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Quality Assurance Project Plan

Little Spokane River PCBs in Fish Tissue Verification Study

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Each study conducted by the Washington State Department of Ecology (Ecology) must have an approved Quality Assurance Project Plan. The plan describes the objectives of the study and the procedures to be followed to achieve those objectives. After completing the study, Ecology will post the final report of the study to the Internet.

This Quality Assurance Project Plan is available on Ecology's website at https://fortress.wa.gov/ecy/publications/SummaryPages/1403127.html

Data for this project will be available on Ecology's Environmental Information Management (EIM) website at www.ecy.wa.gov/eim/index.htm. Search Study ID RCOO0015.

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Quality Assurance Project Plan

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December 2014

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EAP: Environmental Assessment Program

1.0 Title Page and Table of Contents

Table of Contents

List of Figures and Tables

Page

Figures

Tables

2.0 Abstract

Previous studies by the Washington State Department of Ecology (Ecology) have identified elevated levels of polychlorinated biphenyls (PCBs) in fish tissue from the Little Spokane River. As a result, the lower section of the river has been listed in Category 5 of the 303(d) list as being water quality-impaired for PCBs in fish tissue. Recent efforts by the Spokane River Regional Toxics Task Force to characterize and reduce PCB concentrations in the Spokane River have raised questions about PCB concentrations in Spokane River tributaries.

This Quality Assurance Project Plan (QAPP) describes a study to verify the levels of PCBs in Little Spokane River fish. In addition, water samples and streambed sediments will be collected and analyzed to spatially characterize PCB concentrations within the Little Spokane River. Water samples will be collected from upstream and downstream of three permitted dischargers. Ecology will evaluate PCB concentrations in the 303(d) listed portion of the river and compare to those of upstream reaches that are not listed as water quality-impaired for PCBs. Sediment samples will be collected from the mouths of major Little Spokane River tributaries and other locations throughout the river in an effort to spatially characterize PCB concentrations within the drainage.

3.0 Background

The Little Spokane River drains 700 square miles of Spokane, Pend Oreille, and Stevens Counties in northeast Washington, as well as Bonner County in the state of Idaho. The river is one of two major tributaries to the Spokane River (Hangman Creek is the other). The river discharges into the Spokane River at River Mile (RM) 56.3, located in Lake Spokane (Figure 1).

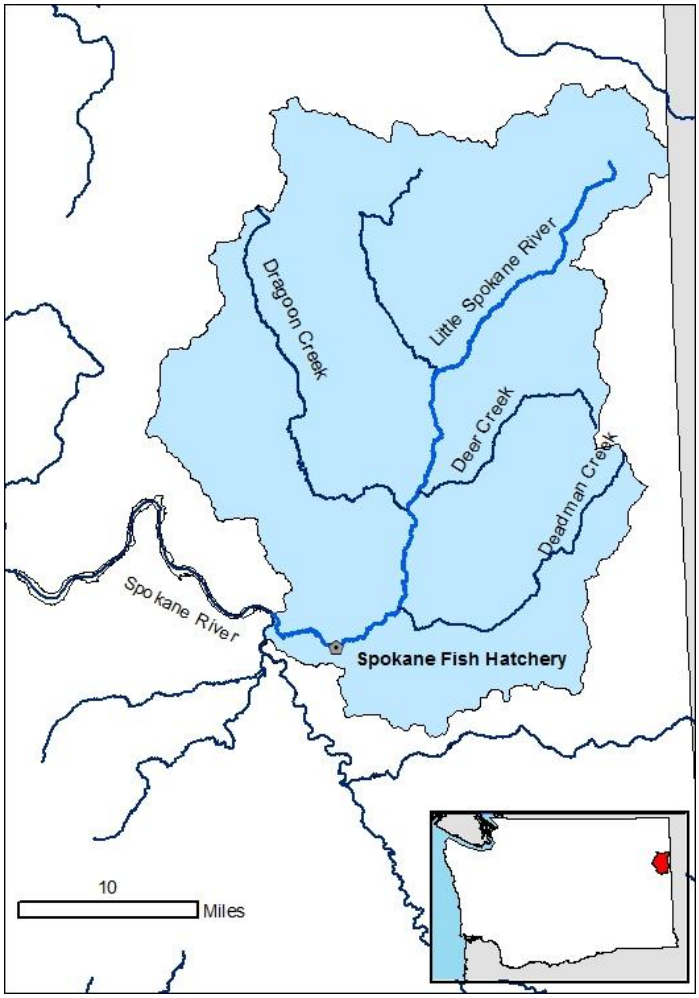

Figure 1. Study Area for Little Spokane River PCB Verification

Previous studies by Ecology have reported elevated PCB levels in fish tissue in 1994 (Ecology, 1995) and again in 1996 (Johnson, 1997). Because of these findings, the Little Spokane River was placed on the 1996 303(d) list (Category 5) as being water quality-impaired for PCBs and has remained on the impaired waters list during subsequent water quality assessments. This study will evaluate if PCBs are still a concern in Little Spokane River fish tissue. PCB contamination in different reaches of the Little Spokane River will be assessed to identify sections of the watershed that may contain sources of PCBs.

Largescale suckers and mountain whitefish were the species of fish that earlier studies reported as having elevated concentrations of PCBs. If PCBs are detected in fish tissue during this study, it would be beneficial if Ecology could identify a source. Since these fish are migratory within river systems (Schmetterling and McFee, 2006; McPhail and Troffe, 1998) and they are free to migrate, their PCB concentrations may not reflect contamination in the Little Spokane River watershed. Sediment and water samples will be tested for PCBs to attempt to determine if PCB contamination originates within the Little Spokane River drainage.

3.1 Study area and surroundings

The study area consists of forest, rangeland, agriculture, and urban development (Ecology, 1995). Urban areas within the watershed are the cities of Spokane and Deer Park, and the Town of Mead. The lower 8 miles of the river were designated a State Scenic River corridor in 1991 by the State Legislature. The upstream section of the river between Highway 2 and 395 is within the Spokane Urban Growth Area. Upstream of Highway 2, land use is rural residential, agricultural, and forest. Groundwater contributes significantly to Little Spokane River flow. See Figures 1 and 2 for maps of the study area.

