



Reassessment of Toxicity of Lake Roosevelt Sediments

December 2001

Publication No. 01-03-043

printed on recycled paper



This report is available on the Department of Ecology home page on the World Wide Web at <http://www.ecy.wa.gov/biblio/0103043.html>

For additional copies of this publication, please contact:

Department of Ecology Publications Distributions Office

Address: PO Box 47600, Olympia WA 98504-7600

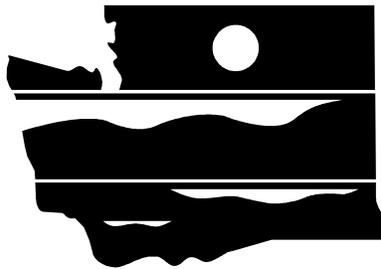
E-mail: ecypub@ecy.wa.gov

Phone: (360) 407-7472

Refer to Publication Number 01-03-043

The Department of Ecology is an equal opportunity agency and does not discriminate on the basis of race, creed, color, disability, age, religion, national origin, sex, marital status, disabled veteran's status, Vietnam era veteran's status, or sexual orientation.

If you have special accommodation needs or require this document in alternative format, please contact Joan LeTourneau, Environmental Assessment Program, at (360)-407-6764 (voice). Ecology's telecommunications device for the deaf (TDD) number at Ecology Headquarters is (360) 407-6006.



WASHINGTON STATE
DEPARTMENT OF
E C O L O G Y

Reassessment of Toxicity of Lake Roosevelt Sediments

by
Brandee Era
and
Dave Serdar

Environmental Assessment Program
Olympia, Washington 98504-7710

December 2001

Waterbody No. WA-CR-1060 (Franklin D. Roosevelt Lake)

Publication No. 01-03-043
printed on recycled paper

This page is purposely blank for duplex printing

Table of Contents

	<u>Page</u>
List of Figures and Tables.....	ii
Abstract	iii
Acknowledgements	iv
Introduction	1
Study Area.....	1
Methods	5
Sampling.....	5
Laboratory Analysis	6
Data Quality	9
Chemical Data	9
Bioassay Data.....	9
Data Evaluation.....	9
Results	11
Conventionals and Metals	11
Bioassay	13
Discussion	15
Longitudinal Distribution of Metals.....	15
Sediment Toxicity Compared to Earlier Studies.....	18
Metals and Sediment Toxicity.....	21
Conclusions	23
Recommendations	24
References	25
Appendices	
A Sample Locations and Descriptions	
B Data Quality	
C Bioassay Methods	
D Data Tables	

List of Figures and Tables

	<u>Page</u>
Figures	
Figure 1. Sampling Site Locations in Lake Roosevelt and the Upper Columbia River, May 2001	2
Figure 2. Inset Views of Sampling Sites	3
Figure 3. Metal Concentration Trends in the Upper Columbia River and Lake Roosevelt Found in the Present and Previous Surveys	16
Figure 4. Sampling Site Locations for the Present Study and Previous Studies with Open Water Grid and Section Grid Overlays	19
Tables	
Table 1. Analytical Methods, Reporting Limits, and Laboratories	6
Table 2. Spearman Ranked Correlation Matrix for Variables Measured in Lake Roosevelt and Upper Columbia River Sediments	10
Table 3. Percent Solids, TOC, and Grain Size for Lake Roosevelt and Upper Columbia River Sediments	11
Table 4. Metals Concentrations in Lake Roosevelt and Upper Columbia River Sediments	12
Table 5. Bioassay Results for Lake Roosevelt and Upper Columbia River Sediments ...	13
Table 6. Comparison of Bioassay Results to Previous Lake Roosevelt Data	17
Table 7. 1998 vs. Proposed 303(d) Listing Decisions for Lake Roosevelt Bioassay data	20
Table 8. Comparison of Lake Roosevelt Metal Concentrations to FSQVs for Metals in Washington State and to Consensus-Based TECs for Freshwater Sediments ...	21

Abstract

Sediments from nine sites within Lake Roosevelt and the upstream reach of the Columbia River were tested for metals concentrations and toxicity by the Washington State Department of Ecology during May 2001. The resulting data were needed to determine if these sites should remain on the federal Clean Water Act Section 303(d) list of impaired waterbodies.

Sediment samples were analyzed for arsenic, cadmium, copper, lead, mercury, and zinc.

- Elevated levels of cadmium and zinc were prevalent throughout the study area, with the exception of the Kettle River, Sanpoil River, and Grand Coulee sites.
- Elevated levels of copper and zinc at the upper Columbia River sites were about three orders of magnitude above reference concentrations and were most likely attributed to sandy slag material (< 2000 - 62 μ m).
- Elevated levels of mercury were found only in the fine sediments (< 62 μ m) of Lake Roosevelt.

Bioassay tests for toxicity included *Chironomus tentans* 20-day survival and growth, *Hyaella azteca* 10-day survival, and Microtox® 100% porewater. With the exception of the Grand Coulee site, all nine sites showed toxicity to at least one of the bioassays. Sediment from the Goodeve Creek site, seven river miles downstream of the Canadian border, was the most toxic to all the bioassay organisms.

Based on the existing 303(d) policy, listing requires only one toxicity hit per segment. Eight of nine sites meet current listing criteria. However, none of the sites meet criteria based on the newly proposed listing policy, which requires toxicity at three separate locations within a segment. Consequently, it is recommended that the eight sites showing toxicity should be reassigned to part 5, *Undetermined Status*, of the proposed 2002 303(d) list. Undetermined Status designation for these sites will allow the Department of Ecology and/or other public and private stakeholders to pursue measures to address toxicity in Lake Roosevelt and the upper Columbia River.

Acknowledgements

We would like to thank the following people for their assistance with this study:

- Julia Beatty and the BC Ministry of Environment, Land and Parks, for their permission to sample sediments in Arrow Lake.
- Stuart Magoon, Pam Covey, Jim Ross, Michelle Lee, and other staff of the Manchester Environmental Laboratory for their work in analyzing samples.
- Rosa Environmental Geotechnical Laboratory; Parametrix, Inc., and Northwestern Aquatic Sciences for their work on the bioassay analysis.
- Cliff Kirchmer for his recommendations on quality assurance and quality control.
- Brett Betts, Sharon Brown, Guy Gregory, Richard Jack, Nora Jewett, Art Johnson, Will Kendra, Dave Knight, Dale Norton, and Carl Nuechterlein, as well as Gary Passmore and Patti Stone of the Colville Confederated Tribes, for their review of the project.
- Randy “Captain” Coots for his assistance with sampling.

Introduction

Franklin D. Roosevelt Lake and the U.S. portion of the upper Columbia River in northeastern Washington constitute a 151-mile reach of the Columbia River stretching from the Canadian border to Grand Coulee Dam. Studies conducted during the past two decades have shown significant contamination of Lake Roosevelt and the upper Columbia with metals, particularly zinc, lead, copper, arsenic, cadmium, and mercury (Lowe et al., 1985; Johnson et al., 1988; Bortelson et al., 1994; Serdar et al., 1994). The Cominco Ltd. lead-zinc smelter in Trail, B.C., located approximately 10 river miles upstream of the international border, is the primary source of metals contamination. Mining activity in the watershed is also a source of metals, especially in lower Lake Roosevelt.

Studies conducted by the Washington State Department of Ecology (Ecology) and the U.S. Geological Survey (USGS) have included bioassay tests on Lake Roosevelt and upper Columbia River sediments (Johnson, 1991; Bortelson et al., 1994). Significant toxicity was reported at seven mainstem Columbia River locations and near the mouths of the Kettle and Sanpoil rivers, which are tributaries to the Columbia. Based upon the toxicity results, these sites were included on the federal Clean Water Act Section 303(d) list of impaired waterbodies for 1998.

Due to questions about the relevance of these decade-old data, the Water Quality Program at Ecology's Eastern Regional Office requested a reassessment of sediment toxicity in Lake Roosevelt and the upstream reach of the Columbia River. Ecology's Environmental Assessment Program conducted the reassessment during May 7-9, 2001 by analyzing sediment metals and toxicity at the nine sites previously found to have sediment toxicity, and at a reference site. Objectives of the survey were to obtain current information on sediment metals chemistry and toxicity, and to determine if the sites should remain on the 303(d) list.

Study Area

The study area covers 151 river miles from the international boundary (site 1) to the Grand Coulee Dam (site 9). The study area is divided into two main parts:

Upper Columbia River, a free-flowing section of the Columbia River:

- | | |
|------------------------|-----------------------|
| Site 1. Boundary | Site 3. Goodeve Creek |
| Site 2. Auxiliary Gage | Site 4. Kettle River |

Lower Lake Roosevelt, the reservoir behind Grand Coulee Dam:

- | | |
|--------------------------|--------------------------|
| Site 5. Castle Rock | Site 8. Swawilla Basin |
| Site 6. Whitestone Creek | Site 9. Grand Coulee Dam |
| Site 7. Sanpoil River | |

The reference site is located in Lower Arrow Lake Canada (site 10), approximately 37 river miles upstream of the International border. Figures 1 and 2 show the sampling site locations. Detailed descriptions and positions for each site are provided in Appendix A-1.

