	70-130	60-135	-	-
3,3',4,4',5'-PeCB	126	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',4,4',6,6'-HxCB	155	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,3',4,4',5'-HxCB ³	156	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,3',4,4',5'-HxCB ³	157	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3',4,4',5,5'-HxCB	167	50	75-125	75-125	25	70-130	70-130	60-135	-	-
3,3',4,4',5,5'-HxCB	169	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',3,4',5,6,6'-HpCB	188	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,3',4,4',5,5'-HpCB	189	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',3,3',5,5',6,6'-OoCB	202	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,3',4,4',5,5',6-OoCB	205	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',3,3',4,4',5,5',6-NoCB	206	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',3,3',4,5,5',6,6'-NoCB	208	50	75-125	75-125	25	70-130	70-130	60-135	-	-
DeCB	209	50	75-125	75-125	25	70-130	70-130	60-135	-	-

1 = Suffix "L" indicates labelled compound.

2 = QC acceptance criteria for IPR, OPR, and samples based on a 20 µL extract final volume

3 = PCBs 156 and 157 are tested as the sum of two concentrations

Table 6. Detailed Measurement quality objectives for PCB congener analyses (cont.)

Congener	Cong. No.	Test conc. ng/mL	CAL/VER (%)		IPR ²		OPR ²		Labelled compound ²	
					(%)		(%)		% recovery in samples	
			Warning limits	Accept. limits	RSD	×	Warning limits	Accept. limits	Warning limits	Accept. limits
Labeled Compounds										
¹³ C ₁₂ -2,2',6-TrCB	19L	100	65-135	50-145	70	20-135	15-145	15-145	30-130	5-145
¹³ C ₁₂ -3,4,4'-TrCB	37L	100	65-135	50-145	70	20-135	15-145	15-145	30-130	5-145
¹³ C ₁₂ -2,2',6,6'-TeCB	54L	100	65-135	50-145	70	20-135	15-145	15-145	30-130	5-145
¹³ C ₁₂ -3,3',4,4'-TCB	77L	100	65-135	50-145	50	45-135	40-145	40-145	30-130	10-145
¹³ C ₁₂ -3,4,4',5-TeCB	81L	100	65-135	50-145	50	45-135	40-145	40-145	30-130	10-145
¹³ C ₁₂ -2,2',4,6,6'-PeCB	104L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
¹³ C ₁₂ -2,3,3',4,4'-PeCB	105L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
¹³ C ₁₂ -2,3,4,4',5-PeCB	114L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
¹³ C ₁₂ -2,3',4,4',5-PeCB	118L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
¹³ C ₁₂ -2',3,4,4',5-PeCB	123L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
¹³ C ₁₂ -3,3',4,4',5-PeCB	126L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
¹³ C ₁₂ -2,2',4,4',6,6'-HxCB	155L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
¹³ C ₁₂ -2,3,3',4,4',5-HxCB3	156L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
¹³ C ₁₂ -2,3,3',4,4',5'-HxCB3	157L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
¹³ C ₁₂ -2,3',4,4',5,5'-HxCB	167L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
¹³ C ₁₂ -3,3',4,4',5,5'-HxCB	169L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
¹³ C ₁₂ -2,2',3,3',4,4',5-HpCB	170L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
¹³ C ₁₂ -2,2',3,4,4',5,5'-HpCB	180L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
¹³ C ₁₂ -2,2',3,4',5,6,6'-HpCB	188L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
¹³ C ₁₂ -2,3,3',4,4',5,5'-HpCB	189L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
¹³ C ₁₂ -2,2',3,3',5,5',6,6'-OcCB	202L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
¹³ C ₁₂ -2,3,3',4,4',5,5',6-OcCB	205L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
¹³ C ₁₂ -2,2',3,3',4,4',5,5',6-NoCB	206L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
¹³ C ₁₂ -2,2',3,3',4,5,5',6,6'-NoCB	208L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
¹³ C ₁₂ -2,2',3,3',4,4',5,5',6,6'-DeCB	209L	100	65-135	50-145	50	45-135	40-145	40-145	40-130	10-145
Cleanup Standards										
¹³ C ₁₂ -2,4,4'-TriCB	28L	100		65-135	70	20-135	15-145	15-145	40-130	5-145
¹³ C ₁₂ -2,3,3',5,5'-PeCB	111L	100		75-125	50	45-135	40-145	40-145	40-130	10-145
¹³ C ₁₂ -2,2',3,3',5,5',6-HpCB	178L	100		75-125	50	45-135	40-145	40-145	40-130	10-145

1 = Suffix "L" indicates labelled compound.

2 = QC acceptance criteria for IPR, OPR, and samples based on a 20 µL extract final volume

3 = PCBs 156 and 157 are tested as the sum of two concentrations

6.2.1.1 Precision

Five duplicate analyses will be conducted for the 30 fish tissue samples in this study. Three of the duplicates will be analyzed as separate aliquots from the same composite samples and will be given separate sample numbers. These types of duplicates are sometimes referred to as split samples and will serve as the field replicates. In addition, the lab will perform two laboratory duplicates at no extra charge. The maximum acceptable relative percent difference is 20% for lipids and % moisture and 50% for PCB congeners (see [Table 5](#)).

6.2.1.2 Bias

Bias is the difference between the population mean and the true value. For this project, bias is measured as acceptable % recovery. Acceptance limits for laboratory verification standards, matrix spikes, and surrogate standards are shown in [Tables 5 and 6](#).

6.2.1.3 Sensitivity

Sensitivity is a measure of the capability of a method to detect a substance above the background noise of the analytical system. Targets for sensitivity for high-resolution PCB congeners are expressed as a method detection limits (MDL) and are shown in [Appendix A, Table A](#).

PCB congeners will be censored if the sample is not above three times the laboratory method blank.

6.2.2 Targets for comparability, representativeness, and completeness

6.2.2.1 Comparability

This study provides a standardized sampling framework and analyses to establish a baseline of PCB concentrations in fish tissue that can be used to help assess the control actions identified in the Comprehensive Plan to Reduce PCBs in the Spokane River ([LimnoTech, 2016](#)). The study utilizes index reaches that are comparable to past studies while including new reaches with similar hydrology for direct comparison across a geographic range.

The study reduces variability by limiting the sampling to a single species with similar residence time in the river. Additionally, fish processing and analysis methods will be standardized to provide directly comparable results over time. The standardization allows the study to be repeated for use as a “yardstick” to monitor PCB concentrations in fish tissue over time. These analyses are expected to provide a direct link to the efficacy of implemented control actions on the bioaccumulation of PCBs in fish tissue in the Spokane River.

Section 8.2 lists the standard operating procedures (SOPs) to be followed for field sampling and tissue processing at the laboratory. All analytical methods used for the project are approved methods commonly used by Ecology for monitoring of PCBs.

6.2.2.2 Representativeness

A total of six survey sections have been identified to ensure representation of the entire study area. Samples will be collected from a single species of sub-adult fish of similar size with similar residence time to reduce variability associated with time of exposure and biological uptake of PCBs.

6.2.2.3 Completeness

WDFW will attempt to collect a total of 25 Redband Trout from each of six survey sections. Fish will be analyzed as composites of five fish each such that each section will have a total of 5 composite samples. If the sample size ($n=25$) is not attainable, a minimum of 3 individual fish can be used in each composite sample. For example, if only 15 fish are collected from a reach, then there would still be a total of 5 composite samples for that reach. For long term trend analysis, it is important to have a higher number of composite samples in order to have enough statistical power ([Seiders, personal communication](#)).