Three permitted facilities discharge water into the Little Spokane River. The Colbert Landfill discharges treated groundwater that was contaminated with organic solvents from 1975 to 1980 (EPA, 2009). The WDFW Spokane Fish Hatchery at Griffith Springs discharges water from the hatchery. The former Kaiser Aluminum Plant in Mead is now NMC Mead LLC and is an inactive industrial site with an active NPDES permit (Joy, 2013). This facility is currently discharging stormwater under their permit. Ecology has documented that this facility has contaminated groundwater with fluoride and cyanide, which flows into the Little Spokane River (Ecology Agreed Order, 2001).

3.1.1 Logistical problems

The land around the Little Spokane River is mostly privately owned. Access to the upper reaches (above the hatchery) will be limited. The upstream portion of the river will have to be fished by using either a backpack shocker or hook and line. These sections will require landowner permission to gain access. Field staff may conduct additional sampling from an electrofishing raft, while being respectful of surrounding private property. Restrictive Riverside Park rules in the lower Little Spokane River must be taken into account.

3.1.2 History of study area

The first human inhabitants of the study area were the Spokane Tribe of Indians. With European settlement in the late 1700s, the population increased and began to impact the watershed. Logging was the first industry in the area. Sawmills in small communities supplied the Great Northern Railroad with wood to construct the trestles and railways.

After depleting easily accessible timber, communities shifted industry from forestry to agriculture. This remains one of the main industries in the watershed today. Many towns and cities in the study area now serve as bedroom communities for the greater Spokane area.

Development of residential and commercial properties have depleted riparian vegetation and increased impervious surfaces. This has contributed to temperature concerns in the Little Spokane River. Agriculture and urbanization have increased fecal coliform bacteria and suspended solids in the river (Joy, 2013). Streamflow has been declining as populations have increased. Withdrawals of groundwater and surface water for irrigation and domestic use have contributed to lower streamflows.

3.1.3 Contaminants of concern

PCBs are the contaminant of concern in the Little Spokane River for this study. This river was placed in Category 5 (on the 303(d) list) of the state Water Quality Assessment as being water quality-limited for PCBs in fish tissue. There are 2 category 5 listings on this river for PCBs in fish tissue. Both listings are for mountain whitefish, the first in 1995 and the second in 1997.

PCBs are legacy pollutants that have been used in electric insulators, flame retardants and heat-transfer fluids, hydraulic fluid, lubricating oil, and additives in paints, carbonless copy paper, adhesives, sealants, and plastics. The most common use of PCBs was in capacitors and transformers. After being recognized as toxic and persistent in the environment, PCBs were banned in the United States in 1979. PCBs have been determined to be carcinogens in laboratory tests on animals and evidence suggests they are human carcinogens (EPA, 2013). Due to the stability of these persistent organic pollutants, PCBs are widely present in sediments and biological organisms. Since PCBs are bioaccumulative, higher levels of PCBs will be detected in organisms than in the environment. Currently the intentional manufacture of PCBs is limited to small amounts for research purposes (Erickson and Kaley, 2010). Inadvertent production of PCBs is permissible at concentrations of less than 50 ppm under the Toxic Substances Control Act.

The Little Spokane River is also 303-d listed for temperature, fecal coliform bacteria, and turbidity. A TMDL assessment addressing these issues was recently completed (Joy, 2013). This TMDL was approved by EPA in 2012. All 303(d) listings for the Little Spokane River watershed are listed in Table 1-A of the Appendix.

3.1.4 Results of previous studies

Ecology studies have identified elevated levels of PCBs in Little Spokane River fish (Table 1) (Ecology, 1995; Johnson, 1997). In 1993, composite samples of mountain whitefish were found to have concentrations of total PCBs ranging from 145 to 285 ug/Kg, while a single cutthroat trout was reported at 118 ug/Kg. In 1996, Ecology collected more mountain whitefish and the levels of PCBs in the fish composites were 53 to 164 ug/Kg. Concentrations of PCBs in largescale suckers (whole fish) collected in 1994 and 1996 were reported to be 440 and 336 ug/Kg, respectively (Ecology, 1995; Johnson, 1997). Table 1 describes the results of previous fish tissue studies on the Little Spokane River.

Table 1. Total PCB concentration in Little Spokane River Fish from Previous Studies

Species	Concentration (ug/kg)	Sample Type	Year Collected	Number in Composite	Data Source
Mountain whitefish	145	Fillet	1994	8	Ecology, 1995
Mountain whitefish	235	Fillet	1994	8	Ecology, 1995
Mountain whitefish	285	Fillet	1994	8	Ecology, 1995
Cutthroat trout	188	Fillet	1994	1	Ecology, 1995
Largescale Sucker	440	Whole Fish	1994	5	Ecology, 1995
Mountain whitefish	164	Fillet	1996	8	Johnson, 1997
Mountain whitefish	130	Fillet	1996	8	Johnson, 1997
Mountain whitefish	53	Fillet	1996	8	Johnson, 1997
Largescale Sucker	336	Whole Fish	1996	5	Johnson, 1997

3.1.5 Regulatory criteria or standards

PCB concentrations will be compared to the appropriate regulatory standards (Table 2). The fish tissue equivalent concentration (FTEC) is the regulatory criteria for several contaminants such as PCBs, DDT, or dioxins and furans. The FTEC is calculated by multiplying the water quality criteria for human health (National Toxics Rule; NTR) with a bioconcentration factor that estimates how much the concentration of a contaminant will increase as it moves to a higher trophic level. Contaminants in fish tissue can then be used to evaluate if water quality criteria have been met for hydrophobic and lipophilic contaminants, which can be difficult to detect in whole water.

Table 2. Regulatory Criteria for Comparison of Total PCBs in Fish, Water, and Sediments.

Analyte	Matrix	Regulatory Criteria	Concentration (ppb)
Total PCBs	water	aquatic toxicity criteria[1] (acute)	2
Total PCBs	water	aquatic toxicity criteria[1] (chronic)	0.014
Total PCBs	water	human health criteria (NTR)	0.00017
Total PCBs	sediment	sediment cleanup objective[2]	110
Total PCBs	fish tissue	human health criteria (NTR)	5.3

[1] WAC 173-201A
[2] WAC 173-204

4.0 Project Description

Elevated levels of PCBs have been detected in Little Spokane River fish. This project will verify if elevated concentrations of PCBs remain a concern in fish from the Little Spokane River. Results from the analysis of water and sediment will attempt to establish if PCB contamination in fish tissue could be a result of PCB levels in the river. Additional sediment samples will be collected to determine if certain reaches or tributaries have higher PCB concentrations. This could potentially indicate PCB sources.