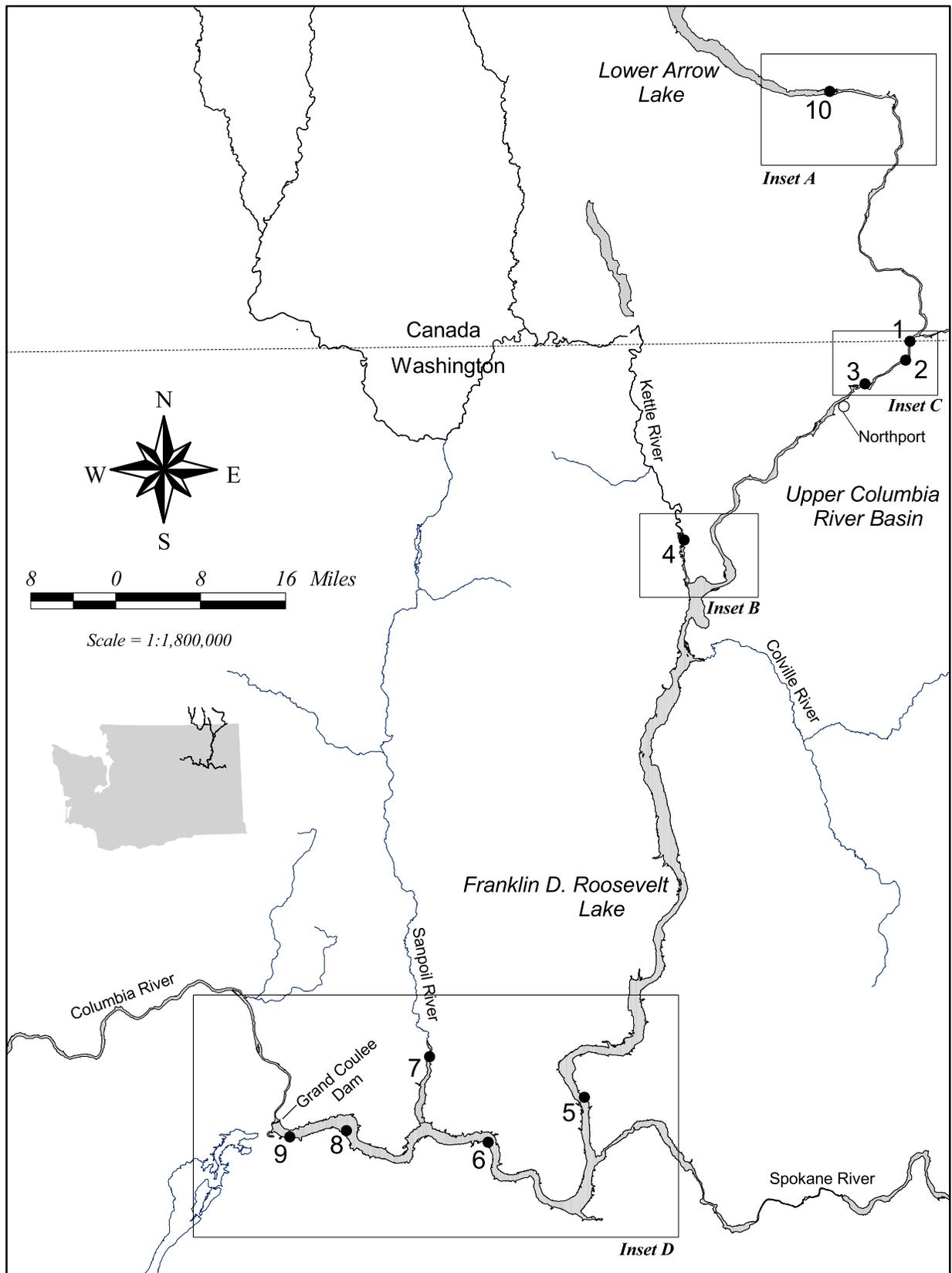
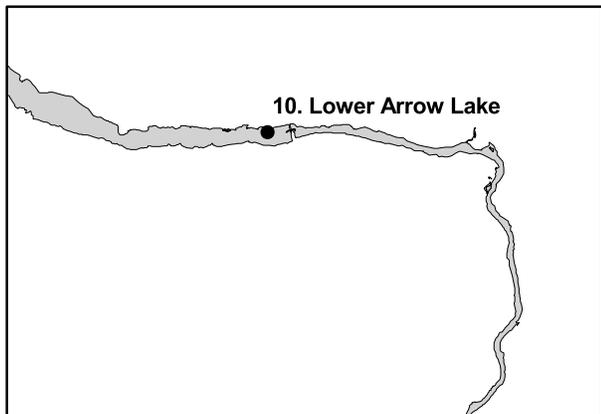
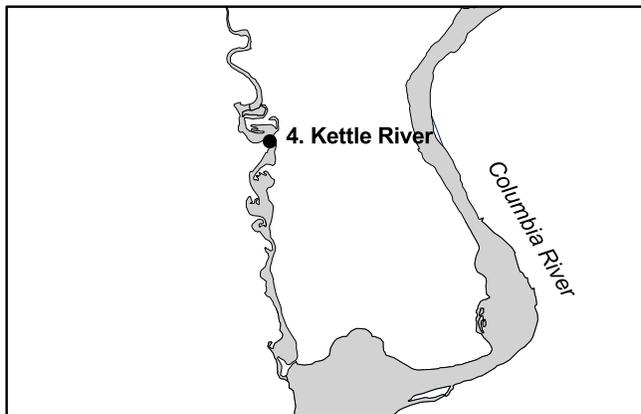


Figure 1. Sampling Site Locations in Lake Roosevelt and the Upper Columbia River, May 2001

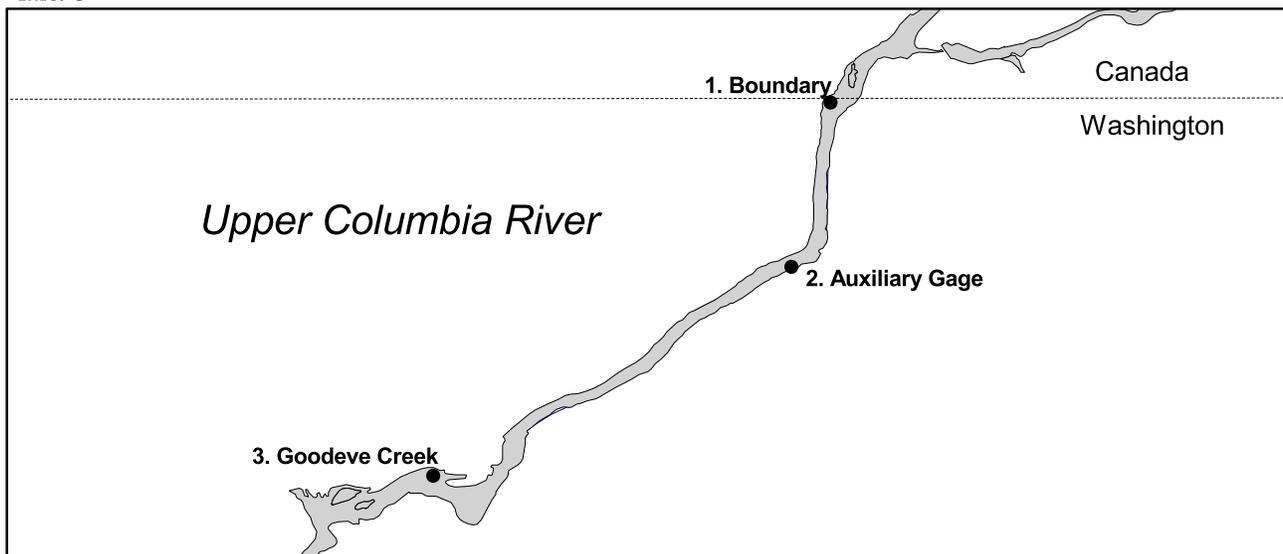
Inset A



Inset B



Inset C



Inset D

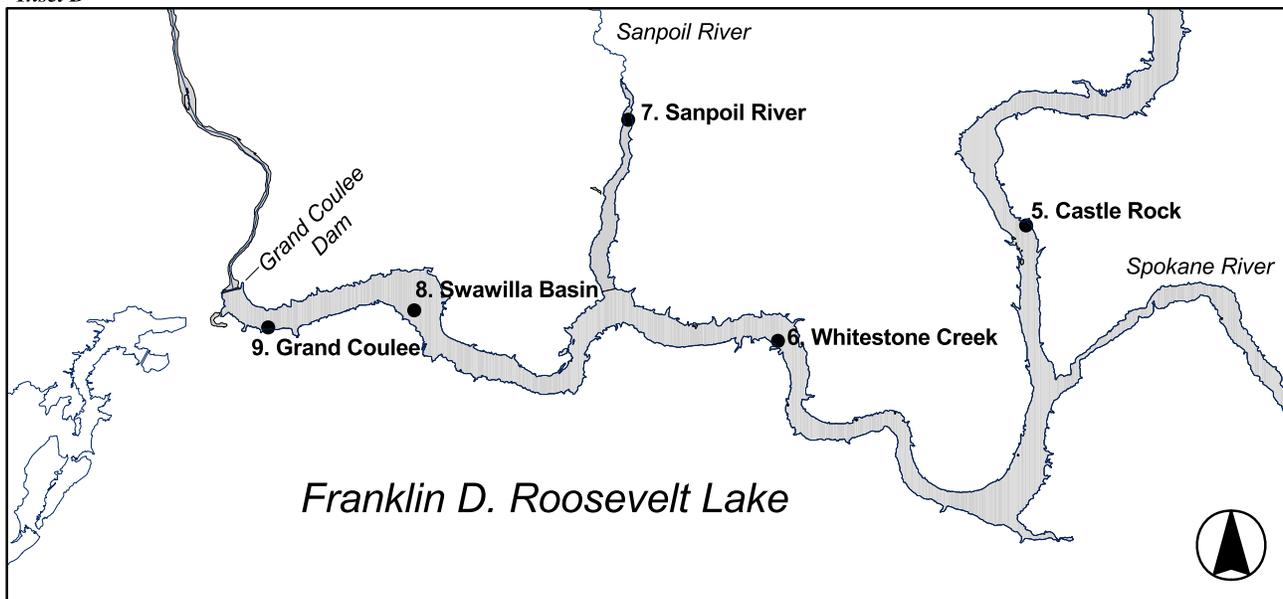


Figure 2. Inset Views of Sampling Sites

This page is purposely blank for duplex printing

Methods

Sampling

Sampling sites for the study were located and recorded using a Magellan GPS and landmarks. Mainstem Columbia River sediments were collected from Ecology's 20-ft skiff using a 0.1 m² stainless steel van Veen grab. The Kettle River and Sanpoil River samples were collected by wading into the water and scooping sediments up with a stainless steel spoon. Field descriptions of sediment samples are included in Appendix A-2.

With the exception of the Kettle River and Sanpoil River sites, each sediment sample was a composite of three grabs. A grab was considered acceptable if not over-filled with sediment, overlying water was present and not excessively turbid, the sediment surface was relatively flat, and if the desired depth penetration could be achieved. Over-filling the van Veen grab with sediment could not be avoided at sites where sediments were soft and the grab over-penetrated. In these cases, the top sediment was scraped off where it contacted the van Veen sampler.

After siphoning off overlying water, the top 10 cm (biologically active layer) of all samples, except the Kettle River sample, were retained for analysis (Ecology, 1995a; EPA, 2000). A depth of only 5 cm could be reached for the Kettle River site due to a rocky substratum. The top 10 cm layer of each grab was removed with stainless steel spoons or scoops, placed in a stainless steel bowl, and homogenized by stirring. Material touching the sidewalls of the grab was not retained for analysis.

For the Microtox® bioassay test, sediment samples were taken with minimum disturbance of the sediment and the sample containers filled completely (no headspace) to minimize changes in porewater chemistry. Ideally, samples for Microtox® analysis should not be homogenized, so as to minimize disturbance to the sediment. Sediment samples from the upper Columbia River and tributary sites were inadvertently homogenized, and samples from Lake Roosevelt were not homogenized. The implications of this difference in the collection of sediment are discussed in further detail in the Data Quality section of this report.

The homogenized sediment was placed in glass jars with Teflon lid liners and cleaned to EPA QA/QC specifications (EPA, 1990). Sample containers, preservation, and holding times are shown in Appendix B-1.

Stainless steel implements used to collect sediments were cleaned by washing with Liquinox detergent, followed by sequential rinses with tap water, 10% nitric acid, and deionized water. The equipment was then air-dried and wrapped in aluminum foil. Between-sample cleaning of the van Veen grab consisted of thorough brushing and rinsing with on-site water.

Puget Sound Estuary Protocols (PSEP) procedures (EPA, 1996) for collection, preservation, transportation, and storage of the sediment samples were followed in an effort to limit sources of bias. Sediment samples were placed on ice immediately after collection and transported to the Ecology/EPA Manchester Environmental Laboratory within two days of collection. The

bioassay samples were shipped to the contract laboratories via Manchester Laboratory. Chain-of-custody was maintained throughout the sampling and analysis.

Laboratory Analysis

Table 1 shows analytical methods, reporting limits, and laboratories.

- Chemical analysis was conducted by Ecology/EPA Manchester Environmental Laboratory, Manchester, Washington.
- Grain size analysis was conducted by Rosa Environmental and Geotechnical Laboratory, Seattle, Washington.
- *Hyalella* and *Chironomus* tests were conducted by Northwestern Aquatic Sciences, Newport, Oregon.
- Microtox® test was conducted by Parametrix, Inc., Kirkland, Washington.

