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Quality Assurance Project Plan

Lake Spokane PCBs in Carp

October 2014

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Publication Information

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/1403123.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, BERA0011.

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Signatures are not available on the Internet version.
EAP: Environmental Assessment Program
EIM: Environmental Information Management database
WQP: Water Quality Program

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Abstract

The Department of Ecology Environmental Assessment Program will conduct a study to quantify concentrations of polychlorinated biphenyls (PCBs) in carp from Lake Spokane. PCBs in several size ranges of carp will be measured, and these results will be used to estimate the bulk quantity of PCBs removed from Lake Spokane as a part of the Avista Utilities carp population reduction project. Carp will be collected in late September through early October 2014. This schedule may be adjusted based on the availability of carp during the planned collection period.

Background

Avista Utilities (Avista) is an energy production and transmission company that supplies electricity and natural gas to customers in eastern Washington and northern Idaho. They own and operate five hydroelectric dams on the Spokane River. Long Lake Dam at the western downstream end creates the reservoir known as Lake Spokane. Ninemile Dam bounds Lake Spokane at the eastern upstream end.

The Washington Department of Ecology (Ecology) determined that the dissolved oxygen (DO) levels in certain portions of the Spokane River and Lake Spokane do not meet Washington's water quality standards, and those portions are listed as impaired water bodies under Section 303d of the Clean Water Act. In response, Ecology developed the *Spokane River and Lake Spokane Dissolved Oxygen Total Maximum Daily Load Water Quality Improvement Report* (DO TMDL) (Moore and Ross, 2010). Within the DO TMDL, Avista was assigned a proportional level of responsibility to improve DO concentrations within Lake Spokane. This requirement was amended into Avista's 401 Water Quality Certification (Certification), included as Appendix B of its Federal Energy Regulatory Commission License for the Spokane River Hydroelectric Project (Lunney, personal communication).

Subsequent to the DO TMDL and the Certification amendment, Avista completed a *Lake Spokane Dissolved Oxygen Water Quality Attainment Plan*, which identified potential measures to improve DO conditions within Lake Spokane (Avista and Golder, 2012). These measures focused on reducing nonpoint sources of phosphorus loading into Lake Spokane. One such measure includes investigating whether a carp population reduction program would improve water quality. Avista will be implementing this investigation starting in the fall of 2013 through the winter of 2014. This investigation will help in understanding population abundance, distribution, and habitat use of carp and will help define a carp population reduction program, if appropriate, that will benefit Lake Spokane water quality (Lunney, personal communication).

Portions of Lake Spokane and the Spokane River are also listed as impaired for PCBs under Section 303d of the Clean Water Act. Carp are known to accumulate high levels of PCBs in their tissue because they have high fat content, they are a long-lived species, and they are bottom feeders. They are frequently near sediments where PCBs settle out in slower moving areas of riverine systems. Because of the carp's tendency to accumulate PCBs, Ecology recognized

Avista's carp population reduction project as an opportunity to remove PCBs from the Spokane River system.

The Ecology Water Quality Program's Eastern Regional Office would like to estimate the reduction of PCBs when the carp population is reduced. The Environmental Assessment Program's Toxics Studies Unit was requested to prepare a quality assurance (QA) project plan to measure PCBs in carp. This information will be used to estimate the mass of PCB removed from Lake Spokane as part of Avista's carp population reduction project.

Study Area

Lake Spokane (formerly known as Long Lake) is located in eastern Washington and several miles to the northwest of the city of Spokane (Figure 1). The lake is a part of the Spokane River and was created by the formation of Long Lake Dam at its western downstream end. The lake boundary for the eastern upstream end is approximately one mile downstream of Nine Mile Dam. The lake covers 24 river miles between the dams. It straddles three counties: Stevens to the north, Lincoln to the west, and Spokane to the south, southeast, and northeast.