4.1 Project goals

The goal of the study is to determine if Little Spokane River trout, mountain whitefish, or other resident fish are within or continue to exceed water quality criteria (as FTEC) for PCBs in fish tissue. A secondary goal is to collect water and sediment samples to compare PCB concentrations in different reaches and tributaries of the Little Spokane River.

4.2 Project objectives

- Collect fish as composites upstream and downstream of the Spokane fish hatchery and analyze for total PCBs.
- Composite sediment samples from eight stream locations and analyze for total PCBs, plus ancillary parameters percent fines and total organic carbon (TOC).
- Collect water by Continuous Low-Level Aquatic Monitoring (CLAM) devices at two stream locations during high and low flow.

4.3 Information needed and sources

Not applicable. Previous data is available in Ecology publications.

4.4 Target population

The study proposes to determine whether fish within the Little Spokane River are impaired by levels of total PCBs in edible tissue. The target population is resident fish from the Little Spokane River. Mountain whitefish and cutthroat trout will be targeted. If target species are not available, other resident species will be substituted. Enough fish will be collected to make up 4 composite samples of three to five fish each, collected above and below the Little Spokane River fish hatchery.

4.5 Study boundaries

All study sampling will occur within the Little Spokane River basin (Figure 1).

Water Resource Inventory Area (WRIA) and 8-digit Hydrologic Unit Code (HUC) numbers for the study area:

WRIA
- 55

HUC numbers
- 170103080305
- 170103080306

4.6 Tasks required

- Preparation
  - Secure river access
  - Secure contract lab for PCB congener analysis
  - Reconnaissance of put in and take out for boat access
  - Reconnaissance of sediment sampling locations
  - Complete rental agreement for CLAM water samplers
  - Compile necessary equipment, e.g., ponar, sample containers, and shock raft
- Sampling
  - Deploy and retrieve CLAM water samplers
    - 2 sample locations
    - 2 deployments- high/low flow
  - Collect sediment samples
    - 8 sample locations
    - 1 composite sample from each location
  - Collect fish samples
    - 2 sample locations
    - 2 composites for each sample location
- Post sampling
  - Process sediment and fish samples, send to lab
  - Verify quality of lab data
  - Data analysis
  - Data entry (EIM)
  - Report preparation

4.7 Practical constraints

Water sampling will be conducted during low flow and high flow conditions. Weather conditions will be followed to ensure water samples are collected during low and high flow conditions.

As mentioned in Section 3.1.1., access to the river will be a concern. Ecology staff will only access the river or tributaries through public right of ways or through property where permission for access has been granted.

Whenever a project involves fish collection, there is uncertainty concerning fish species and availability. Ecology will sample fish opportunistically. The target species are cutthroat trout and mountain whitefish. The collection goal is 10 fish of each species. If target species are not present, other species will be substituted.

4.8 Systematic planning process

This QAPP will be sufficient to address the planning process.

5.0 Organization and Schedule

5.1 Key individuals and their responsibilities

Table 3. Organization of Project Staff and Responsibilities

Staff (all are EAP except client)	Title	Responsibilities
Adriane Borgias Water Quality Program Eastern Regional Office Phone: 509- 329-3515	EAP Client	Clarifies scope of the project. Provides internal review of the QAPP and approves the final QAPP.
Randy Coots Toxics Studies Unit SCS Phone: 360- 407-6690	Project Manager	Writes the QAPP. Oversees field sampling and transportation of samples to the laboratory. Conducts QA review of data, analyzes and interprets data, and enters data into EIM. Writes the draft report and final report.
Michael Friese Toxics Studies Unit SCS Phone: 360- 407-6737	Principal Investigator	Writes the QAPP. Oversees field sampling and transportation of samples to the laboratory. Conducts QA review of data, analyzes and interprets data, and enters data into EIM. Writes the draft report and final report.
Dale Norton Toxics Studies Unit SCS Phone: 360- 407-6765	Unit Supervisor for the Project Manager	Provides internal review of the QAPP, approves the budget, and approves the final QAPP.
Will Kendra SCS Phone: 360- 407-6698	Section Manager for the Project Manager	Reviews the project scope and budget, tracks progress, reviews the draft QAPP, and approves the final QAPP.
Tom Mackie Phone: 509- 454-4244	Section Manager for the Study Area	Reviews the project scope and budget, tracks progress, reviews the draft QAPP, and approves the final QAPP.
Joel Bird Manchester Environmental Laboratory Phone: 360-871-8801	Director	Reviews and approves the final QAPP.
Contract Laboratory	Project Manager	Reviews draft QAPP, coordinates with MEL QA Coordinator.
William R. Kammin Phone: 360-407-6964	Ecology Quality Assurance Officer	Reviews and approves the draft QAPP and the final QAPP.

EAP: Environmental Assessment Program
EIM: Environmental Information Management database
QAPP: Quality Assurance Project Plan
SCS: Statewide Coordination Section

5.2 Special training and certifications

The field lead and field assistant will be familiar with the following Ecology Standard Operating Procedures (SOPs):

- EAP090 - *Decontaminating Field Equipment for Sampling Toxics in the Environment* (Friese, 2014).
- EAP070 - *Minimizing the Spread of Invasive Species* (Parsons et al., 2012).
- EAP009 - *Collection, Processing, and Preservation of Finfish Samples* (Sandvik, 2014).
- EAP040 - *Freshwater Sediment Sampling* (Blakley, 2008).
- EAP015 - *Standard Operating Procedure for Grab Sampling − Freshwater* (Joy, 2006).

All field staff will be current on EAP safety and first aid training.

5.3 Organization chart

See Section 5.1.

5.4 Project schedule

Table 4. Proposed Schedule for Completing Field and Laboratory Work, Data Entry into EIM, and Reports.

Field and laboratory work	Due date	Lead staff
Field work completed	May 2015	Michael Friese
Laboratory analyses completed	July 2015	
Environmental Information System database (EIM)		
EIM Study ID	RCOO0015	
Product	Due date	Lead staff
EIM data loaded	September 2015	Michael Friese
EIM data entry review	October 2015	New staff (NRS1)
EIM complete	November 2015	Michael Friese
Activity Tracker code	15-039	
Author lead	Michael Friese and Randy Coots	
Schedule		
Draft due to supervisor	September 2015	
Draft due to client/peer reviewer	October 2015	
Draft due to external reviewer(s)	November 2015	
Final (all reviews done) due to publications coordinator	December 2015	
Final report due on web	January 2016	

5.5 Limitations on schedule

Not applicable. There are no known limitations on schedule.

