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# Distribution and Significance of Polycyclic Aromatic Hydrocarbons in Lake Washington Sediments Adjacent to Quendall Terminals/J.H. Baxter Site

by
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## ABSTRACT

Priority pollutant polynuclear aromatic hydrocarbon (PAH), pentachlorophenol (PCP), and polychlorinated biphenyl (PCB) concentrations were measured in surface sediments at 18 sites in Lake Washington, near Quendall Terminals/J.H. Baxter site, a historical refining and wood preserving operation. Total PAH concentrations ranged from 0.8-7300 mg/kg, dry weight basis.

The most widespread and severe PAH contamination occurs along a historical T-Pier at Quendall Terminals. PAH levels at two stations along this pier were high enough to adversely effect benthic communities, based on guidelines established by the Ontario Ministry of the Environment for the protection of aquatic biological resources. In addition, sediments were toxic to *Hyallela azteca* in a bioassay and showed reduced diversity in the benthic macroinvertebrate community. Peak PAH concentrations near Quendall Terminals/ J.H. Baxter are in the same range as those reported near Gas Works Park (Lake Union), an area of notably high PAH concentrations. Recommendations for follow-up work included: clarifying the extent of contamination around the T-Pier for remediation purposes, conduct limited sampling in the cove at J.H. Baxter site to allow comparisons with sediment guidelines, and start a Feasibility Study to evaluate options to remediate contaminated sediments.

ACKNOWLEDGMENTS

Many individuals have made valuable contributions to the Quendall Terminals/J.H. Baxter sediment investigation. Although it is not possible to acknowledge all participants, I would like to extend special thanks to the following contributors:

- Laura Chern and Jon Bennett for assisting in the field collections.
- Ron Perrigone of Dunlap Towing for providing access to several of the sampling sites.
- Dickey Huntamer of the Ecology/EPA Manchester Laboratory for his technical advice in designing the analytical scheme and conducting the analysis for the polynuclear aromatic hydrocarbons.
- Jim Cubbage, Bill Yake and Gail Colburn for reviewing the document.
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# INTRODUCTION

Quendall Terminals and the J.H. Baxter site are adjoining parcels located along the southeast shoreline of Lake Washington near the City of Renton (see Figure 1). Between 1917 and 1970, Quendall Terminals (first called Republic Creosote, and then Reilly Tar and Chemical Company) was the site of a refinery that produced creosote and other distillates from various tars. Approximately 500,000 gallons of oil-gas tar from Gas Works Park (Lake Union) was used each month at the plant, in addition to tars from other sources (Munt and Coleman, 1989). The J.H. Baxter property was the site of a wood treating facility. These operations substantially contaminated ground water and onsite soils. In addition, previous studies have identified high concentrations of polynuclear aromatic hydrocarbons (PAH) in Lake Washington sediments along the historical T-pier at Quendall Terminals (EPA, 1983) and from the cove at the southern end of the J.H. Baxter property (Woodward-Clyde, 1989).

Since the areal extent of these studies was somewhat limited, Ecology's Northwest Regional Office (NWRO) requested that the Toxics Investigation Section (TIS) conduct a sediment characterization study in Lake Washington with the following objectives:

- Determine the areal extent and environmental significance of sediment contamination (primarily PAHs) in Lake Washington near Quendall Terminals and the J.H. Baxter site.

- As a secondary objective, provide physical/chemical data and toxicity (bioassay/benthic macroinvertebrate) information for PAHs, to supplement Ecology's efforts to develop numerical freshwater sediment criteria for the State of Washington.

Results from this study, in conjunction with data from previous site investigations will provide information on the extent of sediment remediation needed.

Lake Washington's shorelines and wetlands have been identified as one of the Washington States most valuable natural resources, and as a result they constitute shorelines of state-wide significance (WAC 173-28). In addition, the lake supports important sport and tribal fisheries (Colburn, 1991).

# METHODS

## Sample Collection

To determine the areal distribution of PAHs in Lake Washington near Quendall Terminals/ J.H. Baxter site, surface sediments (top 2 cm) were collected at 18 stations between May 16-17, 1990. Sampling locations are shown in Figure 2. The following criteria were used to select sediment sampling sites: 1) areas that had not been previously studied; 2) areas of

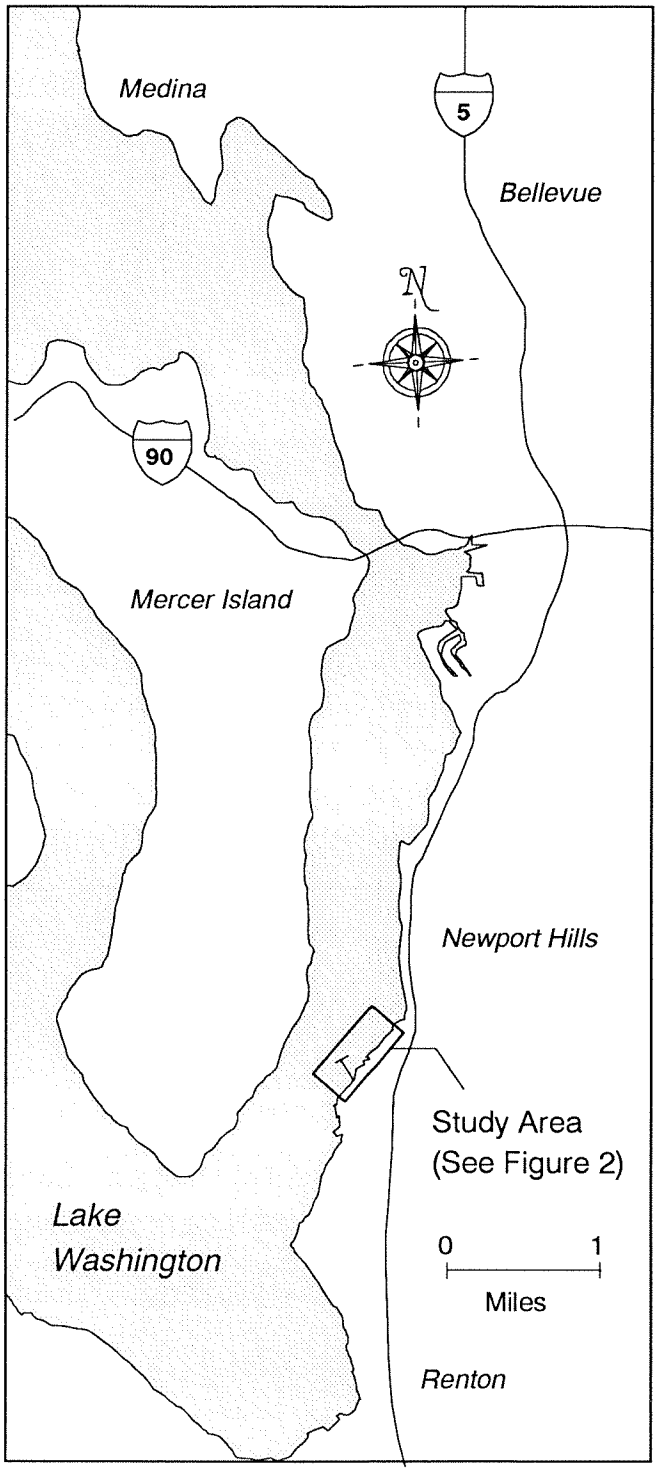


Figure 1: Vicinity Map Quendall Terminals/J.H. Baxter

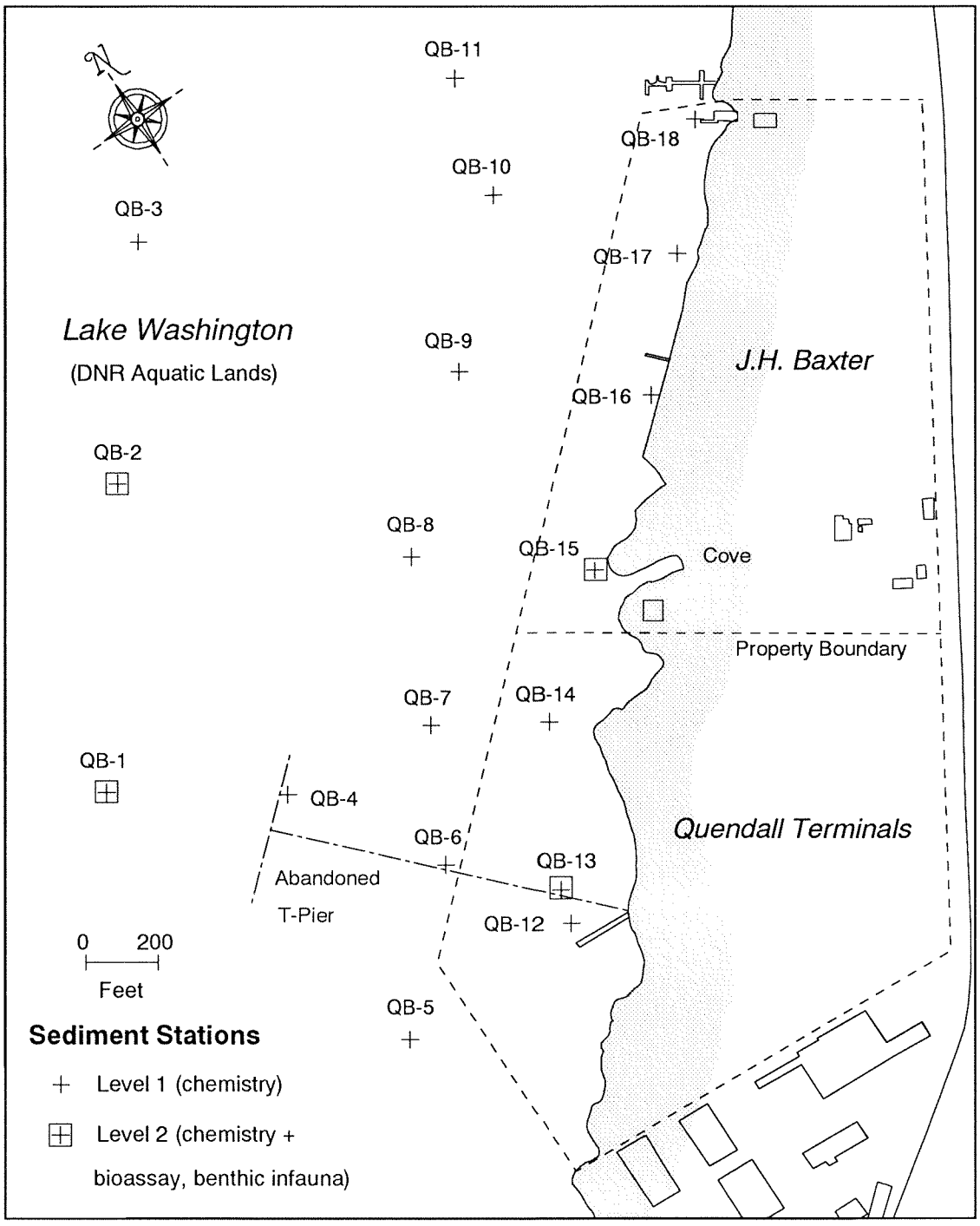


Figure 2: Quendall Terminals/J.H. Baxter sediment sampling locations, Lake Washington.

suspected contamination based on interviews with workers at the site; and 3) sample over increasing depths moving away from the sites into deeper water.

To provide supplemental data for Ecology's efforts to develop numerical criteria for freshwater sediments (Bennett and Cubbage, 1990), aliquots for total sulfides, metals (As, Cd, Cu, Pb and Hg), benthic macroinvertebrate and bioassay (*Hyalella azteca and Daphnia magna*) were prepared from sediments at four stations (1, 2, 13, and 15). These stations were selected to cover a range of PAH concentrations (1, 2-low level, 13, 15-high level) based on data from previous investigations.

Sampling procedures followed Puget Sound Protocols where applicable (Tetra Tech, 1986). Sediments for physical/chemical analyses and bioassay were collected with a modified $0.1 m^2$ stainless steel Van Veen grab. Station positions were located using landmarks and a hand bearing compass, in conjunction with depth readings. To minimize cross-contamination between stations, sediments were collected in order of anticipated increasing contamination levels based on previous data and reconnaissance work.

After retrieving the grab, the top 2 cm layer not in contact with the sidewalls of the sampler was transferred to a stainless steel beaker. The sample was then homogenized by stirring with a stainless steel spoon. Subsamples for individual analyses were taken from this homogenate. All samples were placed in priority pollutant-cleaned glass jars with teflon-lined lids supplied by I-Chem, Hayward, California. An exception being grain size aliquots, which were placed in Whirlpac® bags. All samples were wrapped in polyethylene bags and stored on ice for transport to the laboratory. The grab was decontaminated between stations with onsite lake water. Spoons and beakers were pre-cleaned with sequential washes of hot tap water/liquinox® detergent, 10% nitric acid, distilled/deionized water, pesticide grade acetone, then air dried and wrapped in aluminum foil until used in the field.

To assess sampling/laboratory variability, duplicate sediment samples (e.g. one sample homogenized and split) were prepared at Station 13. In addition, to assess environmental variability, a replicate sample (e.g. two separate samples collected at a similar location), was also prepared at this location.

Benthic macroinvertebrate samples were obtained using a $0.02 m^2$ stainless steel petite ponar grab. The entire contents of each grab were screened through a 30 mesh (0.59mm) sieve. The material retained by the sieve was transferred to one-quart glass jars and immediately fixed with 10% buffered formalin. Five replicate samples were collected at each macroinvertebrate station.

## Analysis and Quality Assurance

The chemical analyses, analytical methods, and laboratories used in this study are listed in Table 1. Quality of the data set was assessed by analysis of method blanks, internal standards, surrogate spikes, duplicate matrix spikes, blind field duplicates, and a standard reference material (SRM-metals only).

Results of metals analysis of SRM-2704 (Buffalo River Sediment) are shown below in Table 2. Acceptable accuracy was obtained for all target metals, being within ±23% of the certified ranges.