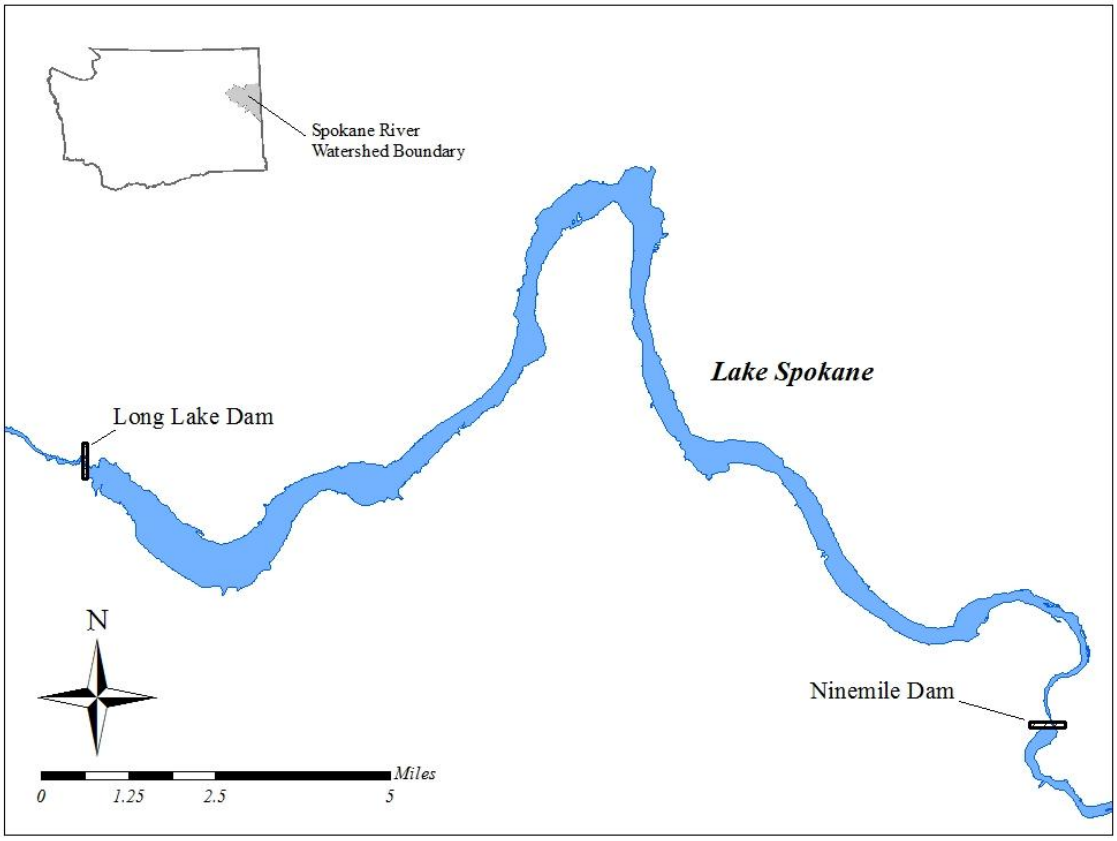

Figure 1. Lake Spokane.

The Water Resource Inventory Area (WRIA) for Lake Spokane is 54 and the Hydrologic Unit Code (HUC) is 17010307.

Project Description

The purpose of the study is to quantify the concentrations of polychlorinated biphenyls (PCBs) in multiple size ranges of common carp (*Cyprinus Carpio*) from Lake Spokane. The data collected will be used in combination with carp population data to estimate the mass of PCBs removed from Lake Spokane as part of Avista's carp population and reduction project.

Ecology will work with Avista and the contractor they have hired to conduct the carp population and reduction project to make sure that sufficient information on the carp population is collected to allow for size class estimates to be made. Ecology will obtain carp between late September and early October 2014, depending on the schedule of the carp population reduction study.

Carp will be analyzed for PCB Aroclors and lipids. A subset of carp will also be analyzed for PCB congeners. Fish will be analyzed on a whole-body basis so that the mass of PCBs can be estimated from the removal of carp from Lake Spokane.

