A Department of Ecology Report

WASHINGTON STATE DEPARTMENT OF ECOLOGY

Metals Concentrations in Spokane River Sediments Collected with USGS in 1998

Abstract

Bioassays and metals analyses were conducted on sediment samples from 14 sites on the Spokane River, obtained from a USGS study on downstream dispersion of trace elements from Lake Coeur d'Alene. Results from the bioassays were unusable due to errors made by the contract laboratory. Metals results were consistent with historical data in showing elevated to extremely high concentrations of zinc, lead, and cadmium in sediment samples from most parts of the river.

Background

As part of the EPA Remedial Investigation/Feasibility Study of the Spokane River Basin, the U. S. Geological Survey assessed the downstream dispersion of sediment-associated trace elements from Lake Coeur d'Alene (USGS, 1998). This work, conducted in October and December 1998 by Dr. Arthur Horowitz, included collection of sediment samples at over 80 sites in the river.

At 14 of these sites, USGS provided samples to the Department of Ecology for toxicity testing (Figure 1). Ecology also analyzed the samples for EPA priority pollutant metals, grain size, and total organic carbon.

Two sediment bioassays were conducted: a 14-day test of survival and growth with the amphipod *Hyalella azteca*, and a Microtox solid phase test using the bacterium *Vibrio fisheri*. *Hyalella* and Microtox have been used to develop freshwater sediment quality criteria for Washington State (Cubbage et al., 1997).

Unfortunately, the laboratory conducting these tests made a series of errors that rendered the *Hyalella* data unusable (Kadlec, 1999). Three of the 14 Microtox tests were also rejected. In light of the extensive problems identified with this laboratory's work, little confidence can be placed in the Microtox results that passed review. As a result, this memorandum is limited to reporting the results from the metals analysis.

by Art Johnson
August 1999

Waterbody Nos. WA-54-1010; 54-1020; 54-2000; 54-9040; 55-1010; 56-1010; 57-1010

Ecology Report #99-330

Historical Data

Between 1988 and 1995, metals have been analyzed in fifteen sediment samples from the Spokane River. Sediment bioassays have been conducted for four locations. The historical data are summarized in Table 1.

With the exception of one site above Nine-Mile Dam, results have consistently shown substantial elevations in zinc, lead, and cadmium. These same metals occur in high concentrations in water and fish tissue samples (Pelletier, 1994; Hopkins and Johnson, 1997; Johnson, Serdar, and Davis, 1994).

Sediment bioassays have been conducted at three locations in the Spokane River Arm of Lake Roosevelt, one location in Long Lake, and one location above Upriver Dam. The tests included the 10-day *Hyalella* amphipod bioassay, which measures survival, and the Microtox test, which measures reduction in the light output of *V. fisheri*. There was evidence of toxicity in one or both tests for the Spokane Arm and Upriver sediments, but not in the Long Lake sample.

1998 Samples

The locations of the USGS samples provided to Ecology in 1998 are shown in Figure 1. Main stem samples were obtained from three sites each in the Spokane Arm and Long Lake, one site in Nine-Mile reservoir, and two sites above Upriver Dam. One sample was taken from each of the three largest tributaries, Little Spokane River, Deep Creek, and Hangman Creek. A sample was also obtained from Liberty Lake, considered by USGS as possibly representing local background concentrations for metals. Appendix A contains sampling site descriptions and coordinates.

The samples were taken from approximately the top 10-cm sediment layer, except for the two upper river samples which were the top 2 cm. Deep and Hangman ceeks were sampled by Ecology. All remaining samples were collected by USGS personnel. The samples provided to Ecology were from additional separate grabs rather than splits of the samples USGS analyzed.

Sampling Methods

Most sediment samples were obtained using stainless steel Ekman or Shipek grabs and consisted of composites from one to five individual grabs. The upper river and Deep Creek samples were collected by hand with stainless steel or plastic scoops. The Hangman Creek sample was taken with a stainless steel pipe dredge.

After draining off overlying water, sediments were removed from the grab samplers with stainless steel scoops, placed in a stainless steel bowls, and homogenized by stirring. Subsamples of the homogenate were placed in glass jars with teflon lid liners, cleaned to EPA QA/QC specifications (EPA, 1990) or Whirl-Pak bags for grain size.