Table 2: Results of analysis of certified reference material for sediment (mg/kg, dry).

| | NBS-2704 | |
|---|---|---|
| Element | Certified Range | AM Test Result |
| Arsenic | 23.4+/-0.8 | 19.5 |
| Cadmium | 3.45+/-0.22 | 2.5 |
| Copper | 98.6+/-5.0 | 75 |
| Lead | 161+/-17 | 120 |
| Mercury | 1.44+/-0.07 | 1.4 |

NBS-2704-Buffalo River Sediment, National Bureau of Standards

Estimates of overall precision (sampling + laboratory) calculated as relative percent difference (RPD) using blind field and laboratory duplicates (range as percent of mean) were as follows: conventionals (±15%); except total sulfides (±70%), grain size (±40%), metals (As, Cu, Pb= ±10%, Cd, Hg= ±40%), organics (±25%).

These data indicate that sample handling procedures and laboratory methods were not significant contributors to data variability for most analytes. An exception being total sulfides, and to a lesser extent grain size and Hg analyses which had lower precision.

Quality assurance review of the organics data (PAHs, pentachlorophenol (PCP) and polychlorinated biphenyls (PCBs) was performed by Dickey Huntamer of the Ecology/EPA Manchester Laboratory. In the opinion of the reviewer, no major analytical problems were encountered in the analysis of these samples. Consequently, the data set is considered acceptable for use, with the accompanying qualifiers noted where appropriate. Case narratives and data reviews for the organics analyses are included in Appendix A1.

Table 1: Summary of analytical methods for Quendall Terminals/J.H. Baxter sediment survey.

| Analysis | Method | Reference | Laboratory |
|---|---|---|---|
| Conventionals | | | |
| Percent Moisture | Dry @ 104°C | Tetra Tech, 1986 | Analytical Resources, Inc. - |
| Total Organic Carbon | Combustion/CO2 measurement | " | Seattle, Wa. |
| Total Sulfides | Spectrophotometric | " | " |
| Grain Size | Seive and pipet | Holme and McIntrye, 1971 | Hart Crowser, Inc.- Seattle, Wa. |
| Biology | | | |
| Daphnia magna | Solid phase 48 hour | Nebeker et al, 1984 | Ecology/EPA Laboratory - Manchester, Wa. |
| Hyalella azteca | 10 day | " | Northwestern Aquatic Sciences - Newport, Or. |
| Benthic infauna | Enumerate (Family/Genus) | - | Taxon - Corvallis, Or. |
| Metals | | | |
| Arsenic | GFAA No. 7060 | EPA, 1986 | AM Test, Inc. - Seattle, Wa. |
| Cadmium | GFAA No. 7131 | " | " |
| Copper | ICP No. 6010 | " | " |
| Lead | GFAA No. 7421 | " | " |
| Mercury | CVAA No. 7471 | " | " |
| Organics | | | |
| PAH | GC/MS No. 3520/3630/8270 | EPA, 1986 | Ecology/EPA Laboratory - Manchester, Wa. |
| PCP | GC/ECD No. 8150 (modified) | " | " |
| PCB | GC/ECD No. 3520/8080 | " | " |

GFAA=Graphite furance atomic absorption
ICP=Inductively coupled plasma-atomic emission
CVAA=Cold vapor atomic absorption

Bioassay results were reviewed by Margaret Stinson of the Ecology/EPA Manchester Laboratory. In the opinion of this reviewer, quality control data were appropriate for the test conditions in both bioassays. Case narratives for *Daphnia magna* and *Hyalella azteca* bioassays are included in Appendix A2.

Benthic macroinvertebrate samples were keyed to the lowest taxonomic level easily obtained. In most cases, individuals were identified to family. Macroinvertebrate results are reported as the average of five replicate samples.

## RESULTS AND DISCUSSIONS

### Conventionals

The results of conventionals analysis of Lake Washington sediments adjacent to Quendall Terminals/ J.H. Baxter site are summarize in Table 3.

In general, sediments near Quendall Terminals/J.H. Baxter site were variable in their physical characteristics. Percent moisture ranged from 21.8 to 81.5. Total organic carbon (TOC) concentrations ranged from 0.3 to 25 percent with a mean of 11.7 percent. The highest TOC value was present at Station 15, located just outside the cove at the southern end of the J.H. Baxter property. Field observations noted a substantial amount of wood debris at this location.

The grain size distribution of Lake Washington sediments is shown in Figure 3. Stations 3,9,10,11,12,14, and 16 were primarily sand (2mm-62$\mu$m). The remaining stations were composed primarily of silt (62$\mu$m-4$\mu$m) and to a lesser extent clay ($<4\mu$m) size particles.

### Organics

The results of organics analysis of Lake Washington sediments near Quendall Terminals/ J.H. Baxter site are summarized in Table 4. After reviewing data from previous site investigations, organics analyses for the present study focused on the following constituents: 16 priority-pollutant PAHs, PCP and PCBs.

PAH levels were extremely variable throughout the study area, with concentrations covering approximately 4 orders of magnitude (total PAH= 0.8-7300 mg/kg, dry). In general, the highest PAH concentrations were present in the vicinity of the historical T-Pier at Quendall Terminals. During the late 1930's to early 1940's, a major spill of unidentified tars occurred along this pier during off-loading of a tanker. It was estimated that 30,000 to 40,000 gallons of material was lost into Lake Washington during this spill. Station 4 (end of the T-Pier) had the highest concentrations of PAHs, at over 0.7% total PAHs on a dry weight basis. The lowest PAH concentrations were measured at Station 14 (0.8 mg/kg, dry). The relatively low PAH

Table 3: Results of conventionals analysis of Lake Washington sediments collected by Ecology May 15–16, 1990 adjacent to Quendall Terminals/J.H. Baxter site.

| Tier | Outer | | | Middle | | | | | | | | Inner | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Station No. QB– | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 13D | 13R | 14 | 15 | 16 | 17 | 18 |
| Property | DNR | DNR | DNR | DNR | DNR | DNR | DNR | DNR | DNR | DNR | DNR | QT | QT | – | – | QT | Bax | Bax | Bax | Bax |
| Sample No. 20– | 8280 | 8281 | 8282 | 8283 | 8284 | 8285 | 8286 | 8287 | 8288 | 8289 | 8290 | 8291 | 8292 | 8293 | 8294 | 8295 | 8296 | 8297 | 8298 | 8299 |
| Depth (ft) | 40 | 40 | 47 | 26 | 20 | 20 | 22 | 23 | 21 | 24 | 33 | 12 | 11 | (Dup) | (Rep) | 10 | 9 | 10 | 10 | 10 |
| Moisture (%) | 73.4 | 74.0 | 65.7 | 80.6 | 76.5 | 81.2 | 80.4 | 81.9 | 69.6 | 75.9 | 40.3 | 60.2 | 70.9 | 69.3 | 79.2 | 21.8 | 79.4 | 71.9 | 80.1 | 81.5 |
| Total Organic Carbon (%) | 3.6 | 6.4 | 6.6 | 12.0 | 13.0 | 11.0 | 14.0 | 13.0 | 23.0 | 13.0 | 24.0 | 6.9 | 7.7 | 7.4 | 11.0 | 0.3 | 25.0 | 16.0 | 9.5 | 11.0 |
| Grain Size (%) | | | | | | | | | | | | | | | | | | | | |
| Gravel (>2mm) | 0 | 1 | 2 | 8 | 5 | 1 | 1 | 4 | 9 | 3 | 2 | 3 | 0 | 3 | 3 | 5 | 2 | 2 | 1 | 0 |
| Sand (2mm–62um) | 11 | 25 | 55 | 19 | 42 | 16 | 21 | 29 | 61 | 60 | 87 | 76 | 47 | 40 | 28 | 93 | 49 | 55 | 26 | 14 |
| Silt (62um–4um) | 68 | 59 | 30 | 53 | 44 | 66 | 62 | 49 | 15 | 31 | 7 | 16 | 35 | 48 | 57 | 1 | 30 | 25 | 43 | 59 |
| Clay (<4um) | 21 | 15 | 13 | 20 | 9 | 17 | 16 | 18 | 16 | 6 | 4 | 5 | 18 | 9 | 12 | 1 | 19 | 18 | 30 | 28 |

DNR= Wash. St. Dept. of Natural Resources

QT= Quendall Terminals

Bax= J.H. Baxter

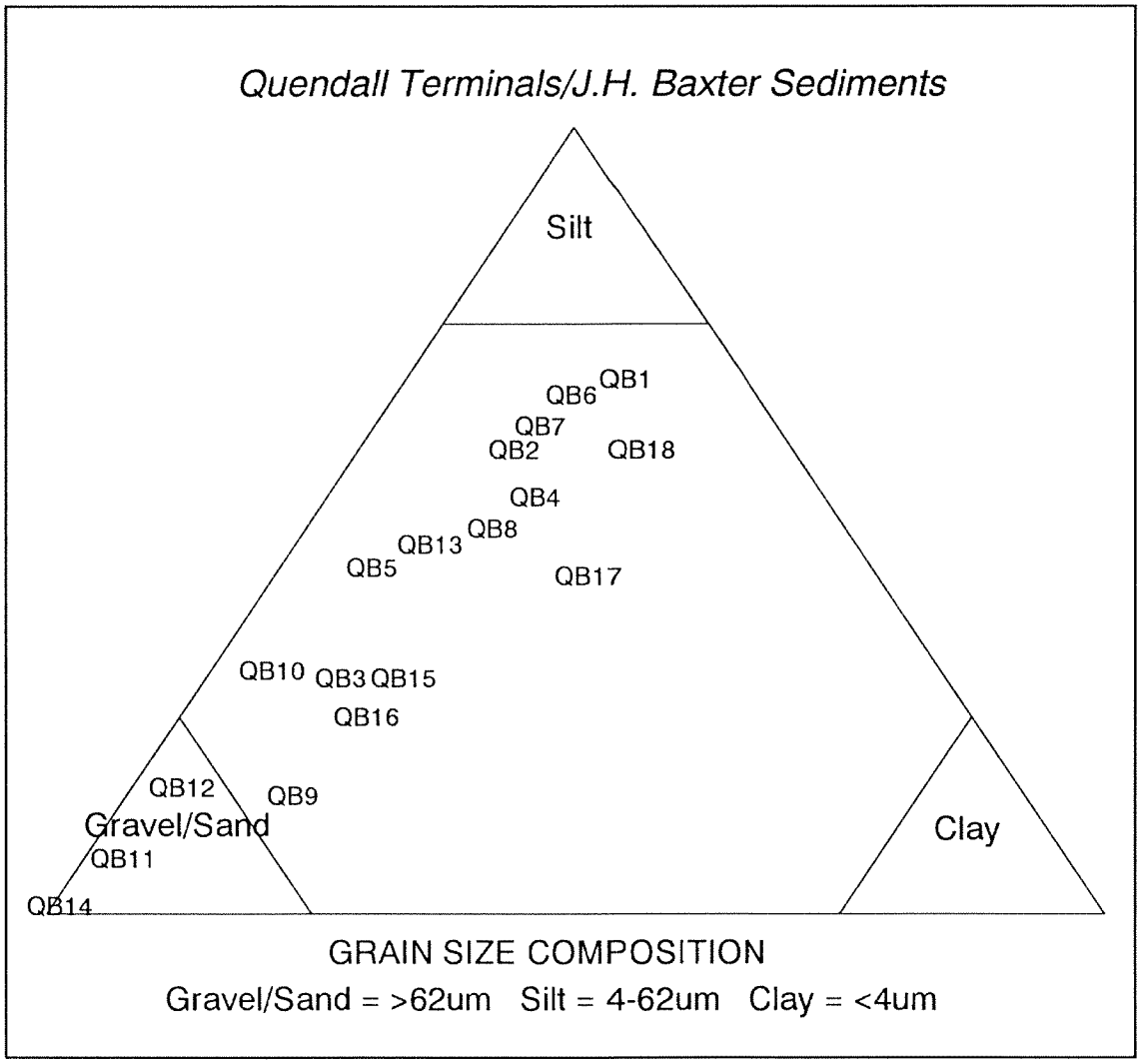


Figure 3: Grain size composition of Lake Washington sediments adjacent to Quendall Terminals/J.H. Baxter.