5.6 Budget and funding

Table 5. Estimated Lab Budget.

Analyte	Samples	QA	Cost($)	MEL($)	Contract($)[1]
Fish					
PCB Congeners	4	2	650	975	3900
Percent Lipids	4	1	n/a		0
Water[2]					
CLAM discs[3]	4	8	160	480	1920
PCB Congeners[4]	4	4	650	1300	5200
TOC	4	1	45	225	
TSS	4	1	12	60	
Sediment					
PCB Congeners	9	2	650	1625	7150
TOC	9	1	45	450	
Grain Size	9	1	100	1000	
		MEL Subtotal		6115	
		Contract Subtotal			18170
		Grand Total			24285

[1] Contract lab prices include a 25% additional contract charge (see MEL column).
[2] Water will be sampled with Continuous Low-Level Aquatic Monitoring (CLAMs) instruments.
[3] CLAM discs will be purchased, preconditioned, and spiked by the contract lab.
[4] Method blank and ongoing precision and recovery (OPR) QA samples will be analyzed free of charge.
TOC: Total organic carbon
TSS: Total suspended solids

6.0 Quality Objectives

6.1 Decision quality objectives (DQOs)

Concentrations of PCBs in fish tissue will be compared to Washington FTECs to determine if the Little Spokane River will remain in Category 5 of the 303(d) list (see Table 1).

6.2 Measurement quality objectives (MQOs)

Table 6. Measurement Quality Objectives.

Analyte	Lab Control Standards (%Recovery)[6]	Laboratory Duplicates (RPD)[1]	Recoveries (%Recovery)	Lowest Concentration of Interest
Fish Tissue				
PCB congeners	Internal Standards and Labeled compounds	≤50%	NA[3]	5 ug/Kg, ww
Lipids	0%-20%	≤20%	NA	0.1%
Water				
PCB Congeners	Labeled congeners	≤50%	25-150%[2]	10 pg/L[4]
TSS	80-120%	≤20%	NA	1 mg/L
TOC	80-120%	≤20%	NA	0.10%
Sediment				
PCB Congeners	Internal Standards and Labeled compounds	≤50%	NA[3]	NA 5 ug/Kg, ww
TOC	75-125%	≤20%	NA	0.1 ug/Kg
Grain Size	NA	NA	<20%[5]	NA

[1] Relative percent difference [2] Labeled compounds [3] Not applicable [4] Congener specific
[5] Relative standard deviation (RSD) for grain size, because it uses triplicate analyses.
[6] The isotopic dilution method used allows for correction for recovery of C_{12} labeled congeners.
TSS: Total suspended solids
TOC: Total organic carbon

6.2.1 Targets for precision, bias, and sensitivity

6.2.1.1 Precision

Precision is a measure of the variability in results of replicate measurements due to random error. Laboratory precision is usually estimated by the analysis of laboratory duplicates (splits) and control samples. Results provide an estimate of analytical precision and matrix homogeneity. Precision of the entire sampling and analysis process can be assessed by analysis of field replicates, which are defined as two samples collected independently at the same time and place.

Overall precision for fish tissue and water samples will be assessed by collection and analysis of field replicates. Duplicate analysis will assess laboratory precision for sediment analyses. Replicates and duplicates are different by their collection methods. Replicates are collected with one sample following another as close to the same time and place as possible. Sediment duplicates (field splits) are created from a single sediment composite of multiple grabs, homogenized and apportioned between two sample jars at the same time. Laboratory duplicates are also possible for fish tissue samples. Following selection of fish to composite and homogenization of sample tissue to a uniform color and consistency, the homogenate can be divided into two sample jars for independent analysis.

Field replicates for fish tissue will consist of the same species of fish, divided into two samples in the field and processed like other composite samples. Replicate samples are collected from the same locations, at the same time, using roughly the same size and number of fish in each composite. Because field replicate samples are collected independently, not from a processed homogenized composite, sample variability would be expected to be slightly higher than for duplicates.

Replication of water samples will consist of two CLAM samplers independently deployed at a site. Location, timing, and processing will be the same for both CLAM samplers from deployment through analysis.

PCB analysis will be completed using EPA 1668C. This method requires samples to be spiked with labeled compounds to evaluate data quality. Standards and blanks will be analyzed to test performance.

6.2.1.2 Bias

Bias is the systematic error due to contamination, sample preparation, calibration, or the analytical process. Most sources of bias are minimized by adherence to established protocols for the collection, preservation, transportation, storage, and analysis of samples. The isotopic dilution method used to analyze for PCBs (EPA, 1668C) requires spiking of labeled congeners into each sample. The method allows for correction of the concentration of target compounds corresponding to the recovery of labeled congeners.

6.2.1.3 Sensitivity

Sensitivity is a measure of the capability of a method to detect a substance. Expectations of sensitivity for this project will be based on the quantitation limit (QL). Often the method detection limit (MDL) is used to describe sensitivity.

Quantitation limits and sample collection schedules are displayed in Table 7.

Table 7. Required Quantitation Limit and Sampling Schedule

Matrix	Analysis	Method	QL[1]	Sample Timing	Sample Number	QC Samples[2]	Sample Total
Fish Tissue	PCBs	EPA 1668C	0.0003-0.01 ug/Kg	Oct 2014	4	2	6
Sediment	PCBs	EPA 1668C	50 ug/Kg	Oct 2014	9	2	11
Water (CLAMs)	PCBs	EPA 1668C	0.01 ng/L[3]	Oct 2014 (low flow)	2	4	6
Water (CLAMs)	PCBs	EPA 1668C		April or May, 2015 (high flow)	2	4	6

[1] Quantitation limit
[2] Duplicates for each matrix. Water also includes OPR, method blank, and trip blank.
[3] Based on approximately 20 to 55 liters of water filtered through the CLAMs.

6.2.2 Targets for comparability, representativeness, and completeness

6.2.2.1 Comparability

Comparability of study results will be ensured by using standard operating procedures and adhering to established data quality criteria consistent with other studies analyzing PCBs. Detection limits will be equal to or better than previous investigations of PCBs conducted in the basin.

6.2.2.2 Representativeness

The sampling design was planned to obtain PCB data representative of fish, sediment, and water in the Little Spokane River. Representativeness will be ensured by using appropriate sampling and sample handling procedures.

Fish tissue samples will be composites of 3 to 5 individual fish. Multiple grabs will be collected and composited for each sediment sample. Sediment and tissue samples will be collected as composites to reduce the variability and better reflect average PCB concentrations.