Scoops and bowls used to manipulate the sediments were cleaned by washing with Liquinox detergent, followed by sequential rinses with tap water, dilute nitric acid, deionized water, and pesticide-grade acetone. The equipment was then air-dried and wrapped in aluminum foil. The same procedures were used to pre-clean the pipe dredge before going into the field. Further details on USGS sediment sampling procedures can be found in USGS (1998).

The sample jars were placed in bubble-wrap and polyethylene bags, stored on ice, and transported to the Ecology/EPA Manchester Laboratory within three days of collection. Chain-of-custody was maintained.

Laboratory Methods

The sample were analyzed by Manchester, except grain size was analyzed by Rosa Environmental & Geotechnical Laboratory, Seattle. Methods of analysis are listed in Table 2.

USGS used a total digestion (aqua regia/HF/$HClO_4$) to analyze their 1998 Spokane River sediment samples, while Ecology used a strong acid, partial digestion (HNO_3/HCl). In spite of the different digestions, results from the two methods agreed closely (Appendix B). The historical data on metals in Spokane River sediments and most other statewide sediment data are from a strong acid digestion.

Data Quality

Manchester prepared written quality assurance reviews of the chemical data for this project. The review included an assessment of sample condition on receipt at the laboratory, compliance with holding times, instrument calibration, procedural blanks, laboratory control samples, standard reference material, matrix spike recoveries, and duplicate sample analyses. The reviews and complete chemical data are on file at EAP.

The only significant data quality issue identified was low matrix spike recoveries for antimony and thallium. Antimony recovery from a laboratory control sample was also low. Therefor, data for these two metals are not reported.

Results and Discussion

Table 3 has the grain size and total organic carbon (TOC) data. The samples obtained from the free flowing reaches of the river and its tributaries were predominantly sand. Long Lake and Spokane Arm sediments were mostly silt and clay. Levels of TOC were generally low (<3%), except in one upper river sample (7.8%) and in Liberty Lake (8.3%).

The metals data are in Table 4. All locations sampled on the main stem had elevated to extremely high concentrations of zinc and most areas had elevated to extremely high concentrations of lead and cadmium. Zinc, lead, and cadmium concentrations in tributary sediments were one to two orders of magnitude lower than in the main stem.

Two locations, one above Upriver Dam and one in lower Long Lake, also showed some elevated mercury. Concentrations of other metals in the main stem were unremarkable and no metals elevations were seen in the tributaries.

By way of comparison, Table 5 summarizes data on metals concentrations typical of Spokane Basin soils, and soils and freshwater sediments statewide. Again, the zinc, lead, and cadmium concentrations in Spokane River main stem sediments are seen to be elevated by one to two orders of magnitude.

Figure 2 shows the distribution of zinc, lead, and cadmium in Ecology's main stem samples. The highest concentrations occurred in the sediments of the upper river and in the lower parts of Long Lake. The relatively low concentrations behind Nine-Mile Dam may reflect dilution by Deep Creek and Hangman Creek sediments. Metals concentrations tend to increase going from the upper to lower portions of Long Lake and the Spokane Arm, a finding supported by the more extensive USGS data.

Table 6 lists three sets of guidelines on metals concentrations in freshwater sediments for protection of aquatic life. The Ontario and Canadian effects levels were developed from simultaneously collected data on benthic invertebrate communities and metals concentrations. The Ecology apparent effects thresholds were calculated from an analysis of bioassay and metals data in the department's FSEDQUAL database on freshwater sediments statewide.

Spokane sediment samples exceeding one or more of these guidelines are indicated with asterisks in Table 5. For the main stem, all samples exceed for zinc, 6 of 10 exceed for cadmium, and 5 of 10 exceed for lead. The elevations in mercury previously noted for two sites on the main stem are still well within the guidelines. No tributary samples approached the guidelines.

Conclusions and Recommendations

Results of this survey are consistent with historical data in showing that the contamination of Spokane River sediments by zinc, lead, and cadmium there is widespread and significant. The failure to directly assess the toxicity of the sediments constitutes a major gap in understanding the metals contamination issue for the river and should be addressed by re-sampling and re-testing.

Acknowledgements

The generous assistance of Art Horowitz and his crew in collecting and providing sediment samples to Ecology is very much appreciated. John Summers of EAP was on hand as Ecology's representative for much of the field work.