Organization and Schedule

Table 1 lists the people involved in this project. All are employees of the Washington State Department of Ecology. Table 2 outlines the proposed schedule for this project.

Table 1. 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.
Brandee Era-Miller Toxics Studies Unit Statewide Coordination Section Phone: 360-407-6771	Project Manager/ 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 Statewide Coordination Section 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 Statewide Coordination Section 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.
Thomas Mackie Eastern Operations Section Phone: 509-457-7136	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	Approves the final QAPP.
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

Table 2. 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	October 2014	Brandee Era-Miller
Processing of samples completed	November 2014	
Laboratory analyses completed	January 2015	
Environmental Information System (EIM) database		
EIM user study ID	BERA0011	
Product	Due date	Lead staff
EIM data loaded	March 2015	Brandee Era-Miller
EIM quality assurance	April 2015	Casey Deligeannis
EIM complete	June 2015	Brandee Era-Miller
Final report		
Author lead / Support staff	Brandee Era-Miller	
Schedule		
Draft due to supervisor	March 2015	
Draft due to client/peer reviewer	April 2015	
Draft due to external reviewer(s)	May 2015	
Final (all reviews done) due to publications coordinator	June 2015	
Final report due on web	July 2015	

Laboratory Quality Objectives

The laboratory measurement quality objectives for the study are shown in Table 3. These are the analytical objectives that both Ecology's Manchester Environmental Laboratory (MEL) and the contract laboratories are required to meet to serve the quality objectives of the study.

Table 3. Measurement Quality Objectives for PCB Analyses.

Parameter	Analytical Method	Lab Control Samples (% Recovery)	Duplicate samples (RPD)	Matrix Spike (% Recovery)	Matrix Spike Duplicates (RPD)	Surrogate Recoveries (% Recovery)
Lipids	MEL SOP 730009	NA	≤20%	NA	NA	NA
PCB Aroclors	EPA 8082	50 – 150	≤40%	50 – 150	40%	50 – 150
PCB congeners	EPA 1668c	compound specific[†]	≤50%	NA	NA	25 – 150[a]

EPA: U.S. Environmental Protection Agency
MEL SOP: Manchester Environmental Laboratory Standard Operating Procedure
RPD: relative percent difference
[†] Per Method for Ongoing Precision and Recovery (OPR), internal standards, and labeled compounds
[a] labeled congeners

Sampling Process Design (Experimental Design)

Carp samples for PCB analysis will cover multiple size classes as shown in Table 4. These size classes were determined using data collected by Avista's contractor during their marking and recapture event that took place in June 2014 (Table 5).

Table 4. Sampling Design for PCBs in Whole Carp from Lake Spokane.

Size Class	Total Length (mm)	Number of Fish in each Composite	Number of Composite Samples	Total Number of Fish
Small	<500	3	3	9
Medium 1	500 - 600	3	3	9
Medium 2	600 - 700	3	3	9
Medium 3	700 - 800	3	3	9
Large	>800	3	3	9
Totals	**--**	**--**	**15**	**45**

Table 5. Carp Population Data from the June 2014 Mark and Recapture Event.

Size Categories (Total Length, mm)	Number of Total Lengths Measured	Number of Fork Lengths Measured	Number of Weights Measured	Number of Males	Number of Females	Number PIT tagged effectively
<100	0	0	0	0	0	0
100-199	9	9	8	0	0	9
200-299	20	14	15	0	0	20
300-399	0	0	0	0	0	0
400-499	4	4	4	0	0	4
500-599	74	31	20	9	1	74
600-699	342	27	23	80	16	327
700-799	182	34	30	15	16	175
800-899	8	7	8	0	0	7
900-999	0	0	0	0	0	0
≥1000	0	0	0	0	0	0
Total	**639**	**126**	**108**	**104**	**33**	**616**

The carp population data in Table 5 represent fish collected at multiple locations within Lake Spokane. For the Ecology PCB composite samples, an effort will be made to include carp from multiple capture locations in order to be representative of the lake at large. GPS coordinates for the capture locations for each fish will be provided to Ecology.

