Final Report

Analysis of Sediments and Soils for Chemical Contamination for the Design of U.S. Navy Homeport Facility at East Waterway of Everett Harbor, Washington

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March 1985

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Final Report

ANALYSIS OF SEDIMENTS AND SOILS
FOR CHEMICAL CONTAMINATION FOR THE
DESIGN OF U.S. NAVY HOMEPORT
FACILITY AT EAST WATERWAY
OF EVERETT HARBOR, WASHINGTON

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Richland, Washington

PREFACE

The U.S. Navy is developing a contaminated sediments assessment program for a proposed Navy homeport facility in the East Waterway of Everett Harbor, Washington. The Navy has asked the U.S. Army Corps of Engineers (COE), Seattle District, to act as its agent to accomplish this work.

The product desired from the COE is a report that describes 1) the horizontal and vertical character of East Waterway sediments and soils, 2) a preliminary assessment of the proposed disposal plan with recommendations for the need for further detailed studies to permit later design of safe and effective containment sites, and 3) an estimate of schedule and cost of those necessary studies within the time frame dictated by the National Environmental Policy Act (NEPA) environmental impact statement (EIS) schedule.

Characterization of sediment chemistry and a preliminary assessment of the concentration of chemicals found in East Waterway sediments and soils is critical to the draft EIS. The key consideration is the completion of work concurrent with the Navy's schedule for preparation of a NEPA EIS for their fiscal year (FY) 1986 and beyond program. The draft EIS is scheduled to be completed and approved by early 1985 to accompany the submission of the project's FY 1986 program to Congress. The Seattle District COE was selected for this work because of its experience and expertise with contaminated sediments in Puget Sound, and specifically at Everett Harbor, and for its ability to rapidly mobilize for sediment characterization studies.

Pacific Northwest Laboratory (PNL) is contracted to provide the Seattle District COE with an expert investigation and analysis of potential contaminated sediments and soils problems. PNL will also identify studies that are needed to predict environmental impacts which could result from the Navy's proposal to dredge and fill portions of Everett Harbor's East Waterway for the Puget Sound homeport facility. A secondary objective is

to aid the COE in the coordinating and planning aspects necessary to reduce adverse environmental effects that might result from disturbance of any contaminated sediments or soils.

SUMMARY

Contaminated sediments in the East Waterway of Everett Harbor, Washington, are extremely localized; they consist of a layer of organically-rich, fine sediments overlying a relatively cleaner, more sandy native material. The contaminated layer varies in thickness throughout the waterway from as much as 2 meters to only a few centimeters. Generally, the layer is thicker and more contaminated at the head of the waterway (northern end) and becomes thinner and less contaminated as one proceeds southerly out of the waterway and into Port Gardner. These sediments contain elevated levels of heavy metals and polynuclear aromatic hydrocarbons (PAH) and scattered concentrations of polychlorinated biphenyls (PCB). Approximately 500,000 cubic yards of material exhibit elevated chemical contamination compared to Puget Sound background levels. The contaminated sediments in this waterway require biological testing before decisions can be made regarding the acceptability of unconfined disposal.

ACKNOWLEDGMENTS

This project was completed on schedule with the utmost quality thanks to the dedication of key personnel including James Young, field collections; James Gurtisen, organic analyses; Charles Apts and Olga Cotter, metals analyses; and Jan Engel, manuscript typing.

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ANALYSIS OF SEDIMENTS AND SOILS FOR CHEMICAL CONTAMINATION
FOR THE DESIGN AND CONSTRUCTION OF A U.S. NAVY HOMEPORT
FACILITY AT EAST WATERWAY OF EVERETT HARBOR, WASHINGTON

INTRODUCTION

At the request of the U.S. Navy, the U.S. Army Corps of Engineers (COE), Seattle District, is helping the Navy develop a program to assess contaminated sediments and soils from the East Waterway of Everett Harbor. The program will provide information necessary to predict environmental impacts that could result from the U.S. Navy's proposal to dredge and fill portions of the East Waterway for a Navy homeport facility. Pacific Northwest Laboratory (PNL) is assisting the COE by providing the chemical analyses for these studies.

In July 1984, members of the Foundation and Materials Branch of the COE collected 19 aquatic sediment cores (E1 through E19), four aquatic sediment grab samples (A1 through A4), and two upland cores (U1 and U2) from the East Waterway (Figure 1). The aquatic cores were collected with a 15-foot-long by 4-inch-diameter Vibricore from the vessel, Puget.

Staff members from the Pacific Northwest Laboratory split the core liners and selected the intervals of sediment to sample for chemical and physical properties. The cores were divided into two or three sections labeled top, middle, and bottom. Evidence from previous sediment coring in the East Waterway indicated that surface sediments would be black, sulfide-containing mud, and subsurface sediments, from approximately 0.5 to 2 meter depths, would be gray, sandy sediments. The sectioning plan was to divide each core into a top section of approximately 1 meter of contaminated sediment, and a bottom section of uncontaminated "native" or background sediment. If the contaminated layer was more than 1.5 meters thick, then a middle section would also be sampled. Only two cores have middle sections; 16 cores have top and bottom sections, and one core has only a top section because no change in sediment properties were observed. Composites of each section were sampled, placed in an ice chest, and

FIGURE 1. Sediment Sampling Locations in the East Waterway (Source: U.S. Army Corps of Engineers)

transported cold to Battelle's Marine Research Laboratory in Sequim, Washington, for analysis.

Aquatic sediment grab samples were taken at four alternate disposal sites by a Van Veen grab sampler set to collect the top 10 to 15 centimeters of sediment. These sediments were placed in appropriate containers and transported to the laboratory.

The two deep upland cores (48 feet long) were drilled by COE personnel using a 4-inch rotary drill with steel casing. Samples of sediment were recovered by a split spoon sampler. The sampler was cleaned before each sample attempt with water and acetone. Three-foot sections of the cored sediment were homogenized and sampled for grain size analysis. Three composite samples (9 to 18 feet in length) were taken from each upland core.

Additional information on the field sample plan is included in the QA/QC plan in the Appendix of this report.

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CONCLUSIONS AND RECOMMENDATIONS

At least two broad areas should be considered for future research. First, biological effects such as bioaccumulation of toxic substances should be examined. This could be done using bioassays with selected sediments from the East Waterway of Everett Harbor. The most acceptable species for such tests are the phoxocephalid amphipods and oyster larvae. PNL has conducted these tests for EPA Region X and the COE and, after discussion with all interested agency representatives, could conduct similar analyses on Everett sediments.

Questions regarding bioaccumulation could be rapidly addressed by conducting analyses of metals and organics in mussel and clam tissues currently frozen at the Sequim laboratory. We recommend that one pooled sample of each species from selected stations (including Sequim control) be analyzed for metals and organics (18 samples). Selection of the stations and toxicants should be agreed upon by discussions with all interested agencies.

The second recommendation is to conduct a more comprehensive analysis of selected sediments for specific contaminants. Metals analyses do not indicate contamination is a problem in surface sediments, with the exception of cadmium in the upper portions of the waterway. Although further research on Cd is not recommended, the sources of Cd should certainly concern EPA Region X. The primary analytical recommendation is to identify the organics present in the unresolved complex mixture. Identities and quantities of these unknown organics can be determined by combining further steps in column chromatography with GC/MS analyses.

The overall recommendation for further research is to determine the possible disposal options and then design analytical (chemical and biological) methods to assess the relative impacts of each option. As noted in the report from the Seattle District office to the Navy (NPSEN-PL-ER, 11/30/84), personnel from the Waterways Experiment Station have been developing a decision-making framework for disposal options. As

the plan develops, it would be well to collect and synthesize all available information on the Everett Waterway and determine what data are still needed.

Based on recent studies at WES and the New York Bight, we believe more consideration should be given to aquatic disposal using the clean native material as a cap to contain the contaminants in the surface sediments. Effectiveness of this strategy can be monitored, which may help alleviate the concerns of regulatory agencies and strengthen the base for future decision making.

ANALYTICAL METHODS

BULK ANALYSES AND TRACE METALS

Sediment samples were analyzed for selected organic chemicals and toxic metals. Bulk chemical analyses were conducted to identify grain size, total and volatile solids, oil and grease, total organic carbon, and sulfides. These analytical procedures are described in the Appendix.

Several sediment samples were analyzed in triplicate to determine the within-sample variability. The results for triplicate analyses of bulk chemical properties are shown in Table 1. Two standard sediments, MESS-1 and NBS 1646, were analyzed in triplicate for metals to verify the accuracy of the analytical methods for metals. These results are given in Table 2, along with the certified values for metals in these standards.

ORGANICS

The detailed analytical procedure used to determine the concentrations of selected polyaromatic hydrocarbons (PAH) and chlorinated organics (PCB as Arochlor 1254) in sediments from the East Waterway are described in the Appendix. The steps used to extract, concentrate, and clean up each sediment sample are summarized in Figure 2. The steps taken to produce recovery data on a known mixture of PAH and a "spike" of Arochlor 1254 are described below to illustrate how the analytical data were obtained.

During a spike recovery experiment, a known quantity of PAH and PCB was introduced to a sample having the same characteristics as the set of samples to be evaluated. The amount of PCB and PAH in the sample was known. The spike was dissolved in a solvent that was miscible with water when added to the wet sample. Mixing, freezing, and equilibration of the spike with the samples were the variables. It should be noted that in the natural world, hydrocarbon pollutant inputs usually have enough time to equilibrate with the physical, chemical, and biological constituents of the sample.