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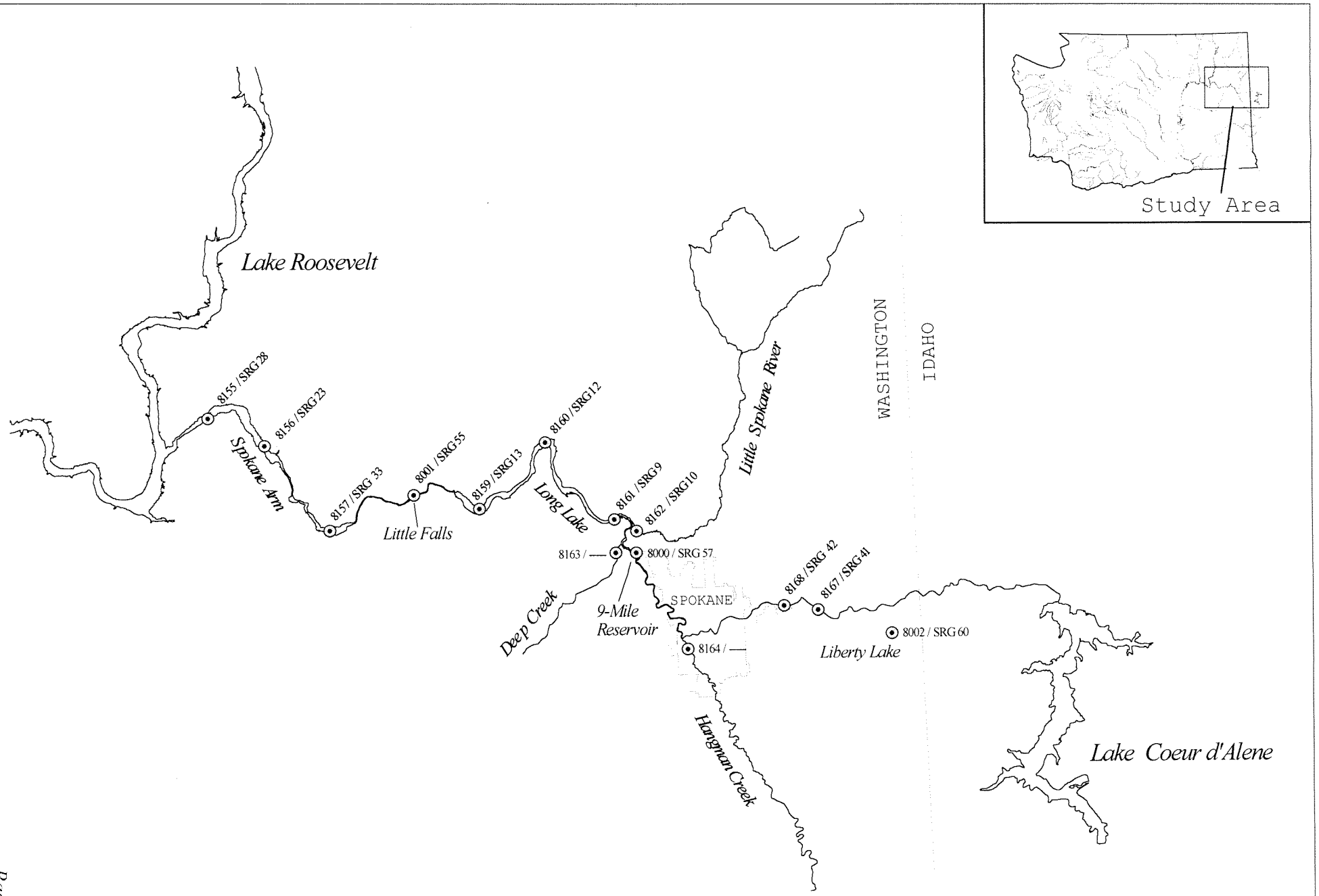

Figure 1. Location of Ecology/USGS Sediment Samples in the Spokane River, October & December, 1998