Water samples will be collected by CLAM sampling technology over a period of approximately 24 hours. Target parameters are reported as an estimated mean concentration over the deployment period. Seasonal variability will be accounted for by collecting water samples during periods of high and low flow.

6.2.2.3 Completeness

Completeness can be defined as the need to collect enough valid data to allow decisions to be made for which the study was designed. The goal of completeness is to collect and analyze 100% of the samples described in the quality assurance plan.

7.0 Sampling Process Design (Experimental Design)

7.1 Study design

This study will produce results for PCB concentrations in fish tissue, water, and sediment from the Little Spokane River. Fish tissue data will be compared to Washington State Water Quality and National Toxics Rule Human Health criteria in the form of FTECs. Study results will determine if the Little Spokane River should remain in Category 5 of the 303(d) list for PCBs.

Water samples collected at 2 locations and sediment samples collected at 8 locations may help to spatially characterize the extent of PCB contamination.

Water and sediment sample results may indicate if fish tissue concentrations are representative of levels of PCBs in the river. Fish tissue PCB concentrations that conflict with sediment and water concentrations may prompt a recommendation for further investigations.

7.1.1 Field measurements

Field measurements made will include the starting and ending flow rates for the CLAM samplers. These volumetric measurements will be used to estimate the total volume of water sampled by the CLAMs.

The length and weight of fish collected will also be measured in the field.

7.1.2 Sampling location and frequency

Fish samples will be collected from two sections of the river. One set of samples will be collected from river mile (RM) 1-7, downstream of the influences of permitted dischargers and major groundwater inputs. The other set of fish samples will be collected from RM 10-20, a segment of the river that includes discharger and groundwater influences. Total numbers of fish targeted for collection will be enough for 3 to 5 fish composites of cutthroat trout and mountain whitefish from each of the two sections of the river. Depending on availability of fish, other species may be collected. Fish samples will be collected during the fall by electrofishing or angling from an inflatable raft. Ecology will obtain permits necessary for fish collection from the appropriate federal and state agencies.

Water samples will also be collected above and below the permitted dischargers and major groundwater inputs. These samples will be collected once in the spring and once in the fall to represent high and low flow river conditions. To assure lower detection limits, water samples will be collected using a technology that will concentrate the PCBs in the water by filtering a large volume of water (≈40-60 L) through a solid phase extraction (SPE) disc. The concentrating method that will be used for this project is called Continuous Low-Level Aquatic Monitoring (CLAM). The CLAM is a water sampling device that can collect a large-volume sample that is filtered in the field. SPE technology has been used for a number of years as a laboratory bench

method. This newer monitoring method is generally a field application of the laboratory technique.

Water samples analyzed for TOC and TSS will be collected following SOP EAP015, *Standard Operating Procedure for Grab sampling – Fresh water* (Joy, 2006).

Sediment samples will be collected from 9 locations in the Little Spokane River. One from the mouth of the river, another just downstream of the hatchery (≈RM8), a sample from above the hatchery, and a reference sample collected from farther upstream. Sediment samples will also be collected from the confluence of the Little Spokane River and its 4 main tributaries: Dragoon Creek, Deer Creek, Little Deep Creek, and the West Branch of the Little Spokane River. Multiple input samples may begin to spatially define the extent of PCB contamination within the watershed. Sediment samples will be composites of multiple grabs when available. Fine sediments (silts and clay) will be targeted. Collection methods will be either hand scooping, using a sediment grab (petite ponar), or a combination of both methods. These samples will be collected during the fall. See Figure 2 in Section 7.2 for an overview of proposed sample locations.

7.1.3 Parameters to be determined

- Fish
  - PCB congeners
  - Percent lipids
- Water
  - PCB congeners
  - TOC
  - TSS
- Sediment
  - PCB congeners
  - TOC
  - Grain Size

