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DEPARTMENT OF ECOLOGY

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TO: Carl Nuechterlein and Pat Hallinan

THROUGH: Larry Goldstein

FROM David Batts and Art Johnson

SUBJECT: Bioassays of Spokane River Sediments (Final)

Summary

Laboratory bioassays of sediment collected at three sites in the Spokane River during August 1994 showed toxicity in samples from behind Upriver Dam and, to a lesser extent, the Spokane Arm Results on a Long Lake sample indicated little or no toxicity This sample, however, had lower metals concentrations than those previously collected in Long Lake Chemicals most likely responsible for the adverse effects were zinc, cadmium, and lead in the Upriver Dam sample, and zinc and lead in the Spokane Arm sample

Background

As part of the Toxics Investigations Section's intensive sampling program in the Spokane River during 1994, toxicity tests and chemical analyses were conducted on sediments collected from one site each in the Spokane Arm, Long Lake, and above Upriver Dam. These areas were selected to encompass the range of PCB, zinc, lead, and cadmium concentrations observed in 1993 (Johnson *et al.*, 1994a; Huntamer, 1994). The sampling sites were identical both years

These data were collected in conjunction with the department's effort to develop a freshwater sediment quality database (FSEDQUAL) and, ultimately, numerical sediment quality criteria (Cubbage and Breidenbach, 1994). The Spokane River data have passed quality assurance review for entry in the database. The present report summarizes these data; the database should be consulted for the complete results.

Methods

Sediment samples were collected August 7 - 10, 1994, using a 0.02 m^2 Ponar grab. Multiple grabs from each site were cornposited to form single site-samples. The grabs sampled the top 5-cm surface layer of sediment.

The composites were homogenized by stirring, split into appropriate containers, chilled, and held on ice for transport to Manchester Environmental Laboratory All sample handling equipment was stainless steel. pre-cleaned by brushing with tap water and Liquinox detergent, and sequential rinses with milli-Q water, nitric acid, milli-Q water, pesticide-grade acetone, and pesticide-grade hexane Field cleaning of the Ponar grab between samples was similar, except acetone was substituted for Liquinox prior to taking the Upriver Dam sample and Dove clear detergent was substituted for Liquinox on subsequent washes

Five tests of sediment toxicity were performed. They can be briefly described as follows:

- *Hyalella* bioassay - *Hyalella azteca* is an amphipod that lives on or in the sediments The test measures ten-day survival and average dry weight of the organisms at the end of the exposure period

- *Hexagenia* bioassay - *Hexagenia limbata* is a burrowing mayfly nymph Twenty-one day survival and organism dry weight are the endpoints

- *Chironomus* bioassay - *Chironomus tentans* is *a* burrowing fly larvae This test also measures survival and weight at the end of ten days

Microtox® - The Microtox® test measures the reduction in light produced by the luminescent marine bacterium *Photobacterium phosphoreum*.

The test was done in two ways:

- Microtox® solid phase and
- Microtox® deionized water extract with osmotic adjustment.

The *Hexagenia* bioassay was conducted at the Ontario Ministry of Environment and Energy, Standards Development Branche Laboratory in Etobicoke, Ontario The other bioassays were done locally by Parametrix, Inc

Chemical analysis of the samples was done by the Manchester laboratory and included PCBs; metals (arsenic, cadmium, chromium, copper, lead, nickel, zinc), and semivolatiles PCBs were analyzed by EPA Method 8080 For metals analysis, samples were digested by EPA Method 3050 and analyzed by ICP (EPA 200 7) Semivolatiles were done by a Manchester modification of SW846 Method 8270. Grain size and total organic carbon determinations were made by Soil Technology, Inc (ASTM D-422 modified with wet preparation) and Sound Analytical Services (Puget Sound Estuary Program method), respectively

Results

The bioassay and associated chemistry results are summarized in Table 1

A marine sediment, washed to remove salt, was used as the laboratory control sample for the *Hyalella*, Microtox® and *Chironomus* tests. Because the control sediments showed some evidence of toxicity to Microtox® and because of uncertainty as to the chemistry of this material, results for Long Lake were used for the statistical comparisons highlighted in Table 1.