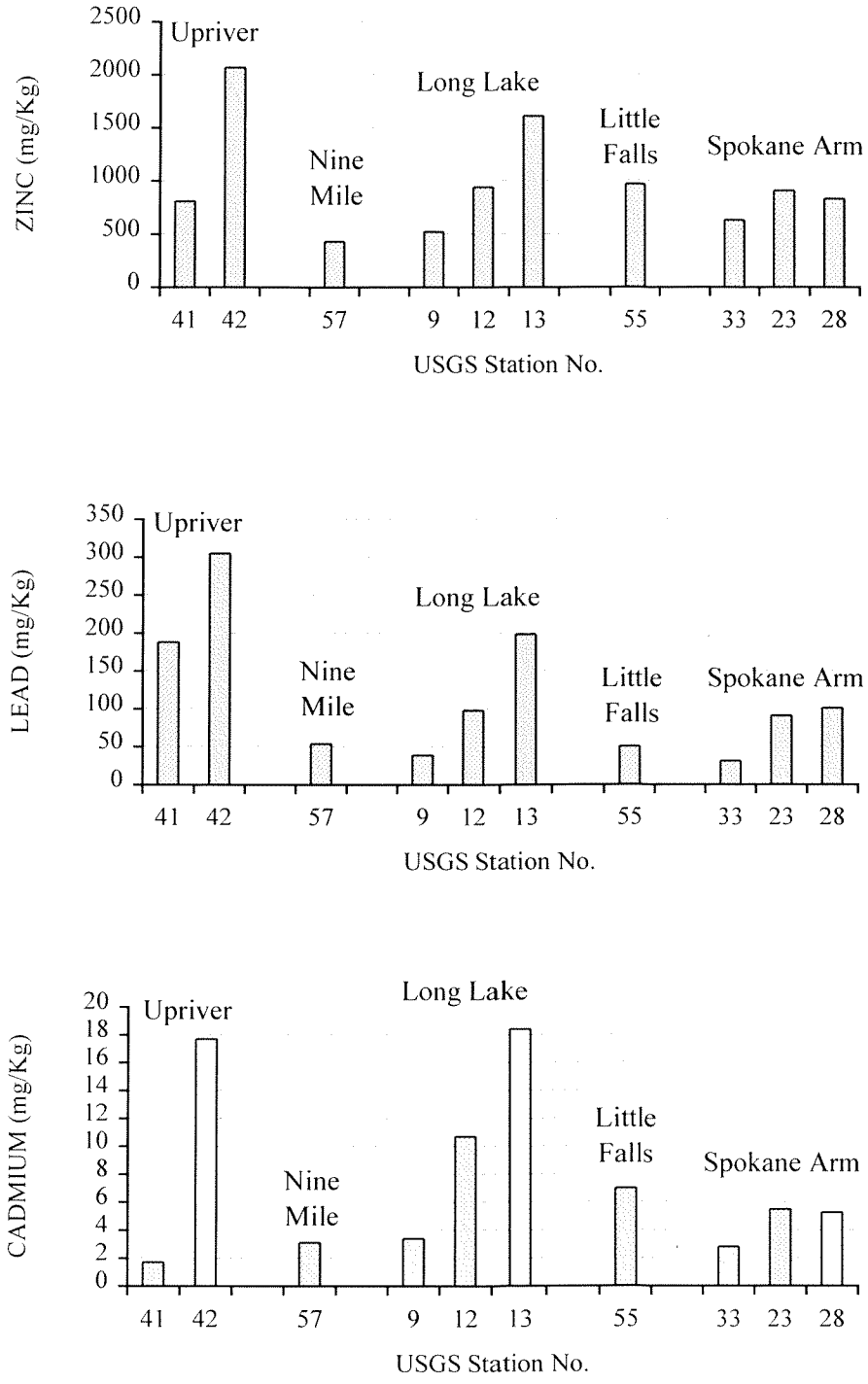

Figure 2. Zinc, Lead, and Cadmium Concentrations in Ecology 1998 Spokane River Sediment Samples (main stem)

Table 1. Summary of Historical Data on Spokane River Sediments.

Location	N =	Metals Concentrations (mg/Kg, dry)			Toxic in Bioassay?		Ref.
		Zinc	Lead	Cadmium	Hyalella	Microtox	
Above Idaho Border	1	1140	366	6.8	- -	- -	(1)
Above Upriver Dam	1	3990	605	33	- -	- -	(1)
	1	4050	542	40	yes	yes	(2)
Above Nine-Mile Dam	1	343	22	1.8	- -	- -	(1)
Long Lake	1	1425	154	16	- -	- -	(1)
	1	520	42	3.9	no	no	(2)
	2	1270 - 1660	76 - 165	14 - 19	- -	- -	(4)
Spokane River Arm (FDR Lake)	1	960	81	7.4	- -	- -	(1)
	1	1180	81	9.1	no	yes	(2)
	1	671	125	11	- -	- -	(3)
	1	1540	128	5.6	- -	- -	(5)
	3	980 - 1800	80 - 150	4.9 - 11	no	yes	(6)

(1) Johnson et al. (1994)
(2) Batts (1995)
(3) Johnson (1991)
(4) Serdar et al. (1994))
(5) Johnson et al. (1988)
(6) Bortleson et al. (1994)

Table 2. Analyical Methods for Ecology 1998 Spokane River Sediment Samples.