6.3 Acceptance criteria for quality of existing data

All data used to support the findings of this project will meet project DQOs. To the extent that any previous data are used, they will also be evaluated for compliance with current DQOs.

6.4 Model quality objectives

N/A

7.0 Study Design

7.1 Study boundaries

Fish sampling will be conducted in WRIA 54 (Lower Spokane River) and 57 (Middle Spokane River). The Spokane River study area will include six survey sections between the Washington/Idaho State Line (rkm 154.5) and the Riverside Water Reclamation Facility (rkm 108.7) (Figure 3). The six survey sections from upstream to downstream will be defined as:

- Section 1 (WA/ID State Line to McMillan Road; rkm 154.5-146.1)
- Section 2 (Flora Road to Donkey Island; rkm 143.1-134.8)
- Section 3 (Upriver Dam to Crestline Street; rkm 129.0-120.2)
- Section 4 (Crestline Street to Division Street; rkm 124.1-120.2)
- Section 5 (Water Street to and T.J. Meenach Bridge; rkm 117.9-112.3) and
- Section 6 (Riverside Water Reclamation Facility to the kayak takeout site approximately 650 m below the effluent pipe rkm 108.7-108.0)

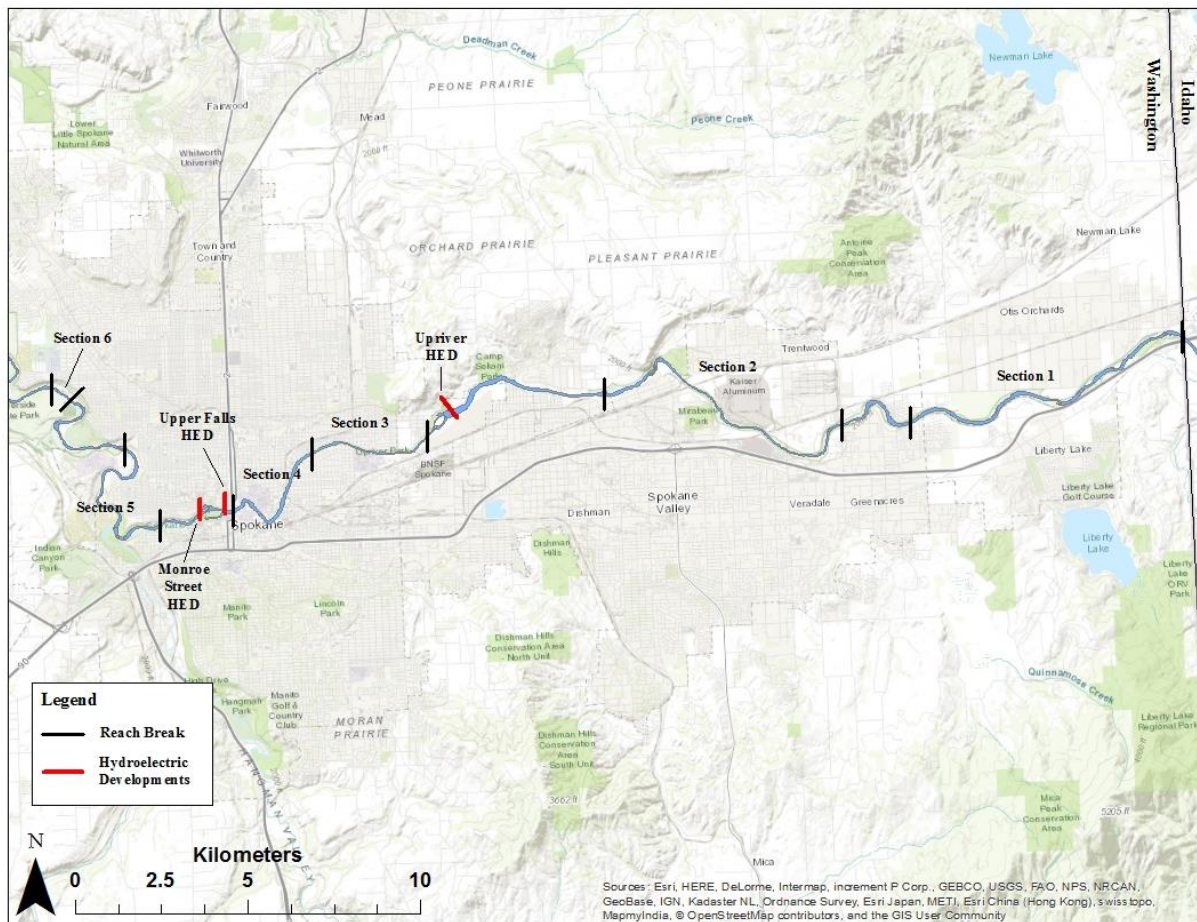


Figure 3. Map of Spokane River Redband Trout PCB Study Area.

7.2 Field data collection

The WDFW will utilize boat electrofishing to capture up to 25 Redband Trout in each of six sections of the Spokane River in Washington state between the Washington/Idaho border to the Nine Mile HED (Figure 3). Drift boat electrofishing will be used in Sections 1, 2, 3, 5 and 6. Power boat electrofishing will be used in Section 4.

7.2.1 Sampling locations and frequency

Sampling will be conducted by boat electrofishing. A crew of 2-3 individuals, one boat captain/rower and 1-2 netters, will conduct the surveys. A maximum of two sampling passes will be conducted at each of the six survey sections. Sampling will be conducted along the left or right shoreline for approximately 600 seconds of “electrofishing on” time. The crew will then anchor and process the samples (if any). The boat crew will then cross the river and sample the opposite shoreline for approximately 600 seconds. This process will be repeated until the full sample ($n=25$) for the survey section has been collected or the end of the section is reached. If necessary, WDFW will conduct a second sampling pass. Sampling will occur during the month of October when river discharge increases from summer low flows allowing safe navigation with a drift boat ($\geq 42.5 \text{ m}^3/\text{s}$).

7.2.2 Field parameters and laboratory analytes to be measured

Biological data collected on each fish will include total length (mm) and weight (g). Fish will not have age or sex determined as the variability presented by those characteristics will already be accounted for based on the targeted total lengths of the fish (200-300 mm) which represent sub-adult and sexually immature fish. Sample collection location data will include GPS coordinates (start and end) of the survey section, date of collection, and time of day. Whole fish tissue composite samples will be analyzed for PCB congeners (Method 1668), % lipids and % moisture.

7.3 Modeling and analysis design

N/A

7.4 Assumptions underlying design

WDFW assumes that low summer discharge flows will increase by the month of October to allow sampling with a drift boat. It is also assumed that a full set ($n=25$) of sub-adult Redband Trout samples can be collected from each of the six survey sections.

7.5 Possible challenges and contingencies

Low Redband Trout density may impede the ability to collect the full sample size from some survey sections. The WDFW will try to collect 25 fish per survey section. The project may have to revise target sample size, revise survey sections or reduce sampling frequency if it is determined that sampling is negatively impacting the Redband Trout populations within the study area.

If the sample size ($n=25$) is not attainable, a minimum of 3 individual fish can be used in each composite sample. For example, if only 15 fish are collected from a reach, then there would still be a total of 5 composite samples for that reach.

Future sampling efforts (years 2022, 2024, etc.) will depend on funding and resources from SRRTTF and others.