Table 1. Analytical Methods, Reporting Limits, and Laboratories

Analysis	Reporting Limit	Method	Laboratory
Chemistry			
Arsenic	4 mg/Kg, dry	ICP/AES – EPA3050B/6010B	Manchester
Cadmium	0.5 mg/Kg, dry	ICP/AES – EPA3050B/6010B	"
Copper	1 mg/Kg, dry	ICP/AES – EPA3050B/6010B	"
Lead	3 mg/Kg, dry	ICP/AES – EPA3050B/6010B	"
Mercury	0.003 mg/Kg, dry	CVAA – EPA7471A/245.5	"
Zinc	0.5 mg/Kg, dry	ICP/AES – EPA3050B/6010B	"
TOC	0.1%	Combustion/CO2 - EPA (1996)	"
% Solids	0.1%	Gravimetric - EPA (1996)	"
Grain Size*	0.1%	Sieve & Pipet - EPA (1996)	Rosa Environmental
Bioassays			
<i>Chironomus tentans</i> 20-day	n/a	Method 100.5 (EPA, 2000)	N.W. Aquatic Sciences
<i>Hyalella azteca</i> 10-day	n/a	ASTM E-1706 and Method 100.1 (EPA, 2000)	N.W. Aquatic Sciences
Microtox porewater	n/a	Ecology Protocol (Adolphson, 2000)	Parametrix

* Gravel, sand, silt, and clay fractions

n/a = reporting limits not applicable to bioassay tests (see Appendix C-2, QC Requirements for Sediment Bioassay)

These bioassay tests have been recently developed, and no laboratories are accredited for the 20-day *Chironomus* and Ecology- modified Microtox® methods. However, Parametrix is accredited for the 1995 PSEP Microtox® test, and Northwestern Aquatic Sciences is accredited for the 10-day ASTM methods for both *Hyaella* and *Chironomus*. In addition, both laboratories have had previous success in conducting the bioassay test methods used in the present study (Johnson and Norton, 2001).

The Microtox® test measures light emitted by the bioluminescent marine bacterium *Vibrio fischeri* on exposure to test sediment porewater for five and fifteen minutes and compared to control and reference sediment porewater. The 100% porewater test (Adolphson, 2000) is an Ecology modification of PSEP procedures that use organic or aqueous extracts (Appendix C-1).

The *Hyaella* test measures survival of the amphipod *Hyaella azteca* after a 10-day exposure to test sediment and is considered an acute toxicity test (ASTM E-1706 and EPA method 100.1; EPA, 2000).

Growth and survival of the midge *Chironomus tentans* are the end points of the 20-day chronic *Chironomus* test. The method is a modification of a 50-to-65 day life-cycle test (EPA method 100.5; EPA, 2000).

This page is purposely blank for duplex printing

Data Quality

Chemical Data

Chemical data met laboratory quality control analysis requirements (Appendix B-2) and are further detailed in the Quality Assurance Project Plan for this study (Era and Serdar, 2001). Quality control samples for the chemical analysis included a laboratory duplicate, one matrix spike, one matrix spike duplicate, method blanks, and laboratory control samples (LCS). A laboratory triplicate analysis was conducted for grain size.

Matrix spike recovery values for the metals data were consistently near 100%, indicating little or no bias due to possible sample matrix interference.

Replicate field samples were compared to determine the overall precision of the data (sampling techniques and laboratory analysis). The relative percent difference (RPD) for each replicate was well within the data quality objective of 25% (Appendix B-3).

Bioassay Data

Bioassay methods and test conditions were followed, and Quality Assurance (QA) and reporting requirements were met (Appendices C-2 and C-3). Results for negative controls and reference toxicants were acceptable for all bioassay tests, with the exception of the negative control for the *Chironomus tentans* survival test which marginally failed to meet quality control requirements for survival (68.6% vs 70%). After careful review of EPA method 100.5 by the laboratory (Northwestern Aquatic Sciences) and the authors of the present study, it was concluded that the negative control met EPA standards for test condition acceptability based upon average dry weight of control organisms at 20 days. The minimum EPA acceptability criterion is 0.48 mg average dry weight for control organisms (EPA, 2000). The control organisms weighed 1.52 mg average dry weight on day 20.

For the Microtox® bioassay test, samples from the upper Columbia River and tributary sites (noted in Appendix A-2) were inadvertently homogenized before filling the sample containers. There were no patterns in the bioassay results indicating that homogenization affected the accuracy of the bioassay toxicity tests. Previous work with Microtox® suggests that field homogenization of sediments may actually reduce toxicity, indicating less of a biological impact than what otherwise might be demonstrated had homogenization not taken place (Adolphson, personal communication).

Data Evaluation

A Spearman Ranked correlation was performed on conventional and metals data using SYSTAT version 9 (Table 2). This type of correlation is a non-parametric test used to determine correlation coefficients for ranked data, rather than the values themselves.

Table 2. Spearman Ranked Correlation Matrix for Variables Measured in Lake Roosevelt and Upper Columbia River Sediments

	% TOC	% Gravel	% Sand	% Silt	% Clay	As	Cd	Cu	Pb	Hg	Zn
% TOC	1.000										
% Gravel	-0.382	1.000									
% Sand	-0.736	0.143	1.000								
% Silt	0.853	-0.353	-0.807	1.000							
% Clay	0.778	-0.259	-0.869	0.871	1.000						
As	0.162	-0.180	-0.097	0.047	0.354	1.000					
Cd	-0.006	-0.612	0.164	-0.075	0.088	0.644	1.000				
Cu	-0.061	-0.591	0.389	-0.282	-0.182	0.530	0.900	1.000			
Pb	-0.043	-0.547	0.212	-0.200	-0.013	0.705	0.964	0.912	1.000		
Hg	0.569	-0.531	-0.512	0.428	0.667	0.777	0.530	0.446	0.543	1.000	
Zn	-0.195	-0.586	0.418	-0.344	-0.169	0.596	0.915	0.948	0.927	0.457	1.000

Boxed values represent significant correlations (p<0.05)

Bioassay results were evaluated to determine sediment toxicity. For the *Hyaella* and *Chironomus* tests, sediment samples were considered toxic if they were significantly different from the reference sediment (i.e., toxicity “hit”). Statistical significance was measured using a t-test at a significance level of 0.05 as recommended by Sediment Management Standards (SMS) for sediment bioassays (WAC Chapter 173-204; Ecology, 1995b). Microtox® toxicity was based on both statistical significance and numerical difference. Statistical significance for Microtox® was determined using the same criteria as *Hyaella* and *Chironomus* with t-test significance of 0.05. Numerical difference for Microtox® was measured by a relative percent difference between the sample and reference sediment $\geq 15\%$ (WAC Chapter 173-204; Ecology, 1995b; Adolphson, personal communication).

The contract laboratories performed both the statistical and numerical tests on the bioassay results. An arcsine square root transformation was performed on both the *Hyaella* and *Chironomus* survival tests prior to t-test analysis. No transformation was performed on *Chironomus* growth. Following determination of normality and homogeneity of variances, a one-tailed Student t-test, Mann Whitney, or Approximate t-test was conducted. The statistical software used was BioStat (Beta v.2.0c) bioassay software developed by the U.S. Army Corps of Engineers, Seattle District. A homoscedastic t-test was used for the Microtox® bioassay data. The data were not transformed.

Results

Conventionals and Metals

Table 3 shows results for percent solids, total organic carbon (TOC), and grain size. Results for TOC analysis conducted at 70° C and at 104° C were similar, therefore only TOC at 104° C is reported in Table 3. TOC was low at all sample sites, ranging from 0.02% - 2.2% with the average value less than 1%. Grain size results indicated that the reference site and upper Columbia River sites (10 and 1-4) consisted mostly of sand (> 84%), and the Lake Roosevelt sites (5-9) consisted mainly of silt and clay fractions (> 89%), with the exception of Grand Coulee Dam (92% sand).

Table 3. Percent Solids, TOC, and Grain Size for Lake Roosevelt Sediments

Site Name	Site No.	Sample No.	% Solids	% TOC (104° C)	Grain Size (%)			
					Gravel ≥ 2000µm	Sand < 2000µm – 62µm	Silt < 62µm – 3.9µm	Clay < 3.9µm
Reference: Lower Arrow Lake	10	198040	77.4	0.2	12.6	84.3	0.0 ^a	0.0 ^a
Boundary	1	198043	69.0	0.5	0.0	96.1	0.0 ^a	0.0 ^a
Auxiliary Gage	2	198042	79.0	0.02 U	0.0	99.6	0.0 ^a	0.0 ^a
Goodeve Creek	3	198041	72.6*	0.5*	0.1	99.3	0.0 ^a	0.0 ^a
Kettle River	4	198044	39.4	1.1	0.6	86.7	10.7	2.1
Castle Rock	5	198046	35.3	1.1	0.0	4.3	64.5	31.2
Whitestone Creek	6	198047	27.4	1.6	0.1*	9.2*	38.7*	52.1*
Whitestone Creek (replicate)	6	198048	27.6	1.6	0.0	7.8	39.6	52.8
Sanpoil River	7	198045	55.5	2.2	0.0	11.0	75.0	13.9
Swawilla Basin	8	198049	25.3	1.7	0.0	4.0	40.8	55.1
Grand Coulee Dam	9	198050	70.8	0.3	0.6	92.3	4.2	3.0

* mean of laboratory duplicate analysis

^a balance of fine material (< 62µm) was too small to quantify

U = The analyte was not detected at or above the reported value

Metals concentrations in sediments are summarized in Table 4. The reference site (10) had low concentrations of all six metals (arsenic, cadmium, copper, lead, mercury, zinc) compared to the other sample sites. Metals were also low in the Kettle River, Sanpoil River, and Grand Coulee (sites 4, 7, 9) samples. Concentrations of cadmium, copper, lead, and zinc were the highest overall in the upper Columbia River (sites 1-3), especially at Auxiliary Gage and Goodeve Creek. Concentrations of copper and zinc at sites 1-3 were generally two to three orders of magnitude above reference concentrations. Arsenic concentrations were low at all nine sites. Mercury concentrations were high in Lake Roosevelt compared to the upper Columbia River.

Table 4. Metals Concentrations in Lake Roosevelt and Upper Columbia River Sediments

Site Name	Site No.	Sample No.	Metals mg/Kg dw					
			As	Cd	Cu	Pb	Hg	Zn
Reference:								
Lower Arrow Lake	10	198040	2.0 U*	0.46*	4*	12*	0.0004 U	27*
Boundary	1	198043	6.6	6.7	494	182	0.10	3730
Auxiliary Gage	2	198042	5.0	18.0	2210	324	0.02	16100
Goodeve Creek	3	198041	20.0	16.2	2210	344	0.08	12200
Kettle River	4	198044	2.0 U	1.0	16	5	0.0007 U	34
Castle Rock	5	198046	8.3	7.1	66	173	0.68	471
Whitestone Creek	6	198047	13.0	11.9	74	285	1.25	952
Whitestone Creek (replicate)	6	198048	13.0	12.4	76	292	1.07	979
Sanpoil River	7	198045	3.5	1.9	20	19	0.03	70
Swawilla Basin	8	198049	11.0	12.4	73	295	1.25	1040
Grand Coulee Dam	9	198050	9.2	1.8	11	17	0.03	86

* mean of laboratory duplicate analysis

U = The analyte was not detected at or above the reported value

A Spearman Ranked correlation performed on conventionals and metals showed several strong correlations: cadmium, copper, lead, and zinc were found to be significantly intercorrelated; arsenic correlated with lead and mercury; and mercury correlated with clay fractions (Table 2).