Analysis	Method	Number
As	HNO_3,HCl digestion/GFAA	EPA 206.2
Pb	HNO_3,HCl digestion/GFAA	EPA 239.2
Se	HNO_3,HCl digestion/GFAA	EPA 7740
Tl	HNO_3,HCl digestion/GFAA	EPA 279.2
Zn,Pb,Cu,Cr,Ni,Cd,Be,Ag,Sb	HNO_3,HCl digestion/ICP	EPA 200.7
Hg	CVAA	EPA 245.5
Total Organic Carbon	Persulfate/UV Ox./FID	PSEP* Method
Grain Size	Sieve & pipette	PSEP Method

* Puget Sound Estuary Program

Table 3. Grain Size and Total Organic Carbon in Sediment Samples (percent).

Reach/Tributary	Ecology Sample Number	USGS Station Number	Gravel	Sand	Silt	Clay	TOC (%)
Upriver	8167	41	0.9	93.5	5.5	0.2	0.28
"	8168	42	1.4	41.7	49.4	7.4	7.8
9-Mile	8000	57	0.0	43.7	48.3	8.0	1.7
Long Lake	8161	9	0.0	86.2	12.0	1.8	1.3
"	8160	12	0.0	1.5	73.5	25.1	2.3
"	8159	13	0.0	2.5	54.0	43.5	2.6
Little Falls	8001	55	0.2	71.1	26.6	2.2	1.1
Spokane Arm	8157	33	0.0	52.6	42.4	5.0	1.6
"	8156	23	0.1	19.1	56.4	24.3	2.2
"	8155	28	2.0	37.6	27.6	32.8	1.6
Hangman Creek	8164	- -	8.8	83.3	7.1	0.8	0.53
Deep Creek	8163	- -	4.5	93.7	1.7	0.1	0.06
Little Spokane R.	8162	10	0.0	49.7	41.7	8.6	3.8
Liberty Lake	8002	60	0.0	21.9	69.7	8.4	8.3

Table 4. Metals Concentrations in Spokane River Sediment Samples (mg/Kg, dry wt.; ppm).

Reach/Tributary	Ecology Sample Number	USGS Station Number	Zn	Pb	Cu	Cr	Ni	Cd	As	Be	Ag	Se	Hg
Upriver	8167	41	807*	188*	8.7	11.5	9.6	1.7	7.0	0.32	<0.5	<0.3	0.012
"	8168	42	2068**	305**	39.1	20.4	15.9	17.7**	9.6	0.64	0.76	0.32	0.25
9-Mile	8000	57	427*	54	20.3	14.4	11.8	3.1	7.6	0.63	<0.3	<0.3	0.026
Long Lake	8161	9	519*	38.9	14.2	12.7	10.7	3.4	6.8	0.45	<0.5	<0.3	0.034
"	8160	12	939**	97.8*	31.6	24.2	18.4	10.7**	7.2	1.0	0.52	<0.3	0.11
"	8159	13	1610**	198*	42.0	28.5	19.9	18.4**	8.6	1.2	1.1	0.31	0.25
Little Falls	8001	55	969**	50.2	17.6	13.9	12.0	7.0*	9.3	0.51	<0.3	0.54	0.030
Spokane Arm	8157	33	630*	30.8	15.3	16.1	13.2	2.8	5.9	0.53	<0.5	<0.3	0.032
"	8156	23	906**	90.8	33.0	25.3	20.7	5.5*	10.1	1.1	<0.5	0.36	0.081
"	8155	28	832**	101*	29.6	23.7	20.9	5.3*	8.6	1.1	<0.5	<0.3	0.096
Hangman Creek	8164	- -	44.6	8.0	12.0	10.3	9.0	<0.5	6.0	0.36	<0.5	<0.3	0.0059
Deep Creek	8163	- -	39.3	8.6	8.9	10.8	9.5	0.53	7.3	0.34	<0.5	<0.3	<0.005
Little Spokane R.	8162	10	60.3	10.0	9.5	15.6	8.7	<0.5	4.4	0.74	<0.5	0.60	0.027
Liberty Lake	8002	60	85.4	45.9	27.4	21.6	15.4	<0.4	4.4	0.80	<0.3	0.56	0.010

* Exceeds lowest sediment quality guideline (see Table 6)
** Exceeds highest sediment quality guideline (see Table 6)

Table 5. Typical Metals Concentrations in Washington Soils and Freshwater Sediments.