Recovery experiments with a direct sample spike have the highest potential for yield, because the spike is readily extracted. A lower yield

TABLE 1. Results of Triplicate Metals Analyses for Metals in Two Everett Harbor Sediments

Sediment Sample	ppm dry wt												
	Cr	Ni	Cu	Zn	Se	Pb	As	Hg	Cd	Ag	Be	Tl	Sb
E-11-T #1	82.5	45.2	101.2	170.6	<0.84	64.8	3.16	0.335	1.03	0.165	0.315	1.61	<4.4
Rep. #2	62.1	42.0	103.0	167.0	<0.82	59.9	4.83	0.383	1.05	0.168	0.383	1.61	<4.3
Rep. #3	67.2	41.9	105.2	158.8	<0.79	64.1	3.22	0.300	1.09	0.180	0.360	1.61	<4.2
A4 GRAB#1	43.7	22.3	33.0	141.3	0.81	19.2	10.29	0.079	1.12	0.088	0.248	2.41	<3.8
Rep. #2	50.8	23.1	31.5	146.9	<0.71	21.0	10.90	0.129	1.14	0.097	-	-	-
Rep. #3	59.4	22.9	31.9	144.0	<0.71	19.3	10.47	0.081	1.04	0.088	-	-	<4.1

TABLE 2. Results of Triplicate Metals Analyses of Standard Marine Sediments

Sediment Standard (a)	ppm dry wt												
	Cr	Ni	Cu	Zn	Se	Pb	As	Hg	Cd	Ag	Be	Tl	Sb
MESS-1 rep. 1	68	29.3	28.1	197	1.05	31.1	11.4	0.178	0.683	0.085	2.00	0.803	-
2	61	29.1	28.9	185.8	<0.92	33.1	11.0	0.178	0.683	0.088	2.12	0.803	-
3	61	35.1	25.3	174.3	1.15	30.4	9.7	0.178	0.695	0.085	1.94	0.803	-
Certified Value	71	29.5	25.1	191	0.3 ^(b)	34.0	10.6	0.171	0.59	(b)	1.9	0.7 ^(b)	-
NBS 1646 rep. 1	92	34.1	20.4	127.5	<0.99	27.8	11.4	0.079	0.348	-	-	-	-
2	93	41.8	21.6	126.4	1.61	28.6	10.4	0.071	0.398	-	-	-	-
3	98	37.2	22.7	134.5	0.96	31.9	9.2	0.073	0.378	-	-	-	-
Certified Value	76	32	18	138	0.5 ^(b)	28.2	11.6	0.63	0.36	-	-	-	-

(a) Standards ME55-1 was provided by the National Research Council of Canada. Standard NBS-1646 was provided by the U.S. National Bureau of Standards.

(b) Not certified for these elements.

FIGURE 2. Sediment Extraction Scheme

or recovery would be expected if the spike could somehow be caused to equilibrate to the natural condition. Recovery is also influenced by other factors during the extraction. These include wall losses, volatility losses, component degradation, contamination, and analytical error. The nature of the sediment dewatering and hydrocarbon extraction scheme used at Pacific Northwest Laboratory decreases the usefulness of the direct addition of a spike to wet sediment. By adding a spike to wet sediment, the effect of other factors in estimating the yield of our extraction method would be excluded. This would most likely result in a high estimate of recovery. Most of the spike would be removed in the initial extractions with methanol and water. A more realistic representation of recovery is achieved if the spike is added to the dewatered sediment, where most of the other factors that produce losses come into play.

This latter procedure, in which the spike is added to the dewatered sediment, was used to determine the percent recovery of each of the selected PAHs analyzed and their values, including the recovery factors (Table 3). As expected, recovery was lower for the more volatile hydrocarbons (alkylbenzenes), but a majority of the recovery percentages were between about 55% to 80%. The recovery factors were used to correct gas chromatographic (GC) peaks to actual values for each PAH in all Everett sediment samples. The same procedure was used to determine the recovery factor for the PCB, Arochlor 1254 (Table 4). It should be noted that in analyses of field samples, there may have been an envelope of unresolved material in the region of Arochlor 1254 (Figure 3). The values presented later in this report should be considered high for this specific PCB because it may include other minor amounts of organics with the same GC retention time.

An additional procedure used in our QA/QC Plan was the analysis of replicate samples from the same container of sediment collected at Everett. The total PAH values for replicates vary by factors of two to four (Table 5). The relative proportion of selected PAHs tends to agree among replicates, but the absolute concentrations vary more than we would like. Part of the problem was the nonhomogeneity of particle sizes within a given sediment sample. Closer replication could be achieved by long-term mixing

TABLE 3. Recovery of PAH from Sediments. One ml of a mixture of PAH Components (1.8 to 15.8 $\mu\text{g/l}$), was added to a 50-g portion of "clean" sediment. The spike was dissolved in 50 ml of methanol and added just prior to soxhlet extraction.

Aromatic Component	Percent Recovery				Std. Dev.	Rec. Fac.
	830A	832A	834A	Avg.		
Ethylbenzene	6.7	26.5	16.1	16.4	9.9	6.1
M-xylene	20.8	52.2	31.7	34.9	15.9	2.9
O-xylene	18.1	26.7	22.1	22.3	4.3	4.5
Isopropylbenzene	16.9	24.4	21.0	20.8	3.8	4.8
1-ethyl-4-methylbenzene	29.7	36.6	33.8	33.4	3.5	3.0
1,2,4-trimethylbenzene	41.7	43.3	44.0	43.0	1.2	2.3
Secbutylbenzene	21.8	31.8	30.4	28.0	5.4	3.6
1-methyl-4-isopropylbenzene	36.3	45.1	42.2	41.2	4.5	2.4
1-3-dimethyl-5-ethylbenzene	56.6	55.4	58.1	56.7	1.4	1.8
1-2-diethylbenzene	51.2	53.3	52.5	52.3	1.1	1.9
1-2-dimethyl-4-ethylbenzene	56.2	57.6	58.6	57.5	1.2	1.7
1,2,3,5-tetramethylbenzene	60.1	60.2	62.2	60.8	1.2	1.6
Naphthalene	60.9	57.7	58.5	59.0	1.7	1.7
Benzothiophene	55.4	55.1	56.5	55.7	0.7	1.8
2-methylnaphthalene	65.0	65.8	66.5	65.8	0.8	1.5
1-methylnaphthalene	66.3	66.6	67.4	66.8	0.6	1.5
Biphenyl	140.0	105.0	129.4	124.8	17.9	0.8
2-6-dimethylnaphthalene	67.8	69.9	69.2	69.0	1.1	1.4
2,3,6-trimethylnaphthalene	69.5	73.8	70.5	71.3	2.3	1.4
Fluorene	68.9	72.5	68.7	70.0	2.1	1.4
Dibenzothiophene	54.7	58.2	55.4	56.1	1.9	1.8
Phenanthrene	71.3	76.7	70.0	72.7	3.6	1.4
Anthracene	73.2	78.6	71.1	74.3	3.9	1.3
1-methylphenanthrene	76.6	83.1	74.9	78.2	4.3	1.3
Fluoranthene	78.4	84.6	76.3	79.8	4.3	1.3
Pyrene	82.9	85.6	76.8	81.8	4.5	1.2
Chrysene	81.3	80.1	77.3	79.6	2.1	1.3
Benzo(e)pyrene	81.4	82.6	78.2	80.7	2.3	1.2
Benzo(a)pyrene	82.8	33.7	69.5	62.0	25.4	1.6
Perylene	96.1	53.7	89.8	79.9	22.9	1.3

TABLE 4. Recovery of Arochlor 1254 from Sediments

<u>Sample</u>	<u>Known Concentration Arochlor 1254 ($\mu\text{g/kg dry}$)</u>	<u>Measured Concentration Arochlor 1254 ($\mu\text{g/kg dry}$)</u>	<u>Percent Recovered</u>
Recov. Rep 1	40	26.6	66.4
Recov. Rep 2	40	27.4	68.4
Recov. Rep 3	40	23.1	57.7
		Average of Reps 1,2,3	64.2
		Standard Deviation	5.7
		Recovery Factor	1.6

FIGURE 3.. Comparison of Unresolved Complex Mixtures. Gas chromatogram of electron capturing components from (A) extract of a 100-g sample from E. Waterway, Everett, near Pier 1 [chromatogram derived from NOAA Tech. Mem. OMPA-19], and (B) extract of a 5-g sample from Station E4-middle, E. Waterway, Everett [analysis performed by PNL].

Legend: 3 CBD-trichlorobutadiene; TCBD-tetrachlorobutadiene; PCBD-Pentachlorobutadiene; HCB-Hexachlorobutadiene; TEC-Technazine (Internal Standard); HCB-Hexachlorobenzene; 2-BRN-Bromonaphthalene (Internal Standard).

TABLE 5. Replicate PAH Analyses of Sediment Samples from Everett Waterway (µg/kg dry sediment)

PAH	E9 Bottom		E19 Bottom		U1 Bottom		U2 Bottom	
Naphthalene	36	138	5	14	281	188	237	603
Benzothiophene	*	10	*	*		8	18	39
2-methylnaphthalene	19	60	4	11	186	255	174	402
1-methylnaphthalene	17	44	4	8	125	171	107	255
Biphenyl	5	14	1	8	21	41	16	40
2-6-dimethylnaphthalene	12	24	*	5	49	85	29	81
2,3,6-trimethylnaphthalene	7	16	*	*	49	99	30	77
Fluorene	23	46	*	*	118	291	83	171
Dibenzothiophene	79	125	18	132	136	406	83	203
Phenanthrene	118	180	3	23	342	997	236	571
Anthracene	61	90	2	8	156	426	81	224
1-methylphenanthrene	90	373	*	121	139	492	102	300
Fluoranthene	159	182	5	24	236	489	101	265
Pyrene	421	677	15	72	85	1402	273	2221
Chrysene	386	610	*	36	341	871	156	346
Benzo(e)pyrene	449	717	*	33	2899	2317	130	*
Benzo(a)pyrene	847	888	*	105	414	2413	106	420
Perylene	*	*	150	303	1133	4891	545	1870
SUM (Naphthalene-Perylene)	2727	4295	207	903	6716	15841	2504	8091

* Less than 1.0 µg/kg dry weight.

(mechanical) or by using a very homogenous sediment type. We purposely chose the less contaminated bottom portions of cores, and we would expect to see greater agreement among replicates when highly contaminated material is analyzed. The objective of this study was to determine the levels of contamination at various depths and sites in Everett. Even with the variability among replicates, we have succeeded in characterizing the level of PAH contamination in different parts of the waterway.

Previous analyses of samples from Everett Waterway indicated that PAH values were relatively low, but unresolved complex mixtures (UCM) indicated that significant amounts of unknown hydrocarbons were present (Malins et al. 1983). Stations that may have contained this material were identified so that more detailed analyses could be used to characterize the unresolved material. Twenty samples that exhibited a

significant UCM are listed in the Appendix (Table A.7). A comparison of our analyses of sediment from Station E4-middle (largest UCM) and an earlier analysis by Malins et al. (1983) produced similar results (see Figure 3).

In addition to the metals and organic analyses conducted by PNL, eleven samples were shipped to a subcontractor, New York Testing, Inc., for EPA priority pollutant analyses. Eight of these were sediment samples from Everett Waterway. The contractor was required to detect individual organics to within 10 to 20 $\mu\text{g}/\text{Kg}$. The methods used (EPA-specified procedures listed in Federal Register 44, 1979, Methods 608, 624, 625), were insufficient for detecting all priority pollutants at this detection limit. The results of the comparisons between these analyses and those by PNL are included in the Appendix (Table A.11).

significant differences listed in the Appendix (Table A.1). A comparison of the mean values of sediment from 1970-1971 (largest 10%) and 1972-1973 (smallest 10%) for the following parameters is shown in Figure 2.