Table 4: Summary of organics analysis of Lake Washington sediments collected by Ecology May 16-17, 1990 adjacent to Quendall Terminals/J.H. Baxter site (mg/kg, dry ie ppm).

| Tier | Outer | | | Middle | | | | | | | | Inner | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Station No. QB- | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 13D | 13R | 14 | 15 | 16 | 17 | 18 |
| Sample No. 20- | 8280 | 8281 | 8282 | 8283 | 8284 | 8285 | 8286 | 8287 | 8288 | 8289 | 8290 | 8291 | 8292 | 8293 | 8294 | 8295 | 8296 | 8297 | 8298 | 8299 |
| Depth (ft) | 40 | 40 | 47 | 26 | 20 | 20 | 22 | 23 | 21 | 24 | 33 | 12 | 11 | (Dup) | (Rep) | 10 | 9 | 10 | 10 | 10 |
| Acenaphthene | 0.037j | 0.023j | 0.028j | 570j | 0.38j | 0.25j | 0.57 | 0.031j | 0.35u | 0.093j | 0.26 | 4.3 | 26 | 25j | 2.4j | 0.15u | 2.8uj | 2.3uj | 0.61u | 0.070j |
| Acenaphthylene | 0.020j | 0.42u | 0.020j | 1.8j | 0.054j | 0.098j | 0.064j | 0.008j | 0.35u | 0.43u | 0.18u | 0.26 | 0.84j | 0.73j | 0.25j | 0.15u | 2.8uj | 2.3uj | 0.61u | 0.62u |
| Naphthalene | 0.10j | 0.10j | 0.12j | 160j | 0.89 | 0.68 | 0.64 | 0.051j | 0.12j | 0.10j | 0.090j | 6.9 | 92j | 67 | 3.6j | 0.034j | 1.3j | 2.3uj | 0.61u | 0.14 |
| Fluorene | 0.043j | 0.030j | 0.031j | 510j | 0.33j | 0.34j | 0.48j | 0.036j | 0.35u | 0.16j | 0.25 | 2.8 | 14j | 14j | 1.2j | 0.15u | 2.8uj | 0.57j | 0.61u | 0.085j |
| Anthracene | 0.063j | 0.42u | 0.063j | 350j | 0.50 | 0.45j | 0.49j | 0.049j | 0.35u | 0.18j | 0.038j | 2.6 | 7.9j | 6.9j | 1.5j | 0.15u | 2.8uj | 0.56j | 0.61u | 0.11j |
| Phenanthrene | 0.29j | 0.21j | 0.26j | 2100j | 2.1 | 2.1 | 2.8 | 0.20 | 0.25j | 1.4 | 1.4 | 12 | 52j | 44 | 8.2j | 0.057j | 1.9j | 6.1j | 0.23j | 0.53j |
| **Sum LPAH** | **0.55j** | **0.36j** | **0.52j** | **3700j** | **4.3j** | **3.9j** | **5.0j** | **0.38j** | **0.37j** | **1.9j** | **2.0j** | **29** | **190j** | **160j** | **17j** | **0.090j** | **3.2j** | **7.2j** | **0.23j** | **0.94j** |
| Fluoranathene | 0.72 | 0.46 | 0.75 | 1400j | 2.5 | 3.7 | 3.7 | 0.54 | 0.56 | 3.2 | 1.1 | 20 | 35j | 43 | 12j | 0.098j | 3.1j | 7.0j | 0.34j | 0.95 |
| Benzo(a)anthracene | 0.57 | 0.28j | 0.27j | 280j | 1.5 | 4.5 | 2.5 | 0.24 | 0.37 | 0.76 | 0.25 | 8.9 | 63j | 43 | 15j | 0.051j | 1.8j | 1.6j | 0.61u | 0.38j |
| Chrysene | 0.85 | 0.57 | 0.70 | 290j | 2.7 | 9.7 | 4.6 | 0.60 | 0.84 | 1.1 | 0.38 | 22 | 110j | 96 | 28j | 0.094j | 3.7j | 1.8j | 0.35j | 0.88 |
| Pyrene | 0.75 | 0.56 | 0.86 | 1200j | 2.7 | 4.4 | 4.0 | 0.60 | 0.80 | 2.4 | 0.90 | 18 | 68j | 65 | 18j | 0.11j | 3.5j | 6.2j | 0.33j | 1.2 |
| Benzo(b)fluoranthene | 1.7 | 1.1 | 1.2 | 270j | 3.4 | 17 | 9.9 | 1.3 | 1.1 | 1.5 | 0.52 | 45 | 280j | 200 | 60j | 0.18 | 5.3j | 2.5j | 0.52j | 1.8 |
| Benzo(k)fluoranthene | 0.38u | 0.42u | 0.30u | 2.1uj | 0.44u | 7.7 | 0.55u | 0.14u | 0.35u | 0.43u | 0.18u | 0.25u | 2.1u | 2.0uj | 2.7u | 0.075j | 2.8uj | 0.94j | 0.61u | 0.62u |
| Benzo(a)pyrene | 0.74 | 0.49 | 0.52 | 87j | 3.2 | 14 | 4.8 | 0.57 | 0.62 | 0.57 | 0.22 | 19 | 140j | 100 | 42j | 0.15u | 2.8j | 1.0j | 0.15j | 0.67 |
| Dibenzo(a,h)anthracene | 0.26j | 0.42u | 0.30u | 13j | 0.5 | 2.8 | 0.91 | 0.14u | 0.35u | 0.43u | 0.18u | 7.1 | 39j | 33j | 7.3j | 0.15u | 0.78j | 2.3uj | 0.61u | 0.62u |
| Indeno(1,2,3-cd)pyrene | 0.45 | 0.31j | 0.28j | 24j | 1.4 | 6.9 | 2.3 | 0.31 | 0.36 | 0.35j | 0.083j | 12 | 84j | 43 | 18j | 0.15u | 1.7j | 0.88j | 0.13j | 0.40j |
| Benzo(g,h,i)perylene | 0.70 | 0.51 | 0.50 | 24j | 2.4 | 11 | 3.7 | 0.48 | 0.57 | 0.46 | 0.14j | 28 | 170j | 93 | 29j | 0.11j | 2.5j | 1.3j | 0.21j | 0.78 |
| **Sum HPAH** | **6.7j** | **4.3j** | **5.1j** | **3600j** | **20** | **82** | **36** | **4.6** | **5.2** | **10j** | **3.6j** | **180** | **990j** | **720j** | **230j** | **0.70j** | **25j** | **23j** | **2.0j** | **7.1j** |
| **Total PAH** | **7.3j** | **4.7j** | **5.6j** | **7300j** | **24j** | **86j** | **41j** | **5.0j** | **5.6j** | **12j** | **5.6j** | **210** | **1200j** | **880j** | **250j** | **0.79j** | **28j** | **30j** | **2.2j** | **8.0j** |
| Carbazole | 0.38uj | 0.42uj | 0.30uj | 370j | 0.44uj | 0.60uj | 0.55uj | 0.14uj | 0.35uj | 0.43uj | 0.18uj | 0.25uj | 2.1uj | 2.0uj | 2.7uj | 0.15uj | 2.8uj | 2.3uj | 610uj | 0.62u |
| Dibenzofuran | 0.38u | 0.42u | 0.30u | 290j | 0.21j | 0.18j | 0.34j | 0.023j | 0.35u | 0.097j | 0.16j | 1.2 | 7.9j | 8.3j | 0.9j | 0.15u | 2.8uj | 2.3uj | 610uj | 0.62u |
| Retene | 8.7 | 1.3 | 15 | 130j | 57j | 24 | 71 | 130j | 190 | 240j | 3.6 | 39 | 27j | 16j | 48j | 0.53 | 810j | 110 | 8.0 | 50 |
| 1-methylnaphthalene | 0.040j | 0.034j | 0.032j | 220j | 0.28j | 0.27j | 0.30j | 0.022j | 0.35u | 0.43u | 0.073j | 4.6 | 64j | 61j | 5.5j | 0.15u | 2.8uj | 2.3uj | 610uj | 0.62u |
| 2-methylnaphthalene | 0.018j | 0.018j | 0.018j | 150j | 0.16j | 0.18j | 0.26j | 0.014j | 0.35u | 0.43u | 0.042j | 2.2 | 42j | 40j | 3.2j | 0.15u | 2.8uj | 2.3uj | 610uj | 0.62u |
| 2-chloronaphthalene | 0.38u | 0.42u | 0.30u | 2.1uj | 0.44u | 0.60u | 0.55u | 0.14u | 0.35u | 0.43u | 0.18u | 0.25u | 2.1uj | 2.0uj | 2.7u | 0.15u | 2.8uj | 2.3uj | 0.61uj | 0.62u |
| Pentachlorophenol | 0.015 | 0.019 | 0.012 | 0.031 | 0.014 | 0.035 | 0.045 | 0.12 | 0.030u | 0.040u | 0.015u | 0.025u | 0.016 | 0.021 | 0.030 | 0.010u | 0.41 | 0.040u | 0.018 | 0.18 |
| 2,3,4,5-tetrachlorophenol | ND | ND | ND | ND | ND | ND | ND | 0.28 | ND | ND | ND | ND | ND | ND | ND | ND | 0.030 | ND | ND | ND |
| PCB's | 0.13u | 0.14u | - | 0.99u | - | 0.18u | - | - | - | - | - | 0.080u | 0.67u | 0.65u | 0.88u | 0.050u | 0.89u | 0.74u | 0.20u | 0.20u |

u=Not detected at detection limit shown

j=Estimated concentration

ND=Not detected at unspecified detection limit

-=Not analyzed

PCB's includes-1016,1221,1232,1242,1248,1254 and 1260

levels, on a dry weight basis, at Station 14 is most likely related to the low fines (see Figure 3) and TOC (Table 3) content of this sample. Hydrocarbons have been shown to preferentially associate with fine grain sediments (Merill and Wade, 1985). At all stations, the sum of individual high molecular weight PAHs (HPAH) were equal to or exceeded the sum of individual low molecular weight PAHs (LPAH). This apparent enrichment of HPAH in sediments is not unexpected, since weathering processes such as evaporation, photochemical oxidation, dissolution, and microbial degradation can preferentially remove PAHs with molecular weights less than that of fluoranthene (Merill and Wade, 1985).

Two chlorophenols, PCP and 2,3,4,5-tetrachlorophenol were present at detectable levels. PCP was present at low levels (0.015$\mu$-0.41 mg/kg, dry), in 14 of the 20 samples analyzed. The highest concentration was measured at Station 15, located adjacent to the cove at the southern end of the J.H. Baxter property. Tetrachlorophenol was also detected at this location (0.03 mg/kg, dry). PCP and tetrachlorophenol are commonly used as wood preservatives (Verschueren, 1983). PCBs were not detected in any of the samples tested.

Retene, which is a naturally occurring resin acid-derived compound (Prahl and Carpenter, 1984), was detected at all sampling sites. Concentrations of retene ranged from 0.53-810 mg/kg dry weight. The presence of retene in the sediments tested is in all probability related to log rafting activities at the two sites.

Forty-four semivolatile compounds were also tentatively identified (TI) in sediments. These compounds, listed in Appendix A3, were primarily alkyl substituted PAHs (i.e.: an alkyl group has been substituted for a hydrogen on one or more of the aromatic rings. Some of the more common alkyl groups are ethyl-; methyl-; isopropyl-; and butyl-). Alkyl substituted naphthalenes and phenanthrenes were the most prevalent TI PAHs. In general, the highest concentrations of all the TI organics occurred at Stations 4 and 13, located along the historical T-Pier at Quendall Terminals. These two stations also contained the highest concentrations of target PAHs (see Table 4). The remaining 26 TI compounds, primarily constituents of petroleum hydrocarbons, were for the most part present at low levels (range 0.039-55 mg/kg, dry).

The distribution of PAHs in sediments normalized for TOC is shown in Figure 4. TOC normalization reduces variability in PAH concentrations associated with differences in sediment TOC content. The distribution of PAHs suggests sediment contamination near Quendall Terminals/J.H. Baxter probably originated from two sources: Past spills along the historical T-Pier and onshore source(s) (via ground water and/or surface drainages) in the vicinity (Stations 14, 15 and 16) of the cove at the southern end of the J.H. Baxter property.

Listed in Table 5 is a relative ranking of sediment stations from the present study based on TOC normalized total PAH concentrations. In general, total PAH concentrations adjacent to Quendall Terminals were in excess of 100 mg PAH/Kg TOC, while concentrations tended to be less than 100 mg PAH/kg TOC at the J.H. Baxter site.

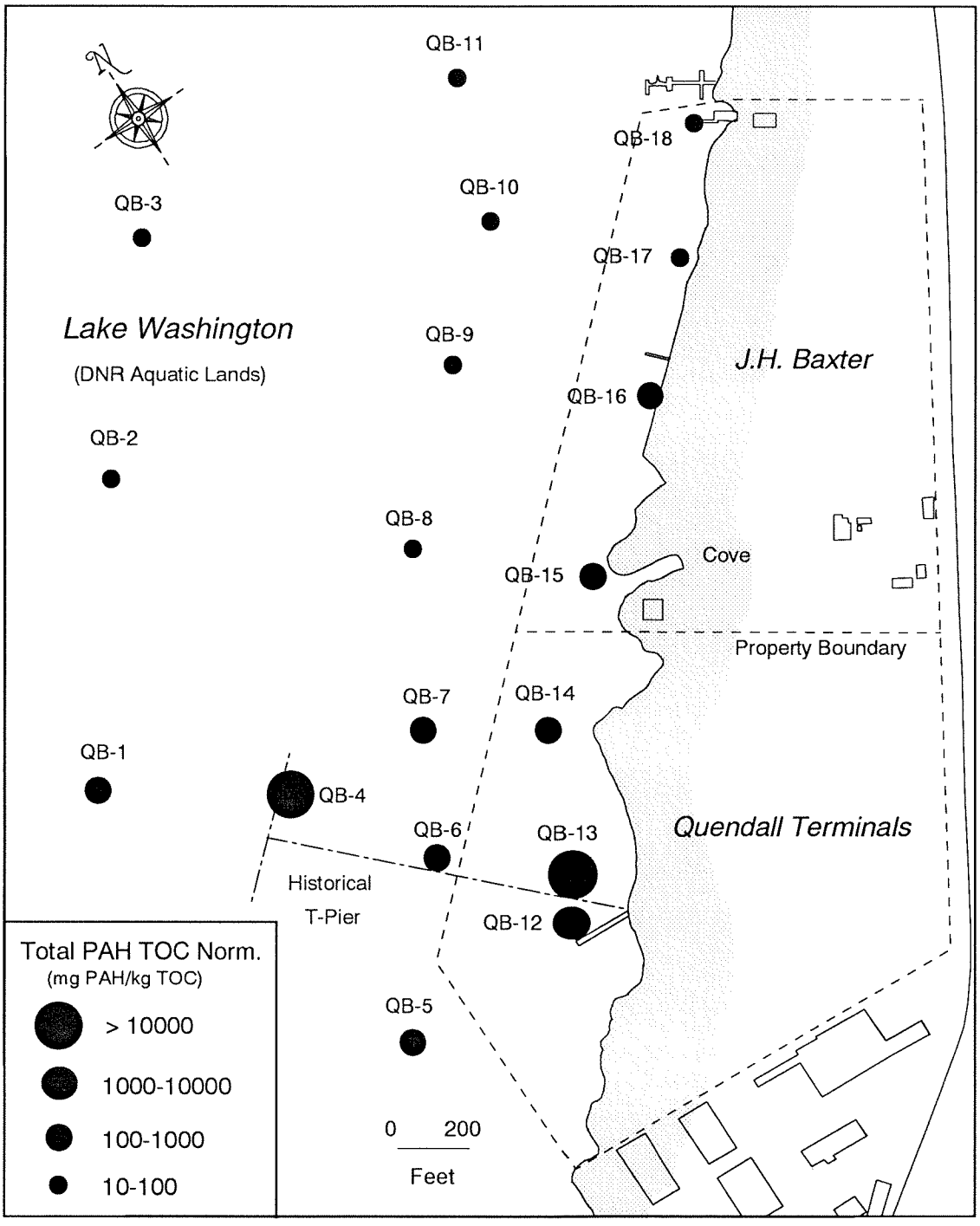


Figure 4: Distribution of Total PAH in Lake Washington Sediments adjacent to Quendall Terminals/J.H. Baxter (mg PAH/kg TOC)