7.2 Maps or diagram

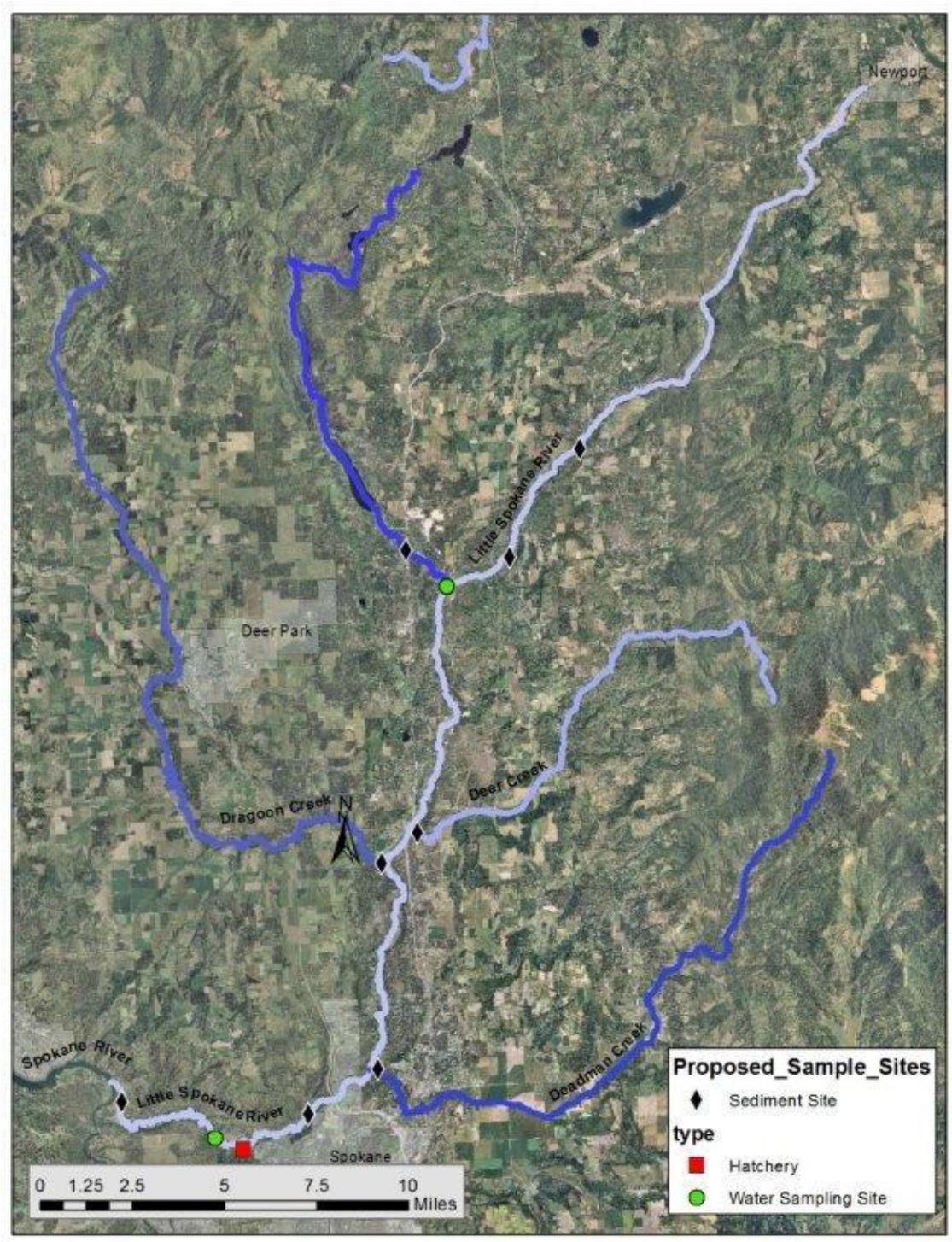

Figure 2. Proposed Sample Sites.

7.3 Assumptions underlying design

There is an assumption that suitable fine, organic sediments will be available from the study area.

There is also an assumption that permission for access will be granted by landowners.

7.4 Relation to objectives and site characteristics

Not applicable.

7.5 Characteristics of existing data

Existing PCB data for fish tissue from the Little Spokane River is 18-20 years old and likely does not reflect current PCB concentrations. Current PCB results for fish tissue must be evaluated to verify 303(d) listings for the river.

Questions still remain as to whether concentrations of PCBs in fish tissue reflect Little Spokane River conditions, since fish species that are present are migratory within the river system. Water and sediment will be collected and analyzed to attempt to evaluate if PCB concentrations in fish are representative of river conditions. If fish, water, and sediment data are contradictory, further investigation may be recommended. Water and sediment samples may also help to spatially evaluate the extent of PCB contamination in the Little Spokane.

8.0 Sampling Procedures

8.1 Field measurement and field sampling SOPs

Field SOPs are listed in Section 5.2, Special training and certifications.

8.2 Containers, preservation methods, holding times

Sampling containers, preservation, and holding times for study samples are shown in Table 8. Pre-cleaned sample containers will be obtained prior to sample processing for fish. Containers will be suitable for the specific analyses to be performed. Containers will be free of contaminants according to EPA (1992) and meet quality assurance certification from the supplier.

Table 8. Containers, Preservation, and Holding Times.

Parameter	Sample Size	Container[1]	Preservation	Holding Time
Fish				
PCB Congeners	30g minimum, 60g preferred	Certified 4 oz Glass w/Teflon Lid Liner	Freeze, -10° C	1 year to extraction, then 40 days to analysis
Lipids	30g minimum, 60g preferred	Certified 4-oz Glass w/Teflon Lid liner	Freeze, -10° C	1 year to extraction, then 40 days to analysis
Sediment				
PCB Congeners	250 g	8-oz Glass	Cool to 4°C or Freeze, -18°C	1 year to extraction, 1 year to analysis
TOC	25 g	Certified 2-oz Glass w/ Teflon Lid Liner	Cool to 4°C	14 days; 6 months frozen
Grain Size	150 g	8-oz Glass or Poly	Cool to 4°C	6 months
Water				
PCB Congeners	C-18 SPE Disc	C-18 SPE discs are placed in provided zip-locked foil bags	Cool to 4° C	14 days
TOC	2-60 mL	60 mL Glass	1:1 HCl to pH<2; Cool to ≤6° C	28 days
TSS	1 L	1 L Poly	Cool to ≤6° C	7 days

[1] Certified sample containers provided by Manchester Environmental Laboratory (MEL) or their contract laboratory
TOC: Total organic carbon
TSS: Total suspended solids

8.3 Invasive species evaluation

Ecology personnel working on this project are required to be familiar with and follow the procedures described in SOP EAP070, *Minimizing the Spread of Invasive Species.*

The sample area is an Area of Moderate Concern. This is a part of Washington State documented as not having established New Zealand Mud Snails or other species of extreme concern. These areas may have other invasive species, including plants, animals, fish, invertebrates, and fish pathogens.

Procedures will be followed to reduce the possibility of moving any potentially harmful organism out of or into the watershed.

8.4 Equipment decontamination

Fish processing equipment will be cleaned following the procedures described in EAP009, *Collection, Processing, and Preservation of Finfish Samples* (Sandvik, 2014). Decontamination procedures for fish processing equipment will include washing with soap and water and rinsing with acetone and hexane. Solvents will be evaporated from equipment under a fume hood.

Sediment samples will be collected with pre-cleaned stainless spoons and composited in pre-cleaned stainless bowls. Cleaning will be completed following the guidance contained in SOP EAP090, *Decontamination of Sampling Equipment for Use in Collecting Toxic Chemical Samples*. When a petite ponar is used for sediment sampling, the pre-cleaned ponar will be scrubbed with a brush and rinsed with ambient water between sample sites. Care will be taken to avoid collecting sediment that has contacted the ponar.

Water samples will be filtered through single-use SPE discs. CLAMs are clean and ready for deployment when they arrive from the contractor.

8.5 Sample ID

Study samples will be assigned unique individual IDs prior to sample collection.

8.6 Chain-of-custody, if required

Chain of custody will be maintained for all samples throughout the project.

8.7 Field log requirements

Field data will be recorded in a bound, waterproof notebook on Rite in the Rain paper. Corrections will be made with single line strikethroughs, initials, and date.

The following information will be recorded in the project field log:

- Name and location of project
- Field personnel names
- Sequence of events
- Any changes or deviations from the QAPP or SOPs
- Environmental conditions
- Date, time, site location, ID, and description of each sample
- Field measurement results
- Identity of QC samples collected
- Unusual circumstances that might affect interpretation of results

8.8 Other activities

Not Applicable. Necessary activities are detailed in other sections of this QAPP.

9.0 Measurement Methods

Table 9 provides information on study parameters, matrix, number of samples, expectations for ranges of results, reporting limits, preparation and analytical methods.

Table 9. Parameters, Number of Samples, Range of Expected Results, Reporting Limits, Sample Preparation, and Analytical Methods for Study Samples.