In addition to the metals and organic analyses conducted by the EPA, eleven samples were shipped to a contractor, New York Testing, Inc., for EPA priority pollutant analysis. Eight of these were sediment samples from the same locations as the EPA samples. The contractor was required to detect individual organic compounds within 10 to 20 days. The method used (EPA approved) was listed in Federal Register, 1970, Volume 35, No. 171. The results of the comparison between these analyses and those by the EPA are included in the Appendix (Table A.1).

RESULTS AND DISCUSSION

Detailed descriptions of the core sample depths are reported in the Appendix. The surface sediments in the East Waterway are primarily a mixture of poorly consolidated sand, mud, wood chips, and tree bark. The average thickness of these contaminated sediments is 1.3 meters or approximately 4 feet. At two sites a middle section approximately 0.5 meters thick was recovered. Below the top or mid-sections is a gray sediment which did not contain noticeable sulfide, wood, and bark. This bottom section is referred to as "native" sediment and is assumed to be sediment below the depth of past dredging activities.

As shown in Table 6 there was generally a 20 to 35 percent reduction in core length relative to the depth of penetration by the barrel of the vibracores. This reduction was probably a result of the base sediment and the native sand and silt being compacted inside the core tube and acting as

TABLE 6. Comparison of Core Length with Depth of Vibracore Penetration

<u>Station</u>	<u>Vibracore Penetration</u>	<u>Core Length (cm)</u>	<u>Difference</u>	<u>Percent Penetration</u>
E1	335	245	90	73
E2	168	127	41	76
E3	366	251	115	69
E4	259	281	--	same
E5	259	198	61	76
E6	366	233	133	64
E7	351	175	176	50
E8	290	214	76	74
E9	259	200	59	77
E10	183	159	24	87
E11	411	303	108	74
E12	381	259	122	68
E13	244	257	--	same
E14	320	234	86	73
E15	274	222	52	81
E16	335	265	70	79
E17	335	123	212	37
E18	351	204	147	58
E19	366	198	168	54

a plug. At stations where the softer upper sediments were deep, the core length and barrel penetration depth were roughly the same. At stations where the core was primarily base sediment, its length was less than 60 percent of the barrel penetration depth.

ORGANICS IN AQUATIC SEDIMENTS

In producing the total PAH values, two steps were taken which strengthen the validity of the reported concentrations. Volatile components with a lower molecular weight than naphthalene were not included in the total. Lower recovery and less accurate reproducibility for these components make the values somewhat suspect. It was frequently (50% of samples) difficult to accurately separate perylene values benzo(e)pyrene and benzo(a)pyrene values. Normally, values are computed based upon a peak for peak match with respect to a standard calibration mixture containing the components of interest. Some chromatograms exhibited an unusually large peak at the normal retention time for perylene. In spite of the problems perylene, benzo(a)pyrene, and benzo(e)pyrene values have been computed and the resultant concentrations included in the summations for each core sample. This was justified using retention time matchups. Absolute confirmation would require the expense of a GC/MS run. A further cleanup step (gel filtration-Sephadex LH20) could provide information as to the nature of the peaks.

In the Appendix (Table A.11) there is a comparison of the data on specific PAH and the PCB 1254 provided by New York Testing, Inc. (NYT) to those produced by Pacific Northwest Laboratory on the same samples. As noted earlier, NYT presented results on a basis of $\mu\text{g/kg}$ wet weight, dry weight by use of the percent water values shown in Table A.2 of the Appendix. It can be seen that rather poor agreement was shown by the two analyses and the majority of the values reported by NYT were considerably less than those of Pacific Northwest Laboratory. The sample with the highest PAH concentrations (E1-top) provided results indicating NYT data were about 20% of Pacific Northwest Laboratory data. Occasionally NYT values were considerably larger than those of Pacific Northwest Laboratory (204 and 456%). Comparison of concentrations for the PCB, Arochlor 1254,

were generally better, with NYT values producing a mean of 32% lower than Pacific Northwest Laboratory concentrations. As noted earlier the chromatograms used by Pacific Northwest Laboratory to determine 1254 concentrations may well have included other hydrocarbons in an envelope at the same retention times. Therefore the lower values reported by NYT using GC/MS may in fact be more accurate for the specific concentration of Arochlor 1254. Since it is better to error on the high side, we have used the 20% for PAH and the 32% for PCB to prepare the contour maps discussed below.

A concern for the hydrocarbon retene was expressed by Don Brown (NOAA). A peak was observed at the expected retention time of retene. The height of this peak follows the general PAH concentrations in the samples. The peak is present in all samples, including bottom core samples. GC/MS analysis should be performed on several samples in order to provide further information regarding this peak.

Contours were prepared for each of the types of analyses conducted on surface sediments (1.3 M) taken in core samples at 19 stations and grab samples taken at four stations. The high, medium and low ranges were arbitrarily chosen after examination of the data for each type of chemical analyses. Figure 4 illustrates the distribution of total organic carbon (TOC) within the Everett East Waterway. Highest concentrations are generally found near loading areas, where both oil and grease and degraded wood tend to add to the total values. The center of the waterway and the grab samples taken at the entrance to the water exhibit low values.

The infrared (IR) analysis of sediment samples measures carbon bonds on organic compounds, so it must be noted that not all of the organics measured can accurately be considered oil or grease. The trends shown in Figure 5 indicate that the upper portion of the waterway is contaminated with petroleum hydrocarbons, particularly Stations E3, E5, E8 and E11. Stations southward from a line between E15 and E14 tend to be relatively clean.

The gas chromatographic analyses of 18 specific aromatic hydrocarbons (naphthalene through perylene) have been summed to produce the PAH values

FIGURE 4. Distribution of % TOC (Total Organic Carbon Dry Wt.) in Surface Sections of Sediment Cores and Surface Grab Samples of the East Waterway. Upland sites (U-1 and U-2) are also shown.

FIGURE 5. Distribution of ppm Oil and Grease (Dry Wt.) in Surface Sections of Sediment Cores and Surface Grab Samples of the East Waterway.

shown in Figure 6. It should be noted that we also quantitated 12 other specific aromatic hydrocarbons in the low molecular weight range. At the time this program was initiated the list of compounds on the Four-Mile Rock Disposal Site criteria was still under discussion. We have since added all the compounds on this list to our analysis program. When this study was conducted 4 of 6 low molecular weight components on the list were quantified in addition to 5 of the 10 higher weight compounds. The values in Figure 6 represent the total of 18 compounds in the same molecular weight range as the 16 compounds listed in the criteria. There is no reason to believe that the components on our previous analysis list do not characterize the level of contamination in marine sediments as well as the nine compounds which have been recently added. The isomers on the criteria list were taken from the EPA priority pollutant list, which was initially prepared in response to findings from mammalian toxicology tests.

The highest concentrations of the aromatics measured were found at Station E5 and the upper waterway (near loading docks) which were in general more contaminated than the middle and southern sections. A relatively high concentration was found in the grab sample from Station A4. Also shown in Figure 6 are four transect lines marked T-1 through T-4. To provide a view of the variations in the thickness of contaminated sediment in various regions of the waterway, Figure 7 has been prepared from data on stations connected by these lines. The open bars indicate the thickness of the surface contaminated layer which is greatest at Station E4 (150 cm). Variable amounts of "native" material was collected at the stations and core at E13 included about 63 cm of this material under 135 cm of contaminated sediment.

Figure 8 demonstrates the relative levels of total PAH at the same stations as Figure 7. The open bars represent the magnitude of contamination in the surface layer, which is greatest for PAH at Station E5 (230 ppm). Only one sample of native material (solid bar) was of sufficient concentration to be illustrated on the scale used in Figure 8. Two stations (E4 and E13) contained a contaminated middle layer which was higher in concentration than the surface sediment.

FIGURE 6. Distribution of ppm PAH (Dry Wt.) in Surface Sections of Sediment Cores and Surface Grab Samples of the East Waterway. Transects shown in Figures 7 and 8 are T-1 through T-4.

FIGURE 7. Depth of Surface Layer in Sample Cores from the East Waterway of Everett Harbor. (Transects 1 through 4 are shown in Figure 6.)

LEGEND: Open bars represent the thickness of the contaminated surface layer; hatched bars represent the mixed layer, including native sediment.

FIGURE 8. Distribution of PAH Along Transects and with Depth at 12 Stations in the Waterway. (Transects 1 through 4 are shown in Figure 6.

LEGEND: Open bars represent the surface layer contamination levels of PAH, and the only significant value for native material is shown in black (E3). Hatched bars represent the concentrations in the middle layer of two stations.

Polychlorinated biphenyls (Figure 9), including an envelope of unresolved compounds, were also found to be highest in the upper region of the waterway (Stations E1, E4 and E5). One of the highest concentrations observed (717 ppb) was at Station E11-top. Most of the remaining stations contained moderate amounts of PCBs, and a low value was observed at Station A3.

In order to place the data obtained in perspective with other analyses in potential disposal sites and the main basin of Puget Sound, Table 7 was prepared. References for these data are Romberg et al. (1984) and Strand et al. (1985). The grab samples are similar to the core tops in bulk properties, except for percent mud (silt plus clay), which is 31% in the grabs compared to 59% in the core tops. The sediments from the core bottoms are different from the tops in both physical and chemical properties. The native sediment contains less water, mud, volatiles, carbon, oil and grease, and sulfides.

The percentages of total solids and mud (silt and clay) do not differ widely between locations. The percentage of volatiles is higher for the top and middle of sediment cores and grab samples than at the Four-Mile Rock site and the main basin. TOC is higher in surface sections of cores and grabs than at the comparison locations. Oil and grease are considerably higher in the top and middle sections of the cores and in the grabs than at other sites and the bottom sections of cores. There are insufficient data at other locations to compare the sulfide values obtained for our stations.

While concentrations of DDTs and PCBs in Everett Harbor are approximately equal to those at Four-Mile Rock and the basin, aromatic hydrocarbons (PAH) values are considerably higher. The bottom of sediment cores generally contain lower amounts of PAH than either Four-Mile Rock or the Puget Sound main basin. However, the top and middle sections of cores and the grab samples contain concentrations which are about three to seven times higher than the Four-Mile Rock site, which is in turn about three times higher than Port Gardner or the main basin of Puget Sound.