Table 5: Ranking of Ecology's Quendall Terminals/J.H. Baxter sediment stations, based on TOC normalized Total PAH concentrations (mg PAH/kg TOC).

| Station | TOC (%) | Total PAH Concentration Dry wt | TOC norm. | Range (mg PAH/kg TOC) |
|---|---|---|---|---|
| 4 | 12j | 7300j | 61000j | |
| 13D | 7.6j | 1040j | 14000j | > 10000 Criteria= 11000 |
| 13R | 11j | 250j | 2300j | |
| 12 | 6.9 | 210 | 3000 | > 1000 |
| 6 | 11j | 86j | 780j | |
| 7 | 14j | 41j | 290j | |
| 14 | 0.3j | 0.8j | 270j | |
| 1 | 3.6j | 7.3j | 200j | |
| 16 | 16j | 30j | 190j | |
| 5 | 13j | 24j | 190j | |
| 15 | 25j | 28j | 110j | > 100 |
| 10 | 13j | 12j | 92j | |
| 3 | 6.6j | 5.6j | 85j | |
| 2 | 6.4j | 4.7j | 73j | |
| 18 | 11j | 8j | 73j | |
| 8 | 13j | 5j | 38j | |
| 9 | 23j | 5.6j | 24j | |
| 11 | 24j | 5.6j | 23j | |
| 17 | 9.5j | 2.2j | 23j | > 10 |

j=Estimated concentration
D=Average of duplicate analysis
R=Replicate sample
=Exceeds guidelines established by Ontario Ministry of Environment (Persaud et al, 1990) Guideline shown is Limit of Tolerance i.e. Concentration of a compound that would be detrimental to the majority of benthic species.

Also included in Table 5 are freshwater sediment quality guidelines developed by the Ontario Ministry of Environment for the protection of aquatic biological resources. These guidelines were included here for comparison because they are the only numerical values currently available to evaluate PAHs levels in freshwater sediments (Bennett and Cubbage, 1990). However, caution should be used in applying these guidelines as cleanup standards for the following reasons. The guidelines were developed on a regional basis and have not been adopted as sediment standards or criteria by the Canadian government. While Washington State is actively developing freshwater sediment standards, including chemical and biological criteria, it will be approximately two years before they are available. In addition, when completed Washington standards may be more or less stringent than the Canadian guidelines. With these caveats in mind, the Canadian guideline shown for total PAHs (11000 mg PAH/kg TOC), represents the limit of tolerance, which is the sediment concentration of a compound that would be detrimental to the majority of benthic species (Persaud et. al, 1990). Stations 4 (61000 mg PAH/kg TOC) and 13 (14000 mg PAH/kg TOC) exceeded the sediment guideline for total PAHs.

To place PAH concentrations near Quendall Terminals/J.H. Baxter site into perspective, Table 6 compares these data to PAH concentrations reported by other investigators in surface sediments from Lake Washington, Lake Union/Ship Canal, Gasworks Park and nine other Washington Lakes. Peak PAH concentrations near Quendall Terminals/J.H. Baxter are in the same range as Lake Union sediments near Gas Works Park, which is considered a heavily contaminated site. In addition, median concentrations near the two sites are comparable to medians for Lake Union/Ship Canal and generally higher than mean values reported for Lake Washington as a whole. Compared to lakes considered uninfluenced by industrial activities, sediments near the two sites contain substantially elevated (up to 4 orders of magnitude) PAH levels.

**Supplemental Data for Freshwater Sediment Criteria Development**

As previously discussed, the following data were not the primary focus of the present study, but were collected to supplement Ecology's efforts to develop numerical freshwater sediment criteria for the State of Washington (Bennett and Cubbage, 1990). Consequently, only a brief evaluation of the data is presented. A more detailed discussion of this data will be presented in a subsequent report which describes Ecology's freshwater sediment criteria development for PAHs.

The results of total sulfides, metals and bioassay analyses at selected sites in Lake Washington near Quendall Terminals/J.H. Baxter are listed in Table 7. The only station that contained detectable levels of sulfides was Station 13, where concentrations ranged from 17.1-44.6 mg/kg. Metals concentrations were low in all sediments analyzed, and in the range of values reported for lakes considered uninfluenced by industrial activities (Johnson and Norton, 1990). Concentrations (mg/kg, dry) of individual elements ranged as follows; arsenic (0.4$\mu$-18), cadmium (0.19$\mu$-0.80), copper (20-41), lead (31-89) and mercury (0.04-0.27). The highest

Table 6: Comparison of PAH concentrations in Lake Washington sediments near Quendall Terminals/ J.H. Baxter site with other data on sediments from Lake Washington, Lake Union/Ship Canal, Gas Works Park and selected Washington Lakes (mg/kg, dry).

| Location | Sampling Interval | No. Samples | Median(Range) LPAH | HPAH | TPAH |
|---|---|---|---|---|---|
| Quendall Terminals/J.H. Baxter | | | | | |
| Present Study | Top 2cm | 20 | 3 (0.09–3700) | 10 (0.7–3600) | 12 (0.8–7300) |
| Woodward–Clyde, 1989 | Top 1 ft | 19 | 0.6 (ND–18000) | 3 (ND–10000) | 4 (ND–28000) |
| EPA, 1983 | Top 1 ft | 13 | 0.2 (0.02–3700) | 2 (ND–12000) | 6 (0.08–16000) |
| Lake Washington | | | | | |
| Romberg, 1984 (1) | Top 2cm | 54 | 0.35 | 3.0 | – |
| Lake Union/Ship Canal | | | | | |
| Cubbage, (in prep) | Top 2cm | 22 | 1.9 (ND–170) | 17 (0.11–630) | 19 (0.11–800) |
| Gas Works Park, Lake Union | | | | | |
| EPA, 1985 | Top 10cm | 33 | 34 (ND–5900) | 180 (ND–25000) | 420 (ND–31000) |
| Other Lakes (2) | | | | | |
| Johnson and Norton, 1990 | Top 2cm | 20 | ND(ND) | ND(ND–1.1) | ND(ND–1.1) |

j=Estimated concentration
ND= Not detected
(1)= Reported values are geometric means
(2)= Includes: Cresent, Samish, American, Black, Wenatchee, Moses, Sprague, Kahlotus and Osoyoos.
References:
Woodward–Clyde, 1989. "Tech Memo. #1– Nearshore Sediment Sampling–J.H. Baxter Facility Renton, Wa.
EPA, 1983."Port Quendall Offshore Sediment Investigation"
Cubbage, (in prep), "Comprehensive Sediment Evaluation of Lake Union Sediments Using the Triad Approach."
EPA, 1985. "Lake Union Sediment Investigation"
Romberg et al, 1984. "Presence, Distribution and Fate of Toxicants in Puget Sound and Lake Washington."
Johnson and Norton, 1990. "Survey of Chemical Contaminants in Ten Washington Lakes."

Table 7: Results of total sulfides, metals and bioassay analysis of selected Lake Washington sediments near Quendall Terminals/J.H. Baxter site.

| Tier | Outer | | Inner | | | | |
|---|---|---|---|---|---|---|---|
| Station No. QB– | 1 | 2 | 13 | 13D | 13R | 15 | Limit |
| Property | DNR | DNR | QT | – | – | Bax | |
| Sample No. 20– | 8280 | 8281 | 8292 | 8293 | 8294 | 8296 | of |
| Depth (ft) | 40 | 40 | 11 | (Dup) | (Rep) | 9 | Tolerance |
| Total Sulfides (mg/kg) | 2.1u | 6.5u | 44.6 | 17.4 | 17.1 | 4.1u | – |
| Metals (mg/kg,dry) | | | | | | | |
| Arsenic | 18 | 7 | 9 | 8 | 4 | 4u | 33 |
| Cadmium | 0.75 | 0.29 | 0.55 | 0.80 | 0.68 | 0.19u | 10 |
| Copper | 41 | 26 | 36 | 35 | 38 | 20 | 110 |
| Lead | 89 | 54 | 36 | 38 | 33 | 31 | 250 |
| Mercury | 0.21 | 0.21 | 0.18 | 0.27 | 0.16 | 0.04 | 2 |
| Bioassay (% survival) | | | | | | | |
| Daphnia magna | 98 | 94 | 98 | – | – | 97 | – |
| Hyella azteca | 70 | 88 | 48 | – | – | 88 | – |

–=Not available
u=Not detected at detection limit shown
Limit of Tolerance= Guideline established by Ontario Ministry of Environment (Persaud et al, 1990)
i.e. Concentration of a compound that would be detrimental to the majority of benthic species
DNR= Wash. St. Dept. of Natural Resources
QT= Quendall Terminals
Bax= J.H. Baxter

concentrations of arsenic, copper and lead were present at Station 1. Cadmium and mercury concentrations were highest at Station 13. None of the metals concentrations measured exceeded Ontario Ministry of Environment guidelines for the protection of aquatic biological resources.

Toxicity of sediments at Stations 1, 2 ,13, and 15 was evaluated with two acute bioassays: *Daphnia magna*, which measures percent survival of the water flea *Daphnia magna* after 48-hours of exposure to the sample; and *Hyalella azteca* (amphipod) which also measures percent survival of the test organism, but is conducted over a 10-day period. Percent survivals for the *Daphnia* bioassay ranged from 94-98, indicating the sediments were non-toxic to *Daphnia*. Percent survival for *Hyalella azteca* ranged from 48-78. Survival at Station 13 was significantly lower than in the control, indicating these sediments were toxic to the test organism. Total PAH concentrations in this sample also exceeded Ontario Ministry of the Environment guidelines for the protection of aquatic biological resources.

The results of benthic macroinvertebrate analysis of selected Lake Washington sediments (stations 1, 2, 13, and 15) are listed in Table 8. A total of 28 taxa were identified in sediments near the two sites. Based on the Shannon-Weiner diversity index (Shannon's H'), Stations 13 and 15 clearly have a less diverse benthic community compared to Stations 1 and 2. Generally speaking, Shannon values of 3-5 indicate clean areas, 1-3 moderately polluted areas, and $<1$ substantially polluted areas (Washington, 1984). Stations 13 and 15 had Shannon H' values of 0.03 and 0.11 respectively. The results of benthic macroinvertebrate analysis and bioassay testing with *Hyalella azteca* seem to support the PAH guidelines established by the Ontario Ministry of the Environment.

## SUMMARY

The distribution of PAHs in Lake Washington sediments near Quendall Terminals/J.H. Baxter site probably originated from two types of sources: Past spills along the historical T-Pier at Quendall Terminals and onshore source(s) (via ground water and/or surface drainages) in the vicinity of the cove at the southern end of the J.H. Baxter property. The most widespread and severe PAH contamination measured during the present study, occurs in the vicinity of the historical T-Pier at Quendall Terminals.

The major findings of the present study can be summarized as follows:

- Based on data from the present study, total PAH concentrations were extremely variable throughout the area sampled, ranging from 0.8-7300 mg/kg, dry weight basis. In general, the highest levels were present in the vicinity of the historical T-Pier at Quendall Terminals.

Table 8: Results of benthic infaunal analysis of Quendall Terminals/J.H. Baxter sediments (reported as average no. of individuals per site from 5 replicates).

| Station No. QB- | 1 | 2 | 13 | 15 |
|---|---|---|---|---|
| Depth (ft) | 40 | 40 | 11 | 9 |
| Nematoda | 0.2 | 0 | 0 | 0 |
| Annelida | | | | |
| Hirudinea | 0.2 | 0.4 | 0 | 0 |
| Oligochaeta | | | | |
| Tubificidae | 2.6 | 1.6 | 0 | 0 |
| Naididae | 12.6 | 6.2 | 4.0 | 11.2 |
| Mollusca | | | | |
| Gastropoda | | | | |
| Planorbella | 0 | 0 | 0 | 0.8 |
| Bivalvia | | | | |
| Pisidium | 16.0 | 12.6 | 0.8 | 4.8 |
| Porifera | 9.0 | 4.4 | 4632.0 | 3433.6 |
| Arachnida | | | | |
| Piona | 0 | 0 | 0.8 | 0 |
| Crustacea | | | | |
| Brachiopoda | 0.6 | 0.6 | 0 | 0 |
| Copepoda | 3.2 | 0.2 | 0 | 4.8 |
| Ostracoda | 0.2 | 0.2 | 0 | 0 |
| Amphipoda | | | | |
| Hyalella azteca | 0 | 0 | 0 | 1.6 |
| Isopoda | | | | |
| Caecidotea | 0 | 0 | 0 | 1.6 |
| Mysidacea | | | | |
| Taphromysis | 0 | 0.2 | 0 | 0 |
| Insecta | | | | |
| Plecoptera | | | | |
| Perlodidae | 0 | 0.2 | 0 | 0 |
| Coleoptera | | | | |
| Psephenus | 0 | 0.2 | 0 | 0 |
| Diptera | | | | |
| Chironomidae | | | | |
| Macropelopia | 0.4 | 0.4 | 2.4 | 1.6 |
| Eukiefferiella | 0.2 | 1.4 | 0 | 0 |
| Heterotrissocladius | 2.0 | 3.0 | 0 | 0 |
| Parakiefferiella | 0 | 2.2 | 0 | 0.8 |
| Chironomus | 0 | 0.4 | 0.8 | 0 |
| Cladopelama | 0 | 0 | 0 | 4.8 |
| Cryptochironomus | 0 | 0.2 | 0 | 0 |
| Dicrotendipes | 0.6 | 1.2 | 0 | 0 |
| Einfeldia | 0 | 0.6 | 0 | 0 |
| Phaenopsectra | 0.8 | 0.6 | 0.8 | 0 |
| Polypedilum | 0 | 0 | 0 | 1.6 |
| Tanytarsus | 0.6 | 3.8 | 0 | 1.6 |
| With sponges | | | | |
| TOTALS | 49.2 | 40.6 | 4641.6 | 3468.8 |
| Number of Taxa | 15 | 21 | 7 | 12 |
| Possible number of Taxa | 28 | 28 | 28 | 28 |
| Shannon's H' | 2.66 | 3.31 | 0.03 | 0.11 |
| J (H'/H'max) | 55.4% | 68.8% | 0.5% | 2.3% |
| Swartz's index | 3 | 6 | 1 | 1 |
| Without sponges | | | | |
| TOTALS | 40.2 | 36.2 | 9.6 | 35.2 |
| Number of Taxa | 14 | 20 | 6 | 11 |
| Possible number of Taxa | 27 | 27 | 27 | 27 |
| Shannon's H' | 2.42 | 3.15 | 2.22 | 2.96 |
| J (H'/H'max) | 50.8% | 66.3% | 46.7% | 62.3% |
| Swartz's index | 3 | 5 | 3 | 5 |

- Total PAH concentrations at Station 4 and 13 along the T-Pier at Quendall Terminals are high enough to adversely affect benthic communities, based on comparisons with Ontario Ministry of Environment guidelines for the protection of aquatic biological resources. This finding is consistent with the *Hyalella azteca* bioassay and benthic macroinvertebrate tests at Station 13, which showed toxicity and reduced benthic community diversity, respectively.