Bioassay

Results for the bioassay tests are summarized in Table 5. With the exception of the Grand Coulee site, all sites had a toxicity hit for at least one of the bioassay tests. The reference sediment from Lower Arrow Lake showed low toxicity in all of the bioassay tests.

Table 5. Bioassay Results for Lake Roosevelt and Upper Columbia River Sediments

Site name	10-day <i>Hyalella azteca</i> % Survival ± SD	20-day <i>Chironomus tentans</i> % Survival ± SD	20-day <i>Chironomus tentans</i> growth* (mg-dry weight)	5-minute Microtox Porewater (percent light reduction from reference)	15-minute Microtox Porewater (percent light reduction from reference)	Toxicity Hit Frequency
Control	90.0 ± 13.1	68.8 ± 13.6	1.52 ± 0.24	--	--	--
Reference: Lower Arrow Lake	71.3 ± 22.3	76.3 ± 10.6	1.18 ± 0.14	--	--	--
Boundary	66.3 ± 21.3	70.0 ± 12.0	1.05 ± 0.19	16	21	2/5
Auxiliary Gage	56.3 ± 15.1	2.5 ± 4.6	1.08 ± 1.36	6	5	2/5
Goodeve Creek	50.0 ± 15.1	0.0 ± 0.0	0.0 ± 0.0	26	38	5/5
Kettle River	68.8 ± 28.0	51.3 ± 36.4	2.55 ± 0.77	67	68	2/5
Castle Rock	72.5 ± 12.8	62.5 ± 10.4	1.55 ± 0.34	15	15	3/5
Whitestone Creek	92.5 ± 11.6	55.0 ± 20.0	1.36 ± 0.18	7	5	1/5
Sanpoil River	70.0 ± 19.3	53.8 ± 27.7	1.08 ± 0.18	2	-1	1/5
Swawilla Basin	75.0 ± 16.0	60.0 ± 16.0	1.25 ± 0.28	8	6	1/5
Grand Coulee Dam	71.3 ± 17.3	63.8 ± 23.3	1.56 ± 0.52	-5	-7	0/5

* Values shown are average growth of surviving organisms. Results of all replicates are used for statistical comparisons. Organism mortality is assigned a value of zero.

boxed values show significant toxicity compared to reference.

-- = Not applicable

Hyalella azteca survival had the fewest number of toxicity hits compared to the other bioassay tests. Mean survival was ≥ 50% at all the sites with the only toxicity hit at Goodeve Creek. The Lake Roosevelt sites generally showed higher survival than the upper Columbia River sites.

Of the two endpoints (survival and growth) for the *Chironomus tentans* test, the survival endpoint appeared to be more sensitive than the growth endpoint, having six toxicity hits compared to two toxicity hits for growth. Mean survival was ≤ 3% at Auxiliary Gage and Goodeve Creek, significantly lower than the other sites which ranged from 51% - 70%.

Chironomus growth at these two sites was also significantly lower than the reference site due to high mortality, although the average weight of the two (of 80) organisms in the Auxiliary Gage sample was similar to the reference.

Sediments were toxic to Microtox® at four sites: Boundary, Goodeve Creek, Kettle River, and Castle Rock.

Table 5 lists the toxicity “hit frequency” for each site. The Auxiliary Gage and Goodeve Creek sediments were the most toxic. The least toxic sediments were from Boundary, Kettle River, Whitestone Creek, Sanpoil River, and Grand Coulee.

Discussion

Longitudinal Distribution of Metals

Sediment samples taken at the upper Columbia River sites (1-3) consisted of a visibly dark sandy mixture, which probably indicates the presence of slag from the Cominco smelter. Previous studies have found the dark sand to contain high levels of slag particles (Bortelson et al., 1994; Serdar et al., 1994). Elevated levels of copper and zinc, and to a lesser extent lead, are associated with slag (Bortelson et al., 1994; Cominco, 1991). Elevated levels of copper and zinc were found at these same three sites, indicating that slag material may still be present in the upper Columbia River.

Another pattern consistently observed is elevated mercury concentrations in the lower part of Lake Roosevelt. Lower Lake Roosevelt has historically contained high percentages of clay sediments ($< 3.9\mu\text{m}$) that have correlated with elevated levels of mercury (Bortelson et al., 1994; Johnson et al., 1988). In the present study, mercury also correlated with the clay sediments of lower Lake Roosevelt (Table 2).

Figure 3 shows metals concentrations in the Columbia River from Lower Arrow Lake, Canada down river to the Grand Coulee Dam, along with results from three previous studies: Johnson et al., 1988; Johnson, 1991; and Bortelson et al., 1994. Total recoverable metals are shown; complete data are in Appendix D-1. River miles in Figure 3 coincide with the sampling sites from the present study. The Kettle River and Sanpoil River sites were not included in the trend comparisons of the mainstem Columbia River.

Differences in exact sample location, sedimentation and erosion, depth of sediment sample (top 10 cm in 2001 and top 2 cm in the other studies), and differences in field and laboratory methods make comparisons difficult. However, the following general longitudinal trends are evident from Figure 3:

- Copper and zinc concentrations are high in the upper Columbia River and low in Lake Roosevelt.
- Mercury levels are low in the upper Columbia River and high in Lake Roosevelt.
- Lead shows no obvious trends, except that lead concentrations have remained relatively high throughout the study area.
- Copper, zinc, and arsenic were lower overall in the present study than in previous studies.
- Cadmium concentrations were higher overall in the present study than in previous studies.

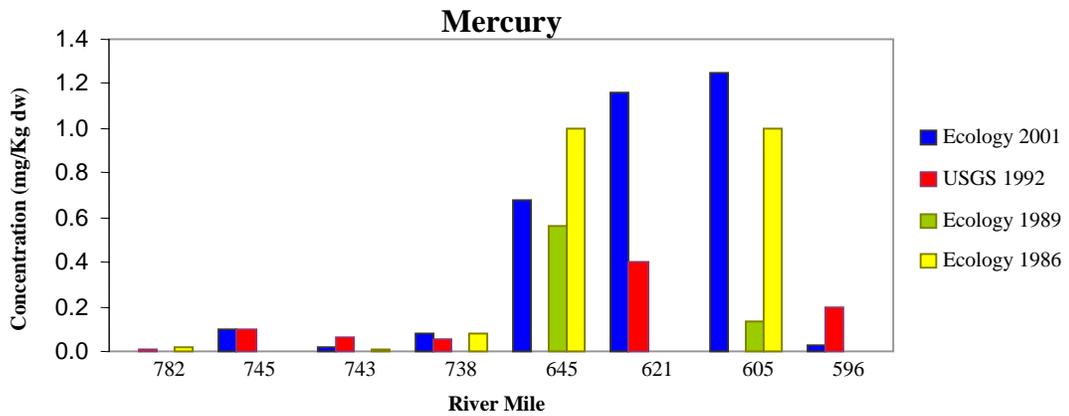
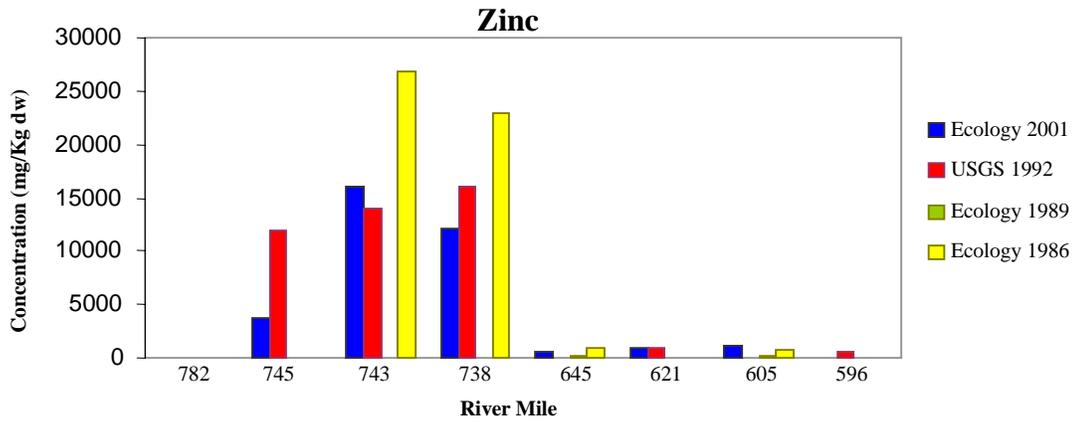
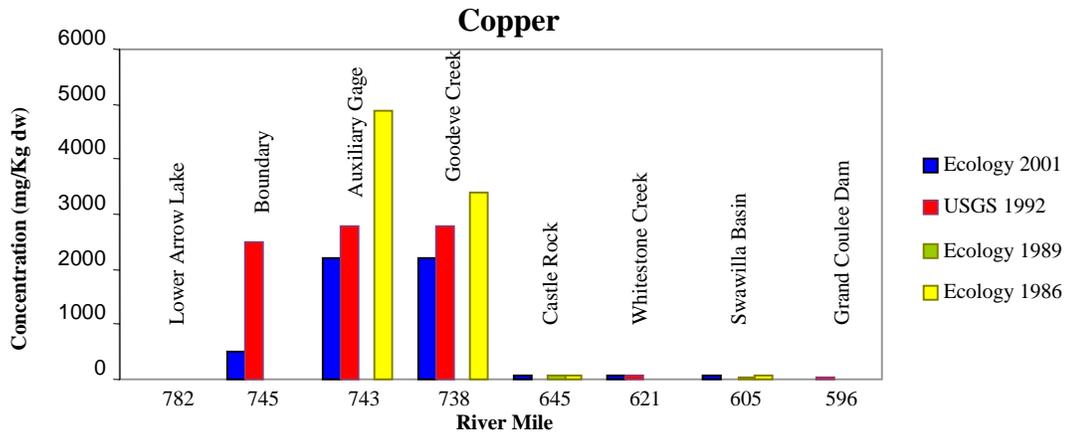


Figure 3. Metal Concentration Trends in the Upper Columbia River and Lake Roosevelt Found in the Present and Previous Surveys