FIGURE 9. Distribution of ppb PCB (Dry Wt.) in Surface Sections of Sediment Cores and Surface Grab Samples of the East Waterway

TABLE 7. Mean Concentrations of Bulk Physical and Chemical Properties and Organic Contaminants in Everett and Puget Sound Sediments

Properties or Contaminants	East Waterway			Grabs, Surface	Disposal Site, Surface		
	Sediment Cores				Port Gardner	Four-Mile Rock	Puget Sound Main Basin
	Top	Mid	Bottom				
Thickness (cm)	132	47	>140	10	10	10	10
----- % Dry Weight -----							
Solids	39	49	71	42	62	58	35
Mud	59	82	52	31	38	37	94
Volatiles	23	13	3.1	21	3.5	4.7	7.4
TOC	12.7	3.6	1.0	10.7	(b)	1.7	2.5
----- ppm (mg/kg dry wt.) -----							
Oil and Grease	4950	4610	92	1850	111	251	41
Sulfides	408	179	31	370		(a)	(a)
PAH	54	72	2	27	3	11	4
PCB	0.4	0.4	<0.01	0.2	0.1	0.6	0.1
DDTs	<0.02	<0.02	<0.02	<0.02	<0.02	0.007	0.002

(a) Mud = silt and clay combined.

(b) Data not available.

METALS IN AQUATIC SEDIMENTS

The mean concentrations of six metals in sections of sediment cores, grabs, and other sites in Puget Sound are shown in Table 8. These data indicate the top and middle layer of sediments in the waterway are contaminated with As, Cd, Cu, Pb, Hg, and Zn compared to the bottom sections, the two disposal sites and the Puget Sound main basin surface sediments. The exceptions are As, Pb, and Zn which are lower in the core tops than in Four Mile Rick Site. Selected sediments sampled were analyzed for all 13 EPA priority pollutant metals. In addition to the six metals above, only Ag was elevated in top sections compared to bottom. The other metals that were not substantially elevated included Cr, Ni, Se, Sb, Be, and Tl. These data are included in the Appendix.

The mean concentrations of four metals in the upper 1 to 2 meters of sediment in East Waterway exceeded those for two potential deep water disposal sites and those for the main basin. Although the mean concentrations of Pb and Zn are less than those for Four Mile Rock, several individual sediments have Pb and Zn concentrations that exceed those at Four- Mile Rock. However, the subsurface "native" sediments in the waterway are generally similar or lower in toxic metals than for sediments at the disposal sites. The "native" sediment can probably pass all tests for deepwater disposal. This uncontaminated sediment could be used for sediment capping or land fill.

TABLE 8. Mean Concentrations (ppm dry weight) of Metals in Everett and Puget Sound Sediments

Metals	East Waterway				Disposal Site, Surface		
	Sediment Cores			Grabs, Surface	Port Gardner	Four-Mile Rock	Puget Sound Main Basin
	Top	Mid	Bottom				
As	7.9	26.4	8.6	12	10	15	10
Cd	1.5	1.5	0.4	1.5	0.3	0.7	0.3
Cu	97	205	41	50	40	92	36
Pb	57	71	9	41	17	126	38
Hg	0.33	3.0	0.06	0.16	0.14	1.1	0.14
Zn	245	186	74	218	85	359	100

Since the pattern of distribution for the six toxic metals was very similar the horizontal distribution of two representative metals (Pb and Hg) in surface sediments of East Waterway are shown in Figures 10 and 11. Both of these figures show a pattern of higher metal concentrations in the northern region of the waterway. This pattern of contamination is generally true for the other toxic metals as well as the organic chemicals.

The metals data for the mid-sections of cores E4 and E13 indicate that subsurface sediments at two locations in the waterway are more contaminated than surface sediments (see Table 8). Previous studies conducted by Crecelius et al. (1984) also reported subsurface maxima for metals and organics in two cores, one taken near E7 the other near E15. These subsurface maxima in contaminant concentrations are of significance when comparing these sediments to other sites in the Sound since the subsurface sediments are generally more contaminated than the surface sediments. The differences between depths and stations along the same transects shown in Figures 6 through 8 can be observed for cadmium in Figure 12. There is relatively little difference in the concentration of Cd in the native material (as shown for PAH in Figure 8) from the 12 stations. Concentrations in the surface layer generally decreases as the transects move from north to south (T-1 toward T-4). The middle layers at E4 and E13 are slightly lower in Cd than surface sediment but considerably higher than native material.

Three other data sets for toxic metals in the East Waterway have reported similar concentrations of metals in surface and subsurface sediments (Crecelius et al. 1984; Malins et al. 1982; and U.S. EPA unpublished data). The EPA data set includes all 13 priority pollutant metals in grab samples from five sites. The EPA data provided by the Manchester, Washington, laboratory will be incorporated in an EPA report now in preparation by Pacific Northwest Laboratory and scheduled for delivery to EPA Region 10 by February 1985.

BULK CHEMISTRY AND CONTAMINANTS IN UPLAND SEDIMENT CORES

The bulk chemistry and contaminants in the two upland cores are shown in Table 9. The upland sediments are generally much less contaminated

FIGURE 10. Distribution of ppm Pb (Dry Wt.) in Surface Sections of Sediment Cores and Surface Grab Samples of the East Waterway

FIGURE 11. Distribution of ppb Hg (Dry Wt.) in Surface of Sediment Cores and Surface Grab Samples of the East Waterway

FIGURE 12. Distribution of Cd (ppm) Along Transects and with Depth. (Transects 1 through 4 are shown in Figure 6.)

LEGEND: Open bars represent the levels of Cd in the surface layer; solid bars indicate the concentrations in native material; hatched bars represent mid-sections.

TABLE 9. Mean Concentrations of Bulk Physical and Chemical Properties of Upland Cores from Everett Harbor

Properties	Upland Cores		
	Top	Mid	Bottom
Thickness, feet	12	18	18
	----- % Dry Weight -----		
Solids	71	74	71
Mud	21	31	36
Volatiles	13	2.9	4.9
TOC	5.0	1.0	1.8
	----- ppm Dry Weight -----		
Oil and Grease	1405	85	213
Sulfides	14	13	88
PCB	0.08	<0.01	<0.01
PAH	3.53	0.53	4.61
DDTs	<0.02	<0.02	<0.02
As	4.8	8.7	10.5
Cd	0.31	0.36	0.32
Cu	40	34	43
Pb	24	8.7	9.3
Hg	0.046	0.052	0.062
Zn	99	70	72

than the aquatic surface sediments. The upland sediments are similar to the "native" sediments in the waterway. This similarity is probably due to the origin of the upland sediments which may have been fill material from previous river sand dredging activities. The upland sediments are less contaminated (with all pollutants) than the Four-Mile Rock sediments and approximately the same as that at Port Gardner disposal site.

BIOACCUMULATION

The procedures used in this phase of our research are outlined in the Scope of Work prepared for the Seattle District COE. Basically, sediments from the top section of 22 stations (E1-E18 and A1-A4) were placed in the bottom of separate aquaria. Three additional aquaria were prepared (E1-B, E4-B and Sequim Bay), which may be considered control exposures. Ten Macoma inquinata were placed in the sediments of each tank and 10 mussels (Mytilus edulis) were placed on plastic racks, 8 cm above the sediment. Since mortality of mussels was occurring, the exposure time was limited to

13 days. The only significant mortality at the time of termination was in Tank E-11-T which contained sediments with the highest PCB concentration (717 ppb). Only one mussel remains from this tank for use in chemical analysis, but sufficient numbers of mussels and clams are available from all other tanks.

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APPENDIX

DETAILED ANALYSES CONDUCTED BY PACIFIC NORTHWEST LABORATORY

APPENDIX

DETAILED ANALYSES CONDUCTED BY PACIFIC NORTHWEST LABORATORY

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Table A1. Core samples from Everett Harbor (T = top, M = middle, B = bottom)

Station	Water Depth (ft)	Core Length (cm)	Zone ¹ Intervals (cm)	Zones Sampled	Zone Interval Sampled ¹ (cm)
E-10	-30.5	159	0-100 (T) 100-115 (M) 115-159 (B)	T,B	0-90 (T) 115-159 (B)
E-13	-30.0	257	0-137 (T) 137-197 (M) 197-257 (B)	T,M,B	0-90 (T) 137-197 (M) 197-257 (B)
E-1	-39.5	245	0-65 (T) 65-95 (M) 95-245 (B)	T,B	0-65 (T) 170-237 (B)
E-4	-45	281	0-154 (T) 154-200 (M) 200-281 (B)	T,M,B	0-90 (T) 157-190 (M) 220-270 (B)
E-15	-33.2	222	0-65 (T) 65-85 (M) 85-222 (B)	T,B	0-65 (T) 152-210 (B)
E-8	-24.9	214	0-73 (T) 73-214 (B)	T,B	0-73 (T) 150-200 (B)
E-11	-14.8	303	0-112 (T) 112-303 (B)	T,B	0-90 (T) 220-270 (B)
E-7	-36.0	175	0-60 (T) 60-175 (B)	T,B	0-60 (T) 100,150 (B)
E-12	-32.3	259	0-41 (T) 41-56 (M) 56-259 (B)	T,B	0-41 (T) 140-190 (B)
E-9	-28.5	200	0-50 (T) 50-58 (M) 58-200 (B)	T,B	0-50 (T) 115-160 (B)
E-6	-27.5	233	0-122 (T) 122-233 (B)	T,B	0-30 (T) 180-220 (B)
E-18	-34.7	204	0-204 (T) ²	T	10-60 (T)

Table A1, Continued

Station	Water Depth (ft)	Core Length (cm)	Zone ¹ Intervals (cm)	Zones Sampled	Zone Interval Sampled ¹ (cm)
E-17	-22.2	123	0-30 (T) 30-123 (B)	T,B	0-30 (T) 60-116 (B)
E-19	-40.2	198	0-15 (T) 15-198 (B)	T,B	0-15 (T) 107-157 (B)
E-16	-34.2	265	0-53 (T) 53-265 (B) ³	T,B	0-53 (T) 205-245 (B)
E-14	-33.5	234	0-44 (T) 44-234 (B)	T,B	0-44 (T) 150-200 (B)
E-5	-33.5	198	0-42 (T) 42-198 (B)	T,B	0-42 (T) 130-185 (B)
E-2	-28.5	127	0-73 (T) 73-127 (B)	T,B	23-73 (T) 90-125 (B)
E-3	-28.5	251	0-38 (T) 38-251 (B)	T,B	0-38 (T) 150-200 (B)
A-1	-27.8				Top 10
A-2	-36.8				Top 10
A-3	-3.5				Top 10
A-4	-3.5				Top 10

¹Where sufficient sediment was available for analysis in the middle layer (E-4 and E-13) it was taken in addition to samples from the top and bottom layers. The majority of sediment was sampled in both top and bottom layers, but transition zones were avoided.