7.5.1 Logistical problems

During drought years, low flows may delay sampling or hinder the ability to access the entire study area. WDFW may postpone the survey until flows increase to an acceptable level.

7.5.2 Practical constraints

Equipment failure could lead to a disruption in sampling. WDFW will take all precautions to ensure sampling equipment is in safe working order prior to sampling and will expedite equipment repair if necessary to complete the sampling in a timely manner.

Fish collection will take place while Spokane County and the rest of Washington State are under restricted operations due to the COVID-19 pandemic. WDFW has developed a COVID-19 field work protocol that they will carefully follow during fish collection and processing in October of 2020 ([WDFW, 2020](#)).

7.5.3 Schedule limitations

WDFW does not foresee any schedule limitations for field sampling.

8.0 Field Procedures

8.1 Invasive species evaluation

Field personnel for this project are required to be familiar with and follow the procedures described in SOP EAP070 ([Parsons et al., 2018](#)), Minimizing the Spread of Invasive Species.

All WDFW sampling equipment is inspected for invasive species after each sampling session when used in different water bodies. Personal protective gear is drainage specific or decontaminated between uses in different drainages. Virkon® is used to decontaminate boots, waders and nets when used between different bodies of water.

8.2 Measurement and sampling procedures

Two SOPs will cover the collection and processing of fish samples:

- [Sandvik, P. \(2018a\)](#) – Standard Operating Procedure EAP009, Version 1.2: Field Collection, Processing, and Preservation of Finfish Samples at the Time of Collection in the Field.
- [Sandvik, P. \(2018b\)](#) – Standard Operating Procedure EAP007, Version 1.2: Resecting Finfish Whole Body, Body Parts, or Tissue Samples.

PCB congener analysis will follow EPA Method 1668C ([EPA, 2010](#)). SGS-AYXS has their own internal modified version of Method 1668C: MLA-010.

8.3 Containers, preservation methods, holding times

Fish samples will be preserved after field collection according to [Sandvik \(2018a\)](#). Fish samples will be processed by SGS-AXYS following [Sandvik \(2018b\)](#) and their internal methods as appropriate. Sample containers, preservation and holding times are shown in [Table 7](#).

Table 7. Sample containers, preservation, and holding times.

Parameter	Matrix	Minimum Quantity Required	Container*	Preservative	Holding Time
PCB congeners, % Lipids and % Moisture	tissue	30 grams wet weight	8 oz. glass jar, certified clean	freeze	1 year frozen

*Containers will be provided by SGS-AXYS Laboratory during processing. SGS-AXYS conducts batch proofing of the certified clean jars they receive from the manufacturer.

8.4 Equipment decontamination

Fish collection equipment (electrofishing boat, live well, nets and gloves) will be rinsed with on-site water prior to collection. Processing of fish in the field will follow Ecology SOP EAP009, Version 1.2: Field Collection, Processing, and Preservation of Finfish Samples at the Time of Collection in the Field ([Sandvik, 2018a](#)). SOP EAP009 contains steps for minimizing fish sample contamination such as double wrapping the fish with clean foil (dull side in) and then

placing them in clean sealable plastic bags in a clean cooler on ice prior to transport from the field.

8.5 Sample ID

Individual fish samples will be identified by survey year, survey section and fish sample number. Fish sample numbers will be 01 through 25 for each survey section. (e.g. 2020_SR1_01 for fish sample number 01 collected in Spokane River Section 1 during the 2020 survey). SGS-AXYS will give their own sample numbers for the fish tissue composite samples to be analyzed for PCBs congeners. Additionally, SGS-AXYS will provide documentation on which individual fish samples they combine for composite analysis.

8.6 Chain of custody

Chain of custody will be maintained throughout the project. WDFW will provide the necessary documentation for international shipping of biological samples to SGS-AXYS.

8.7 Field log requirements

Data will be recorded in pencil on standardized data sheets printed on “Rite in the Rain” waterproof paper. Data will include date (mm/dd/yyyy), time (24 hr.), survey section (1-6), section starting and ending GPS coordinates (Lat. ddd.ddddd°, Lon. ddd.ddddd°), initials of field personnel. Biological data collection will include species, total length (mm), and weight (g). A comments column will be used to describe any anomalies or descriptive notes.

8.8 Other activities

No additional activities require description.

9.0 Laboratory Procedures

9.1 Lab procedures table

Table 8 details the laboratory procedures for the Spokane River Redband Trout PCB Study. Typical detection limits, method detection limits, low calibration limits, and reporting limits for SGS-AXYS's Method MLA-010, their modified version of EPA Method 1668C, are detailed in Appendix A, Table A-1, on a per congener basis.

Table 8. Measurement methods (laboratory).

Analyte	Sample Matrix	Samples (Number/Arrival Date)	Expected Range of Results*	Reporting Limit	Sample Prep Method	Analytical (Instrumental) Method
PCB Congeners	Tissue	≤150 fish to be composited into 30 samples +3 duplicates	4 – 28 ug/Kg total PCBs	0.00000005 ug/Kg per congener	EPA 1668C (AXYS Method MLA-010)	
Lipids			3 – 15%	0.1%	SOP SLA-020 ¹	Gravimetric
Moisture		October 2020	60 – 80%	0.1%	SOP SLA-015 ¹	Gravimetric

* Ranges for PCB congeners and lipids came from the Spokane Hatchery Study where sub-adult whole Rainbow Trout were analyzed for PCBs (Wong, 2018).

¹ = The standard operating procedures (SOPs) for lipids and moisture are proprietary SGS-AXYS methods. SOP SLA-020 is based on dioxin method 1613b.

9.2 Sample preparation method(s)

Sample preparation methods for both PCB congeners and lipids are contained in Method MLA-010 (EPA Method 1668C).

9.3 Special method requirements

There are no special method requirements.

9.4 Laboratories accredited for methods

SGS-AXYS is accredited for analysis of PCB congeners (EPA Method 1668C) in fish tissue.

10.0 Quality Control Procedures

WDFW staff will coordinate with SGS-AXYS Laboratory prior to field activities to coordinate the shipping of samples to ensure that samples are packaged correctly and that the proper international shipping forms are included. Field data will be reviewed for completeness at the end of each sampling occasion. The data will be entered into an Excel spreadsheet and double checked for quality assurance. Copies of the field data sheets and the electronic data set will be provided to SGS-AXYS and LimnoTech following data entry and QAQC. Samples will be labeled in the field and checked against the field data sheet.

10.1 Table of field and laboratory quality control

Each type of QC sample listed in [Table 9](#) has MQOs associated with it (see *Section 6.2*) that will be used to evaluate the quality and usability of the results for this project.

Table 9. Quality control samples, types, and frequency.

Parameter	Field		Laboratory			
	Blanks	Replicates*	Check Standards	Method Blanks	Analytical Duplicates	Matrix Spikes
PCB Congeners	NA	3	1/batch Initial cal. ver. and ongoing cal. ver. if needed (12 hour frequency)	1/batch	2	1/batch
Lipids	NA	3	NA	1/batch	2	NA

*Separate aliquots from three different composite samples and will be given their own sample numbers and analyzed as separate samples, thus serving as field replicates. In addition, the lab will perform two analytical duplicates.

10.2 Corrective action processes

The SGS-AXYS analysts will document whether project data meets method QC criteria. Any departures from normal analytical methods will be documented by the laboratory and described in the data package from the laboratory as well as in the final report for the project. If any samples do not meet QC criteria, the project manager will determine whether data should be reanalyzed, rejected, or used with appropriate qualification.