Carp will be analyzed as whole body composite samples. Each composite sample will consist of 3 fish and 3 samples will analyzed for each size class for a total of 15 composite samples and 45 fish total for the study (Table 4).

Ideally, each composite sample will follow the *75% Rule* where the length of the smallest fish is at least 75% the length of the largest fish as recommended by *EPA's Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories* (EPA, 2000).

All 15 samples will be analyzed for lipids and PCB Aroclors. A subset of 10 samples (2 composite samples per size class) will be analyzed for the more expensive PCB congeners.

Estimates of PCB Body Burden and Population

Two possible methods may be used to estimate the body burden of PCBs in an individual carp and then from that data estimate mass removal of PCBs for future carp removal efforts in Lake Spokane.

1. **Regression Analysis**
   Once PCB concentrations have been determined for each composite sample, the results will be plotted against total length, weight, and lipid content, using regression analysis. If there is an acceptable correlation ($R^2 > 0.75$) between PCBs and any of the measurements, the regression equation can be used for any carp with the appropriate measurement data. Estimated PCB concentrations can then be summed for a mass population estimate.

2. **Size Class**
   PCB concentrations for the composite samples within each size class will give an estimated range of PCBs for that size class. Any carp that falls within a given size class can then be assigned a range of PCB concentrations. Estimated PCB concentration ranges can then be summed for a mass population estimate.

Older fish often contain higher concentrations of bioaccumulative chemicals like PCBs, since they have been around longer in the environment to accumulate chemicals. Age data along with lipid content will be used to help interpret the results of the study such that if concentrations vary between samples of the same size class, fish age and lipid content may account for some of the differences.

Data Reduction

For both PCB Aroclor and congener analyses, results will be considered non-detects ("U") if the concentrations are less than three times the concentration of the associated laboratory method blanks. The result values (qualified as non-detects) will then be reported at the estimated quantitation limit (EQL) or at the level of detection, whichever is higher.

Data Qualifier Definitions:

U	The analyte was not detected at or above the reported sample quantitation limit.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ	The analyte has been "tentatively identified" and the associated numerical value represents its approximate concentration.
ND	Not Detected.

Results for PCB congener analysis (EPA 1668c) that do not meet the isotopic abundance ratio and retention time criteria for positive identification will be qualified by MEL with an "NJ" and considered to be tentatively identified. NJ qualified data will also be blank corrected or censored using the five times blank rule as described above.

For summing of total PCB Aroclors and total PCB congeners, non-detected results will be assigned a value of zero. If only non-detected results comprise a total value, then the final total result was simply reported as "ND" for not detected. Sample totals will be assigned a qualifier of "J" (estimated) if more than 10% of the result concentrations are composed of results containing "J" qualifiers.

Sampling Procedures

Fish will be collected by a contractor hired by Avista. Ecology staff will meet the contractor in the field to receive fish during collection. Ecology will process the fish in the field by following SOP EAP009 *Standard Operating Procedure for Field Collection, Processing, and Preservation of Finfish Samples at the Time of Collection in the field* (Sandvik, 2010a).

Field processing includes assigning each fish a unique sample number, recording lengths and weights, double wrapping in foil, and placing in plastic bags in coolers on ice for transport to Ecology headquarters. Collection locations and times will also be recorded.

Once fish are transported back to Ecology headquarters they will be partially processed by removing scales, rinsing with deionized water, filleting, and cutting the heads into several smaller portions. Each fish (as multiple pieces) will then be re-packaged in clean aluminum foil and frozen prior to the rest of the tissue processing. This two-step processing will be conducted so that the carp will more easily fit into the Hobart meat grinder.

Processing by Ecology staff will follow SOP EAP007 *Standard Operating Procedure for Resecting Finfish Whole Body, Body Parts or Tissue Samples* (Sandvik, 2010b). This procedure is briefly described in the following paragraphs.