²Entire core contained the same sandy silt.

³Observed layering in core.

Table A1 (Cont)

CORE SAMPLES FROM UPLAND SITES

<u>Samples</u>	<u>Core Length</u>	<u>Zones Sampled¹ for Chemistry</u>
U-1	48 feet	
U-1-T		0-9 feet
U-1-M		9-30 feet
U-1-B		30-48 feet
U-2	48 feet	
U-2-T		0-15 feet
U-2-M		15-30 feet
U-2-B		30-48 feet

¹ Sixteen samples taken from each site for grain size analysis.

Table A2

BULK CHEMISTRY OF EVERETT HARBOR SEDIMENTS

Station No.	Water Depth MLLW Feet	Core Sect. Cm	Dry Wt %	Volatiles %	TOC %	Oil ppm	Sulfides ug/g
E-1-T	39.5	0-65	34.43	26.55	6.84	5245	325
E-1-B	39.5	170-237	74.78	2.09	0.76	46	30.1
E-2-T	28.5	23-73	57.60	8.68	4.83	6750	418
E-2-B	28.5	90-125	70.91	3.84	1.54	123	70.6
E-3-T	28.5	0-38	28.20	21.03	10.50	7250	612
E-3-B	28.5	150-200	70.43	2.52	0.75	87	23.6
E-4-T	45	0-90	29.26	17.65	6.87	2700	335
E-4-M	45	157-190	39.28	12.58	4.65	8630	214
E-4-B	45	220-270	71.00	2.84	0.72	98	12.6
E-5-T #1	33.5	0-42	31.56	17.61	8.12	9090	497
E-5-T #2	33.5	0-42	31.56	-	-	8600	445
E-5-T #3	33.5	0-42	31.56	-	-	8520	516
E-5-B #1	33.5	130-185	76.38	2.21	0.68	26	16.1
E-5-B #2	33.5	130-185	-	2.27	-	-	-
E-5-B #3	33.5	130-185	-	2.23	-	-	-
E-6-T	27.5	0-30	28.95	19.27	8.69	3980	431
E-6-B	27.5	180-220	67.59	3.21	1.50	102	31.8
E-7-T	36.0	0-60	39.51	32.49	9.58	6020	545
E-7-B	36.0	100-150	75.32	2.29	0.68	50	13.1
E-8-T	24.9	0-73	31.83	19.92	8.85	7820	537
E-8-B	24.9	150-200	69.12	5.11	1.98	452	58.6
E-9-T	28.5	0-50	33.83	18.31	7.96	4730	325
E-9-B	28.5	115-160	54.37	7.53	2.55	229	61.8
E-10-T	30.5	0-90	44.52	32.82	5.41	2540	300
E-10-B	30.5	115-159	72.43	3.58	0.99	61	19.0
E-11-T #1	14.8	0-90	30.54	48.85	18.22	1400	649.5
E-11-T #2	14.8	0-90	-	49.88	16.83	-	-
E-11-T #3	14.8	0-90	-	51.51	20.32	-	-
E-11-B #1	14.8	220-270	70.01	2.51	1.03	73	28.2
E-11-B #2	14.8	220-270	70.01	-	0.87	105	-
E-11-B #3	14.8	220-270	70.01	-	0.84	60	-
E-12-T	32.3	0-41	31.38	22.43	10.80	5930	712
E-12-B	32.3	140-190	72.83	2.36	0.94	41	14.4
E-13-T	30.0	0-90	33.45	32.82	6.35	3820	400
E-13-M	30.0	137-197	58.89	12.02	2.41	2100	144
E-13-B	30.0	197-257	74.59	2.94	0.69	65	18.6
E-14-T	33.5	0-44	34.88	19.42	8.36	3500	394
E-14-B	33.5	150-200	72.34	2.88	0.88	79	45.7
E-15-T	33.2	0-65	46.55	44.41	7.84	5580	555
E-15-B	33.2	152-210	72.54	2.48	0.68	63	35.3

Table A2 (cont.)

Station No.	Water Depth MLLW Feet	Core Sect. Cm	Dry Wt %	Volatiles %	TOC %	Oil ppm	Sulfides ug/g
E-16-T #1	34.2	0-53	59.91	6.43	2.42	783	111
E-16-T #2	34.2	0-53	-	-	-	-	-
E-16-T #3	34.2	0-53	-	-	-	-	-
E-16-B	34.2	205-245	71.46	3.04	0.85	42	19.3
E-17-T	22.2	0-30	31.62	30.41	15.64	2590	193
E-17-B	22.2	60-116	74.45	2.19	0.49	18	29.2
E-18-T	34.7	10-60	79.41	1.31	0.39	25	30.2
E-19-T	40.2	0-15	37.76	20.21	7.68	1660	388
E-19-B	40.2	107-157	77.33	1.46	0.43	8	31.5
A-1 Grab	27.8	-	37.71	20.34	8.72	2730	520
A-2 Grab	36.8	-	38.48	15.66	6.89	2360	421
A-3 Grab	3.5	-	65.32	4.19	1.98	537	105
A-4 Grab	-	-	-	-	-	-	-
Rep #1	3.5	-	28.73	42.68	24.75	1780	429
Rep #2	3.5	-	-	42.62	26.55	-	-
Rep #3	3.5	-	-	42.38	23.97	-	-
U-1-T	-	0-9 feet	62.52	21.17	8.55	2700	40.3
U-1-M #1	-	9-30 feet	72.27	3.74	1.03	114.0	16.2
U-1-M #2	-	-	-	-	-	-	-
U-1-M #3	-	-	-	-	-	-	-
U-1-B	-	30-48 feet	69.2	5.44	2.11	256	92.1
U-2-T	-	0-15 feet	78.50	3.99	1.45	110	11.9
U-2-M	-	15-30 feet	75.32	2.13	1.04	55	9.31
U-2-B	-	30-48 feet	72.67	4.28	1.53	171	84.9
Blank #1	-	-	-	0.00	-	<4	<5.0
Blank #2	-	-	-	0.00	-	<4	<5.0
Blank #3	-	-	-	0.00	-	<4	<5.0
MESS #1	-	-	-	-	2.56	-	-
MESS #2	-	-	-	-	2.51	-	-
MESS #3	-	-	-	-	2.57	-	-

Table A3

Grain Size Analysis of Everett Harbor Sediments

Station No.	Grain Size (%)			
	Gravel	Sand	Silt	Clay
E-1-T	13.03	24.77	41.47	20.73
E-1-B	0.00	4.42	67.66	27.92
E-2-T #1	22.93	46.89	19.33	10.85
E-2-T #2	28.13	43.40	19.33	9.14
E-2-T #3	21.03	50.66	18.52	9.79
E-2-B	0.31	58.70	31.02	9.97
E-3-T	8.76	25.48	39.22	26.54
E-3-B	0.00	58.92	31.77	9.31
E-4-T	5.77	19.18	53.07	21.98
E-4-M	3.40	2.70	65.08	28.81
E-4-B	0.00	1.98	59.18	38.83
E-5-T #1	13.60	29.69	32.22	24.48
E-5-T #2	8.19	37.28	29.62	24.91
E-5-T #3	7.79	31.67	39.91	20.62
E-5-B	0.02	66.98	22.16	10.85
E-6-T	2.80	9.33	58.47	29.39
E-6-B	0.46	70.16	21.07	8.32
E-7-T	3.89	39.68	36.43	20.01
E-7-B	0.00	70.88	18.27	10.85
E-8-T	0.46	26.41	48.13	25.00
E-8-B	0.68	67.28	21.70	10.34
E-9-T	11.40	32.84	38.01	17.76
E-9-B	0.06	8.18	50.66	41.10
E-10-T	12.82	0.87	58.01	28.30
E-10-B	0.00	30.22	53.67	16.12
E-11-T	20.97	44.43	18.45	16.15
E-11-B #1	0.00	62.58	25.60	11.82
E-11-B #2	0.02	55.30	33.93	10.76
E-11-B #3	0.01	57.43	31.43	11.13
E-12-T	20.46	23.68	34.30	21.57
E-12-B	0.01	45.09	36.85	18.05
E-13-T	0.07	22.19	51.97	25.77
E-13-M	0.46	29.11	50.81	19.61
E-13-B	0.17	51.80	38.14	9.90
E-14-T	5.68	37.43	37.41	19.49
E-14-B	0.00	37.28	44.53	18.19
E-15-T	6.86	26.08	41.37	25.70
E-15-B	0.01	30.38	50.44	19.17
E-16-T	0.90	33.78	48.82	16.49
E-16-B	0.00	50.12	36.29	13.59

Table A3 (cont)

Station No.	Grain Size (%)			
	Gravel	Sand	Silt	Clay
E-17-T	22.05	27.62	29.53	20.80
E-17-B	1.02	65.64	25.10	8.23
E-18-T	0.40	75.58	18.84	5.18
E-19-T	14.34	39.37	32.10	14.19
E-19-B	1.69	69.84	21.29	7.19
A-1 GRAB	10.97	44.07	29.26	15.69
A-2 GRAB	5.21	39.12	33.18	22.49
A-3 GRAB	0.29	93.98	2.16	3.58
A-4 GRAB	33.59	48.91	10.92	6.58
U1 0-3 Ft	39.07	48.76	6.21	5.96
U1 3-6	8.60	69.54	18.62	3.24
U1 6-9	8.31	5.61	62.49	23.59
U1 9-12	9.31	80.88	9.81	0.00
U1 12-15	0.66	83.22	11.71	4.41
U1 15-18	1.23	68.60	20.91	9.26
U1 18-21	0.20	77.20	16.29	6.31
U1 21-24	0.09	70.72	20.96	8.23
U1 24-27	0.22	67.59	22.43	9.76
U1 27-30	0.28	45.92	36.50	17.30
U1 30-33	3.92	86.35	3.97	5.76
U1 33-36	5.14	75.10	12.59	7.17
U1 36-39	14.36	57.55	18.63	9.46
U1 39-42	9.90	73.60	10.18	6.32
U1 42-45	2.88	56.34	29.27	11.51
U1 45-48	0.03	42.36	42.03	15.58
U2 0-3 #1	55.07	38.81	3.27	2.85
U2 0-3 #2	51.92	42.90	2.64	2.55
U2 0-3 #3	47.74	45.58	2.34	4.34
U2 3-6	18.29	76.19	5.06	0.46
U2 6-9	23.20	70.48	6.06	0.26
U2 9-12	53.82	22.17	17.29	6.7
U2 12-15	11.33	64.61	18.28	5.78
U2 15-18	0.73	50.93	33.26	15.09
U2 18-21	0.34	89.37	6.94	3.35
U2 21-24	1.50	86.38	9.29	2.82
U2 24-27	4.60	74.61	14.82	5.97
U2 27-30	4.52	19.69	55.37	20.42
U2 30-33	4.92	71.53	15.99	7.57
U2 33-36	0.36	40.20	46.66	12.78
U2 36-39	5.47	66.96	23.11	4.47
U2 39-42	7.00	67.60	19.92	5.49
U2 42-45	2.19	44.23	40.14	13.44
U2 45-48	0.00	34.95	54.07	10.97