- On a TOC normalized basis, PAH levels in Lake Washington sediments at Quendall Terminals exceeded 100 mg PAH/kg TOC, while concentrations at J.H. Baxter tended to be less than 100 mg PAH/kg TOC.

- Peak PAH concentrations near Quendall Terminals/J.H. Baxter site are in the same range as those near Gas Works Park (Lake Union). In addition, median concentrations near the two sites are comparable to medians for Lake Union/ Ship Canal and generally higher than values reported for Lake Washington as a whole.

- Retene, a naturally occurring resin acid-derived compound, was detected at all stations. The presence of retene in this area is, in all probability related to log rafting activities.

## RECOMMENDATIONS

- Collect additional samples for physical and chemical analyses in the vicinity of the historical T-Pier at Quendall Terminals to clarify the areal and vertical extent of PAH contamination for remediation purposes. Sampling should supplement existing data and concentrate on the area within approximately 300 feet around the T-Pier.

- Simultaneously determine TOC and PAH levels in sediments from the cove at the southern end of the J.H. Baxter site. These data would allow comparisons to be made with sediment guidelines established by the Ontario Ministry of the Environment for the protection of aquatic biological resources.

- Start a Feasibility Study to evaluate remediation options for problem sediments identified in Lake Washington adjacent to Quendall Terminals/J.H. Baxter site.

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# Appendix A1

# Appendix A1: Organics Analysis Case Narrative and Quality Assurance Review.

## MANCHESTER ENVIRONMENTAL LABORATORY

7411 Beach Drive S.E. Port Orchard, WA 98366

### CASE NARRATIVE

**September 24 , 1990**

Subject: Quendall/Baxter- Polynuclear Aromatic Hydrocarbons

Samples: 90 - 208280, 208281, 208282, 208283, 208284, 208285, 208286, 208287, 208288, 208289, 208290, 208291, 208292, 208293, 208294, 208295, 208296, 208297, 208298, 208299

Case No.: DOE- 038Z

By: Dickey D. Huntamer
Organic Analysis Unit

***POLYNUCLEAR AROMATIC HYDROCARBONS***

**ANALYTICAL METHODS:**

Semivolatile water samples were soxhlet extracted with acetone using SW 846 3520/8270 procedure with capillary GC/MS analysis of the sample extracts. Normal Manchester surrogate spike compounds were added to the sample. The sample was cleaned up using Silica Gel (SW 846 Method 3630).

**HOLDING TIMES:**

The samples were extracted within 13 days of collection and analyzed within 40 days of extraction. All recommended holding times were met.

**BLANKS:**

Polynuclear Aromatic Hydrocarbons (PAH) target compounds were not detected in the laboratory reagent blanks.

**SURROGATES:**

The normal Manchester Laboratory acid/base-neutral semivolatile surrogate compounds were added to the sample prior to extraction. Because of the silica gel cleanup performed on the samples three of the surrogates, 2-Fluorophenol, d5-Phenol and d5-Nitrobenzene were lost and only 2-Fluorobiphenyl, d14-Terphenyl and d10-Pyrene were recovered in the samples. Only d10-Pyrene is chemically related to the PAH target analytes. Surrogate spike recoveries in the highly contaminated samples,208283, 208290, 208292, 208293, 208294, 208296 and 208297 tend to be higher than the Contract Laboratory Program (CLP) QC limits for 2-Fluorobiphenyl and d14-Terphenyl. The high surrogate recoveries may be due to interference from co-eluting compounds or the effects of small errors in quantitation being magnified by the dilution factor. The d10-Pyrene surrogate recoveries should be considered as advisory only. This compound is not a CLP surrogate but is used by the Manchester Laboratory. Only two samples, the blank duplicate EBS0144D at 19% and sample 208297 at 144% were outside the

advisory QC limits of 34% to 142%. The other two surrogates were within CLP limits except for the samples which were heavily contaminated and required dilution.

## MATRIX SPIKE AND MATRIX SPIKE DUPLICATE (MS/MSD):

The matrix spike recoveries and Relative Percent Differences (RPD) were all within acceptable QC limits. Spike recoveries ranged from 23% to 116% and RPD from 0% to 12%.

## SPECIAL ANALYTICAL PROBLEMS:

Due to the high hydrocarbon content of the samples silica gel cleanup was performed to enhance the PAH analysis. Consequently the other non-PAH analytes normally seen were removed and the data was reported as NAR (No Analytical Result). Since the analysis for PAH was done by GC/MS Tentatively Identified Compounds (TIC) which co-eluted with the PAH fraction in the silica gel cleanup are also reported. These are primarily substituted PAH compounds.

One target compound, Retene, was found at high levels in a number of samples. The high concentrations may be biased high since it is very likely that other tetra methyl; dimethyl-ethyl and methyl isopropyl phenanthrenes may be included. After discussion with the Project Officer, Dale Norton, one possible explanation for the high Retene concentrations in the sediment samples could be the presence of log booms near the shore and layers of bark on the underlying sediment at some sample locations. The breakdown and decay of the bark can release resin acids (Abietic acid) and bacterial action can lead to the formation of Retene.

# *HERBICIDES*

## ANALYTICAL METHODS:

Extraction and analysis was accomplished following Manchester Lab modified EPA Method 8150 for Herbicides and Pentachlorophenol (PCP), Tetrachlorophenol and Trichlorophenol.

## BLANKS:

No significant blank contamination was found.

## SURROGATES:

No surrogate recovery limits have been established for this method. Surrogate recoveries for this sample set ranged from 10% to 81% for 2,4,6-Tribromophenol except for samples 208284 and 208297 which had 2% and 0% respectively. PCP was not detected in sample 208297 but due to the failure to recover the surrogate the quantitation limit should have the "J" flag added to show that it is estimated. Possible explanations for the low surrogate recoveries are discussed in the attached Case Narrative Note.

## HOLDING TIMES:

All sample extraction and analysis holding times were met. Samples were extracted within the seven day holding time and extracts were analyzed within the 40 day extract holding time.

## MATRIX SPIKE AND MATRIX SPIKE DUPLICATE:

Matrix spike and matrix spike duplicate recoveries were 36% and 45% respectively. The RPD was 22%. Currently there are no established QC limits for this method.

## SPECIAL ANALYTICAL PROBLEMS:

There were no significant analytical problems other than that caused by the hydrocarbon contamination present in the sample. Two factors may have contributed to the low surrogate spike recoveries. The high concentrations of hydrocarbons in the samples could have reduced the efficiency of the derivitization and this may have led to a subsequent loss of the underivitized PCP on the Florisil column.

### *POLYCHLORINATED BIPHENYLS*

## ANALYTICAL METHODS:

The sample was extracted in a soxhlet extractor using acetone as the solvent. Analysis was done by EPA Method 3520/8080 using capillary GC analysis.

## HOLDING TIMES:

All sample extraction and analysis holding times were met.

## BLANKS:

None of the laboratory blanks contained any target analytes.

## SURROGATES:

All surrogate spike recoveries were within normal QC limits.

## MATRIX SPIKE AND MATRIX SPIKE :

The matrix spikes were spiked with PCB-1242 and 1260. Matrix spike recoveries and RPD were within acceptable QC limits.

## SPECIAL ANALYTICAL PROBLEMS:

There were no significant problems with the PCB analysis.

## DATA QUALIFIERS

U - The material was analyzed for, but was not detected. The associated numerical value is the sample quantitation limit.

J - The associated numerical value is an estimated quantity.

R - The data are unusable (compound may or may not be present). Resampling and reanalysis is necessary for verification.

NAR No Analytical Result.

ND Not Detected

CASE NARRATIVE NOTE

PROJECT CODE: DOE - 0382 SAMPLE #: 90208280

TO: 90208299

PARAMETER: Pentachlorophenol (PCP)

DATE: 7-11-90

BY: RK Hammer

ANALYSIS COMMENTS:

The samples were extracted by the Manchester laboratory's herbicide extraction method. After the extraction was completed, the pentachlorophenol, PCP (or the surrogate 2,4,6-Tribromophenol, TBP) was derivatized to the methyl ester form, florisiled using the microcolumn technique (one gram of florisil eluted with 10ml 50% preserved diethyl ether in isooctane) and treated with acid. If any of the PCP or TBP was left underivatized due to lack of diazomethane or contact time, it was lost during the florisil step, thus accounting for the reduced or lost surrogate recoveries. Another mechanism causing low surrogate recoveries is a matrix constituent(s) that interferes with the derivation process. The relatively higher surrogate recoveries for the method blanks, as opposed to the samples, suggests a matrix effect is the most plausible mechanism for reduced surrogate recoveries.

Samples positive for PCP having an injected amount of between two and six picograms (pg) were given as estimated quantities (j) because of the uncertainty of values not within the calibration curve. Samples with injected amounts less than two picograms were flagged as non-detects (u) having a detection limit corresponding to about 5.5pg (10% less than the lowest calibration standard) injected.

Sample number 90208297 had no surrogate recovery. Since the sample set was relatively large and the surrogate is dispensed with a rather cumbersome 100ul syringe and only one sample had 0% surrogate recovery, the most likely exclamation for this is that no surrogate was added.

# Appendix A2

# Appendix A2: Case Narratives for *Hyallela azteca and Daphnia magna* bioassays.

DATA REVIEW

BY: Margaret Stinson 7/12/90

FOR: Quendall/Baxter Sediments, 20-8280, -81, -92, -96

DATE: July 15, 1990

Northwestern Aquatic Science has submitted the attached results of a Freshwater Amphipod Bioassay using Hyalella azteca, on sediment samples from the Quendall/Baxter Sediment Study. Survival in Sample 20-8292 was significantly different than the control survival. No significant differences in survival were observed between the other test sediments and controls. Quality Control data were appropriate for the test.

NORTHWESTERN AQUATIC SCIENCES

P.O. Box 1437, Newport, Oregon 97365 (503) 265-7225

June 26, 1990

Ms. Margaret Stinson
Washington Department of Ecology
Manchester Laboratory
P.O. Box 307
Manchester, WA 98353

Dear Margaret:

Enclosed are the following test reports and associated protocol(s):

1) Test No. 386-1, Hyalella azteca 10-day sediment toxicity test (WDOE Samples No. 20-8280, 20-8281, 20-8292, 20-8296; NAS Samples No. 3343D, 3344D, 3345D, 3346D).

2) Test No. 386-4, Hyalella azteca 96-hr reference toxicant test.

Also enclosed is an invoice (No. 6872) for these tests and a copy of the Request for Contract Laboratory Services/Chain of Custody form.

If you have any questions, please don't hesitate to call me.

Sincerely,

Dick

Richard S. Caldwell, Ph.D.
Director

Enclosures

# TEST REPORT

## TEST IDENTIFICATION

Test No.: 386-1
Title: Hyalella azteca 10-day freshwater sediment toxicity test.
Protocol No.: NAS-386-HA-1, May 24, 1990. Based on Nebeker et al. 1984. Env. Toxicol. Chem. 3:617-630.

## STUDY MANAGEMENT

Study Sponsor: Washington Department of Ecology, Manchester Laboratory, P.O. Box 346, Manchester, WA 98353.
Sponsor's Study Monitor: Ms. Margaret Stinson
Testing Laboratory: Northwestern Aquatic Sciences, P.O. Box 1437, Newport, OR 97365.
Test Location: Newport Laboratory.
Laboratory's Study Personnel: R.S. Caldwell, Ph.D., Proj. Man./Study Dir.; D.R. Buhler, Ph.D., QA Officer; Linda K. Garrison, B.A., Aq. Biol.; Valerie L. Shaffer, B.S., Aq. Biol.
Study Schedule:
Test Beginning: 5-26-90, 2:00 p.m.
Test Ending: 6-5-90, 2:00 p.m.
Disposition of Study Records: All specimens, raw data, reports and other study records are stored according to Good Laboratory Practice regulations at: Northwestern Aquatic Sciences, Yaquina Bay Rd., P.O. Box 1437, Newport, OR 97365.
Good Laboratory Practices: The test was conducted following the principles of Good Laboratory Practices (GLP) as defined in the EPA/TSCA Good Laboratory Practice regulations effective December 29, 1983 (40 CFR Part 792).

## TEST MATERIAL

Description: Four freshwater sediments: 1) WDOE # 20-8280; 2) WDOE # 20-8281; 3) WDOE # 20-8292; 4) WDOE # 20-8296.
Dates of Collection: Samples were collected on 5-16-90 and 5-17-90.