All processing utensils will be cleaned in order to prevent contamination of the samples. Utensils include stainless steel bowls and knives and tissue grinding appliances having plastic, wood or stainless steel parts. The cleaning steps are: (1) Liquinox soap and hot water wash, (2) 10% nitric acid rinse, (3) deionized water rinse, and (4) acetone and hexane rinses.

Whole fish tissue will be processed using a commercial-grade Hobart food grinder. Fish will first be partially thawed then thoroughly rinsed with deionized water to remove any adhering debris, aging structures removed, and sex determined by cutting into the gut cavity of the fish. Aging structures will be removed following SOP EAP008 *Standard Operating Procedures for Resecting DNA Samples and Aging for Finfish* (Sandvik, 2010c). Structures will be sent to the Washington State Department of Fish and Wildlife (WDFW) for age determination.

Whole fish will then be cut into appropriate size pieces for fitting into the Hobart grinder opening. Tissue from each fish will be passed three times through the grinder and finally homogenized to a consistent color and texture. Equal-weighted aliquots from the homogenized tissue of all three fish will be combined into a large stainless steel bowl and once again homogenized to a consistent color and texture. Sample containers will then be filled with the composited fish tissue. Sample containers and holding times for the fish tissue samples are shown in Table 6.

Table 6. Sample Containers, Preservations, and Holding Times.[1]

Parameter	Container	Preservation	Holding Time
Lipids	From same jar as PCB Aroclors (4-oz jar)	Transport at 4°C; can store frozen at -18°C	1 year extraction; 14 days analysis
PCB Aroclors	Certified 4-oz amber glass w/ Teflon lid liner		1 year extraction; 1 year analysis
PCB congeners	Certified 4-oz amber glass w/ Teflon lid liner		

[1] Information in table was adapted from MEL, 2008.

Laboratory Measurement Procedures

The desired reporting limits, expected concentrations, analytical methods, and laboratories for the project are shown in Table 7. The PCB Aroclor and lipid analysis will be conducted by MEL and the PCB congener analysis will be contracted with another accredited laboratory through MEL.

Table 7. Reporting Limits and Expected Concentrations for the Analytical Methods.

Parameter	Desired Laboratory Reporting Limits	Expected Concentrations	Analytical Method	Laboratory
Lipids (%)	0.1	1 – 20	MEL SOP 730009	MEL
PCB Aroclors (ug/Kg ww)	1.1 – 44 per Aroclor	50 – 3,000 total	EPA 8082	MEL
PCB congeners (ug/Kg ww)	0.005 – 0.8 per congener	50 – 3,000 total	EPA 1668C	Contract

EPA: U.S. Environmental Protection Agency
MEL SOP: Manchester Environmental Laboratory Standard Operating Procedure

Quality Control Procedures

Field

Quality control in the field will be maintained by following as closely as possible Ecology's SOP EAP009 *Standard Operating Procedure for Field Collection, Processing, and Preservation of Finfish Samples at the Time of Collection in the Field* (Sandvik, 2010a).

Having 3 to 5 replicate composite samples per size range will give a good estimate of natural variability among similarly-sized samples.

Laboratory

The laboratory quality control procedures routinely followed by MEL and the chosen contract laboratory will be satisfactory for the purposes of this project. MEL will follow SOPs as described in the Manchester Environmental Laboratory Quality Assurance Manual (MEL, 2012).

Laboratory quality control samples are given in Table 8. Two samples will be analyzed in duplicate as a measure of analytical precision.

Table 8. Laboratory Quality Control Samples.

Parameter	Laboratory Control Sample	Method Blank	Surrogate Spikes	Matrix Spike	Matrix Spike Duplicate	Laboratory Duplicate
Lipids	--	1/batch	--	--	--	2/project
PCB Aroclors	1/batch	1/batch	All samples	1/batch	1/batch	2/project
PCB congeners	1/batch	1/batch	All samples	--	--	2/project

Laboratory Budget

The analytical costs for the project are $14,386, as shown in Table 9.

Table 9. Laboratory Budget.