11.0 Data Management Procedures

11.1 Data recording and reporting requirements

The WDFW will enter field data into an Excel spreadsheet. The data will be double checked for quality assurance. Copies of the field data sheets and the electronic data set will be provided to Ecology and LimnoTech. LimnoTech will provide electronic copies of the final validated PCB congener data to both Ecology and Spokane County.

SRRTTF is responsible for entering the project data into Ecology's Environmental Information System (EIM). An EIM Study ID has already been created for the project: **SRRTTF-RedBT**. Ecology will provide technical assistance if needed for EIM data entry. EIM does not accept QA/QC information such as method blanks, therefore only the final validated and censored data will be entered into EIM.

Amy Sumner from Spokane County will enter the data into the SRRTTF database, which is maintained by the County. The SRRTTF database accepts method blank information and as such it will be entered into the database.

11.2 Laboratory data package requirements

SGS-AXYS will provide a level IV data package that includes a case narrative and final laboratory results. The case narrative will provide QC results, discuss any problems encountered during the analyses, and discuss corrective actions made. This information will be used to help evaluate data quality and determine whether MQOs for this project were met.

11.3 Electronic transfer requirements

Laboratory data will be delivered in the form of an Electronic Data Deliverable (EDD) that meets both LimnoTech's formatting requirements and requirements for uploading into the SRRTTF database.

11.4 EIM/STORET data upload procedures

Data for this project will be loaded into both the SRRTTF database and EIM.

11.5 Model information management

N/A

12.0 Audits and Reports

12.1 Field, laboratory, and other audits

Audits are conducted as a regular part of laboratory operating procedures. Upon request, results of the audits will be made available. No field audits are planned for this project.

12.2 Responsible personnel

The quality assurance manager for SGS-AXYS is responsible for any routine laboratory audits.

12.3 Frequency and distribution of reports

After all data have been received, reviewed, and analyzed, the results of this project will be presented in the form of a draft final report summarizing the study and describing the assessment of current PCB concentrations in sub-adult Redband Trout in the Spokane River study area. The draft will be distributed to the Task Force and Ecology for review, and revised in response to comments.

12.4 Responsibility for reports

The report will be authored by David Dilks of LimnoTech.

13.0 Data Verification

EPA defines data verification as “the process of evaluating the completeness, correctness, and conformance/compliance of a specific data set against the method, procedural, or contractual requirements.”

13.1 Field data verification, requirements, and responsibilities

One field team member from WDFW will enter the field notes into Excel spreadsheets and another team member will review the field notes after entry. Oversight will be provided by the WDFW field lead.

13.2 Laboratory data verification

SGS-AXYS will review and verify laboratory results prior to submitting the data level IV package.

13.3 Validation requirements

The LimnoTech Quality Assurance Coordinator will serve as an independent third party validator, and will review the complete PCB congener data package submitted by the external lab following EPA guidelines ([EPA, 2016](#)), this QAPP, and QC requirements of EPA Method 1668C. The LimnoTech Quality Assurance Coordinator will prepare a report of the Level III data validation, which includes an overall assessment of data quality, usability, and whether project MQOs were met.

13.4 Model quality assessment

NA

14.0 Data Quality (Usability) Assessment

14.1 Process for determining project objectives were met

After data have been independently validated, the project manager will review the data and assess whether project MQOs outlined in [Tables 5 and 6](#) were met. The data will either be accepted, accepted with qualification, or rejected. If MQOs were not met, the project manager will discuss whether any samples should be re-analyzed, or if any other corrective actions should be taken with the laboratory.

14.2 Treatment of non-detects

Non-detected congener results (those qualified as U, UJ, or NUJ) will not be included in calculations of total PCBs. Results qualified as “NJ” (evidence that the analyte is present; result is an estimate) will be included in homolog and total PCB calculations.

EPA Method 1668C allows for low-level detection of PCB congeners. However, PCB congeners may be present in laboratory method blanks at higher concentrations than the detection limit. Different censoring methods can be used to censor results due to method blank contamination. The choice of method depends on study objectives. For example, censoring at <10 times the detected method blank concentration provides the most numerically conservative approach to quantification. It provides the greatest assurance that the analyte present in the sample represents actual sampling site conditions; however, it may lead to the censoring of true positive results. Censoring at <3 times the detected method blank concentration is a useful approach that helps in the ability to detect trends.

For this project, congener results that are less than three times the detected method blank concentration will be qualified as non-detect. Application of this qualification rule aligns with this study’s main objective of identifying trends, and with previous and ongoing work conducted by SRRTTF.

14.3 Data analysis and presentation methods

As these data are designed to define baseline conditions for future trend assessments, no specific numerical analyses are necessary for this project.

14.4 Sampling design evaluation

Five composite samples at each of the six sampling locations should yield enough statistical power to evaluate trends against future monitoring events. This is especially true because the target size of the Redband Trout are from a sub-adult population that should be uniform in age (sexual immaturity), feeding behavior and other factors.

14.5 Documentation of assessment

Data results and discussion will be documented in the final report by LimnoTech.

15.0 References

- Davies, H., 2015. PCB Chemical Action Plan. Washington State Department of Ecology and the Washington State Department of Health, Olympia. Publication No. 15-07-002. <https://fortress.wa.gov/ecy/publications/SummaryPages/1507002.html>
- EPA, 1999. Polychlorinated biphenyls (PCBs) update—impact on fish advisories: Washington, D.C., United States Environmental Protection Agency, EPA 823-F-99-019, 7 p.
- EPA, 2010. Method 1668C: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids and Tissue by HRGC/HRMS. Office of Water, Office of Science and Technology, Engineering and Analysis Division. EPA-820-R-10-005.
- EPA, 2016. National Functional Guidelines for High Resolution Superfund Methods Data Review. EPA Report Number 542-B-16-001. United States Environmental Protection Agency, Washington D.C. https://www.epa.gov/sites/production/files/2016-05/documents/hrsm_nfg.pdf
- Johnson, A., D. Serdar, and D. Davis. 1994. Results of 1993 screening survey on PCBs and metals in the Spokane River. Washington Department of Ecology, Toxics, Compliance and Groundwater Investigations Section, Olympia, WA.
- LimnoTech, 2016. 2016 Comprehensive Plan to Reduce Polychlorinated Biphenyls (PCBs) in the Spokane River. Prepared for the Spokane River Regional Toxics Task Force. http://srrttf.org/wp-content/uploads/2016/04/2016_Comp_Plan_Final_Approved.pdf
- LimnoTech, 2020. Spokane River Regional Toxics Task Force 2020-2021 Water Column PCB Monitoring. Prepared for the Spokane River Regional Toxics Task Force.
- MacCoy, D.E., 2001, PCBs in tissue of fish from the Spokane River, Washington, 1999: U.S. Geological Survey Fact Sheet FS-067-01, 6 p.
- Maret, T.R., and Dutton, D.M., 1999, Summary of information on synthetic organic compounds and trace elements in tissue of aquatic biota, Clark Fork-Pend Oreille and Spokane River Basins, Montana, Idaho, and Washington, 1974-96: U.S.
- Newell, A.J., Johnson, D.W., and Allen, L.K., 1987, Niagara River biota contamination project—fish flesh criteria for piscivorous wildlife: New York State Department of Environmental Conservation, Division of Fish and Wildlife, Bureau of Environmental Protection, Technical Report 87-3, 182 p.
- Parsons, P., D. Hallock, K. Seiders, B. Ward, C. Coffin, E. Newell, C. Deligeannis and K. Welch, 2018. Standard Operating Procedure EAP070, Version 2.2: Minimize the Spread of Invasive Species. Publication 18-03-201. Washington State Department of Ecology, Olympia. <https://fortress.wa.gov/ecy/publications/SummaryPages/1803201.html>
- Sandvik, P. 2018a. Standard Operating Procedure EAP009, Version 1.2: Field Collection, Processing, and Preservation of Finfish Samples at the Time of Collection in the Field. Publication 18-03-237. Washington State Department of Ecology, Olympia. <https://fortress.wa.gov/ecy/publications/SummaryPages/1803237.html>.