Hyalella and Microtox® bioassays showed significant toxicity (greater than 95% probability) in the samples from behind Upriver Dam and, to a lesser extent, the Spokane Arm. There was only 50% survival of *Hyalella* in Upriver Dam sediments compared to 80% and 92% survival elsewhere Sediments from both Upriver Dam and the Spokane Arm reduced bacterial luminescence in the Microtox® test, EC_{50} (extract concentrations causing 50% less light production) were 28% and 41%, respectively

Spokane Ann and Long Lake sediments appeared to have an adverse effect on *Chironomus* survival These results, however, are subject to question Five pupating individuals were found at test termination Growth results did not indicate a problem but different life-stages could affect acute toxicity. The lab previously noted a problem with cannibalism, but ran these tests with five organisms per chamber The lab has experience with a thirty-day emergence test, but did not have much experience with the ten-day acute/chronic test.

It is not possible to determine with certainty which of the chemical contaminants detected in these samples were responsible for the toxicity observed Cubbage and Breidenbach (1994) used FSEDQUAL to derive preliminary estimates of apparent effects thresholds (AETs) for the *Hyalella* and Microtox® (standard) bioassays The chemical data in Table 1 were compared to these AETs and to freshwater sediment criteria that have been developed in Canada (Persaud *et al.*, 1993) Zinc, cadmium, and lead in the Upriver Dam sample, and zinc and lead in the Spokane Arm sample exceeded tentative freshwater AET values or Provincial guidelines severe effect levels (SELs), or both These metals were the most likely cause of the toxic response in the *Hyalella* and/or Microtox® tests It is possible that other materials contributed to toxicity as noted below

Other organic compounds detected in the Spokane River sediments included fifteen polyaromatic hydrocarbons (PAHs, e.g. naphthalene), 4-methylphenol, and retene (Table 1). These are among the more commonly detected semivolatiles in freshwater sediments. Sources include, but are not limited to, fossil fuels and their combustion (PAHs), and tars and wood waste (methylphenol and retene). The tentatively identified compounds referred to in Table 1 footnotes appear to be mostly naturally occurring sterols and aryl and polycyclic acids and alcohols.

Based on guidelines and criteria, the following chemicals may have contributed to toxicity in the 1994 Spokane River samples. Marine guidance is only shown where no freshwater guidance exists.

Above Up-River Dam,

Arsenic:	Exceeds:	Provincial SQG LEL[1]	
Cadmium:	Exceeds:	Provincial SQG LEL and SEL[2]	
		Preliminary Freshwater AET[3]	
Chromium:	Exceeds:	Provincial SQG LEL	
Copper:	Exceeds:	Provincial SQG LEL	
Lead:	Exceeds:	Provincial SQG LEL and SEL	
		Preliminary Freshwater AET	
Nickel:	Exceeds:	Provincial SQG LEL	
Zinc:	Exceeds:	Provincial SQG LEL and SEL	
		Preliminary Freshwater AET	
PCB 1248:	Exceeds[4]:	Provincial SQG LEL	(raw chemical conc)
4-methylphenol:	Exceeds:	Marine AET SQSL[5]	(raw and TOC normalized)
		Marine AET CSL[6]	(raw and TOC normalized)
		(no Provincial guidelines, no Freshwater AET for this chemical)	

At Lake Roosevelt Spokane Arm,

> arsenic, cadmium. copper, lead, nickel, and zinc exceed the Provincial SQG LELs; zinc exceeds the Provincial SEL as well.

At Long Lake,

> arsenic, cadmium, lead, and zinc exceed the Provincial SQG LELs

As described recently (Johnson *et al.*, 1994b), the deposits from which the Upriver Dam sample were obtained are of limited extent and are not typical of bed material in the upper river. Recent