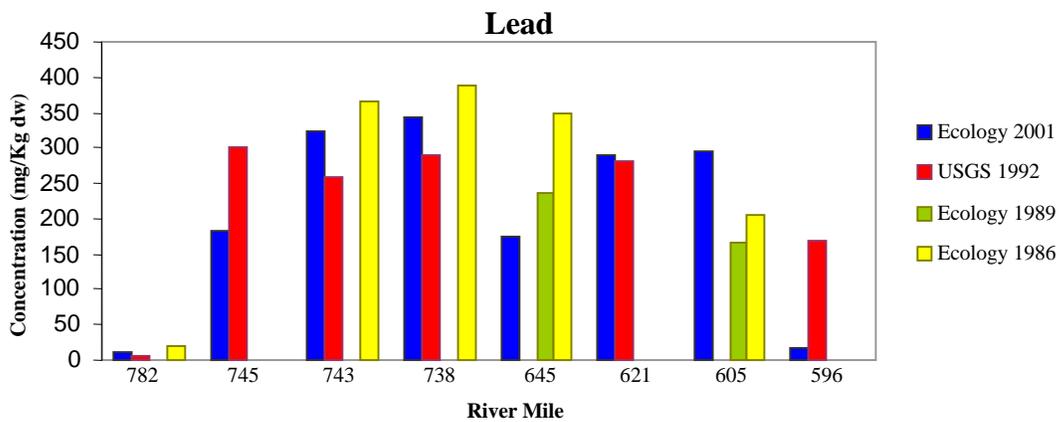
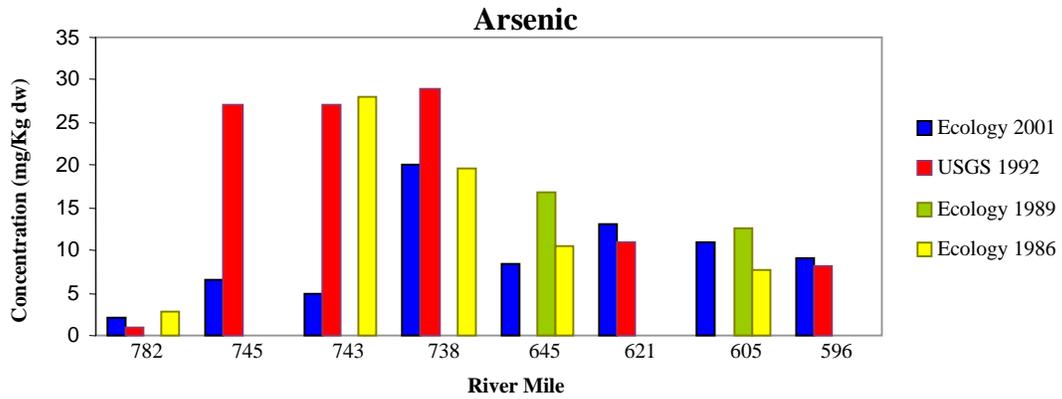
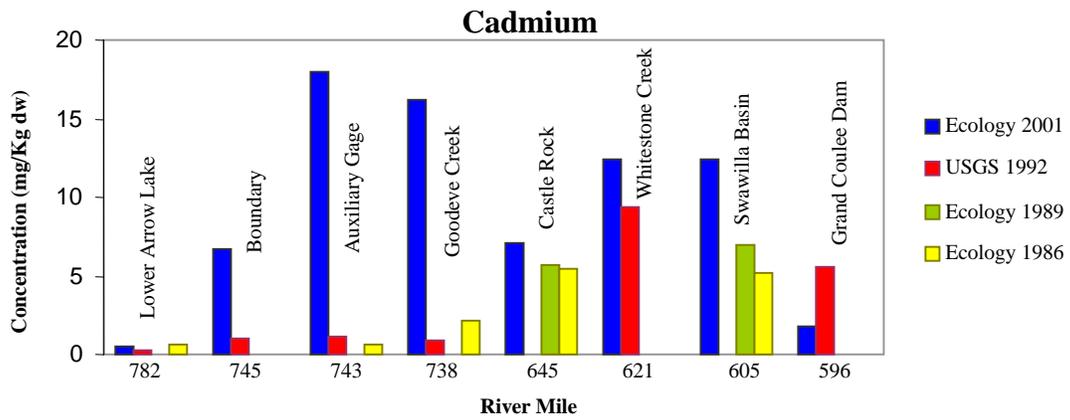


Figure 3. (continued)

Sediment Toxicity Compared to Earlier Studies

Table 6 compares bioassay results to previous studies by Johnson (1991) and Bortelson et al. (1994). It is difficult to draw conclusions about specific changes in sediment toxicity due to the differences in types of bioassay tests and methods, time (season and year), and exact locations. It is, however, apparent that some level of toxicity remains at all nine sites, with the exception of the Grand Coulee site. The upper Columbia River sites are generally more toxic to bioassay organisms than the lower Lake Roosevelt sites. The Grand Coulee site was originally listed on the 303(d) list based on the results of the 1992 USGS study (Bortelson et al., 1994), in which the sample sediments consisted of finer material (< 62µm). In the present study, sediments for the Grand Coulee site were sandy and lower in metals, which may explain the difference in sediment toxicity. The 2001 Grand Coulee site was also located approximately 7000 feet from the original 1992 station location (Figure 4).

Table 6. Comparison of Bioassay Results to Previous Lake Roosevelt Data

	Lower Arrow Lake	Boundary	Auxiliary Gage	Goodeve Creek	Kettle River	Castle Rock	Whitestone Creek	Sanpoil River	Swawilla Basin	Grand Coulee Dam
Ecology/ 2001 ^a										
<i>Hyalella</i> Survival	R	n	n	Y	n	n	n	n	n	n
<i>Chironomus</i> Survival	R	n	Y	Y	n	Y	Y	Y	Y	n
<i>Chironomus</i> Growth	R	n	Y	Y	n	n	n	n	n	n
Microtox Porewater	R	Y	n	Y	Y	Y	n	n	n	n
USGS/ 1992 ^b										
<i>Hyalella</i> Survival	R	Y*	Y*	Y*	n	--	n	Y*	--	n
<i>Ceriodaphnia</i> Survival	R	Y*	Y*	n	Y*	--	n	n	--	n
<i>Ceriodaphnia</i> Reproduction	R	Y*	Y*	Y*	Y*	--	Y*	n	--	Y*
Microtox Porewater	R	--	--	--	Y	--	Y	n	--	n
Ecology/ 1989 ^c										
<i>Hyalella</i> Survival	--	--	--	--	--	Y*	--	--	Y*	--
<i>Daphnia</i> Solid Phase	--	--	--	--	--	Y*	--	--	Y*	--
<i>Daphnia</i> Elutriate	--	--	--	--	--	n	--	--	n	--
Microtox Porewater	--	--	--	--	--	n	--	--	n	--

^a Present study

^b Bortelson et al., 1994

^c Johnson, 1991

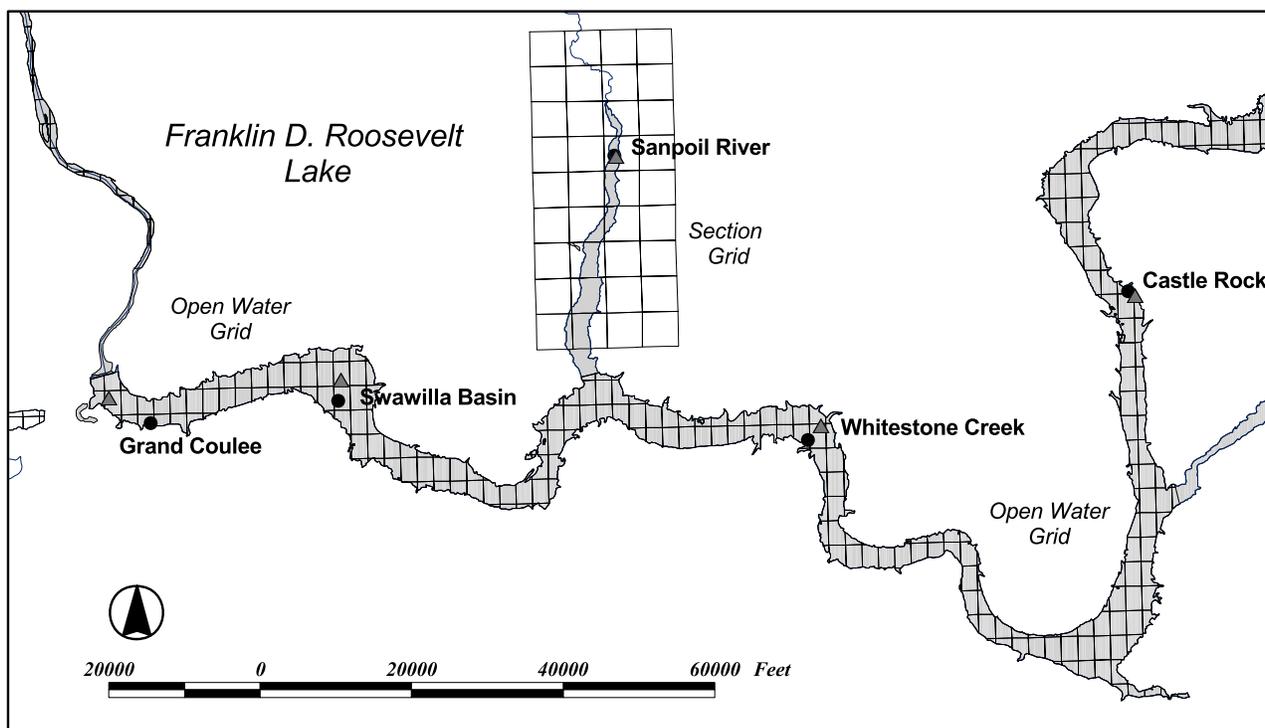
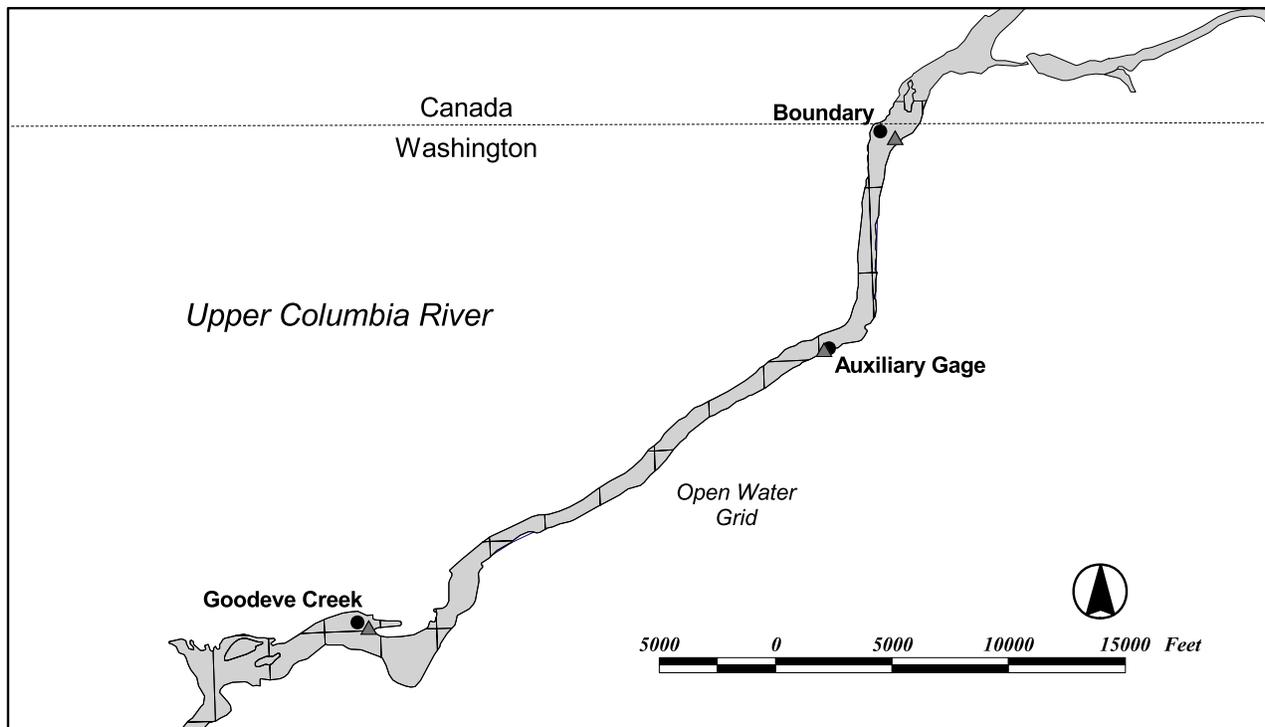
* Basis for listings on the 1998 303(d) list

Y = Sediment toxic to bioassay organisms

n = Sediment not toxic to bioassay organisms

R = Reference sediments

-- = no data



- 2001 Station Locations
- ▲ Previous Station Locations (Ecology, 1989 or USGS, 1994)

Figure 4. Sampling Site Locations for the Present Study and Previous Studies with Open Water Grid and Section Grid Overlays

1998 303(d) vs. Proposed 303(d) Listing Policy

The present study set out to determine if currently listed sites should remain on the 303(d) list. However, since completion of sampling for the present study, changes have been proposed for the 2002 303(d) listing policy (Ecology, 2001).