- Sandvik, P. 2018b. Standard Operating Procedure EAP007, Version 1.2: Resecting Finfish Whole Body, Body Parts, or Tissue Samples. Publication 18-03-235. Washington State Department of Ecology, Olympia.
<https://fortress.wa.gov/ecy/publications/SummaryPages/1803235.html>.
- Seiders, K. Personal Communication on June 22, 2020 between Keith Seiders, Program Manager for Ecology's Freshwater Fish Contaminant Monitoring Program and Brandee Era-Miller from Ecology's Environmental Assessment Program, Toxics Studies unit.
- Seiders, K., C. Deligeannis, P. Sandvik, and M. McCall. 2014. Freshwater Fish Contaminant Monitoring Program – 2012 Results. Publication No. 04-03-020. Washington State Department of Ecology, Olympia.
<https://fortress.wa.gov/ecy/publications/SummaryPages/1403020.html>
- Serdar, D. and A. Johnson. 2006. PCBs, PBDEs, and Selected Metals in Spokane River Fish, 2005. Publication No. 06-03-025. Washington State Department of Ecology, Olympia.
<https://fortress.wa.gov/ecy/publications/summarypages/0603025.html>
- WDFW, 2020. Washington Department of Fish and Wildlife – Fish Program: COVID-19 Fieldwork Protocol. Washington State Department Fish and Wildlife, Olympia, WA. Updated June 15, 2020.
- Wong, S. 2018. Evaluation of Fish Hatcheries as Sources of PCBs to the Spokane River. Publication No. 18-03-014. Washington State Department of Ecology, Olympia.
<https://fortress.wa.gov/ecy/publications/documents/1803014.pdf>

16.0 Appendices

Appendix A. Reporting Limits for PCBs in Fish Tissue

Table A-1. Typical Detection Limits, Method Detection Limits, Low Calibration Limits, and Reporting Limits for PCBs using SGS-AXYS Method MLA-010 for EPA 1668C.

Matrix	TISSUE					
Units/Sample Size	pg/g based on 10g sample					
Default Extract Volume	20uL					
Analyte	Typical SDL	MDL*	LOQ	LMCL based on CS-1	LMCL based on High Sensitivity CS-0.2	RL ¹
CL1-PCB-1	0.1	1.24	3	2	0.4	0.05
CL1-PCB-2	0.1	0.63	3	2	0.4	0.05
CL1-PCB-3	0.1	1	3	2	0.4	0.05
CL2-PCB-4	0.2	1.07	3	2	0.4	0.05
CL2-PCB-5	0.2	0.68	3	2	0.4	0.05
CL2-PCB-6	0.2	0.89	3	2	0.4	0.05
CL2-PCB-7	0.2	1.88	3	2	0.4	0.05
CL2-PCB-8	0.2	0.94	3	2	0.4	0.05
CL2-PCB-9	0.2	1.02	3	2	0.4	0.05
CL2-PCB-10	0.2	0.96	3	2	0.4	0.05
CL2-PCB-11	0.2	5.61	6	2	0.4	0.05
CL2-PCB-12/13	0.2	0.86	3	2	0.4	0.05
CL2-PCB-14	0.2	0.42	3	2	0.4	0.05
CL2-PCB-15	0.2	0.86	3	2	0.4	0.05
CL3-PCB-16	0.1	1.72	3	2	0.4	0.05
CL3-PCB-17	0.1	1.18	3	2	0.4	0.05
CL3-PCB-19	0.1	1.31	3	2	0.4	0.05
CL3-PCB-21/33	0.1	0.69	3	2	0.4	0.05
CL3-PCB-22	0.1	0.86	3	2	0.4	0.05
CL3-PCB-23	0.1	0.67	3	2	0.4	0.05
CL3-PCB-24	0.1	1.78	3	2	0.4	0.05
CL3-PCB-25	0.1	0.69	3	2	0.4	0.05
CL3-PCB-26/29	0.1	0.69	3	2	0.4	0.05
CL3-PCB-27	0.1	1.21	3	2	0.4	0.05
CL3-PCB-28/20	0.1	2.1	3	2	0.4	0.05
CL3-PCB-30/18	0.1	1.18	3	2	0.4	0.05
CL3-PCB-31	0.1	0.81	3	2	0.4	0.05
CL3-PCB-32	0.1	0.99	3	2	0.4	0.05

Matrix	TISSUE					
Units/Sample Size	pg/g based on 10g sample					
Default Extract Volume	20uL					
Analyte	Typical SDL	MDL*	LOQ	LMCL based on CS-1	LMCL based on High Sensitivity CS-0.2	RL ¹
CL3-PCB-34	0.1	0.61	3	2	0.4	0.05
CL3-PCB-35	0.1	0.6	3	2	0.4	0.05
CL3-PCB-36	0.1	0.72	3	2	0.4	0.05
CL3-PCB-37	0.1	0.84	3	2	0.4	0.05
CL3-PCB-38	0.1	0.54	3	2	0.4	0.05
CL3-PCB-39	0.1	0.22	3	2	0.4	0.05
CL4-PCB-41/40/71	0.1	1.83	3	2	0.4	0.05
CL4-PCB-42	0.1	1.83	3	2	0.4	0.05
CL4-PCB-43	0.1	1.41	3	2	0.4	0.05
CL4-PCB-44/47/65	0.1	1.83	3	2	0.4	0.05
CL4-PCB-45/51	0.1	1.38	3	2	0.4	0.05
CL4-PCB-46	0.1	1.38	3	2	0.4	0.05
CL4-PCB-48	0.1	1.38	3	2	0.4	0.05
CL4-PCB-50/53	0.1	1.38	3	2	0.4	0.05
CL4-PCB-52	0.1	1.5	3	2	0.4	0.05
CL4-PCB-54	0.1	0.9	3	2	0.4	0.05
CL4-PCB-55	0.1	1.11	3	2	0.4	0.05
CL4-PCB-56	0.1	0.49	3	2	0.4	0.05
CL4-PCB-57	0.1	0.69	3	2	0.4	0.05
CL4-PCB-58	0.1	0.93	3	2	0.4	0.05
CL4-PCB-59/62/75	0.1	1.83	3	2	0.4	0.05
CL4-PCB-60	0.1	0.49	3	2	0.4	0.05
CL4-PCB-61/70/74/76	0.1	1.16	3	2	0.4	0.05
CL4-PCB-63	0.1	0.85	3	2	0.4	0.05
CL4-PCB-64	0.1	1.41	3	2	0.4	0.05
CL4-PCB-66	0.1	0.79	3	2	0.4	0.05
CL4-PCB-67	0.1	0.86	3	2	0.4	0.05
CL4-PCB-68	0.1	0.8	3	2	0.4	0.05
CL4-PCB-69/49	0.1	1.41	3	2	0.4	0.05
CL4-PCB-72	0.1	0.81	3	2	0.4	0.05
CL4-PCB-73	0.1	1.36	3	2	0.4	0.05
CL4-PCB-77	0.1	1.06	3	2	0.4	0.05