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1 Provincial Sediment Quality Guidelines Lowest Effects Level – tolerated by most, but not all, benthic organisms
2 Provincial Sediment Quality Guidelines Severe Effects Level -- detrimental to most benthic organisms
3 Apparent Effects Level -- the level at which a toxic effect is always observed (Washington)
4 While PCB 1248 is listed here. its TOC normalized value is considerably lower than the Provincial SQG SEL. Also, PCBs are more likely to exhibit chronic toxicity than the acute toxicity indicated by the *Hyalella* test. Finally, some metals levels were considerably higher than the Provincial SELs; these have a much higher probability of causing the demonstrated acute toxicity than the PCBs do.
5 Sediment Quality Screening Level (Washington)
6 Cleanup Screening Level (Washington)

analysis at Manchester Laboratory indicates the source of organic carbon at this site is charcoal from burned pine trees (Huntamer, 1995). It should be noted that most metals concentrations in the 1994 Long Lake sample, including zinc and cadmium, were one-quarter to one-half the concentrations in the 1993 sample. Metals data from two sites sampled in Long Lake during a 1992 Ecology survey (Serdar et ***al.,*** 1994) are in line with the 1993 results. The 1992/93 surveys sampled the top 2 cm of sediment as opposed to the top 5 cm in 1994. It is likely that bioassays of the 1992 and 1993 Long Lake sediment samples would have shown some toxicity

References

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Table 1. Results of Bioassay and Chemical Tests of Spokane River Sediment Collected August 1994

	Test	Parameter(s)		Above Upriver Dam	Lake Roosevelt Spokane Ann	Long Lske (Reference)
				001	003	002
	Bioassays					
	Hyalella	acute	% survival	[50]	80	92
		chronic **	dry wt (mg)	0.11	0 11	0 13
	® Microtox	*di* extract	EC50 (% ext)	[28]	[41]	>52 9
*		solid phase	EC50 (ppm)	*45359*	*6620*	*13566*
*	*Chironomus*	acute	% survival	*84*	*[32]*	*48*
*		chronic **	dry wt (mg)	*2 20*	*2 21*	*2 84*
*	*Hexagenia*	acute	% survival	*100*	*100*	*100*
*		chronic **	dry wt (mg)	*9.22*	*11.98*	*26.74*
	Conventionals (%)					
	Fines			33	93	19
	TOC			13	1.8	0.81
	Metals (ppm dry weight)					
	As			18 P	29 P	9.6 P
	Cd			39.6	9.07	3.91
	Cr			29.8	20.1	9.58
	Cu			63.8	33.8	11.7 B
	Pb			542	81	42.3
	Ni			18.4	17.9	9 P
	Zn			4050	1180	520
	Organics (ppb dry weight)					
	2-Methylnaphthalene			94 J	—	11.7 J
	4-Methylphenol			3590 J	—	599
	Acenaphthene			53 J	—	—
	Acenaphthylene			296 J	—	—
	Anthracene			—	—	20.5 J
	Benzo(a)anthracene			—	—	52.7 J
	Benzo(a)pyrene			—	—	35.9 J
	Benzo(b)fluoranthene			—	—	53.1 J
	Benzo(ghi)perylene			—	—	23.2 J
	Benzo(k)fluoranthene			—	—	23.5 J
	Chrysene			—	—	57.3 J
	Fluoranthene			291 J	27.2 J	52.7 J
	Indeno(1,2,3-cd)pyrene			—	—	35.9 J
	Naphthalene			1270 J	—	53.1 J
	Phenanthrene			394 J	—	23.2 J
	Pyrene			359 J	—	23.5 J
	Retene			6020 J	77.1 J	57.3 J
	PCBs (ppb dry weight)					
	PCB1248			4500	35	21
	PCB1254			—	—	—
	PCB1260			—	—	—
	Total PCBs			4500	35	21
	Total PCBs (mg/kg OC)			34 6	1 9	2 6

* We consider Microtox (R) solid-phase, Chironomous, and Hexagenia tests to be experimental and not definitive for freshwater sediments

** Chronic dry wt (mg) is average per organism in each test group

di de-ionized water

[ ] Significant effect (p<0 05) compared to reference Reference sediment is Long Lake

[ ] - Apparent effect (p<0 05) compared to reference Reference sediment is Long Lake, but we're not convinced of the validity of this bioassay

— - not found above the quantification limit

P - measurement is above the detection limit, but below the quantification limit

B may have been contaminated during lab preparation

J - estimate

Tentatively Identified Compounds (TICs) are not shown in this able

LG/DB/AJ:krc

cc: Bill Sake, Jim Cubbage, Dale Norton