Parameter	Sample Number + QA[1]	Expected Range of Results	Reporting Limits	Cleanup and Extraction Methods	Analytical Method
Fish					
PCB Congeners	10	0.005-300 ug/Kg	0.003-0.01[2] ug/Kg, ww	EPA 1668C (HiRes GC/MS)	
Lipids	9	0.1-15%	0.10%	EPA 1668	
Sediment					
PCB Congeners	11	1-500 ug/Kg	0.20-0.50[3] ug/Kg, dw	EPA 1668C (HiRes GC/MS)	
TOC	5	1-10%	0.1%	PSEP-TOC, Combustion NDIR	
Grain Size	5	NA	0.1%	PSEP 1986, Sieve and Pipet	
Water[2]					
PCB Congeners	8	10-1,000 pg/L	10 pg/L[1]	EPA 3535	EPA 1668C (HiRes GC/MS)
TOC	5	1-2 mg/L	1 mg/L	SM 5310B	
TSS	5	1-10 mg/L	1 mg/L	SM 2540D	

[1] QA includes lab and field replicates.
[2] Reporting limits are congener specific.
[3] Water will be sampled using Continuous Low-Level Aquatic Monitoring instruments.

9.1 Field procedures table/field analysis table

Field procedures are described in SOPs (see Section 5.2).

9.2 Lab procedures table.

See table 7.

9.2.1 Analyte

PCBs.

9.2.2 Matrices

Fish, sediment, and water.

9.2.3 Number of samples

See Table 9.

9.2.4 Expected range of results

See Table 9.

9.2.5 Analytical method

See Table 9.

9.2.6 Sensitivity/Method Detection Limit (MDL)

Quantitation Limits are in Table 9.

10.0 Quality Control (QC) Procedures

Table 10 provides information on quality control (QC) samples to be analyzed. These may include laboratory blanks, duplicates, laboratory control samples, or labeled compounds. Evaluation criteria as MQOs are included for QC samples as the expectations for fully useable data.

Table 10. Laboratory Quality Control Samples for Fish Tissue, Sediments, and Water.

Parameter	Method Blank	Check Standard	Duplicates	Labeled Compounds	MS/MSD	OPR[1] Standards
Fish Tissue						
PCB Congeners	1/batch	1/batch	1/batch	all samples	--	--
Lipids	1/batch	1/batch	1/batch	--	--	--
Sediment						
PCB Congeners	1/batch	1/batch	--	all samples	--	each batch
TOC	1/batch	1/batch	--	--	--	--
Grain Size	--	--	1/batch[2]	--	--	--
Water						
PCB Congeners	1/batch	1/batch	1/batch	all samples	--	each batch
TOC	1/batch	1/batch	1/batch	--	1/batch	--
TSS	1/batch	1/batch	1/batch	--	--	--

[1] Ongoing Precision and Recovery
[2] Triplicates are analyzed for grain size analyses.

10.1 Table of field and lab QC required

See Table 10.

10.2 Corrective action processes

When a significant number of analytical results fall outside established MQOs, the laboratory analyst will contact the project manager for guidance on how to proceed. This may entail re-running samples, application of a clean-up method, or following recommendations listed under the analytical method for corrective action. Any departure from the normal analytical method will be documented by the laboratory analyst. Method departures will be described in detail in the data package from the laboratory and the study report.

11.0 Data Management Procedures

11.1 Data recording/reporting requirements

All field data and observations will be recorded in notebooks on waterproof paper. The information contained in field notebooks will be transferred to Excel spreadsheets after return from the field. Data entries will be independently verified for accuracy by another member of the project team.

Case narratives included in the data package from MEL will discuss any problems encountered with the analyses, corrective action taken, changes to the requested analytical method, and a glossary for data qualifiers. Laboratory QC results will also be included in the data package. This will include results for surrogate recoveries, laboratory duplicates, matrix spikes, and laboratory blanks. The information will be used to evaluate data quality, determine if the MQOs were met, and act as acceptance criteria for project data.

Field and laboratory data for the project will be entered into Ecology's Environmental Information Management database (EIM). Laboratory data will be downloaded directly into EIM from MEL's data management system. Data from contract laboratories will be submitted in electronic format for inclusion into EIM.

11.2 Laboratory data package requirements

The laboratories will provide a standard deliverable package after completing their work. The laboratories will provide all relevant quality control data. The data package will be delivered electronically via email.

11.3 Electronic transfer requirements

See Section 11.2.

11.4 Acceptance criteria for existing data

The fish tissue data that will be used for comparison are the data that resulted in the 303(d) listing.

11.5 EIM/STORET data upload procedures

All result transmittals from laboratories must be provided in an electronic data deliverable (EDD) format that meets Ecology requirements for loading into EIM. Data generated with the use of CLAMs will be stored in a data repository for results that require calculation. EIM only holds raw data.

12.0 Audits and Reports

MEL participates in performance and system audits of their routine procedures. Results of these audits are available upon request.

A draft report of the study findings will be completed by the principal investigator in October 2015 and a final report in January 2016. The report will include, at a minimum, the following:

- Map showing all sampling locations and any other pertinent features of the study area.
- Coordinates of each sampling site.
- Description of field and laboratory methods.
- Discussion of data quality and the significance of any problems encountered.
- Summary tables of the chemical and physical data.
- Results of the toxic contaminants relative to available criteria and determination if PCB tissue results warrant continued 303(d) listing for the Little Spokane River.
- Discussion of any seasonal significance from data concentrations of toxic chemicals reported for CLAM samplers and surface waters.
- Results compared from PCBs in sediment to available freshwater sediment criteria.
- Recommendations for follow-up actions, based on study results.
- Complete set of chemical and physical data in the Appendix.

Upon study completion, all project data will be entered into EIM, except for the water quality data generated using the CLAM systems. The CLAM system is still considered under development. Until SOPs for this method have been approved, this data will not be included in EIM. However, it can be obtained by contacting the study author. Public access to electronic data and the final report for the study will be available through Ecology's Internet homepage (www.ecy.wa.gov).

12.1 Number, frequency, type, and schedule of audits

An audit will not be required for this project.

12.2 Responsible personnel

See Section 5.1.

12.3 Frequency and distribution of report

This report will be produced and generated once.

12.4 Responsibility for reports

See Section 5.1.

13.0 Data Verification

13.1 Field data verification, requirements, and responsibilities

The principal investigator is responsible for the final acceptance of the project data. The complete data package, along with MEL's written report, will be assessed for completeness and reasonableness. Based on these assessments, the data will either be accepted, accepted with qualifications, or rejected and re-analysis considered.