## DILUTION WATER

Source: NAS spring water.
Date of Collection: 5-25-90.
Pretreatment: Filtered, aerated.

## TEST ORGANISMS

Species: Hyalella azteca, amphipod.
Age: Adult
Source: NAS culture; stocks were originally purchased from ESE, Gainesville, FL.
Acclimation: Temperature, 24.4 $\pm$ 0.4°C; Dissolved oxygen, 7.7 $\pm$ 0.3 mg/L; pH, 7.5 $\pm$ 0.2; and conductivity, 284 $\pm$ 15 µmhos/cm for the two week period prior to testing.

## TEST PROCEDURES AND CONDITIONS

Test Chambers: 1 L borosilicate glass beakers.
Test Volumes: 200 ml of test sediment and 800 ml of dilution water per beaker.
Replicates/Treatment: 5
Organisms/Treatment: 50

Water Volume Changes per 24 hr: None.
Effects Criteria: Mortality, defined as the lack of movement of body or appendages in response to tactile stimulation.
Water Quality and Other Test Conditions: Temperature, 20.2 ± 0.4°C; dissolved oxygen, 8.6 ± 0.5 mg/L; pH, 7.8 ± 0.3; conductivity, 247 ± 49 µmhos/cm; hardness, 64 ± 8 mg/L as $CaCO_3$; and alkalinity, 46 ± 21 mg/L as $CaCO_3$. Photoperiod 16:8 hr, L:D.

## DATA ANALYSIS METHODS

ANOVA and Dunnett's test (Dunnett 1957, Weber et al. 1988) were used to compare the means survival between each test sediment and the control. An arcsine transformation was performed on the survival data prior to statistical analysis. The statistical software employed for these calculations was Crunch Statistical Package (Crunch Software Corp.).

## PROTOCOL DEVIATIONS

None

## TEST RESULTS

A detailed tabulation of the test results is given in Table 1. Survival in WDOE Sample No. 20-8292 was significantly ($P<0.05$) less than in the control. Survival was not significantly ($P<0.05$) less for any of the remaining test sediments compared to the control sediment.

## STUDY APPROVAL

Richard D. Caldwell 6/25/90

Study Director Date

Table 1. Survival of Hyalella azteca exposed for ten days to freshwater sediments in Test No. 386-1.

| Sample No. | Repl. | Number of amphipods | | Percent survival | Mean* percent survival |
|---|---|---|---|---|---|
| | | Exposed | Surviving | | |
| Control | 1 | 10 | 10 | 100.0 | |
| | 2 | 10 | 7 | 70.0 | |
| | 3 | 10 | 9 | 90.0 | |
| | 4 | 10 | 10 | 100.0 | |
| | 5 | 10 | 9 | 90.0 | 90.0 |
| 20-8280 | 1 | 10 | 5 | 50.0 | |
| | 2 | 10 | 6 | 60.0 | |
| | 3 | 10 | 8 | 80.0 | |
| | 4 | 10 | 9 | 90.0 | |
| | 5 | 10 | 7 | 70.0 | 70.0 |
| 20-8281 | 1 | 10 | 10 | 100.0 | |
| | 2 | 10 | 10 | 100.0 | |
| | 3 | 10 | 10 | 100.0 | |
| | 4 | 10 | 6 | 60.0 | |
| | 5 | 10 | 8 | 80.0 | 88.0 |
| 20-8292 | 1 | 10 | 3 | 30.0 | |
| | 2 | 10 | 2 | 20.0 | |
| | 3 | 10 | 5 | 50.0 | |
| | 4 | 10 | 10 | 100.0 | |
| | 5 | 10 | 4 | 40.0 | 48.0* |
| 20-8296 | 1 | 10 | 9 | 90.0 | |
| | 2 | 10 | 9 | 90.0 | |
| | 3 | 10 | 9 | 90.0 | |
| | 4 | 10 | 9 | 90.0 | |
| | 5 | 10 | 8 | 80.0 | 88.0 |

* An asterisk (*) next to the treatment mean indicates that the latter was significantly ($P<0.05$) different from the control mean.

# TEST PROTOCOL

## 96-HR REFERENCE TOXICANT TEST USING THE AMPHIPOD HYALELLA AZTECA AND CADMIUM CHLORIDE

1. INTRODUCTION

1.1 Purpose of Study:
The purpose of this test is to measure the toxicity of cadmium as cadmium chloride using the amphipod, Hyalella azteca.

1.2 Summary of Method:
The test is performed using adult amphipods, Hyalella azteca, obtained from a biological supplier or laboratory culture. This non-renewal test employs a 96-hr static exposure to 1000 ml of test solution in 1 L beakers. A standard experimental design is employed consisting of exposure of the test animals to five cadmium concentrations, and a dilution water control. Two replicate vessels, each containing 10 amphipods, are used at each test level. Mortality is the effect criterion. The data analysis includes caiculation of the LC50 and the determination of a NOEC and LOEC.

2. STUDY MANAGEMENT

2.1 Sponsor's Name and Address:
Washington Department of Ecology
P.O. Box 307
Manchester, WA 98353

2.2 Sponsor's Study Monitor:
Ms. Margaret Stinson

2.3 Name of Testing Laboratory:
Northwestern Aquatic Sciences
Yaquina Bay Road
P.O. Box 1437
Newport, OR 97365.

2.4 Test Location:
Tests will be performed at the firm's laboratory at Newport, OR.

2.5 Laboratory's Key Personnel to be Assigned to the Study:
Project Manager/Technical Director: Richard S. Caldwell, Ph.D.
Quality Assurance Officer: Donald R. Buhler, Ph.D.
Aquatic Biologist: Linda K. Garrison, B.A.
Aquatic Biologist: Valerie Shaffer, B.S.

2.6 Proposed Study Schedule: Test to begin either during or within two weeks of the actual sample test.

2.7 Good Laboratory Practices:
The tests are conducted following the principles of Good Laboratory Practices (GLP) as defined in the EPA/TSCA Good Laboratory Practice regulations effective December 29, 1983 (40 CFR Part 792).

3. TEST MATERIAL:

Cadmium as cadmium chloride. A 1.0 mg/ml stock solution is used to make the concentrations daily.

4. TEST WATER:

The test water is laboratory spring water.

5. TEST ORGANISMS:

5.1 Source:
Cultured at NAS. Originally purchased from ESE in Gainesville, FL.

5.2 Age:
Adults

5.3 Acclimation and Pretest Observation:
No special procedures employed. Cultures are maintained at 20-25°C under a 16:8 L:D photoperiod. The culture water is NAS spring water (Hardness and alkalinity approximately 80-120 and 20-50 mg/L as $CaCO_3$, respectively). Amphipods are fed dried maple leaves with occasional Tetramin® flake supplements until 24 hours prior to testing. Mortality during the 48-hr prior to testing should not exceed 5%.

6. DESCRIPTION OF TEST SYSTEM:

6.1 Test Chambers and Environmental Control:

Test chambers used in the toxicity test are 1 liter Pyrex beakers holding 1000 ml of test solution. Test chambers are maintained at constant temperature by partial immersion in a temperature-controlled water bath. Test containers are not aerated during the test. The system is maintained in a photoperiod-controlled room or enclosure.

6.2 Cleaning:
All laboratory glassware, including test chambers, is cleaned as described in ASTM Standard No. E-729 (ASTM 1980), paragraph 6.6. New glassware and test systems are washed with laboratory detergent, followed by rinses with tap water, pesticide-free acetone, water, 5% nitric or hydrochloric acid, and twice with tap water. Following every test, reusable glassware is rinsed with water, cleaned using a procedure appropriate for removing the toxicant tested (i.e., acid to remove metals, bases, and mineral deposits; detergent and pesticide-free acetone to remove organic compounds; and a 200 mg/L solution of hypochlorite to aid in removal of organic matter and to disinfect glassware), and rinsed twice with tap water. Test systems and chambers are rinsed again with dilution water just before use.

7. EXPERIMENTAL DESIGN AND TEST PROCEDURES:

7.1 Experimental Design:
The test involves exposure of amphipods to five cadmium concentrations in a logarithmic series and a dilution water control.

Exposures are for 96 hours. Each treatment consists of two replicate test containers each containing 10 amphipods. A stratified random design is used for the placement of beakers in the water bath. Test organisms are impartially distributed to the test chambers by adding one or two animals to each chamber and repeating the process until each contains 10 organisms.

7.2 Effect Criterion:
The effect criterion used in the amphipod bioassay is mortality, defined as the lack of movement of body or appendages on response to tactile stimulation.

7.3 Test Conditions:
The dissolved oxygen concentration in each test container must be greater than 60% saturation throughout the 96-hr test. The test temperature employed is 20 ± 2°C. A 16:8, L:D photoperiod is used. Illumination is supplied by daylight fluorescent lamps. The test solution is not replaced throughout the test.

7.4 Beginning the Test:
The test is begun by adding the organisms to the equilibrated test containers as previously described.

7.5 Feeding:
Animals are not fed during the test period.

7.6 Test Duration, Type and Frequency of Observations, and Methods:
The duration of the acute toxicity test is 96-hours. The type and frequency of observations to be made are summarized as follows:

| Type of Observation | Times of Observation |
|---|---|
| Biological Data | |
| Survival | At end of test. |
| Physical and Chemical Data | |
| Dissolved oxygen, pH, temperature, and conductivity (in one replicate of each test level and the control) | Daily |
| Hardness and alkalinity (in one replicate of the highest concentration and control) | Beginning and end of test |

Dissolved oxygen is measured directly in test beakers using a polarographic oxygen probe calibrated in air. The pH is measured using a properly calibrated meter with scale divisions of 0.1 pH units. Temperature is measured using a mercury thermometer or a telethermometer calibrated with a mercury thermometer. Conductivity, hardness, and alkalinity are measured using EPA approved or suggested methods (EPA 1979).

7.7 Criteria of Test Acceptance:
For the test to be considered acceptable, the minimum survival of organisms in the control treatment at the end of the test must be

90%.

8. DATA ANALYSIS

Percent survival is calculated for each treatment replicate from the raw data and the means are obtained for each treatment level. The LC50 is calculated using the EPA TOXDAT Multi-Method program (EPA 600/4-85/013). NOEC and LOEC values for survival are computed using ANOVA and Dunnett's test (Dunnett 1957, Weber et al. 1988). An arcsine transformation is performed prior to statistical analysis.

9. REPORTING

A report of the test results must include the following information: name and identification of the test; the investigator and laboratory; information on the test material; information on the test water; detailed information about the test organisms including acclimation conditions; a description of the experimental design and test chambers and other test conditions including water quality; information about any aeration that may have been required; definition of the effect criteria and other observations; responses, if any, in the control treatment; the concentration, if any, at which survival is less than in the control; any unusual information about the test or deviations from procedures.

10. STUDY DESIGN ALTERATION:

Amendments made to the protocol must be approved by the sponsor and study director and should include a description of the change, the reason for the change, the date the change took effect, and the dated signatures of the study director and sponsor. Any deviations in the protocol must be described and recorded in the study raw data.

11. REFERENCE PROCEDURES:

ASTM. 1980. Standard practice for conducting acute toxicity tests with fishes, macroinvertebrates and amphibians. ASTM Standard Method No. E-729.

Dunnett, C.W. 1957. New tables for multiple comparisons with a control. Biometrics 20:482-491.

EPA. 1979. Methods for chemical analysis of water and wastes. Environmental Protection Agency, Cincinnati. EPA 600/4-79-020.

Nebeker, A.V. et al. 1984. Biological methods for determining toxicity of contaminated freshwater sediments to invertebrates. Environ Toxicol. Chem. 3:617-630.

Nebeker, A.V., and C.E. Miller. 1988. Use of the amphipod crustacean Hyalella azteca in freshwater and estuarine sediment toxicity tests. Environ. Toxicol. Chem. 7:1027-1033.

Peltier, W.H. and C.I. Weber. 1985. Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms (Third Edition). EPA/600-4-85-013.

Weber, C.E., et al. 1988. Short-term methods for estimating the chronic toxicity of effluents and receiving waters to marine and estuarine organisms. EPA-600/4-87/028.

13. APPROVALS:

| Name | Date | |
|---|---|---|
| | | for Washington Department of Ecology |
| Rod D. Caldwell | 6/2/90 | for Northwestern Aquatic Sciences |

# TEST REPORT

## TEST IDENTIFICATION

Test No.: 386-4

Title: Hyalella azteca 96-hr reference toxicant test using cadmium as cadmium chloride.

Protocol No.: NAS-386-HA-2, June 2, 1990. Based on Nebeker et al. 1984. Env. Toxicol. Chem. 3:617-630 and EPA/600/4-85-013.

## STUDY MANAGEMENT

Study Sponsor: Washington Department of Ecology, Manchester Laboratory, P.O. Box 346, Manchester, WA 98353.

Sponsor's Study Monitor: Ms. Margaret Stinson

Testing Laboratory: Northwestern Aquatic Sciences, P.O. Box 1437, Newport, OR 97365.

Test Location: Newport Laboratory.

Laboratory's Study Personnel: R.S. Caldwell, Ph.D., Proj. Man./Study Dir.; D.R. Buhler, Ph.D., QA Officer; Linda K. Garrison, B.A., Aq. Biol.; Valerie L. Shaffer, B.S., Aq. Biol.

Study Schedule:

Test Beginning: 6-2-90, 12:40 p.m.

Test Ending: 6-6-90, 11:30 a.m.

Disposition of Study Records: All specimens, raw data, reports and other study records are stored according to Good Laboratory Practice regulations at: Northwestern Aquatic Sciences, Yaquina Bay Rd., P.O. Box 1437, Newport, OR 97365.

Good Laboratory Practices: The test was conducted following the principles of Good Laboratory Practices (GLP) as defined in the EPA/TSCA Good Laboratory Practice regulations effective December 29, 1983 (40 CFR Part 792).