Table A4

CONCENTRATION OF METALS IN EVERETT HARBOR SEDIMENT

Station No.	Ppm (Dry Weight Basis)												
	Cr	Ni	Cu	Zn	Se	Pb	As	Hg	Cd	Ag	Be	Tl	Sb
E-1-T	123	82.2	127.4	317	1.20	85.0	10.7	0.608	1.55	0.338	0.878	1.61	<4.4
E-1-B	153	63.9	44.8	80.5	<0.93	9.0	8.99	0.053	0.200	-	-	-	-
E-2-T	98	60.3	89.2	118.4	<0.90	49.4	4.9	0.348	1.31	0.203	0.788	0.803	4.8
E-2-B	100	53.0	36.3	67.9	<0.92	10.1	9.19	0.031	0.325	-	-	-	-
E-3-T	113	70.6	112.9	255	<0.93	90.6	7.2	0.445	2.05	-	-	-	-
E-3-B	141	52.5	35.6	64.2	<0.92	11.6	6.94	0.055	0.532	-	-	-	-
E-4-T	119	67.4	97.8	240	<0.97	55.8	8.5	0.264	2.44	-	-	-	-
E-4-M	149	72.4	165.4	205	1.03	73.4	13.6	3.482	1.47	-	-	-	-
E-4-B	163	72.5	52.7	92.4	<0.97	10.1	9.3	0.073	0.273	-	-	-	-
E-5-T	136	63.1	110.1	286	<0.96	138.7	<2.5	0.652	2.36	-	-	-	-
E-5-B #1	122	61.4	38.9	74.4	1.23	8.3	7.80	0.064	0.222	-	-	-	-
E-5-B #2	-	-	-	-	-	-	-	0.053	0.222	-	-	-	-
E-5-B #3	-	-	-	-	-	-	-	0.040	0.236	-	-	-	-
E-6-T	113	66.3	154.2	202	<0.94	74.4	12.8	0.287	2.18	0.424	0.810	<0.803	<4.3
E-6-B	142	57.2	37.7	64.4	1.09	9.9	15.6	0.064	0.399	-	-	-	-
E-7-T	111	61.6	106.3	198	<0.92	55.6	7.6	0.529	1.14	-	-	-	-
E-7-B	150	61.2	35.5	71.2	<0.94	5.7	9.93	0.047	0.236	-	-	-	-
E-8-T	119	63.0	121.6	319	<0.92	68.5	7.5	0.370	1.97	-	-	-	-
E-8-B	121	48.3	35.8	64.6	<0.91	16.3	6.10	0.077	0.325	-	-	-	-
E-9-T	155	70.2	114.9	364	<1.0	67.9	7.4	0.318	1.42	-	-	-	-
E-9-B	173	91.0	82.4	118.3	<1.0	20.1	15.0	0.134	0.650	-	-	-	-
E-10-T	124	65.8	88.1	520	1.04	55.9	7.4	0.194	1.65	-	-	-	-
E-10-B	141	58.5	44.2	76.8	<0.94	11.0	6.94	0.062	0.406	-	-	-	-
E-11-T #1	82.5	45.2	101.2	170.6	<0.84	64.8	3.16	0.335	1.03	0.165	0.315	1.61	<4.4
E-11-T #2	62.1	42.0	103.0	167.0	<0.82	59.9	4.83	0.383	1.05	0.168	0.383	1.61	<4.3
E-11-T #3	67.2	41.9	105.2	158.8	<0.79	64.1	3.22	0.300	1.09	0.180	0.360	1.61	<4.2
E-11-B #1	143	57.1	41.1	72.3	<0.93	10.5	7.50	0.064	1.03	0.029	0.900	0.803	<4.2
E-11-B #2	135	54.5	41.2	73.5	1.14	7.6	9.29	-	0.281	0.029	0.900	0.803	<4.6
E-11-B #3	108	61.0	40.2	75.4	<0.92	11.2	7.38	-	-	-	-	-	<4.4
E-12-T	115	69.0	104.2	185.7	<0.92	56.9	8.9	0.344	1.57	-	-	-	-
E-12-B	161	69.5	46.7	83.9	1.12	9.6	8.29	0.058	0.273	-	-	-	-
E-13-T	111	74.6	107.9	226	1.10	59.7	10.0	0.194	1.71	-	-	-	-
E-13-M	146	67.4	224	166.7	1.04	69.9	39.2	2.603	1.52	-	-	-	-
E-13-B	151	52.3	37.1	70.8	1.23	9.8	7.01	0.053	0.370	-	-	-	-
E-14-T	108	87.4	92.3	157.8	1.01	56.4	6.2	0.376	1.91	-	-	-	-
E-14-B	155	59.7	41.5	76.6	<0.93	8.8	10.9	0.061	0.303	-	-	-	-
E-15-T	111	73.9	96.9	145.8	<1.0	67.3	7.7	0.389	1.55	-	-	-	-
E-15-B	130	61.8	42.9	77.0	<0.92	10.4	6.84	0.064	0.259	-	-	-	-
E-16-T #1	158	61.4	54.8	87.8	<0.94	21.0	8.9	0.204	0.739	0.085	0.743	1.61	<4.5
E-16-T #2	-	-	-	-	-	-	-	0.201	0.746	0.085	-	-	-
E-16-T #3	-	-	-	-	-	-	-	0.206	0.665	0.088	-	-	-
E-16-B	152	65.5	44.1	80.1	<0.94	8.8	8.63	0.066	0.296	-	-	-	-

Table A4 (cont.)

Station No.	Ppm (Dry Weight Basis)												
	Cr	Ni	Cu	Zn	Se	Pb	As	Hg	Cd	Ag	Be	Tl	Sb
E-17-T	97	59.8	77.2	696	<0.89	31.6	14.9	0.129	1.62	-	-	-	-
E-17-B	104	36.0	21.2	47.7	<0.91	8.1	3.92	0.035	0.229	-	-	-	-
E-18-T	97	37.6	20.0	44.9	<0.89	6.1	5.33	0.028	0.244	-	-	-	-
E-19-T	95	50.6	66.9	113.1	1.19	34.4	8.36	0.276	1.48	0.235	0.720	0.803	<4.2
E-19-B	104	38.9	20.1	44.8	0.96	5.0	5.62	0.028	0.214	-	-	-	-
A1 Grab	133	63.3	76.7	441	1.01	75.3	16.1	0.301	1.90	-	-	-	-
A2 Grab	149	63.1	75.3	237	<0.93	55.7	13.5	0.234	2.60	-	-	-	-
A3 Grab	94	30.2	15.0	53.2	<0.85	14.5	6.41	0.030	0.244	-	-	-	-
A4 Grab #1	43.7	22.3	33.0	141.3	0.81	19.2	10.29	0.079	1.12	0.088	0.248	2.41	<3.8
A4 Grab #2	50.8	23.1	31.5	146.9	<0.71	21.0	10.90	0.129	1.14	0.097	-	-	-
A4 Grab #3	59.4	22.9	31.9	144.0	<0.71	19.3	10.47	0.081	1.04	0.088	-	-	<4.1
U-1-T	97	40.0	42.9	111.1	<0.85	135.1	4.7	0.048	0.288	-	-	-	-
U-1-M #1	123	54.1	38.4	73.7	<0.92	10.3	9.96	0.060	0.421	-	-	-	-
U-2-M #2	-	-	-	-	-	-	-	0.057	0.362	-	-	-	-
U-1-M #3	-	-	-	-	-	-	-	0.055	0.362	-	-	-	-
U-1-B	137	54.3	44.4	72.0	0.92	11.0	10.22	0.064	0.310	-	-	-	-
U-2-T	149	50.2	35.5	85.7	<0.92	43.1	4.84	0.044	0.340	-	-	-	-
U-2-M	117	45.4	30.4	64.7	<0.91	7.0	7.37	0.044	0.303	-	-	-	-
U-2-B	100	51.1	42.3	71.8	1.12	7.5	10.80	0.060	0.325	-	-	-	-
MESS-1 #1	68	29.3	28.1	197	1.05	31.1	11.4	0.178	0.683	0.085	2.00	0.803	-
MESS-1 #2	61	29.1	28.9	185.8	<0.92	33.1	11.0	0.178	0.683	0.088	2.12	0.803	-
MESS-1 #3	61	35.1	25.3	174.3	1.15	30.4	9.7	0.178	0.695	0.085	1.94	0.803	-
NBS #1	92	34.1	20.4	127.5	<0.99	27.8	11.4	0.079	0.348	-	-	-	-
NBS #2	93	41.8	21.6	126.4	1.61	28.6	10.4	0.071	0.398	-	-	-	-
NBS #3	98	37.2	22.7	134.5	<0.96	31.9	9.2	0.073	0.378	-	-	-	-
BCSS-1 #1	-	-	-	-	-	-	-	0.135	-	-	-	-	-
BCSS-1 #2	-	-	-	-	-	-	-	0.133	-	-	-	-	-
BCSS-1 #3	-	-	-	-	-	-	-	0.136	-	-	-	-	-
Blank #1	-	-	-	-	-	-	-	N.D.	0.015	<0.003	0.045	<0.803	-
Blank #2	-	-	-	-	-	-	-	N.D.	0.015	<0.003	0.045	<0.803	-
Blank #3	-	-	-	-	-	-	-	N.D.	0.015	<0.003	0.045	<0.803	-

Table A5. Summary of Capillary GC analysis of Arochlor 1254 in Sediments from the East waterway of Everett Harbor, Washington

Sample I.D.	Measured Concentration Arochlor 1254 $\mu\text{g/kg dry sed}$	
A-4-GRAB	255
A-3-GRAB	39
A-2-GRAB	291
A-1-GRAB	273
E-1-Top	585
E-1-Bottom	*	
E-2-Bottom	*	
E-3-Top	391
E-3-Bottom	*	
E-4-Top	391
E-4-Middle	581
E-4-Bottom	*	
E-5-Top	668
E-5-Bottom	*	
E-6-Bottom	*	
E-7-Top	364
E-7-Bottom	*	
E-8-Top	336
E-8-Bottom	13
E-9-Top	451
E-9-Bottom	*	
E-10-Top	272
E-10-Bottom	*	
E-11-Top	717
E-12-Top	281
E-12-Bottom	*	