The current listing policy (1998 303(d) list) requires only one toxicity “hit” per sample and station location within a waterbody segment. Based on this current policy, all but the 2001 Grand Coulee site should remain listed. The Grand Coulee site from the prior USGS (Bortelson et al., 1994) study should remain listed.

The proposed listing policy is more stringent in that it requires a toxicity “hit” at three separate station locations within the same segment. For rivers and streams, the portion of the waterbody lying within the section of a township and range defines a segment. For open water areas, a grid 2,460 feet longitude by 3,650 feet latitude defines a segment. Lake Roosevelt and upper Columbia River sites fall under the open water segment designation, and the Sanpoil and Kettle River sites fall under the river and stream segment designation. According to the policy, none of the sites are close enough to meet this “three station segment” condition.

Figure 4 shows open water and section segments (grids) for upper Columbia River and lower Lake Roosevelt sites. Even if site locations from the previous listings were counted as separate station locations from the present study, there would still not be the three stations required within the same segment. Table 7 shows how the results for the present study would be interpreted for listing or de-listing on the 303(d) list, based on the current and proposed listing criteria.

Table 7. 1998 vs. Proposed 303(d) Listing Decisions for Lake Roosevelt Bioassay data

Site Name	Ecology 1989		USGS 1992		Present Study	
	1998	Proposed 2002	1998	Proposed 2002	1998	Proposed 2002
Boundary	--	--	Yes	No	Yes	No
Auxiliary Gage	--	--	Yes	No	Yes	No
Goodeve Creek	--	--	Yes	No	Yes	No
Kettle River	--	--	Yes	No	Yes	No
Castle Rock	Yes	No	--	--	Yes	No
Whitestone Creek	--	--	Yes	No	Yes	No
Sanpoil River	--	--	Yes	No	Yes	No
Swawilla Basin	Yes	No	--	--	Yes	No
Grand Coulee Dam	--	--	Yes	No	No	No

-- = no data

Metals and Sediment Toxicity

Previous surveys of the upper Columbia River and Lake Roosevelt have suggested that sediment toxicity was likely due to metals contamination of sediments. Although the bioassay listing decisions for the present study do not depend on the results of metal concentrations for Lake Roosevelt sediments, it is worth noting the relationship between sediment toxicity and metals concentrations. This relationship may be best evidenced by comparing metals concentrations to Freshwater Sediment Quality Values (FSQVs) for metals in Washington State and to Consensus-Based Threshold Effects Concentrations (TECs) for freshwater sediments shown in Table 8 (Cubbage et al., 1997; MacDonald et al., 2000).

Table 8. Comparison of Lake Roosevelt Metal Concentrations to FSQVs for Metals in Washington State and to Consensus-Based TECs for Freshwater Sediments

Site Name	Site No.	Sample No.	Metals mg/Kg dw					
			As	Cd	Cu	Pb	Hg	Zn
Reference:			2.0					
Lower Arrow Lake	10	198040	U*	0.46*	4*	12*	0.0004 U	27*
Boundary	1	198043	6.6	6.7	494	182	0.10	3730
Auxiliary Gage	2	198042	5.0	18.0	2210	324	0.02	16100
Goodeve Creek	3	198041	20.0	16.2	2210	344	0.08	12200
Kettle River	4	198044	2.0 U	1.0	16	5	0.0007 U	34
Castle Rock	5	198046	8.3	7.1	66	173	0.68	471
Whitestone Creek	6	198047	13.0	11.9	74	285	1.25	952
Whitestone Creek (replicate)	6	198048	13.0	12.4	76	292	1.07	979
Sanpoil River	7	198045	3.5	1.9	20	19	0.03	70
Swawilla Basin	8	198049	11.0	12.4	73	295	1.25	1040
Grand Coulee Dam	9	198050	9.2	1.8	11	17	0.03	86
FSQVs	--	--	57	5.1	390	450	0.41	410
Consensus-Based TECs	--	--	9.8	0.99	32	36	0.18	121

* mean of laboratory duplicate analysis

U = The analyte was not detected at or above the reported value

Shaded and **bolded** values exceed both FSQVs and Consensus-Based TECs

White-boxed and **bolded** values exceed Consensus-Based TECs

There are no Washington State standards or EPA national criteria for metal contamination in freshwater sediments. There have, however, been many different freshwater sediment quality guidelines created in the United States. FSQVs and TECs represent the spectrum of both moderately conservative and highly conservative guidelines and have been used in Ecology studies to evaluate the relationship between metals concentrations and the possibility of effects to benthic life (Johnson et al., 2000; Serdar et al., 2000).

The FSQVs were derived by analyzing freshwater bioassay and chemistry data sets collected in Washington, and by reviewing freshwater and marine sediment criteria developed in Canada and the U.S., including Washington standards for marine waters. The creators of the FSQVs concluded that, when applied to freshwater, the existing SMS for marine waters provided the best mix of sensitivity and efficiency in predicting effects to the bioassay organism *Hyaella azteca* and essentially minimum chemical concentrations expected to cause adverse effects on biological resources. It was concluded that the FSQVs predict biological effects better than other sets of values, including sediment quality criteria and guidelines developed by other regulatory agencies.

The TECs were recently developed and integrate work done by a number of investigators including Cabbage et al. TECs are concentrations below which harmful effects on sediment dwelling organisms are not expected to occur, and are therefore more conservative values.

Metal concentrations from Lake Roosevelt sediments exceed both the FSQVs (moderately conservative) and TECs (highly conservative) for at least three different metals at six sites (Table 8). All six sites also had bioassay failures. Based on freshwater sediment quality guideline exceedences and bioassay failures for these six sites, it is reasonable to assume that the bioassay toxicity may be due to the high levels of metals.

Conclusions

Metals concentrations and toxicity levels in the upper Columbia River and Lake Roosevelt sediments remain relatively high. All but the Grand Coulee site had at least one toxicity hit out of the suite of bioassay tests performed on the sediments. In the upper Columbia River reach, above the town of Northport and below the Canadian border, cadmium, copper, lead, and zinc concentrations were elevated. In Lake Roosevelt, cadmium, mercury, and zinc concentrations were elevated. Based on these findings and the findings of previous studies, bioassay toxicity for the upper Columbia River and Lake Roosevelt may be attributed to metals contamination.

Although the Kettle River and Sanpoil River sites showed toxicity to some of the bioassay tests, metal concentrations were very low. The cause of the apparent sediment toxicity for these sites is unknown and could possibly be attributed to other toxic parameters not tested for in the present study.

The primary objective of this study was to reassess metals concentrations and toxicity of sediments through bioassay tests and to make recommendations on the continued listing or de-listing of the upper Columbia River and Lake Roosevelt sites on the 303(d) list. Based on the existing policy for 303(d) listings (one toxicity hit per segment), eight of the nine sites (1-8) should be listed. Based on the newly proposed listing criteria (toxicity at three separate locations within a segment), none of the nine sites should be placed on the 2002 303(d) list.

Regardless of the 303(d) listing status, there is sufficient toxicity at the majority of sites in the upper Columbia River and Lake Roosevelt to warrant further investigation.

Recommendations

The nine sites tested for sediment toxicity in Lake Roosevelt and the upper Columbia River do not meet criteria for listing on part 1 of the 2002 303(d) list, based on the proposed Water Quality Policy 1-11 for freshwater sediment bioassay failure. However, due to significant toxicity in the majority of the sites, it is recommended that eight of the nine sites be reassigned to part 5, *Undetermined Status*, of the proposed 2002 303(d) list. This list will allow Ecology and/or other public and private stakeholders to track these waterbodies and pursue additional sampling, incorporate a waterbody into existing studies, or find other means to confirm or address the problem.

Additional research to re-evaluate these sites for listing on the proposed 2002 303(d) list should be designed to encapsulate bioassay tests for at least three separate stations within the same segment. For some of the nine sites, the addition of only one new station may meet the data collection criteria of the proposed policy.

References

- Adolphson, P., 2000 (draft final). Microtox® 100 Percent Sediment Porewater Toxicity Assessment. Washington State Department of Ecology, Olympia, WA.
- Adolphson, P., 2001. Personal communication. Washington State Department of Ecology, Olympia, WA.
- Bortelson, G., S.E. Cox, M.D. Munn, R.J. Schumaker, E.K. Block, L.R. Lucy, S.B. Cornelius, 1994. Sediment-Quality Assessment of Franklin D. Roosevelt Lake and the Upstream Reach of the Columbia River, Washington, 1992. U.S. Geological Survey. Open File Rept. 94-315.
- Cominco, 1991. Environmental Management Program; Slag Disposal Options – Environmental and Engineering Studies. Trail, B.C.
- Cabbage, J., D. Batts, and S. Breidenbach, 1997. Creation and Analysis of Freshwater Sediment Quality Values in Washington State. Washington State Department of Ecology, Olympia, WA. Pub. No. 97-323a.
- Ecology, 1995a (draft). Guidance on the Development of Sediment Sampling and Analysis Plans Meeting the Requirements of the Sediment Management Standards. Chapter 173-204 WAC. Washington State Department of Ecology, Olympia, WA.
- Ecology, 1995b. Sediment Management Standards. Chapter 173-204 WAC. Washington State Department of Ecology, Olympia, WA.
- Ecology, 2001 (draft). Assessment of Water Quality for the Section 303(d) List. Water Quality Program Policy 1-11. Washington State Department of Ecology, Olympia, WA.
- EPA, 1990. Specifications and Guidance for Obtaining Contaminant-Free Sample Containers. U.S. Environmental Protection Agency, OSWER Directive #93240.0-05.
- EPA, 1996. Puget Sound Estuary Program (PSEP): Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound. U.S. Environmental Protection Agency, Region 10, Office of Puget Sound, Seattle, WA.
- EPA, 2000. Methods for Measuring the Toxicity and Bioaccumulation of Sediment Associated Contaminants with Freshwater Invertebrates, Second Edition. U.S. Environmental Protection Agency. EPA-600-R-99-064.
- Era, B. and D. Serdar, 2001. Quality Assurance Project Plan for Reassessment of Toxicity of Lake Roosevelt Sediments. Washington State Department of Ecology, Olympia, WA.
- Johnson, A., D. Norton, and B. Yake, 1988. An Assessment of Metals Contamination in Lake Roosevelt. Washington State Department of Ecology, Olympia, WA. Pub. No. 89-e26.