Matrix	TISSUE					
Units/Sample Size	pg/g based on 10g sample					
Default Extract Volume	20uL					
Analyte	Typical SDL	MDL*	LOQ	LMCL based on CS-1	LMCL based on High Sensitivity CS-0.2	RL ¹
CL4-PCB-78	0.1	0.42	3	2	0.4	0.05
CL4-PCB-79	0.1	0.8	3	2	0.4	0.05
CL4-PCB-80	0.1	0.43	3	2	0.4	0.05
CL4-PCB-81	0.1	1.18	3	2	0.4	0.05
CL5-PCB-82	0.1	0.86	3	2	0.4	0.05
CL5-PCB-83/99	0.1	1.4	3	2	0.4	0.05
CL5-PCB-84	0.1	1.29	3	2	0.4	0.05
CL5-PCB-88/91	0.1	1.29	3	2	0.4	0.05
CL5-PCB-89	0.1	0.76	3	2	0.4	0.05
CL5-PCB-92	0.1	1.03	3	2	0.4	0.05
CL5-PCB-94	0.1	0.94	3	2	0.4	0.05
CL5-PCB-95/100/93/102/98	0.1	1.29	3	2	0.4	0.05
CL5-PCB-96	0.1	1.24	3	2	0.4	0.05
CL5-PCB-103	0.1	1.16	3	2	0.4	0.05
CL5-PCB-104	0.1	0.84	3	2	0.4	0.05
CL5-PCB-105	0.1	1.32	3	2	0.4	0.05
CL5-PCB-106	0.1	0.73	3	2	0.4	0.05
CL5-PCB-108/124	0.1	0.48	3	2	0.4	0.05
CL5-PCB-109/119/86/97/125/87	0.1	1.4	3	2	0.4	0.05
CL5-PCB-107	0.1	0.48	3	2	0.4	0.05
CL5-PCB-110/115	0.1	0.86	3	2	0.4	0.05
CL5-PCB-111	0.1	0.58	3	2	0.4	0.05
CL5-PCB-112	0.1	1.4	3	2	0.4	0.05
CL5-PCB-113/90/101	0.1	1.4	3	2	0.4	0.05
CL5-PCB-114	0.1	1.3	3	2	0.4	0.05
CL5-PCB-117/116/85	0.1	0.86	3	2	0.4	0.05
CL5-PCB-118	0.1	1.18	3	2	0.4	0.05
CL5-PCB-120	0.1	0.7	3	2	0.4	0.05
CL5-PCB-121	0.1	0.67	3	2	0.4	0.05
CL5-PCB-122	0.1	0.23	3	2	0.4	0.05
CL5-PCB-123	0.1	1.26	3	2	0.4	0.05
CL5-PCB-126	0.1	1.33	3	2	0.4	0.05

Matrix	TISSUE					
Units/Sample Size	pg/g based on 10g sample					
Default Extract Volume	20uL					
Analyte	Typical SDL	MDL*	LOQ	LMCL based on CS-1	LMCL based on High Sensitivity CS-0.2	RL ¹
CL5-PCB-127	0.1	0.24	3	2	0.4	0.05
CL6-PCB-128/166	0.1	0.85	3	2	0.4	0.05
CL6-PCB-130	0.1	1.13	3	2	0.4	0.05
CL6-PCB-131	0.1	0.76	3	2	0.4	0.05
CL6-PCB-132	0.1	1.13	3	2	0.4	0.05
CL6-PCB-133	0.1	0.75	3	2	0.4	0.05
CL6-PCB-134/143	0.1	1.02	3	2	0.4	0.05
CL6-PCB-136	0.1	1.3	3	2	0.4	0.05
CL6-PCB-137	0.1	0.8	3	2	0.4	0.05
CL6-PCB-138/163/129/160	0.1	0.85	3	2	0.4	0.05
CL6-PCB-139/140	0.1	0.76	3	2	0.4	0.05
CL6-PCB-141	0.1	1.52	3	2	0.4	0.05
CL6-PCB-142	0.1	1.17	3	2	0.4	0.05
CL6-PCB-144	0.1	1.02	3	2	0.4	0.05
CL6-PCB-145	0.1	1.69	3	2	0.4	0.05
CL6-PCB-146	0.1	1	3	2	0.4	0.05
CL6-PCB-147/149	0.1	1.02	3	2	0.4	0.05
CL6-PCB-148	0.1	0.84	3	2	0.4	0.05
CL6-PCB-150	0.1	1.39	3	2	0.4	0.05
CL6-PCB-151/135/154	0.1	1.02	3	2	0.4	0.05
CL6-PCB-152	0.1	1.35	3	2	0.4	0.05
CL6-PCB-153/168	0.1	1.52	3	2	0.4	0.05
CL6-PCB-155	0.1	1	3	2	0.4	0.05
CL6-PCB-156/157	0.1	1.36	3	2	0.8	0.05
CL6-PCB-158	0.1	0.85	3	2	0.4	0.05
CL6-PCB-159	0.1	0.84	3	2	0.4	0.05
CL6-PCB-161	0.1	1.15	3	2	0.4	0.05
CL6-PCB-162	0.1	0.76	3	2	0.4	0.05
CL6-PCB-164	0.1	0.96	3	2	0.4	0.05
CL6-PCB-165	0.1	0.88	3	2	0.4	0.05
CL6-PCB-167	0.1	1.36	3	2	0.4	0.05
CL6-PCB-169	0.1	1.02	3	2	0.4	0.05

Matrix	TISSUE					
Units/Sample Size	pg/g based on 10g sample					
Default Extract Volume	20uL					
Analyte	Typical SDL	MDL*	LOQ	LMCL based on CS-1	LMCL based on High Sensitivity CS-0.2	RL ¹
CL7-PCB-170	0.1	0.77	3	2	0.4	0.05
CL7-PCB-171/173	0.1	1.21	3	2	0.4	0.05
CL7-PCB-172	0.1	0.91	3	2	0.4	0.05
CL7-PCB-174	0.1	1.25	3	2	0.4	0.05
CL7-PCB-175	0.1	1.3	3	2	0.4	0.05
CL7-PCB-176	0.1	1.35	3	2	0.4	0.05
CL7-PCB-177	0.1	0.84	3	2	0.4	0.05
CL7-PCB-178	0.1	0.74	3	2	0.4	0.05
CL7-PCB-179	0.1	1.08	3	2	0.4	0.05
CL7-PCB-180/193	0.1	0.79	3	2	0.4	0.05
CL7-PCB-181	0.1	1.21	3	2	0.4	0.05
CL7-PCB-182	0.1	1.38	3	2	0.4	0.05
CL7-PCB-183/185	0.1	1.38	3	2	0.4	0.05
CL7-PCB-184	0.1	1.1	3	2	0.4	0.05
CL7-PCB-186	0.1	1.24	3	2	0.4	0.05
CL7-PCB-187	0.1	1.11	3	2	0.4	0.05
CL7-PCB-188	0.1	1.02	3	2	0.4	0.05
CL7-PCB-189	0.1	0.6	3	2	0.4	0.05
CL7-PCB-190	0.1	0.75	3	2	0.4	0.05
CL7-PCB-191	0.1	0.83	3	2	0.4	0.05
CL7-PCB-192	0.1	0.79	3	2	0.4	0.05
CL8-PCB-194	0.1	0.83	3	2	0.4	0.05
CL8-PCB-195	0.1	1.01	3	2	0.4	0.05
CL8-PCB-196	0.1	1.21	3	2	0.4	0.05
CL8-PCB-197/200	0.1	0.93	3	2	0.4	0.05
CL8-PCB-198/199	0.1	1.21	3	2	0.4	0.05
CL8-PCB-201	0.1	1.21	3	2	0.4	0.05
CL8-PCB-202	0.1	1.14	3	2	0.4	0.05
CL8-PCB-203	0.1	1.04	3	2	0.4	0.05
CL8-PCB-204	0.1	0.93	3	2	0.4	0.05
CL8-PCB-205	0.1	1.03	3	2	0.4	0.05
CL9-PCB-206	0.1	1.11	3	2	0.4	0.05