* Arochlor 1254 not detectable

Table A5 (Cont). Summary of Capillary GC analysis of Arochlor 1254 in sediments from the East waterway of Everett Harbor, Washington

Sample	Measured Concentration Arochlor 1254 I.D. $\mu\text{g/kg dry sed}$	
E-13-Top	232
E-13-Middle	120
E-13-Bottom	*	
E-14-Top	286
E-14-Bottom	*	
E-15-Top	377
E-15-Bottom	*	
E-16-Bottom	*	
E-17-Top	238
E-17-Bottom	*	
E-18-Top	*	
E-19-Bottom	*	
U-1-Top	143
U-1-Mid	*	
U-1-Bottom	*	
U-2-Top	16
U-2-Mid	*	
U-2-Bottom	*	

* Arochlor 1254 not detectable

Table A6. Summary of Capillary GC analysis of PAH's in sediments from the East waterway of Everett Harbor, Washington

Sample I.D.	Measured Concentration Total Select PAH's mg/kg dry sediment	
A-4-GRAB	24.62
A-3-GRAB	3.85
A-2-GRAB	27.07
A-1-GRAB	53.38
E-1-Top	103.13
E-1-Bottom	0.90
E-2-Bottom	5.32
E-3-Top	84.97
E-3-Bottom	9.60
E-4-Top	41.15
E-4-Middle	106.35
E-4-Bottom	0.27
E-5-Top	222.92
E-5-Bottom	0.75
E-6-Bottom	2.16
E-7-Top	37.00
E-7-Bottom	0.36
E-8-Top	46.68
E-8-Bottom	2.73
E-9-Top	69.80
E-9-Bottom	2.73
E-10-Top	17.98
E-10-Bottom	0.13
E-11-Top	69.40
E-12-Top	25.53
E-12-Bottom	0.10

* Arochlor 1254 not detectable

Table A6 (Cont). Summary of Capillary GC analysis of PAH's in sediments from the East waterway of Everett Harbor, Washington

Sample I.D.	Measured Concentration Total Select PAH's mg/kg dry sediment	
E-13-Top	27.10
E-13-Middle	38.25
E-13-Bottom	0.12	
E-14-Top	20.19
E-14-Bottom	0.16	
E-15-Top	41.29
E-15-Bottom	0.23	
E-16-Bottom	0.21	
E-17-Top	10.71
E-17-Bottom	0.79
E-18-Top	0.02	
E-19-Bottom	0.21	
U-1-Top	3.79
U-1-Mid	0.61	
U-1-Bottom	6.72
U-2-Top	3.26
U-2-Mid	0.45	
U-2-Bottom	2.50

* Arochlor 1254 not detectable

Sample I.D.	E-1-Top	
Wet weight extracted (grams)	5	
Dry:Wet weight ratio	0.28	
Concentration units	$\mu\text{g/kg dry}$	
Ethylbenzene	*	
M-xylene	*	
O-xylene	*	
Isopropylbenzene	*	
1-ethyl-4-methylbenzene	170	
1,2,4-trimethylbenzene	440	
Secbutylbenzene	354	
1-methyl-4-isopropylbenzene	3611	
1-3-dimethyl-5-ethylbenzene	*	
1-2-diethylbenzene	*	
1-2-dimethyl-4-ethylbenzene	450	
1,2,3,5-tetramethylbenzene	95	
Naphthalene	1980
Benzo(b)thiophene	126	
2-methylnaphthalene	1625	
1-methylnaphthalene	1117	
Biphenyl	370	
2,6-dimethylnaphthalene	1072	
2,3,6-trimethylnaphthalene	2006	
Fluorene	1992	
Dibenzothiophene	4863	
Phenanthrene	9103	
Anthracene	6481	
1-methylphenanthrene	6837	
Fluoranthene	9828	
Pyrene	21390	
Chrysene	12775	
Benzo(e)pyrene	8323	
Benzo(a)pyrene	4733	
Perylene	8511	
SUM(Naphthalene-Perylene)	103132	

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D. E-1-Bottom

Wet weight extracted (grams) 50
 Dry:Wet weight ratio 0.75
 Concentration units $\mu\text{g/kg dry}$

Ethylbenzene	*
M-xylene	*
O-xylene	*
Isopropylbenzene	*
1-ethyl-4-methylbenzene	*
1,2,4-trimethylbenzene	*
Secbutylbenzene	*
1-methyl-4-isopropylbenzene	2
1-3-dimethyl-5-ethylbenzene	*
1-2-diethylbenzene	*
1-2-dimethyl-4-ethylbenzene	*
1,2,3,5-tetramethylbenzene	*
Naphthalene	*
Benzothiophene	*
2-methylnaphthalene	*
1-methylnaphthalene	*
Biphenyl	*
2-6-dimethylnaphthalene	*
2,3,6-trimethylnaphthalene	*
Fluorene	*
Dibenzothiophene	*
Phenanthrene	*
Anthracene	*
1-methylphenanthrene	*
Fluoranthene	*
Pyrene	*
Chrysene	*
Benzo(e)pyrene	*
Benzo(a)pyrene	*
Perylene	898

SUM(Naphthalene-Perylene) 898

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E-2-Bottom

Wet weight extracted (grams)

50

Dry:Wet weight ratio

0.71

Concentration units

 $\mu\text{g/kg dry}$

Ethylbenzene	*	
M-xylene	*	
O-xylene	*	
Isopropylbenzene	6	
1-ethyl-4-methylbenzene	11	
1,2,4-trimethylbenzene	3	
Secbutylbenzene	*	
1-methyl-4-isopropylbenzene	155	
1-3-dimethyl-5-ethylbenzene	5	
1-2-diethylbenzene	*	
1-2-dimethyl-4-ethylbenzene	14	
1,2,3,5-tetramethylbenzene	3	
Naphthalene	169	
Benzothiophene	9	
2-methylnaphthalene	51	
1-methylnaphthalene	35	
Biphenyl	13	
2,6-dimethylnaphthalene	11	
2,3,6-trimethylnaphthalene	13	
Fluorene	71	
Dibenzothiophene	148	
Phenanthrene	246	
Anthracene	116	
1-methylphenanthrene	72	
Fluoranthene	219	
Pyrene	461	
Chrysene	442	
Benzo(e)pyrene	464	
Benzo(a)pyrene	1065	
Perylene	1693	
SUM(Naphthalene-Perylene)	5319	

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E-3-Top

Wet weight extracted (grams)

5

Dry:Wet weight ratio

0.26

Concentration units

 $\mu\text{g/kg dry}$

Ethylbenzene

1192

M-xylene

2006

O-xylene

*

Isopropylbenzene

*

1-ethyl-4-methylbenzene

219

1,2,4-trimethylbenzene

329

Secbutylbenzene

621

1-methyl-4-isopropylbenzene

2744

1-3-dimethyl-5-ethylbenzene

93

1-2-diethylbenzene

*

1-2-dimethyl-4-ethylbenzene

1159

1,2,3,5-tetramethylbenzene

95

Naphthalene

1509

Benzothiophene

*

2-methylnaphthalene

461

1-methylnaphthalene

841

Biphenyl

470

2,6-dimethylnaphthalene

918

2,3,6-trimethylnaphthalene

266

Fluorene

1171

Dibenzothiophene

2975

Phenanthrene

3487

Anthracene

3477

1-methylphenanthrene

5791

Fluoranthene

5249

Pyrene

11341

Chrysene

8920

Benzo(e)pyrene

15236

Benzo(a)pyrene

9465

Perylene

13391

SUM(Naphthalene-Perylene)

84967

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E-3-Bottom

Wet weight extracted (grams)

50

Dry:Wet weight ratio

0.69

Concentration units

 $\mu\text{g/kg dry}$

Ethylbenzene

*

M-xylene

*

O-xylene

*

Isopropylbenzene

*

1-ethyl-4-methylbenzene

7

1,2,4-trimethylbenzene

*

Secbutylbenzene

*

1-methyl-4-isopropylbenzene

81

1-3-dimethyl-5-ethylbenzene

2

1-2-diethylbenzene

*

1-2-dimethyl-4-ethylbenzene

3

1,2,3,5-tetramethylbenzene

2

Naphthalene

277

Benzothiophene

10

2-methylnaphthalene

76

1-methylnaphthalene

52

Biphenyl

19

2-6-dimethylnaphthalene

*

2,3,6-trimethylnaphthalene

17

Fluorene

149

Dibenzothiophene

119

Phenanthrene

378

Anthracene

132

1-methylphenanthrene

69

Fluoranthene

345

Pyrene

692

Chrysene

423

Benzo(e)pyrene

2812

Benzo(a)pyrene

810

Perylene

3224

SUM(Naphthalene-Perylene)

9604

* < 1.0 $\mu\text{g/kg dry weight}$

Sample 1.8.