## TEST MATERIAL

Description: Cadmium as cadmium chloride. $CdCl_2$-$2.5H_2O$ stock solution (1.00 mg/ml as Cd) prepared 12-11-89. The stock solution was used daily to prepare test solutions.

## DILUTION WATER

Source: NAS spring water.

Date of Collection: 6-2-90.

Pretreatment: Filtered, aerated.

## TEST ORGANISMS

Species: Hyalella azteca, amphipod.

Age: Adult

Source: NAS culture; stocks were originally purchased from ESE, Gainesville, FL.

Acclimation: Temperature, 24.4 ± 0.4°C; Dissolved oxygen, 7.8 ± 0.4 mg/L; pH, 7.6 ± 0.3; and conductivity, 287 ± 11 µmhos/cm for the two week period prior to testing.

## TEST PROCEDURES AND CONDITIONS

Test Chambers: 1 L borosilicate glass beakers.

Test Concentrations: 10, 3.3, 1.0, 0.33, 0.10, and 0.0 µg/L.

Replicates/Treatment: 2

Organisms/Treatment: 20

Water Volume Changes per 24 hr: None.
Effects Criteria: Mortality, defined as the lack of movement of body or appendages in response to tactile stimulation.
Water Quality and Other Test Conditions: Temperature, 20.6 ± 0.2°C; dissolved oxygen, 8.9 ± 0.4 mg/L; pH, 7.7 ± 0.2; conductivity, 249 ± 19 µmhos/cm; hardness, 60 ± 0 mg/L as $CaCO_3$; alkalinity, 30 ± 5 mg/L as $CaCO_3$. Photoperiod 16:8 hr, L:D.

## DATA ANALYSIS METHODS

Percent survival was calculated for each treatment replicate from the raw data and the means were obtained for each treatment level. The LC50 was calculated using the EPA TOXDAT Multi-Method program (*EPA 600/4-85/013*). NOEC and LOEC values for survival were computed using ANOVA and Dunnett's test (Dunnett 1957, Weber et al. 1988). An arcsine transformation was performed prior to statistical analysis. The statistical software employed for these calculations was Crunch Statistical Package (Crunch Software Corp.).

## PROTOCOL DEVIATIONS

One replicate at 10 µg/L failed to receive animals at the initiation of the test.

## TEST RESULTS

A detailed tabulation of the test results is given in Table 1. The biological effects given as the NOEC, LOEC, and LC50 are as shown below.

| | |
|---|---|
| NOEC (ug/L) | 1.0 |
| LOEC (ug/L) | 3.3 |
| LC50 (ug/L) | 0.91 |
| (95% C.I.) | (0.64-1.41) |
| Method | moving average |

STUDY APPROVAL

Rick D. Caldwell 6/25/90

Study Director Date

Table 1. Survival of Hyalella azteca exposed for 96-hr to cadmium as cadmium chloride in Test No. 386-4.

| Cadmium concentration (µg/L) | Repl. | Number of amphipods | | Percent survival | Mean* percent survival |
|---|---|---|---|---|---|
| | | Exposed | Surviving | | |
| 10 | 1 | -- | -- | -- | |
| | 2 | 10 | 0 | 0.0 | 0.0* |
| 3.3 | 1 | 9 | 0 | 0.0 | |
| | 2 | 8 | 0 | 0.0 | 0.0* |
| 1.0 | 1 | 8 | 4 | 50.0 | |
| | 2 | 7 | 7 | 100.0 | 75.0 |
| 0.33 | 1 | 9 | 6 | 66.7 | |
| | 2 | 9 | 9 | 100.0 | 83.3 |
| 0.10 | 1 | 9 | 9 | 100.0 | |
| | 2 | 10 | 9 | 90.0 | 95.0 |
| Control | 1 | 10 | 8 | 80.0 | |
| | 2 | 10 | 10 | 100.0 | 90.0 |

* An asterisk (*) next to the treatment mean indicates that the latter was significantly ($P<0.05$) different from the control mean.

# TEST PROTOCOL

## STATIC 10-DAY FRESHWATER SEDIMENT TOXICITY TEST USING THE AMPHIPOD HYALELLA AZTECA

### 1. INTRODUCTION

1.1 Purpose of Study:
The purpose of this test is to measure the toxicity of freshwater sediments using the amphipod, Hyalella azteca.

1.2 Summary of Method:
The 10-day static test is performed using adult amphipods, Hyalella azteca, obtained from a biological supplier or laboratory culture. Clean sand is obtained from an uncontaminated stream bed for use as the control sediment. Test or control sediments (200 ml) are placed in the bottom of 1 liter borosilicate glass beakers used as test vessels to which are added 800 ml of clean test water. Five replicate containers of each test and control sediment, each containing 10 test organisms, are employed. Cumulative mortality after 10 days is the response criterion used. Between-treatment comparisons are made using an analysis of variance, and Dunnett's post hoc test is used to compare individual test sediments with the control. Test observations are made daily.

### 2. STUDY MANAGEMENT

2.1 Sponsor's Name and Address:
Washington Department of Ecology
P.O. Box 307
Manchester, WA 98353

2.2 Sponsor's Study Monitor:
Ms. Margaret Stinson

2.3 Name of Testing Laboratory:
Northwestern Aquatic Sciences
Yaquina Bay Road
P.O. Box 1437
Newport, OR 97365.

2.4 Test Location:
Tests will be performed at the firm's laboratory at Newport, OR.

2.5 Laboratory's Key Personnel to be Assigned to the Study:
Project Manager/Technical Director: Richard S. Caldwell, Ph.D.
Quality Assurance Officer: Donald R. Buhler, Ph.D.
Aquatic Biologist: Linda K. Garrison, B.A.
Aquatic Biologist: Valerie Shaffer, B.S.

2.6 Proposed Study Schedule: Test to begin May 26, 1990.

2.7 Good Laboratory Practices:
The tests are conducted following the principles of Good Laboratory Practices (GLP) as defined in the EPA/TSCA Good Laboratory Practice regulations effective December 29, 1983 (40 CFR Part 792).

3. TEST MATERIAL:

Unidentified freshwater test sediments.

4. TEST WATER:

The test water is laboratory spring water.

5. TEST ORGANISMS:

5.1 Source:
Cultured at NAS. Originally purchased from ESE in Gainesville, FL.

5.2 Age:
Adults

5.3 Acclimation and Pretest Observation:
No special procedures employed. Cultures are maintained at 20-25°C under a 16:8 L:D photoperiod. The culture water is NAS spring water (Hardness and alkalinity approximately 80-120 and 20-50 mg/L as $CaCO_3$, respectively). Amphipods are fed dried maple leaves with occasional Tetramin® flake supplements until 24 hours prior to testing. Mortality during the 48-hr prior to testing should not exceed 5%.

6. DESCRIPTION OF TEST SYSTEM:

6.1 Test Chambers and Environmental Control:
Test chambers used in the toxicity test are 1 liter Pyrex beakers holding a 200 ml layer of test or control sediment and 800 ml of test water. Test chambers are maintained at constant temperature by partial immersion in a temperature-controlled water bath. Minimal aeration is supplied to avoid oxygen depletion. The system is maintained in a photoperiod-controlled room or enclosure.

6.2 Cleaning:
All laboratory glassware, including test chambers, is cleaned as described in ASTM Standard No. E-729 (ASTM 1980), paragraph 6.6. New glassware and test systems are washed with laboratory detergent, followed by rinses with tap water, pesticide-free acetone, water, 5% nitric or hydrochloric acid, and twice with tap water. Following every test, reusable glassware is rinsed with water, cleaned using a procedure appropriate for removing the toxicant tested (i.e., acid to remove metals, bases, and mineral deposits; detergent and pesticide-free acetone to remove organic compounds; and a 200 mg/L solution of hypochlorite to aid in removal of organic matter and to disinfect glassware), and rinsed twice with tap water. Test systems and chambers are rinsed again with dilution water just before use.

## 7. EXPERIMENTAL DESIGN AND TEST PROCEDURES:

### 7.1 Experimental Design:

The test involves exposure of amphipods to test and control sediments. The sediments are placed on the bottom of the test containers and are overlain with test water. The test exposure is for 10 days. Neither sediments nor test water are replaced during the test. Each treatment consists of five replicate test containers, each containing 10 amphipods. Randomization of test containers in the water bath involves random assignment of one test chamber for each treatment in one row, followed by random assignment of a second test chamber for each treatment in a second row and so on until all rows have been placed. Test organisms are impartially distributed to the test chambers by adding one or two animals to each chamber and repeating the process until each contains 10 organisms.

### 7.2 Setup of Test Containers:

Sediment (200 ml) is placed into each of five replicate beakers. After addition of the sediment, 800 ml of test water is gently poured into each beaker, bringing the total contents to 1000 ml. Beakers should be left unaerated overnight to reduce turbidity and to allow more time for water-sediment contact prior to addition of test animals. Before adding animals, the beakers should be aerated for 30 min using glass-tipped plastic airlines. Gentle aeration with the tip 3 cm below the water surface is used to aerate the water, avoiding any disturbance of the sediment.

### 7.3 Effect Criterion:

The effect criterion used in the amphipod bioassay is mortality, defined as the lack of movement of body or appendages on response to tactile stimulation.

### 7.4 Test Conditions:

The dissolved oxygen concentration in each test container must be greater than 60% saturation throughout the 10-day test. Test containers are gently aerated to maintain the oxygen level. The test temperature employed is 20 $\pm$ 2°C. A 16:8, L:D photoperiod is used. Illumination is supplied by daylight fluorescent lamps. The test solution is not replaced throughout the test.

### 7.5 Beginning the Test:

The test is begun by adding the organisms to the equilibrated test containers as previously described.

### 7.6 Feeding:

Animals are fed 0.1 g of crushed (1-5 mm) dried maple leaves per beaker at the beginning of the test and not fed again unless all of the food is eaten.

### 7.7 Test Duration, Type and Frequency of Observations, and Methods:

The duration of the acute toxicity test is 10 days. The type and frequency of observations to be made are summarized as follows:

| Type of Observation | Times of Observation |
|---|---|
| **Biological Data** | |
| Survival | Day 10 |
| **Physical and Chemical Data** | |
| Dissolved oxygen, pH, temperature, and conductivity (in one replicate of each test sediment and control) | Daily |
| Hardness and alkalinity (in one replicate of each test sediment and control) | Beginning and end of test |

Dissolved oxygen is measured directly in test aquaria using a polarographic oxygen probe calibrated in air. The pH is measured using a properly calibrated meter with scale divisions of 0.1 pH units. Temperature is measured using a mercury thermometer or a telethermometer calibrated with a mercury thermometer. Conductivity, hardness, and alkalinity are measured using EPA approved or suggested methods (EPA 1979).

7.8 Criteria of Test Acceptance:
For the test to be considered acceptable, the minimum survival of organisms in the control treatment at the end of the test must be 90%.

## 8. DATA ANALYSIS

ANOVA and Dunnett's test (Dunnett 1957, Weber et al. 1988) are used to compare the survival between any test sediment and the control. If only one test sediment is being employed, Student's t test may be used to make the comparison.

## 9. REPORTING

A report of the test results must include the following information: name and identification of the test; the investigator and laboratory; information on the test material; information on the test water; detailed information about the test organisms including acclimation conditions; a description of the experimental design and test chambers and other test conditions including water quality; information about any aeration that may have been required; definition of the effect criteria and other observations; responses, if any, in the control treatment; the concentration, if any, at which survival is less than in the control; any unusual information about the test or deviations from procedures.

## 10. STUDY DESIGN ALTERATION:

Amendments made to the protocol must be approved by the sponsor and study director and should include a description of the change, the reason for the change, the date the change took effect, and the dated signatures of the study director and sponsor. Any deviations in the protocol must be described and recorded in the study raw data.

11. REFERENCE TOXICANT

Reference toxicant testing should be included with each study as defined in the Quality Assurance Program of the laboratory.

12. REFERENCE PROCEDURES:

ASTM. 1980. Standard practice for conducting acute toxicity tests with fishes, macroinvertebrates and amphibians. ASTM Standard Method No. E-729.

Dunnett, C.W. 1957. New tables for multiple comparisons with a control. Biometrics 20:482-491.

EPA. 1979. Methods for chemical analysis of water and wastes. *Environmental Protection Agency, Cincinnati. EPA 600/4-79-020.*

Nebeker, A.V. et al. 1984. Biological methods for determining toxicity of contaminated freshwater sediments to invertebrates. Environ Toxicol. Chem. 3:617-630.

Nebeker, A.V., and C.E. Miller. 1988. Use of the amphipod crustacean Hyalella azteca in freshwater and estuarine sediment toxicity tests. Environ. Toxicol. Chem. 7:1027-1033.

Peltier, W.H. and C.I. Weber. 1985. Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms (Third Edition). EPA/600-4-85-013.

Weber, C.E., et al. 1988. Short-term methods for estimating the chronic toxicity of effluents and receiving waters to marine and estuarine organisms. EPA-600/4-87/028.

13. APPROVALS:

| | |
|---|---|
| _____ | for Washington Department of Ecology |
| Name Date | |
| 5-24-90 | for Northwestern Aquatic Sciences |
| Name Date | |

WASHINGTON STATE DEPARTMENT OF ECOLOGY
ENVIRONMENTAL INVESTIGATIONS AND LABORATORY SERVICES
MANCHESTER LABORATORY

M E M O R A N D U M

TO: Dale Norton

FROM: Margaret Stinson

SUBJECT: Quendall/Baxter
Results of Daphnia magna Sediment Bioassays

DATE: June 22, 1990

## Introduction

Sediment samples were collected at four sites near the Quendall/Baxter site on Lake Union. Sediment toxicity was assessed using Daphnia magna and Hyalella azteca solid phase tests. This report describes the results of a 48-hour test of survival using Daphnia magna.