13.2 Lab data verification

Data verification is a process conducted by producers of data. Normally a MEL unit supervisor or an analyst experienced with the method verifies laboratory data. It involves a detailed examination of the data package using professional judgment to determine whether the MQOs have been met.

Data verification involves examining the data for errors, omissions, and compliance with QC acceptance criteria. MEL's SOPs for data reduction, review, and reporting will meet the needs of the project. Data packages, including QC results for analyses conducted by MEL, will be assessed by laboratory staff using the EPA Functional Guidelines for Organic Data Review.

MEL staff will provide a written report of their data review which will include a discussion of whether (1) MQOs were met, (2) proper analytical methods and protocols were followed, (3) calibrations and controls were within limits, and (4) data were consistent, correct, and complete, without errors or omissions.

13.3 Validation requirements, if necessary

Independent data validation will not be required.

14.0 Data Quality (Usability) Assessment

14.1 Process for determining whether project objectives have been met

After the project data have been reviewed and verified, the principal investigator will determine if the data are of sufficient quality to make determinations and decisions for which the study was conducted. The data from the laboratory's QC procedures, as well as results from laboratory control standards and duplicates, and labeled standard recoveries, will provide information to determine if MQOs have been met. A review of sample results will be performed following each of the two seasonal sampling events to assess the need for modifications to the sampling or analysis program. Laboratory and QA staff familiar with assessment of data quality may be consulted. The project final report will discuss data quality and whether the project objectives were met. If limitations in the data are identified, they will be noted.

Some analytes will be reported near the detection capability of the selected methods. MQOs may be difficult to achieve for these results. MEL's SOP for data qualification and best professional judgment will be used in the final determination of whether to accept, reject, or accept the results with qualification. The assessment will be based on a review of laboratory QC results. This will include assessment of laboratory precision, contamination (blanks), accuracy, matrix interferences, and the success of laboratory QC samples meeting MQOs.

14.2 Data analysis and presentation methods

Not applicable.

14.3 Treatment of non-detects

Results for PCB congeners that are not detected at the practical quantitation limit (PQL) or estimated detection limit (EDL), whichever is higher, will not be included in PCB totals. Only detected congeners will be included in PCB sample totals.

14.4 Sampling design evaluation

The number of fish samples will be sufficient to verify the 303(d) listing for PCBs in fish tissue on the Little Spokane River. Sediment and water samples are sufficient for this level of screening. Additional sampling for source assessment may occur at another phase of this project if PCB contamination is determined to exceed water quality criteria in the Little Spokane River. The project schedule provides sufficient time to evaluate analytical results and adapt the project plan between sampling events if needed.

14.5 Documentation of assessment

This will occur in the final report.

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Appendices

Appendix A. 303(d) Category 5 Listings in the Little Spokane River Watershed

Parameter	Medium	Listing ID	2012 Category†
PCBs	Fish tissue	9051	5
pH	Water	50416	5
pH	Water	11388	5
Bacteria	Water	46144	5
pH	Water	50434	5
Dissolved Oxygen	Water	41981	5
pH	Water	50410	5

Category 5- Segment is on the 303(d) list as water quality-impaired.

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.

Endocrine disruptor: Chemicals that may interfere with an organism's endocrine system and result in negative developmental, reproductive, neurological, and immune system effects.

Estimated Detection Limit (EDL): Minimum concentration required to produce a specified signal to noise ratio.

Fish Tissue Equivalent Concentration (FTEC): The concentration of a contaminant in fish tissue that equates to Washington's water quality standard for toxic substances for the protection of human health. Washington uses the National Toxics Rule Water Quality Criteria for the protection of human health. The FTEC is calculated by multiplying the contaminant-specific Bioconcentration Factor (BCF) times the contaminant-specific National Toxics Rule Water Quality Criterion for water.

Hydrophobic: Literally, scared of water. These are non-polar molecules or compounds that do not interact with water molecules or polar compounds.

Lipophilic: Literally, fat loving. These are substances that dissolve or combine with fats, oils, lipids, and non-polar solvents. Lipophilic also means not soluble in water.

PCBs: Polychlorinated biphenyls. A synthetic organic compound that consists of chlorine attached to biphenyl, which is a molecule made up of 2 benzene rings. PCB congeners are any one of 209 unique, well-defined PCB compounds.

Ponar: A grab sampler used to sample sediments.

Practical Quantitation Limit (PQL): Lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operations.

Reach: A specific portion or segment of a stream.

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

Streamflow: Discharge of water in a surface stream (river or creek).

Surface waters of the state: Lakes, rivers, ponds, streams, inland waters, salt waters, wetlands and all other surface waters and water courses within the jurisdiction of Washington State.

Thalweg: The deepest and fastest moving portion of a stream.

Total suspended solids (TSS): Portion of solids retained by a filter.

Trophic level: The position an organism occupies in a food web or chain. Primary producers usually start at the first level, followed by herbivores, carnivores, and apex or top predators are at the highest trophic level.

Turbidity: A measure of water clarity. High levels of turbidity can have a negative impact on aquatic life.

Watershed: A drainage area or basin in which all land and water areas drain or flow toward a central collector such as a stream, river, or lake at a lower elevation.

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

Ecology	Washington State Department of Ecology
EIM	Environmental Information Management database
EPA	U.S. Environmental Protection Agency
EQL	Estimated Quantitation Limit
GIS	Geographic Information System software
GPS	Global Positioning System
MEL	Manchester Environmental Laboratory
MQO	Measurement quality objective
NTR	National Toxics Rule
PBT	Persistent, bioaccumulative, and toxic substance
PCB	Polychlorinated biphenyls
PQL	Practical Quantitation Limit
QA	Quality assurance
RM	River mile
SOP	Standard operating procedures
TOC	Total organic carbon
TSS	Total suspended solids
WAC	Washington Administrative Code
WRIA	Water Resource Inventory Area

Units of Measurement

kg	kilograms, a unit of mass equal to 1,000 grams
pg/L	picograms per liter (parts per quadrillion)
ug/Kg	micrograms per kilogram (parts per billion)
ug/L	micrograms per liter (parts per billion)

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 population 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 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): Data Quality Indicators (DQIs) are 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): Data Quality Objectives are 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 is usable for intended purposes.
- J (or a J variant), data is estimated, may be usable, may be biased high or low.
- REJ, data is 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: The term split sample denotes when a discrete sample is further 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

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