Metal	Terrestrial Soils (90th percentile)[1] Spokane Basin n = 79	Terrestrial Soils (90th percentile)[1] Statewide n = 490	Freshwater Sediments (median) Background[2] n = 25 - 42
Zinc	66	86	84
Copper	22	36	24
Chromium	18	42	58
Nickel	16	38	- -
Lead	15	17	41
Arsenic	9.0	7.0	3.4
Cadmium	1.0	1.0	0.5
Beryllium	0.8	2	- -
Mercury	0.02	0.07	- -
Silver	- -	- -	- -

[1]San Juan (1994)

[2]PTI (1989)

Table 6. Guidelines on Metals in Freshwater Sediments (mg/Kg, dry wt.; ppm).

Metal	Ontario[1] Severe Effects Level	Canada[2] Probably Effects Level	Ecology[3] Apparent Effects Threshold
Zinc	820	310	520
Lead	250	91	260
Copper	110	200	840
Chromium	110	90	280
Nickel	75	36	46
Cadmium	10	3.5	7.6
Arsenic	33	17	40
Beryllium	- -	- -	- -
Silver	- -	- -	4.5
Selenium	- -	- -	- -
Mercury	2.0	0.49	0.56

[1]Persaud et al. (1993)

[2]Environment Canada (1994)

[3]Cubbage et al. (1997)

Appendix A. Location of Ecology/USGS 1998 Spokane River Sediment Samples.

Ecology Sample Number	USGS Station Number	Date	Description	Approx. River Mile	Depth (meters)	Latitude	Longitude
Upriver Reach							
43-8167	SRG-41	10/22/98	Between Trent Rd.& Sullivan Park	86.5	1.0	47 41 23	117 16 00
43-8168	SRG-42	10/22/98	Between Myrtle Pt. & Felts Field	83.5	1.3	47 41 38	117 18 02
Nine-Mile Reservoir							
49-8000	SRG-57	12/01/98	Approx. one mile above Deep Cr.	60.0	1.5	47 45 24.5	117 31 37
Long Lake							
43-8161	SRG-9	10/19/98	Upper lake, bw Little Spokane R.	54.5	3.7	47 47 55	117 34 07
43-8160	SRG-12	10/19/98	Middle lake @ Tum Tum		8.5	47 53 25	117 39 53
43-8159	SRG-13	10/19/98	Lower lake, above Long Lake Dam	36.7	14.9	47 48 52	117 47 47
Little Falls Reservoir							
49-8001	SRG-55	12/01/98	Above Little Falls Dam		2.0	47 49 28	117 54 48.5
Spokane River Arm of Lake Roosevelt							
43-8157	SRG-33	10/21/98	Upper Arm, off Mill Canyon	21	3.5	47 47 38	118 03 32
43-8156	SRG-23	10/20/98	Middle Arm @ Porcupine Bay	11.5	35.9	47 54 09	118 09 42
43-8155	SRG-28	10/20/98	Lower Arm near Five-Mile Point	5.0	23.1	47 55 55	118 15 43
Tributaries							
43-8164	- -	10/18/98	Hangman Cr ab. Riverside Avenue	- -	0.5	47 39 21	117 27 13
43-8163	- -	10/18/98	Deep Creek @ mouth	- -	0.3	47 45 47	117 32 55
43-8162	SRG-10	10/19/98	Little Spokane R. 0.2 mi ab. mouth	- -	2.4	47 47 37	117 31 54
Liberty Lake (USGS background site)							
49-8002	SRG-60	12/01/98	North end	- -	8.0	47 38 07	117 04 07

Appendix B. Comparison of Ecology (43-) and USGS Analytical Results for Sediments from Selected Sites in the Spokane River Basin (prepared by A. Horowitz, USGS).