Parameter	Number of Samples	Number of QA Samples[1]	Total Number of Samples	Cost per Sample	Subtotal
Lipids	15	2	17	33	$ 561
PCB Aroclors	15	4	19	175	$ 3,325
PCB congeners	10	2	12	875[†]	$ 10,500
				Project Total Laboratory Costs	**$ 14,386**

[1] Includes 2 laboratory duplicates and a matrix spike/matrix spike duplicate for PCB Aroclors

[†] Includes a 25% contracting fee from MEL

Cooler and plastic bags for carp storage and transfer will cost $1,200, making the total project cost $15,586.

Data Management Procedures

Fish tissue processing information will be carefully transferred to electronic data sheets and reviewed for potential transfer errors.

The data packages from MEL and the contract laboratories will include case narratives discussing any problems encountered during analysis, corrective actions taken, and an explanation of data qualifiers. The project manager will then review the data packages to determine if analytical MQOs (laboratory control samples, laboratory duplicates, and matrix spikes) were met.

All project data will be entered into Ecology's Environmental Information Management (EIM) database for availability to the public and interested parties. Data entered into EIM follows a formal data review process where data are reviewed by the project manager, the person entering the data, and an independent reviewer.

Audits and Reports

MEL participates in performance and system audits of their routine procedures. The results of these audits are available on request.

A draft report will be provided to internal Ecology reviewers, collaborating entities, external reviewers, and other interested parties by May 2015. The final technical report will be completed in July 2015 and will include the following elements:

- Information about the sampling locations, including geographic coordinates and maps.
- Descriptions of field and laboratory methods.
- Tables presenting all the data.
- Discussion of project data quality.
- Summary of significant findings.
- Recommendations for future follow-up work.

Upon completion of the study, the data will be entered into Ecology's EIM database. Electronic data and the final report for the study will be available to the public on Ecology's Internet homepage (www.ecy.wa.gov).

Data Verification and Validation

The project manager will review laboratory data packages and data verification reports. Based on these assessments, the data will either be accepted, accepted with appropriate qualifications, or rejected and re-analysis considered.

To determine if analytical MQOs (Table 3) have been met, the project manager will compare results of the field and laboratory quality control samples to MQOs.

Formal (third party) validation of the data will not be necessary for this project.

Data Quality (Usability) Assessment

Once the data have been reviewed and verified, the project manager will determine if the data are useable for the purposes of the project and whether the data should be accepted, accepted with additional qualification, or rejected and re-analysis considered. Data quality and usability will be discussed in the final report.

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SOP Number EAP008. www.ecy.wa.gov/programs/eap/quality.html

Appendix. Glossary, Acronyms, and Abbreviations

Glossary

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.

Dissolved oxygen (DO): A measure of the amount of oxygen dissolved in water.

Parameter: A physical chemical or biological property whose values determine environmental characteristics or behavior.

Total Maximum Daily Load (TMDL): A distribution of a substance in a water body designed to protect it from not meeting (exceeding) water quality standards. A TMDL is equal to the sum of all of the following: (1) individual wasteload allocations for point sources, (2) the load allocations for nonpoint sources, (3) the contribution of natural sources, and (4) a margin of safety to allow for uncertainty in the wasteload determination. A reserve for future growth is also generally provided.

303(d) list: Section 303(d) of the federal Clean Water Act requires 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 standard and are not expected to improve within the next two years.

Acronyms, Abbreviations, and Units of Measurement

DO	(See Glossary above)
Ecology	Washington State Department of Ecology
EIM	Environmental Information Management database
EPA	U.S. Environmental Protection Agency
MEL	Manchester Environmental Laboratory
MQO	Measurement quality objective
PCB	polychlorinated biphenyls
QA	Quality assurance
RM	River mile
SOP	Standard operating procedures
TMDL	(See Glossary above)
WRIA	Water Resource Inventory Area
°C	degrees centigrade
ft	feet
g	gram, a unit of mass
kg	kilograms, a unit of mass equal to 1,000 grams
ug/Kg	micrograms per kilogram (parts per billion)
ww	wet weight