Johnson, A., 1991. Review of Metals, Bioassay, and Macroinvertebrate Data from Lake Roosevelt Benthic Samples Collected in 1989. Washington State Department of Ecology, Olympia, WA. Pub. No. 91-e23.

Johnson, A. and D. Norton, 2001. Chemical Analysis and Toxicity Testing of Spokane River Sediments Collected in October 2000. Washington State Department of Ecology, Olympia, WA. Pub. No. 01-03-019.

Lowe, T.P., T.W. May, W.G. Brumbaugh, and D.A. Kane, 1985. *National Contaminant Biomonitoring Program: Concentration of Seven Elements in Freshwater Fish, 1978-1981.* Arch. Environ. Contam. Toxicol. 14:363-388.

MacDonald, D.D., C.G. Ingersoll, and T.A. Berger, 2000. *Development of and Evaluation of Consensus-Based Sediment Quality Guidelines for Freshwater Ecosystems.* Arch. Environ. Contam. Toxicol. 39:20-31.

Serdar, D., B. Yake, and J. Cabbage, 1994. Contaminant Trends in Lake Roosevelt. Washington State Department of Ecology, Olympia, WA. Pub. No. 94-185.

Serdar, D., J. Cabbage, and D. Rogowski, 2000. Concentrations of Chemical Contaminants and Bioassay Response to Sediments in Salmon Bay, Seattle. Results of Phase III Sampling. Washington State Department of Ecology, Olympia, WA. Pub. No. 00-03-053.

Appendices

This page is purposely blank for duplex printing

Appendix A

Sample Locations and Descriptions

This page is purposely blank for duplex printing

Appendix A-1. Sediment Sampling Locations

Site Name	Site No.	Date	River Mile/ Tributary Mile	Water Depth (Ft)	Description	Latitude	Longitude
Boundary	1	5/08/01	745	20	Near right bank below sharp bend in river	48° 59.810'	117° 38.324'
Auxiliary Gage	2	5/08/01	743	10	20 ft off left bank, just above gaging station	48° 58.282'	117° 38.842'
Goodeve Creek	3	5/08/01	738	3	Just off right bank sand bar	48° 56.493'	117° 44.157'
Kettle River	4	5/08/01	707/4.3	1	Off left bank and 200 ft downstream of Napoleon Bridge	48° 44.067'*	118° 06.950'*
Castle Rock	5	5/09/01	645	63	160 yards off left bank	47° 58.817'	118° 21.329'
Whitestone Creek	6	5/09/01	621	75	Off left bank	47° 55.373'	118° 33.320'
Sanpoil River	7	5/08/01	616/7.2	2	0.1 mile off right bank and campground	48° 02.433'	118° 40.154'
Swawilla Basin	8	5/09/01	605	112	Middle of reservoir (closer to left bank)	47° 56.611'	118° 50.517'
Grand Coulee Dam	9	5/09/01	596	14	60 ft off left bank	47° 56.169'	118° 57.140'
Lower Arrow Lake (reference site)	10	5/07/01	782	7	20 ft off left bank and 0.1 miles upstream of boat ramp	49° 20.379'	117° 52.452'

*GPS reading not taken at time of sampling. Locations from Bortelson et al., 1994.

All positions acquired from GPS (NAD83 datum), except Kettle River site Pool height (above sea level) for Franklin D. Roosevelt Lake was 1231.91 ft on 5/9/01

Appendix A-2. Sediment Sample Descriptions

Site Name	Collection Date	Sample No.	Collection Method	depth (cm)	No. of grabs in composite	Description of sediment
Boundary	5/08/01	198043	0.1 m ² van Veen Grab	10	3	1. yellow-brown sand with some organic material ^a 2. same 3. same
Auxiliary Gage	5/08/01	198042	0.1 m ² van Veen Grab	10	3	1. dark gray-coarse sand ^a 2. same 3. same
Goodeve Creek	5/08/01	198041	0.1 m ² van Veen Grab	10	3	1. dark gray-coarse sand 2. same as above 3. dark gray sand with some pockets of finer yellow sand and fine woody debris ^a
Kettle River	5/08/01	198044	Stainless steel spoon	5	2	1. fine/fluffy material with some fine organic debris 2. light brown sandy sediment (The composite sample had sulfurous odor.) ^a
Castle Rock	5/09/01	198046	0.1 m ² van Veen Grab	10	3	1. the top .5 cm consisted of orange fine sediment, the rest consisted of gray clay with some black streaking ^b 2. same 3. same
Whitestone Creek	5/09/01	198047	0.1 m ² van Veen Grab	10	3	1. the top .5 cm consisted of orange fine sediment, the rest consisted of gray clay with some black streaking ^b 2. same 3. same
Sanpoil River	5/08/01	198045	Stainless steel spoon	10	1	1. silt and mud
Swawilla Basin	5/09/01	198049	0.1 m ² van Veen Grab	10	3	1. the top .5 cm consisted of orange fine sediment, the rest consisted of gray clay with some black streaking ^b 2. same 3. same
Grand Coulee Dam	5/09/01	198050	0.1 m ² van Veen Grab	10	3	1. light brown sand 2. same as 1 3. consisted of 1 cm of orange fine sediment on top of light brown sand ^b
Lower Arrow Lake (reference site)	5/07/01	198040	0.1 m ² van Veen Grab	10	3	1. yellow sand 2. yellow sand with some minor amounts of blue-gray sand 3. consisted of 6 cm of yellow sand on top of blue-gray sand ^a

^a Composite samples homogenized for Microtox® bioassay

^b Individual sediment grabs were full to the top of the 0.1 m² van Veen grab

Appendix B

Data Quality

This page is purposely blank for duplex printing

Appendix B-1. Sample Containers, Preservation, and Holding Times

Analysis	Container	Preservation	Holding Time
Chemistry			
Metals	8 oz glass; TFE-lined lid	4 °C in the dark	6 months
TOC	4 oz glass; TFE-lined lid	4 °C in the dark	28 days (1 year frozen)
% Solids	8 oz glass; TFE-lined lid	4 °C in the dark	7 days
Grain size	8 oz glass; TFE-lined lid	4 °C in the dark	6 months
Bioassays			
<i>Chironomus</i>	1-liter glass; TFE-lined lid	4 °C in the dark	14 days
<i>Hyalella</i>	1-liter glass; TFE-lined lid	4 °C in the dark	14 days
Microtox	1-liter glass; TFE-lined lid	4 °C in the dark	14 days

Appendix B-2. Quality Control Analysis Requirements for Sediment Chemistry

Sample Type	TOC	Grain Size	Metals
Matrix spikes	N/A	N/A	75-125% recovery
Matrix spike duplicates	N/A	N/A	≤ 25% RPD
Laboratory control samples	N/A	N/A	Within method control limits
Laboratory duplicates	≤ 20% RPD	≤ 20% RPD	≤ 20% RPD
Standard reference material	N/A	N/A	Accepted range of certified values

N/A = Not Applicable

RPD = Relative Percent Difference

Appendix B-3. Analysis of Replicate Samples

	Sample No. 198047	Sample No. 198048	% RPD*
Solids (%)	27.4	27.6	0.5
TOC 70 (%)	1.6	1.7	4.3
TOC 104 (%)	1.6	1.6	0
Grain Size (%)			
Gravel	0	0	0
Sand	8.3	7.8	4.4
Silt	37	39.6	4.8
Clay	54.8	52.8	2.6
Metals mg/Kg dw			
As	13	13	0
Cd	11.9	12.4	2.9
Cu	73.7	75.9	2.1
Pb	285	292	1.7
Hg	1.25	1.07	11.0
Zn	952	979	2.0

* Relative Percent Difference

Appendix C
Bioassay Methods

This page is purposely blank for duplex printing

Appendix C-1. Department of Ecology Microtox Porewater Protocol

(Draft Final 8/15/00, Peter Adolphson)

Microtox® 100 Percent Sediment Porewater Toxicity Assessment

Background

Microtox is a rapid method of assessing toxicity in aqueous media by utilizing the bioluminescent properties of the marine bacteria *Vibrio fischeri*. The test method assumes that light emitted by the bacteria can be used as an accurate assessment of the overall biological condition of the bacteria exposed to chemical compounds and mixtures. Light emitted by the bacteria exposed to potentially toxic samples is compared to light emitted to unexposed bacterial controls. Differences in luminescence are therefore deemed an indication of relative toxicity.

EPA has recommended Microtox for TIE/TRE applications (EPA/600/2-88/070) as well as stormwater investigations. Successful applications also include NPDES compliance and sediment evaluations in freshwater, estuarine, and marine applications. Washington State PSEP (Puget Sound Estuarine Protocols) uses both an organic and an aqueous extraction protocol to assess sediment toxicity.

Recognizing that the goal of most sediment toxicity studies is to determine if ecologically/toxicologically significant differences exist between reference and investigative site sediments, four significant differences exist between the PSEP protocol and this revised protocol.

1) Extraction procedures are 100% pore water extraction rather than complex organic and aqueous extractions; 2) No serial dilutions are performed because LC50 calculations are not required to assess sediment toxicity between reference and site sediments; 3) No MOAS (Microtox Osmotic Adjusting Solution) is utilized; and 4) Statistical procedures utilize standard Analysis of Variance (ANOVA) or t-test procedures.

Microtox Test Procedure

Porewater Extraction and Adjustment

The general Microtox procedure involves centrifugation of 500ml of both reference and test sediments at approximately 4500G in for 30 minutes resulting in approximately 50 ml of pore water. Approximately 25mls of pore water is then pipetted into a clean glass container. The remaining porewater volume is set aside if needed for reducing salinity should the initial salinity adjustments steps outlined below result in the sample exceeding 22ppt.

The sample is then adjusted for salinity, dissolved oxygen, and pH *in the following order*.

1) Salinity is adjusted to 20 ± 2 ppt using commercially available dry bulk marine aquarium reef salts (e.g. Forty Fathoms Reef®). [Note: The salinity adjustment step is omitted for Marine and estuarine sediments whose porewater exceeds 20ppt salinity.]

- 2) The dissolved oxygen (DO) is then adjusted by gentle aeration or agitation until it is between 50-100% saturation.
- 3) The pH of the salinity and DO adjusted reference and test sediment pore water should not differ from each other by more than 0.4 pH units. The pH is adjusted to 7.9-8.2 (if necessary) using a micropipette and a dilute solution (0.5 N) NaOH or HCl. Total volume of NaOH and/or HCl should be recorded. Final concentration [compared with 100% porewater extracted] can then be calculated using these data. Final dilution should not be reduced below 90% of the pore water extract. *[Note: The control solution is prepared by using deionized or distilled water and adjusting salinity, DO, and pH as described above.]*

Preparation of Bacterial Suspension and Bioassay Test Setup

A vial of freeze-dried bacteria is rehydrated with 1.0 ml of Microtox® Reconstitution solution and allowed to equilibrate for 30-90 minutes in the 4-degree Microtox Analyzer well.

[NOTE: Mixing of the reconstituted bacteria is essential. Mix the reconstituted solution with a 1 ml pipette a minimum of 20 times by pipetting. First pipette the solution from the bottom of the cuvette and deposit the pipetted solution on the surface of the liquid remaining in the cuvette. Then pipette 1 ml of solution from the bottom of the cuvette and slowly pipette the liquid into the bottom of the cuvette.]