Matrix	TISSUE					
Units/Sample Size	pg/g based on 10g sample					
Default Extract Volume	20uL					
Analyte	Typical SDL	MDL*	LOQ	LMCL based on CS-1	LMCL based on High Sensitivity CS-0.2	RL ¹
CL9-PCB-207	0.1	2.69	3	2	0.4	0.05
CL9-PCB-208	0.1	0.99	3	2	0.4	0.05
CL10-PCB-209	0.1	0.96	3	2	0.4	0.05

* = Federal Register 40 CFR Part 136, Appendix B Rev.1, MDLs determined according to Rev. 2 (2017).

1 = Reporting Limit (RL) is the lowest concentration routinely reported for the method. RLs are set to minimize potential for false positive detection or the requirement to qualify results very close to detection limit and in some cases may exceed the sample specific detection limit (SDL) achieved.

Appendix B. Glossaries, Acronyms, and Abbreviations

Glossary of General Terms

Clean Water Act: A federal act passed in 1972 that contains provisions to restore and maintain the quality of the nation's waters. Section 303(d) of the Clean Water Act establishes the TMDL program.

Effluent: An outflowing of water from a natural body of water or from a human-made structure. For example, the treated outflow from a wastewater treatment plant.

Reach: A specific portion or segment of a stream.

Salmonid: Fish that belong to the family *Salmonidae*. Species of salmon, trout, or char.

Sediment: Soil and organic matter that is covered with water (for example, river or lake bottom).

Stormwater: The portion of precipitation that does not naturally percolate into the ground or evaporate but instead runs off roads, pavement, and roofs during rainfall or snow melt. Stormwater can also come from hard or saturated grass surfaces such as lawns, pastures, playfields, and from gravel roads and parking lots.

303(d) list: Section 303(d) of the federal Clean Water Act, requiring Washington State to periodically prepare a list of all surface waters in the state for which beneficial uses of the water – such as for drinking, recreation, aquatic habitat, and industrial use – are impaired by pollutants. These are water quality-limited estuaries, lakes, and streams that fall short of state surface water quality standards and are not expected to improve within the next two years.

Acronyms and Abbreviations

e.g.	For example
Ecology	Washington State Department of Ecology
EIM	Environmental Information Management database
EPA	U.S. Environmental Protection Agency
et al.	And others
GPS	Global Positioning System
HED	Hydroelectric Development
i.e.	In other words
LOQ	Level of quantification
MQO	Measurement quality objective
PBT	Persistent, bioaccumulative, and toxic substance
PCB	Polychlorinated biphenyls
ppm	part per million
QA	Quality assurance
QC	Quality control
rkm	river kilometer
RM	River mile
RPD	Relative percent difference
RSD	Relative standard deviation
SOP	Standard operating procedures

SRRTTF	Spokane River Regional Toxics Taskforce
WDFW	Washington Department of Fish and Wildlife
WRIA	Water Resource Inventory Area

Units of Measurement

°C	degrees centigrade
Ft	feet
g	gram, a unit of mass
Kg	kilograms, a unit of mass equal to 1,000 grams
km	kilometer, a unit of length equal to 1,000 meter
mm	millimeter
m ³ /s	meters cubed per second (a measure of river discharge or flow)
ng/mL	nanograms per milliliter
oz	once
pg/g	picograms per gram (parts per trillion)
TL	total length
µg/kg	micrograms per kilogram (parts per billion)
µL	microliter
WT	weight
ww	wet weight

Quality Assurance Glossary

Accreditation: A certification process for laboratories, designed to evaluate and document a lab’s ability to perform analytical methods and produce acceptable data. For Ecology, it is “Formal recognition by (Ecology)...that an environmental laboratory is capable of producing accurate analytical data.” [WAC 173-50-040] (Kammin, 2010)

Accuracy: The degree to which a measured value agrees with the true value of the measured property. USEPA recommends that this term not be used, and that the terms *precision* and *bias* be used to convey the information associated with the term *accuracy* (USGS, 1998).

Analyte: An element, ion, compound, or chemical moiety (pH, alkalinity) which is to be determined. The definition can be expanded to include organisms, e.g., fecal coliform, *Klebsiella* (Kammin, 2010).

Bias: The difference between the sample mean and the true value. Bias usually describes a systematic difference reproducible over time and is characteristic of both the measurement system and the analyte(s) being measured. Bias is a commonly used data quality indicator (DQI) (Kammin, 2010; Ecology, 2004).

Blank: A synthetic sample, free of the analyte(s) of interest. For example, in water analysis, pure water is used for the blank. In chemical analysis, a blank is used to estimate the analytical response to all factors other than the analyte in the sample. In general, blanks are used to assess possible contamination or inadvertent introduction of analyte during various stages of the sampling and analytical process (USGS, 1998).

Calibration: The process of establishing the relationship between the response of a measurement system and the concentration of the parameter being measured (Ecology, 2004).

Check standard: A substance or reference material obtained from a source independent from the source of the calibration standard; used to assess bias for an analytical method. This is an obsolete term, and its use is highly discouraged. See Calibration Verification Standards, Lab Control Samples (LCS), Certified Reference Materials (CRM), and/or spiked blanks. These are all check standards but should be referred to by their actual designator, e.g., CRM, LCS (Kammin, 2010; Ecology, 2004).

Comparability: The degree to which different methods, data sets and/or decisions agree or can be represented as similar; a data quality indicator (USEPA, 1997).

Completeness: The amount of valid data obtained from a project compared to the planned amount. Usually expressed as a percentage. A data quality indicator (USEPA, 1997).

Continuing Calibration Verification Standard (CCV): A quality control (QC) sample analyzed with samples to check for acceptable bias in the measurement system. The CCV is usually a midpoint calibration standard that is re-run at an established frequency during the course of an analytical run (Kammin, 2010).

Control chart: A graphical representation of quality control results demonstrating the performance of an aspect of a measurement system (Kammin, 2010; Ecology 2004).

Control limits: Statistical warning and action limits calculated based on control charts. Warning limits are generally set at ± 2 standard deviations from the mean, action limits at ± 3 standard deviations from the mean (Kammin, 2010).

Data integrity: A qualitative DQI that evaluates the extent to which a data set contains data that is misrepresented, falsified, or deliberately misleading (Kammin, 2010).

Data quality indicators (DQI): Commonly used measures of acceptability for environmental data. The principal DQIs are precision, bias, representativeness, comparability, completeness, sensitivity, and integrity (USEPA, 2006).