The samples were identified as follows:

| | |
|---|---|
| Sample No. 20-8280: | "QB-1; DN; 5/16/90"<br>Grey-brown silty mud |
| Sample No. 20-8281: | "QB-2; DN; 5/16/90"<br>Grey-brown silty mud |
| Sample No. 20-8292: | "QB-13; DN; 5/17/90"<br>Grey-brown mud with oily sheen |
| Sample No. 20-8296: | "QB-15; DN; 5/17/90"<br>Brown mud with some organic material |

## Methods

Samples were collected May 16-17, 1990, and were held on ice until received at Manchester Laboratory May 18, 1990.

Tests were conducted following the method of Nebeker, et al. (1984). Test organisms were from a culture maintained at Manchester Laboratory.

Each sample was thoroughly homogenized and approximately 200 ml of sediment was placed in each of five 1-Liter Pyrex beakers. The beakers were then carefully filled to 950 ml with dechlorinated Manchester city water that had been aerated for four hours. Five beakers of 1000 ml dechlorinated water were prepared as controls. Test containers were covered with watchglasses and placed in random order in a 20 degree C environmental chamber overnight to equilibrate. The following day aeration was initiated two hours before introduction of test organisms.

To initiate the test, ten 5-day-old Daphnia were isolated and added to each replicate beaker. The water/sediment volume was adjusted to 1000 ml. Dissolved oxygen and pH were measured for each sample at initiation and termination of the test.

Cadmium chloride, provided by EPA/EMSL Cincinnati, was used as reference toxicant for the test. Three replicates were prepared for each concentration, using dechlorinated Manchester city water and no sediment.

After 48-hours, the Daphnids were removed from the test beakers, counted, and number of survivors was recorded. A sample was collected from the water fraction to analyze for hardness, alkalinity, and conductivity. The test began on May 25, and was terminated May 27, 1990.

Data were analyzed using software provided by the EPA Biological Methods Branch, Cincinnati, Ohio. Survival data were tested using analysis of variance (of arcsine square root transformed data) followed by Dunnett's Test. The LC50 for the reference toxicant was estimated graphically.

## Results

Survival ranged from 94% to 98% in the sediment samples, and was 100% in the control. Using Dunnett's test, survival was not statistically different from the control in any of the samples.

The estimated LC50 for the cadmium chloride reference toxicant was 17.5 ug/L. This is slightly above the upper end of the range of values normally observed in this laboratory for Daphnia magna. Water quality measurements were appropriate for survival of the test organisms throughout the test.

Test results and water chemistry data are tabulated in Table 1. Printouts from statistical analyses are in Appendix I.

Table 1. Results of bioassays on sediments from Quendall/Baxter, using *Daphnia magna*.

| Sample | Survivors Replicate 1 | 2 | 3 | 4 | 5 | Percent Survival | pH 0-Hour | pH 48-Hour | Dissolved Oxygen (mg/L) 0-Hour | Dissolved Oxygen (mg/L) 48-Hour | Alkalinity (mg/L CaCO3) 48-Hour | Hardness (mg/L CaCO3) 48-Hour | Conductivity (umhos/cm) 48-Hour |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 20-8280 | 10 | 10 | 10 | 9 | 10 | 98.0 | 7.50 | 7.82 | 6.65 | 7.8 | 52 | 47 | 193 |
| 20-8281 | 8 | 10 | 9 | 10 | 10 | 94.0 | 7.62 | 8.03 | 7.40 | 8.2 | 63 | 54 | 204 |
| 20-8292 | 10 | 10 | 10 | 9 | 10 | 98.0 | 7.62 | 7.80 | 7.60 | 7.7 | 61 | 51 | 215 |
| 20-8296 | 20* | 8 | 10 | 10 | 10 | 96.7 | 7.21 | 7.96 | 7.00 | 8.1 | 75 | 58 | 217 |
| Control | 10 | 10 | 10 | 10 | 10 | 100.0 | 8.22 | 8.17 | 8.55 | 8.5 | 79 | 64 | 241 |

* Test organisms apparently introduced twice

# References

Nebeker, Alan V., Michael A. Cairns, Jack H. Gakstatter, Kenneth W. Malueg, Gerald S. Schuytema, and Daniel F. Krawczyk. 1984. "Biological Methods for Determining Toxicity of Contaminated Freshwater Sediments to Invertebrates." Environ. Toxicol. and Chem., Vol. 3, pp. 617-630.

# APPENDIX I

Quendall/Baxter, Daphnia magna sediment test: 20-8280 - 20-8296

Summary Statistics for Raw Data

| Group | n | Mean | s.d. | cv% |
|---|---|---|---|---|
| 1 = control | 5 | 1.0000 | .0000 | .0 |
| 2 | 5 | .9400 | .0894 | 9.5 |
| 3 | 5 | .9800 | .0447 | 4.6 |
| 4 | 5 | .9800 | .0447 | 4.6 |
| 5 | 1 | .9670 | .0000 | .0 |

Quendall/Baxter, Daphnia magna sediment test: 20-8280 - 20-8296

Summary Statistics and ANOVA

Transformation = Arcsine Square Root

| Group | n | Mean | s.d. | cv% |
|---|---|---|---|---|
| 1 = control | 5 | 1.5708 | .0000 | .0 |
| 2 | 5 | 1.4137 | .2209 | 15.6 |
| 3 | 5 | 1.5064 | .1439 | 9.6 |
| 4 | 5 | 1.5064 | .1439 | 9.6 |
| 5 | 1 | 1.3881 | .0000 | .0 |

*) the mean for this group is significantly less than the control mean at alpha = 0.05 (1-sided) by a t - test with Bonferroni adjustment of alpha level

Minumum detectable difference for
t-tests with Bonferroni adjustment = -.234899
This corresponds to a difference of -.054170 in original units
This difference corresponds to -5.42 percent of control

```
**************************************************
*                                                *
* Note - the above value for the minimum         *
* detectable difference is approximate as        *
* the sample sizes are not the same for all of   *
* the groups.                                    *
*                                                *
**************************************************
```

Between groups sum of squares = .074474 with 4 degrees of freedom.

Error mean square = .022547 with 16 degrees of freedom.

```
**************************************************
*                                                *
* Warning - the test for equality of variances   *
* could not be computed as 1 or more of the      *
* variances is zero.                             *
*                                                *
**************************************************
```

# Appendix A3

Appendix A3: Summary of tentatively identified semivolatile organics detected in Quendall Terminals/J.H. Baxter sediments (mg/kg, dry)
(all values shown are estimated concentrations only, based on presumptive evidence of material)

| Tier | Outer | | | Middle | | | | | | | | Inner | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Station No. QB- | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | - | - | 14 | 15 | 16 | 17 | 18 |
| Property | DNR | DNR | DNR | DNR | DNR | DNR | DNR | DNR | DNR | DNR | DNR | QT | QT | - | - | QT | Bax | Bax | Bax | Bax |
| Sample No. 20- | 8280 | 8281 | 8282 | 8283 | 8284 | 8285 | 8286 | 8287 | 8288 | 8289 | 8290 | 8291 | 8292 | 8293 | 8294 | 8295 | 8296 | 8297 | 8298 | 8299 |
| Depth (ft) | 40 | 40 | 47 | 26 | 20 | 20 | 22 | 23 | 21 | 24 | 33 | 12 | 11 | (Dup) | (Rep) | 10 | 9 | 10 | 10 | 10 |
| **Substituted PAH's** | | | | | | | | | | | | | | | | | | | | |
| Naphthalenes | - | 3.9 | 1.2 | 160 | 4.9 | 0.76 | 5.2 | 0.47 | 4.4 | 4.7 | 3.7 | 13 | 84 | 62 | 3.6 | - | 12 | - | - | 0.24 |
| Fluorenes | - | - | - | 18 | - | 2.8 | - | - | - | - | - | 1.9 | 20 | - | - | - | - | - | - | - |
| Anthracenes | - | - | - | - | - | 0.41 | - | - | - | - | - | 4.1 | 43 | - | - | - | - | - | - | - |
| Phenanthrenes | - | 2.3 | 1.2 | 160 | 12 | 1.2 | - | 2.5 | 9.3 | 21 | 0.90 | 9.3 | 26 | 17 | 25 | 0.13 | 5.1 | 1.5 | - | 1.9 |
| Fluoranthenes | - | - | - | 18 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| Chrysenes | - | - | - | 3.7 | - | 3.3 | - | - | - | - | - | 5.3 | 21 | 28 | 5.8 | - | - | - | - | - |
| Pyrenes | - | - | - | 33 | - | 3.2 | - | - | - | - | - | 4.7 | 72 | 36 | 9.4 | - | - | - | - | - |
| **Non-Substituted PAH's** | | | | | | | | | | | | | | | | | | | | |
| Dihydro-naphthacene | - | - | - | 23 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| Benzo(a)fluorene | - | - | - | 49 | - | 1.0 | - | - | - | - | - | - | 20 | 11 | 5.9 | - | - | - | - | - |
| Benzo(b)fluorene | - | - | - | - | - | 1.8 | - | - | - | - | - | 4.7 | - | - | 2.1 | - | - | - | - | - |
| Dihydroanthracene | - | - | - | 27 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| Benzo(c)phenanthrene | - | - | - | - | - | 0.71 | - | - | - | - | - | 3.0 | - | - | - | - | - | - | - | - |
| Cyclopenta(DEF)phenanthrene | - | - | - | - | - | 0.61 | - | - | - | - | - | 3.8 | - | - | - | - | - | - | - | - |
| Benzo(j)fluoranthene | - | - | - | 18 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| Benzo(g,h,i)fluoranthene | - | - | - | - | - | 0.53 | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| Benzo(e)pyrene | 1.2 | 0.89 | 0.85 | 59 | 3.2 | 15 | 6.4 | 0.92 | 1.2 | 1.0 | 0.35 | 19 | 170 | 110 | 50 | 0.18 | 5.0 | 2.2 | 0.34 | 1.3 |
| Cyclopenta(cd)pyrene | - | - | - | 17 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| Perylene | 1.0 | 0.73 | 0.88 | - | 1.1 | 3.1 | 1.5 | 0.29 | 0.66 | 0.56 | 0.29 | 3.7 | 34 | - | 9.9 | - | - | - | 1.6 | 0.82 |
| **Substituted Semivolatiles** | | | | | | | | | | | | | | | | | | | | |
| Aceanthrylene | - | - | - | - | - | - | - | - | - | - | - | - | - | 20 | - | - | - | - | - | - |
| Azulenes | - | 0.15 | - | - | - | - | 0.32 | 0.063 | 0.16 | 0.41 | 0.039 | - | - | - | - | - | - | - | - | - |
| Benzenes | - | 5.2 | 19 | - | 0.58 | - | 3.4 | 0.26 | 1.7 | 15 | 4.0 | - | 46 | 42 | 7.3 | - | 5.8 | - | - | - |
| Cyclo-hexane | - | - | - | - | - | - | - | - | - | - | 1.5 | - | - | - | - | - | - | - | - | - |
| Cyclo-hexene | - | - | - | - | - | 0.23 | 1.9 | - | 0.84 | 2.1 | 1.5 | - | - | - | - | - | - | - | - | - |
| Cyclo-hexenol | - | - | - | - | 0.19 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| Cyclo-heptenes | - | - | - | 50 | 0.38 | - | 1.4 | 0.094 | 0.41 | - | 0.70 | - | - | - | - | - | - | - | - | - |
| Cyclo-hexadiene | - | - | - | - | - | - | - | 0.21 | - | 1.3 | 0.76 | - | - | - | - | - | - | - | - | - |
| Cyclo-octene | - | 0.15 | - | - | - | - | - | - | - | 0.39 | - | - | - | - | - | - | - | - | - | - |
| Cyclo-tetradecatetraene | - | - | - | - | - | - | 1.7 | 0.54 | - | 1.5 | - | - | - | - | - | - | - | - | - | - |
| Furans | - | - | - | 4.9 | - | 0.11 | - | - | - | - | - | 0.41 | 3.9 | 2.2 | - | - | - | - | - | - |
| Indenes | - | - | - | - | - | - | - | - | - | - | - | 0.75 | 18 | 17 | 2.0 | - | - | - | - | - |
| Methanols | - | - | - | - | 0.090 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| Thiophenes | - | - | - | - | - | - | - | - | - | - | - | - | - | 0.70 | - | - | - | - | - | - |
| Triphenylenes | - | - | - | 17 | - | 1.8 | - | - | - | - | - | 2.8 | - | 10 | - | - | - | - | - | - |
| **Non-Substituted Semivolatiles** | | | | | | | | | | | | | | | | | | | | |
| Alpha-cubebene | - | 0.083 | - | - | 0.20 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| Beta-phellandrene | - | - | - | - | - | - | - | 0.13 | - | - | - | - | - | - | - | - | - | - | - | - |
| Biphenyl | - | - | - | - | - | - | - | - | - | - | - | - | - | 2.3 | - | - | - | - | - | - |
| 3-carene | 0.33 | 1.4 | 0.36 | - | 1.3 | 1.0 | 3.3 | 0.50 | 1.9 | 5.8 | 8.2 | - | - | - | - | - | - | - | - | 0.59 |
| 4-carene | - | - | - | - | - | - | - | - | - | - | - | 0.31 | - | - | 2.1 | - | 6.1 | - | - | - |
| Benzo(b)naphtho(2,3-D)furan | - | - | - | 23 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| Patchoulene | - | - | - | - | 0.16 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| Terphenyl | - | - | - | 45 | - | - | - | - | - | - | - | 3.2 | - | - | - | - | - | - | - | - |
| Benzo(b)thiophene | - | - | - | - | - | - | - | - | - | - | - | - | 1.8 | 1.8 | - | - | - | - | - | - |
| Dibenzothiophene | - | - | - | 55 | - | - | - | - | - | - | 0.091 | 0.12 | 3.9 | 2.9 | - | - | - | - | - | - |
| Benzo(b)naphtho(2,1-D)thiophene | - | - | - | 25 | - | 0.58 | - | - | - | - | - | 1.3 | 6.7 | 6.4 | 2.5 | - | - | - | - | - |

-= Not detected at unspecified detection limit
DNR= Wash. St. Dept. of Natural Resources
QT= Quendall Terminals
Bax= J.H. Baxter