One (1.0) ml of control solution is then placed in each of 5 test cuvettes and placed into the 15-degree incubation chambers. This procedure is followed for the laboratory control solution, reference sediment porewater samples, and test sediment porewater samples, for up to 4 test sediments/batch (5 pseudo-replicates per site).

In each of the test, reference, and control sample cuvettes, 10 uL of rehydrated bacteria suspension is added at 30 second intervals, immediately mixed using a 1ml pipette and allowed to incubate for 5 minutes. Used pipette tips are replaced with clean tips after each series of 5 pseudo-replicates (ref, control, and each test series ex: A1-A5).

[NOTE: Extreme care must be used when pipetting these low volumes as slight residual amounts or presence of air bubbles in the pipette may cause variation due to error by as much as 100%.]

Data collection

At the initial (I_0) 5 minute mark, the first control vial is placed into the read chamber to “set” the instrument. At 30-second intervals each cuvette (inclusive of A1) is placed into the read chamber for the initial reading (I_0). After 5 additional minutes a second reading (I_5) is obtained following the above procedure. A 15-minute (I_{15}) is obtained in an additional 10 minutes.

Data analysis

Statistical calculations are performed using a standard t-test by comparing reference with test site data. No gamma correction is required. Statistically significant differences with $\alpha = 0.05$ and the following relative differences are indications of test failure.

Control output should exceed 80 percent at the 5-minute reading and 65% at the 15-minute reading.

Appendix C-2. QC Requirements for Sediment Bioassay

(Modified from Table B-1 in Ecology, 1995b)

Sediment Toxicity Test Conditions

Test Species	Frequency of Water Quality Monitoring		Control Limits		Control Samples		Test Acceptability	
	Hardness, alkalinity, conductivity, pH, ammonia	Temp., D.O.	Temp (C)	Dissolved Oxygen (% sat.)	Negative Control	Positive Control		Reference Sediment
Amphipod <i>Hyalella azteca</i>	Beginning and end of test	Daily	23±1	>40%	Clean sediment*	Reference toxicant Cadmium	Yes	Mean survival in control sediment ≥80 percent. Mean weight of surviving controls ≥ 0.1 mg
Midge <i>Chironomus tentans</i>	Beginning and end of test	Daily	23±1	>40%	Clean sediment*	Reference toxicant Cadmium	Yes	Mean control survival ≥ 70% and minimum weight of survivors 0.6 mg
Microtox® (100% pore water) <i>Vibrio fischeri</i>	NA	NA	15	NA	Control solution*	Reference toxicant Cadmium	Yes	Control output > 80% @ 5 minutes and >65% @ 15 minutes

* Negative control sediments provided by the laboratory

Appendix C-3. Reporting Requirements for Sediment Bioassays (from EPA, 2000)

16.4 Reporting

16.4.1 The record of the results of an acceptable sediment test should include the following information either directly or by referencing available documents:

16.4.1.1 Name of test and investigator(s), name and location of laboratory, and dates of start and end of test.

16.4.1.2 Source of control or test sediment, and method for collection, handling, shipping, storage and disposal of sediment.

16.4.1.3 Source of test material, lot number if applicable, composition (identities and concentrations of major ingredients and impurities if known), known chemical and physical properties, and the identity and concentration(s) of any solvent used.

16.4.1.4 Source and characteristics of overlying water, description of any pretreatment, and results of any demonstration of the ability of an organism to survive or grow in the water.

16.4.1.5 Source, history, and age of test organisms; source, history, and age of brood stock, culture procedures; and source and date of collection of the test organisms, scientific name, name of person who identified the organisms and the taxonomic key used, age or life stage, means and ranges of weight or length, observed diseases or unusual appearance, treatments used, and holding procedures.

16.4.1.6 Source and composition of food; concentrations of test material and other contaminants; procedure used to prepare food; and feeding methods, frequency and ration.

16.4.1.7 Description of the experimental design and test chambers, the depth and volume of sediment and overlying water in the chambers, lighting, number of test chambers and number of test organisms/treatment, date and time test starts and ends, temperature measurements, dissolved oxygen concentration ($\mu\text{g/L}$) and any aeration used before starting a test and during the conduct of a test.

16.4.1.8 Methods used for physical and chemical characterization of sediment.

16.4.1.9 Definition(s) of the effects used to calculate LC50 or EC50s, biological endpoints for tests, and a summary of general observations of other effects.

16.4.1.10 A table of the biological data for each test chamber for each treatment, including the control(s), in sufficient detail to allow independent statistical analysis.

16.4.1.11 Methods used for statistical analyses of data.

16.4.1.12 Summary of general observations on other effects or symptoms.

16.4.1.13 Anything unusual about the test, any deviation from these procedures, and any other relevant information.

16.4.2 Published reports should contain enough information to clearly identify the methodology used and the quality of the results.

Appendix D

Data Tables

This page is purposely blank for duplex printing

Appendix D-1. Comparison of Conventional and Metal Concentration (Total Recoverable and Total) Data from Lake Roosevelt Sediment Studies

	Lower Arrow Lake	Boundary	Auxiliary Gage	Goodeve Creek	Kettle River	Castle Rock	Whitestone Creek	Sanpoil River	Swawilla Basin	Grand Coulee Dam
River Mile	782	745	743	738	707	645	621	616	605	596
TOC (%)										
Ecology 2001 ^a	0.2	0.5	.02 U	.5*	1.1	1.1	1.6**	2.2	1.7	0.3
USGS 1992 ^b	0.2	0.1	0.1	0.2	1.3	--	1.6	2.3	1.6	1.3
Ecology 1989 ^c	--	--	--	--	--	0.81	--	1.15	1.04	--
Ecology 1986 ^d	1.6	--	7.1	3.7	2.4	2.7	--	3.3	1.5	--
Percent Fines (≤ 62 μm)										
Ecology 2001 ^a	0.0	0.0	0.0	0.0	12.8	95.7	91.6**	88.9	95.9	7.2
USGS 1992 ^b	10.0	1.0	0.0	5.0	44.0	--	99.0	85.0	99	95
Ecology 1989 ^c	--	--	--	--	--	95.8	--	96.4	84.2	--
Ecology 1986 ^d	33.12	--	0.6	1.2	76.3	89.3	--	86.26	79.98	--
Arsenic (mg/Kg dw)										
Ecology 2001 ^a / Total Recov	2.0 U*	6.6	5.0	20.0	2.0 U	8.3	13**	3.5	11	9.2
USGS 1992 ^b / Total	<2	34.0	35.0	32.0	<4.0	--	16	7.3	21	18.0
USGS 1992 ^b / Total Recov	<0.9	27	27	29	--	--	11	3.4	--	8.1
Ecology 1989 ^c / Total	--	--	--	--	--	14.3	--	7.9	13.8	--
Ecology 1989 ^c / Total Recov	--	--	--	--	--	16.7	--	6.2	12.6	--
Ecology 1986 ^d / Total Recov	2.75	--	27.9	19.7	2.8	10.4	--	4.1	8	--
Cadmium (mg/Kg dw)										
Ecology 2001 ^a / Total Recov	0.46*	6.7	18.0	16.2	0.96	7.09	12.2**	1.9	12.4	1.8
USGS 1992 ^b / Total	<0.50	1.0	1.1	0.9	0.50	--	9.8	1.4	9.9	6.2
USGS 1992 ^b / Total Recov	<0.2	<2.8	<3.8	<2.4	--	--	9.4	<1.3	--	5.6
Ecology 1989 ^c / Total	--	--	--	--	--	6.4	--	2.2	7.2	--
Ecology 1989 ^c / Total Recov	--	--	--	--	--	5.7	--	2.7	6.9	--
Ecology 1986 ^d / Total Recov	0.6	--	0.6	2.1	0.3	5.5	--	0.7	5.2	--

	Lower Arrow Lake	Boundary	Auxiliary Gage	Goodeve Creek	Kettle River	Castle Rock	Whitestone Creek	Sanpoil River	Swawilla Basin	Grand Coulee Dam
River Mile	782	745	743	738	707	645	621	616	605	596
Copper (mg/Kg dw)										
Ecology 2001 ^a / Total Recov	3.6*	494	2210	2210	15.6	66.4	74.8**	19.5	73.2	10.7
USGS 1992 ^b / Total	9.0	2700	3000	2900	23.0	--	76.0	36	73	61.0
USGS 1992 ^b / Total Recov	6.9	2500	2800	2800	--	--	66	25	--	51
Ecology 1989 ^c / Total	--	--	--	--	--	62.0	--	39	57.0	--
Ecology 1989 ^c / Total Recov	--	--	--	--	--	61	--	38	49	--
Ecology 1986 ^d / Total Recov	15	--	4870	3390	29	67.0	--	34	65.0	--
Lead mg/Kg dw										
Ecology 2001 ^a / Total Recov	11.5*	182	324	344	4.6	173	289**	19	295	17
USGS 1992 ^b / Total	17.0	280	310	310	20.0	--	290	23.0	310	190
USGS 1992 ^b / Total Recov	<4.8	300	260	290	--	--	280	15	--	170
Ecology 1989 ^c / Total	--	--	--	--	--	251	--	43	195	--
Ecology 1989 ^c / Total Recov	--	--	--	--	--	236	--	32	165	--
Ecology 1986 ^d / Total Recov	19	--	365	389	5	349	--	10	206	--
Mercury mg/Kg dw										
Ecology 2001 ^a / Total Recov	0.0004 U	0.102	0.016	0.0769	0.0007 U	0.678	1.16**	0.029	1.25	0.029
USGS 1992 ^b / Total	<0.05	0.2	0.3	0.1	<0.05	--	1.40	0.06	1.6	0.800
USGS 1992 ^b / Total Recov	0.006	0.1	0.06	0.05	--	--	0.4	0.03	--	0.2
Ecology 1989 ^c / Total	--	--	--	--	--	1.05	--	0.11	0.78	--
Ecology 1989 ^c / Total Recov	--	--	--	--	--	0.56	--	0.05	0.13	--
Ecology 1986 ^d / Total Recov	0.015	--	0.01	0.08	0.02	1.0	--	0.04	1.00	--
Zinc mg/Kg dw										
Ecology 2001 ^a / Total Recov	26.9*	3730	16100	12200	33.9	471	966**	69.9	1040	86.1
USGS 1992 ^b / Total	47	13000	16000	17000	70	--	1000	160	1100	730
USGS 1992 ^b / Total Recov	29	12000	14000	16000	--	--	950	120	--	610
Ecology 1989 ^c / Total	--	--	--	--	--	539	--	263	702	--
Ecology 1989 ^c / Total Recov	--	--	--	--	--	188	--	142	242	--
Ecology 1986 ^d / Total Recov	48	--	26840	22920	60	954	--	100	757	--

^a Present study; top 10 cm of sediments collected and analyzed in 2001

^b Bortelson et al., 1994; top 2 cm of sediments collected and analyzed in 1992

^c Johnson, 1991; top 2 cm of sediments collected and analyzed in 1989

^d Johnson, 1988; top 2 cm of sediments collected and analyzed in 1986

* mean of laboratory duplicate analysis

** mean of replicate sample analysis

U = The analyte was not detected at or above the reported value

-- = no data