Data quality objectives (DQO): Qualitative and quantitative statements derived from systematic planning processes that clarify study 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 data needed to support decisions (USEPA, 2006).

Data set: A grouping of samples organized by date, time, analyte, etc. (Kammin, 2010).

Data validation: An analyte-specific and sample-specific process that extends the evaluation of data beyond data verification to determine the usability of a specific data set. It involves a detailed examination of the data package, using both professional judgment and objective criteria, to determine whether the MQOs for precision, bias, and sensitivity have been met. It may also include an assessment of completeness, representativeness, comparability, and integrity, as these criteria relate to the usability of the data set. Ecology considers four key criteria to determine if data validation has actually occurred. These are:

- Use of raw or instrument data for evaluation.
- Use of third-party assessors.
- Data set is complex.
- Use of EPA Functional Guidelines or equivalent for review.

Examples of data types commonly validated would be:

- Gas Chromatography (GC).
- Gas Chromatography-Mass Spectrometry (GC-MS).
- Inductively Coupled Plasma (ICP).

The end result of a formal validation process is a determination of usability that assigns qualifiers to indicate usability status for every measurement result. These qualifiers include:

- No qualifier – data are usable for intended purposes.
- J (or a J variant) – data are estimated, may be usable, may be biased high or low.
- REJ – data are rejected, cannot be used for intended purposes.

(Kammin, 2010; Ecology, 2004).

Data verification: Examination of a data set for errors or omissions, and assessment of the Data Quality Indicators related to that data set for compliance with acceptance criteria (MQOs).

Verification is a detailed quality review of a data set (Ecology, 2004).

Detection limit (limit of detection): The concentration or amount of an analyte which can be determined to a specified level of certainty to be greater than zero (Ecology, 2004).

Duplicate samples: Two samples taken from and representative of the same population, and carried through and steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variability of all method activities including sampling and analysis (USEPA, 1997).

Field blank: A blank used to obtain information on contamination introduced during sample collection, storage, and transport (Ecology, 2004).

Initial Calibration Verification Standard (ICV): A QC sample prepared independently of calibration standards and analyzed along with the samples to check for acceptable bias in the measurement system. The ICV is analyzed prior to the analysis of any samples (Kammin, 2010).

Laboratory Control Sample (LCS): A sample of known composition prepared using contaminant-free water or an inert solid that is spiked with analytes of interest at the midpoint of the calibration curve or at the level of concern. It is prepared and analyzed in the same batch of regular samples using the same sample preparation method, reagents, and analytical methods employed for regular samples (USEPA, 1997).

Matrix spike: A QC sample prepared by adding a known amount of the target analyte(s) to an aliquot of a sample to check for bias due to interference or matrix effects (Ecology, 2004).

Measurement Quality Objectives (MQOs): Performance or acceptance criteria for individual data quality indicators, usually including precision, bias, sensitivity, completeness, comparability, and representativeness (USEPA, 2006).

Measurement result: A value obtained by performing the procedure described in a method (Ecology, 2004).

Method: A formalized group of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, data analysis), systematically presented in the order in which they are to be executed (EPA, 1997).

Method blank: A blank prepared to represent the sample matrix, prepared and analyzed with a batch of samples. A method blank will contain all reagents used in the preparation of a sample, and the same preparation process is used for the method blank and samples (Ecology, 2004; Kammin, 2010).

Method Detection Limit (MDL): This definition for detection was first formally advanced in 40CFR 136, October 26, 1984 edition. MDL is defined there as the minimum concentration of an analyte that, in a given matrix and with a specific method, has a 99% probability of being identified, and reported to be greater than zero (Federal Register, October 26, 1984).

Percent Relative Standard Deviation (%RSD): A statistic used to evaluate precision in environmental analysis. It is determined in the following manner:

$$\%RSD = (100 * s)/x$$

where s is the sample standard deviation and x is the mean of results from more than two replicate samples (Kammin, 2010).

Parameter: A specified characteristic of a population or sample. Also, an analyte or grouping of analytes. Benzene and nitrate + nitrite are all parameters (Kammin, 2010; Ecology, 2004).

Population: The hypothetical set of all possible observations of the type being investigated (Ecology, 2004).

Precision: The extent of random variability among replicate measurements of the same property; a data quality indicator (USGS, 1998).

Quality assurance (QA): A set of activities designed to establish and document the reliability and usability of measurement data (Kammin, 2010).

Quality Assurance Project Plan (QAPP): A document that describes the objectives of a project, and the processes and activities necessary to develop data that will support those objectives (Kammin, 2010; Ecology, 2004).

Quality control (QC): The routine application of measurement and statistical procedures to assess the accuracy of measurement data (Ecology, 2004).

Relative Percent Difference (RPD): RPD is commonly used to evaluate precision. The following formula is used:

$$[Abs(a-b)/((a + b)/2)] * 100$$

where “Abs()” is absolute value and a and b are results for the two replicate samples. RPD can be used only with 2 values. Percent Relative Standard Deviation is (%RSD) is used if there are results for more than 2 replicate samples (Ecology, 2004).

Replicate samples: Two or more samples taken from the environment at the same time and place, using the same protocols. Replicates are used to estimate the random variability of the material sampled (USGS, 1998).

Representativeness: The degree to which a sample reflects the population from which it is taken; a data quality indicator (USGS, 1998).

Sample (field): A portion of a population (environmental entity) that is measured and assumed to represent the entire population (USGS, 1998).

Sample (statistical): A finite part or subset of a statistical population (USEPA, 1997).

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 (Ecology, 2004).

Spiked blank: A specified amount of reagent blank fortified with a known mass of the target analyte(s); usually used to assess the recovery efficiency of the method (USEPA, 1997).

Spiked sample: A sample prepared by adding a known mass of target analyte(s) to a specified amount of matrix sample for which an independent estimate of target analyte(s) concentration is available. Spiked samples can be used to determine the effect of the matrix on a method's recovery efficiency (USEPA, 1997).

Split sample: A discrete sample subdivided into portions, usually duplicates (Kammin, 2010).

Standard Operating Procedure (SOP): A document which describes in detail a reproducible and repeatable organized activity (Kammin, 2010).

Surrogate: For environmental chemistry, a surrogate is a substance with properties similar to those of the target analyte(s). Surrogates are unlikely to be native to environmental samples. They are added to environmental samples for quality control purposes, to track extraction efficiency and/or measure analyte recovery. Deuterated organic compounds are examples of surrogates commonly used in organic compound analysis (Kammin, 2010).

Systematic planning: A step-wise process which develops a clear description of the goals and objectives of a project, and produces decisions on the type, quantity, and quality of data that will be needed to meet those goals and objectives. The DQO process is a specialized type of systematic planning (USEPA, 2006).

References for QA Glossary

Ecology, 2004. Guidance for the Preparation of Quality Assurance Project Plans for Environmental Studies. Washington State Department of Ecology, Olympia, WA.

<https://fortress.wa.gov/ecy/publications/SummaryPages/0403030.html>.

Kammin, B., 2010. Definition developed or extensively edited by William Kammin, 2010. Washington State Department of Ecology, Olympia, WA.

USEPA, 2006. Guidance on Systematic Planning Using the Data Quality Objectives Process EPA QA/G-4. <http://www.epa.gov/quality/qs-docs/g4-final.pdf>.

USGS, 1998. Principles and Practices for Quality Assurance and Quality Control. Open-File Report 98-636. U.S. Geological Survey.
<http://ma.water.usgs.gov/fhwa/products/ofr98-636.pdf>.