	Hangman Creek		Little Spokane		Liberty Lake		Upriver Sites				Nine-Mile		Long Lake	
	43-8164	SRH-06	43-8162	SRG-10	49-8002	SRG-60	43-8167	SRG-41	43-8168	SRG-42	49-8000	SRG-57	43-8161	SRG-9
Antimony	<4	1.3	<4	0.8	<3	1.0	<4	2.2	<4	6.5	<3	1.1	<4	1.3
Beryllium	0.36	1.6	0.74	2.0	0.8	1.4	0.32	2.0	0.64	1.6	0.63	1.7	0.45	1.8
Cadmium	<0.5	<0.1	<0.5	<0.1	<0.4	0.4	1.7	2.2	17.7	23	3.1	3.3	3.4	2.7
Chromium	10.3	37	15.6	36	21.6	40	11.5	33	20.4	43	14.4	14	12.6	40
Copper	12.0	13	9.53	8	27.4	33	8.7	9	39.1	45	20.3	24	14.2	13
Lead	8.0	33	10.0	16	45.9	54	188	350	305	360	54.0	63	38.9	43
Nickel	9.0	13	8.7	13	15.4	25	9.6	14	15.9	22	11.8	21	10.7	16
Silver	<0.5	<0.5	<0.5	<0.5	<0.3	<0.5	<0.5	<0.5	0.76	0.7	<0.3	<0.5	<0.5	<0.5
Zinc	44.6	64	60.3	66	85.4	120	807	900	2068	2300	427	500	519	530
Arsenic	5.99	8.3	4.38	5.7	4.41	5.6	7.04	9.9	9.56	12	7.61	11	6.77	7.9
Selenium	<0.3	0.2	0.60	0.8	0.56	0.5	<0.3	0.1	0.32	0.5	<0.3	0.2	<0.3	<0.1
Thallium	<0.3	<50	<0.3	<50	<0.3	<50	<0.3	<50	<0.3	<50	<0.3	<50	<0.3	<50
Mercury	0.0059	0.01	0.027	0.01	0.010	0.07	0.0123	0.02	0.247	0.20	0.026	0.04	0.034	0.03
TOC	0.53	1.4	3.8	2.3	8.3	8.0	0.28	0.3	7.8	7.2	1.7	1.9	1.3	0.9
%<63-µm	7.9	16	50.3	19	78.1	16	5.7	1	56.8	32	56.3	62	13.8	6

	Long Lake				Little Falls		Spokane River Arm					
	43-8160	SRG-12	43-8159	SRG-13	49-8001	SRG-55	43-8156	SRG-23	43-8155	SRG-28	43-8157	SRG-33
Antimony	<4	1.6	<4	2.4	<3	1.4	<4	2	<4	2.1	<4	1.8
Beryllium	1.00	2.0	1.16	2.2	0.51	1.7	1.10	2.1	1.06	2.2	0.53	1.8
Cadmium	10.7	8.2	18.5	11	7.0	5.6	5.5	4.9	5.3	4.3	2.8	3.3
Chromium	24.2	52	28.7	57	13.9	37	25.3	53	23.7	50	16.1	40
Copper	31.6	34	42.4	43	17.6	16	33.0	35	29.6	26	15.3	20
Lead	97.8	110	201	190	50.2	50	90.8	100	101	80	30.8	39
Nickel	18.4	28	20	30	12.0	17	20.7	28	20.9	23	13.2	19
Silver	0.52	<0.5	1.1	0.5	<0.3	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
Zinc	939	840	1635	1200	969	860	906	980	832	780	630	690
Arsenic	7.23	13	8.88	15	9.34	8.4	10.1	20	8.62	15	5.86	7.1
Selenium	<0.3	0.2	0.3	0.4	0.54	0.2	0.36	0.3	<0.3	0.2	<0.3	0.2
Thallium	<0.3	<50	<0.3	<50	<0.3	<50	<0.3	<50	<0.3	<50	<0.3	<50
Mercury	0.112	0.10	0.25	0.15	0.030	0.04	0.081	0.06	0.096	0.04	0.032	0.07
TOC	2.3	2.20	2.6	2.8	1.1	1.2	2.2	1.9	1.6	1.4	1.6	1.6
%<63-µm	98.6	70	97.0	49	28.8	13	80.7	27	60.4	9	47.4	47