E-4-Top

Wet weight extracted (grams) 5

Dry:Wet weight ratio 0.24

Concentration units $\mu\text{g/kg dry}$

Ethylbenzene 10907

M-xylene 12797

O-xylene 2206

Isopropylbenzene 238

1-ethyl-4-methylbenzene 276

1,2,4-trimethylbenzene 702

Secbutylbenzene 1576

1-methyl-4-isopropylbenzene 4693

1-3-dimethyl-5-ethylbenzene 31

1-2-diethylbenzene *

1-2-dimethyl-4-ethylbenzene

1,2,3,5-tetramethylbenzene 117

Naphthalene 989

Benzo(b)thiophene *

2-methylnaphthalene 337 1-methylnaphthalene 139 Biphenyl 135 2,6-dimethylnaphthalene 169 2,3,6-trimethylnaphthalene 196 Fluorene 552 Dibenzothiophene 1761 Phenanthrene 1734 Anthracene 1307 1-methylphenanthrene 3246 Fluoranthene 5491 Pyrene 12026 Chrysene 7745 Benzo(e)pyrene 2416 Benzo(a)pyrene 1136 Perylene 1749

SUM(Naphthalene-Perylene) 41146

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E-4-Mid

Wet weight extracted (grams)

15

Dry:Wet weight ratio

0.35

Concentration units

 $\mu\text{g/kg dry}$

Ethylbenzene

2030

M-xylene

2120

O-xylene

*

Isopropylbenzene

*

1-ethyl-4-methylbenzene

*

1,2,4-trimethylbenzene

271

Secbutylbenzene

273

1-methyl-4-isopropylbenzene

2927

1-3-dimethyl-5-ethylbenzene

*

1-2-diethylbenzene

*

1-2-dimethyl-4-ethylbenzene

865

1,2,3,5-tetramethylbenzene

499

Naphthalene

13280

Benzo(b)thiophene

1728

2-methylnaphthalene

5578

1-methylnaphthalene

3993

Biphenyl

2492

2,6-dimethylnaphthalene

5984

2,3,6-trimethylnaphthalene

2957

Fluorene

5676

Dibenzothiophene

7464

Phenanthrene

14519

Anthracene

4290

1-methylphenanthrene

4680

Fluoranthene

7800

Pyrene

15398

Chrysene

3457

Benzo(e)pyrene

2478

Benzo(a)pyrene

1286

Perylene

3290

SUM(Naphthalene-Perylene)

106350

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E-4-Bottom

Wet weight extracted (grams) 50

Dry:Wet weight ratio 0.72

Concentration units $\mu\text{g/kg dry}$

Ethylbenzene 12

M-xylene *

O-xylene *

Isopropylbenzene 4

1-ethyl-4-methylbenzene 11

1,2,4-trimethylbenzene 1

Secbutylbenzene *

1-methyl-4-isopropylbenzene 10

1-3-dimethyl-5-ethylbenzene *

1-2-diethylbenzene *

1-2-dimethyl-4-ethylbenzene 6

1,2,3,5-tetramethylbenzene *

Naphthalene 24

Benzothiophene *

2-methylnaphthalene 12

1-methylnaphthalene 9

Biphenyl 2

2-6-dimethylnaphthalene 5

2,3,6-trimethylnaphthalene *

Fluorene 6

Dibenzothiophene 31

Phenanthrene 17

Anthracene *

1-methylphenanthrene 10

Fluoranthene 9

Pyrene 17

Chrysene *

Benzo(e)pyrene 24

Benzo(a)pyrene *

Perylene 99

SUM(Naphthalene-Perylene) 266

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E-5-Top

Wet weight extracted (grams) 5

Dry:Wet weight ratio 0.32

Concentration units $\mu\text{g/kg dry}$

Ethylbenzene 3344

M-xylene 5404

O-xylene 461

Isopropylbenzene 13

1-ethyl-4-methylbenzene 259

1,2,4-trimethylbenzene 810

Secbutylbenzene 979

1-methyl-4-isopropylbenzene 11671

1-3-dimethyl-5-ethylbenzene 237

1-2-diethylbenzene *

1-2-dimethyl-4-ethylbenzene 1621

1,2,3,5-tetramethylbenzene 277

Naphthalene 6337

Benzothiophene 605

2-methylnaphthalene 3257

1-methylnaphthalene 2493

Biphenyl 1285

2-6-dimethylnaphthalene 3353

2,3,6-trimethylnaphthalene 1105

Fluorene 6756

Dibenzothiophene 6626

Phenanthrene 20116

Anthracene 9123

1-methylphenanthrene 2278

Fluoranthene 27554

Pyrene 45330

Chrysene 23773

Benzo(e)pyrene 24592

Benzo(a)pyrene 24915

Perylene 11219

SUM(Naphthalene-Perylene) 222922

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D. E-5-Bottom

Wet weight extracted (grams) 50

Dry:Wet weight ratio 0.76

Concentration units $\mu\text{g/kg dry}$

Ethylbenzene *

M-xylene *

O-xylene *

Isopropylbenzene *

1-ethyl-4-methylbenzene *

1,2,4-trimethylbenzene 8

Secbutylbenzene *

1-methyl-4-isopropylbenzene 5

1-3-dimethyl-5-ethylbenzene *

1-2-diethylbenzene *

1-2-dimethyl-4-ethylbenzene 6

1,2,3,5-tetramethylbenzene *

Naphthalene 471

Benzothiophene 79

2-methylnaphthalene 6

1-methylnaphthalene 6

Biphenyl 3

2-6-dimethylnaphthalene *

2,3,6-trimethylnaphthalene *

Fluorene 7

Dibenzothiophene 32

Phenanthrene 21

Anthracene 9

1-methylphenanthrene 29

Fluoranthene 17

Pyrene 33

Chrysene 36

Benzo(e)pyrene *

Benzo(a)pyrene *

Perylene *

SUM(Naphthalene-Perylene) 749

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E-6-Bottom

Wet weight extracted (grams) 50

Dry:Wet weight ratio 0.55

Concentration units $\mu\text{g/kg dry}$

Ethylbenzene *

M-xylene *

O-xylene *

Isopropylbenzene *

1-ethyl-4-methylbenzene 19

1,2,4-trimethylbenzene 11

Secbutylbenzene 154

1-methyl-4-isopropylbenzene *

1-3-dimethyl-5-ethylbenzene 12

1-2-diethylbenzene *

1-2-dimethyl-4-ethylbenzene 11

1,2,3,5-tetramethylbenzene 4

Naphthalene 242

Benzothiophene 11

2-methylnaphthalene 160

1-methylnaphthalene 105

Biphenyl 19

2-6-dimethylnaphthalene 26

2,3,6-trimethylnaphthalene 22

Fluorene 92

Dibenzothiophene 70

Phenanthrene 279

Anthracene 79

1-methylphenanthrene 53

Fluoranthene 107

Pyrene 278

Chrysene 82

Benzo(e)pyrene *

Benzo(a)pyrene 111

Perylene 421

SUM(Naphthalene-Perylene) 2155

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E-7-Top

Wet weight extracted (grams)

5

Dry:Wet weight ratio

0.34

Concentration units

 $\mu\text{g/kg dry}$

Ethylbenzene

804

M-xylene

1479

O-xylene

*

Isopropylbenzene

*

1-ethyl-4-methylbenzene

485

1,2,4-trimethylbenzene

1635

Secbutylbenzene

*

1-methyl-4-isopropylbenzene

22044

1-3-dimethyl-5-ethylbenzene

434

1-2-diethylbenzene

*

1-2-dimethyl-4-ethylbenzene

*

1,2,3,5-tetramethylbenzene

225

Naphthalene

3512

Benzothiophene

*

2-methylnaphthalene

931

1-methylnaphthalene

633

Biphenyl

613

2-6-dimethylnaphthalene

1050

2,3,6-trimethylnaphthalene

166

Fluorene

751

Dibenzothiophene

1317

Phenanthrene

3485

Anthracene

977

1-methylphenanthrene

820

Fluoranthene

3143

Pyrene

6626

Chrysene

3614

Benzo(e)pyrene

5129

Benzo(a)pyrene

640

Perylene

1390

SUM(Naphthalene-Perylene)

36998

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D. E-7-Bottom

Wet weight extracted (grams) 50
 Dry:Wet weight ratio 0.75
 Concentration units $\mu\text{g/kg dry}$

Ethylbenzene	165
M-xylene	244
O-xylene	24
Isopropylbenzene	2
1-ethyl-4-methylbenzene	13
1,2,4-trimethylbenzene	*
Secbutylbenzene	3
1-methyl-4-isopropylbenzene	*
1-3-dimethyl-5-ethylbenzene	1
1-2-diethylbenzene	*
1-2-dimethyl-4-ethylbenzene	2
1,2,3,5-tetramethylbenzene	*
Naphthalene	*
Benzothiophene	*
2-methylnaphthalene	2
1-methylnaphthalene	2
Biphenyl	1
2-6-dimethylnaphthalene	*
2,3,6-trimethylnaphthalene	*
Fluorene	*
Dibenzothiophene	7
Phenanthrene	3
Anthracene	*
1-methylphenanthrene	*
Fluoranthene	*
Pyrene	*
Chrysene	*
Benzo(e)pyrene	50
Benzo(a)pyrene	*
Perylene	295
SUM(Naphthalene-Perylene)	359

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E-8-Top

Wet weight extracted (grams)

5

Dry:Wet weight ratio

0.30

Concentration units

 $\mu\text{g/kg dry}$

Ethylbenzene

*

M-xylene

1594

O-xylene

*

Isopropylbenzene

*

1-ethyl-4-methylbenzene

*

1,2,4-trimethylbenzene

*

Secbutylbenzene

352

1-methyl-4-isopropylbenzene

7575

1-3-dimethyl-5-ethylbenzene

56

1-2-diethylbenzene

*

1-2-dimethyl-4-ethylbenzene

396

1,2,3,5-tetramethylbenzene

339

Naphthalene

2006

Benzothiophene

*

2-methylnaphthalene

944

1-methylnaphthalene

721

Biphenyl

298

2-6-dimethylnaphthalene

808

2,3,6-trimethylnaphthalene

431

Fluorene

841

Dibenzothiophene

2589

Phenanthrene

3275

Anthracene

1447

1-methylphenanthrene

3445

Fluoranthene

3200

Pyrene

7996

Chrysene

3619

Benzo(e)pyrene

5389

Benzo(a)pyrene

2301

Perylene

7369

SUM(Naphthalene-Perylene)

46680

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E-8-Bottom

Wet weight extracted (grams)

50

Dry:Wet weight ratio

0.71

Concentration units

 $\mu\text{g/kg dry}$

Ethylbenzene

*

M-xylene

*

O-xylene

*

Isopropylbenzene

*

1-ethyl-4-methylbenzene

4

1,2,4-trimethylbenzene

8

Secbutylbenzene

*

1-methyl-4-isopropylbenzene

302

1-3-dimethyl-5-ethylbenzene

1

1-2-diethylbenzene

*

1-2-dimethyl-4-ethylbenzene

4

1,2,3,5-tetramethylbenzene

*

Naphthalene

347

Benzothiophene

*

2-methylnaphthalene

34

1-methylnaphthalene

22

Biphenyl

11

2,6-dimethylnaphthalene

14

2,3,6-trimethylnaphthalene

10

Fluorene

41

Dibenzothiophene

61

Phenanthrene

243

Anthracene

59

1-methylphenanthrene

35

Fluoranthene

143

Pyrene

389

Chrysene

71

Benzo(e)pyrene

1035

Benzo(a)pyrene

*

Perylene

213

SUM(Naphthalene-Perylene)

2730

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E-9-Top

Wet weight extracted (grams)

5

Dry:Wet weight ratio

0.29

Concentration units

 $\mu\text{g/kg dry}$

Ethylbenzene

*

M-xylene

*

O-xylene

*

Isopropylbenzene

*

1-ethyl-4-methylbenzene

275

1,2,4-trimethylbenzene

*

Secbutylbenzene

*

1-methyl-4-isopropylbenzene

1920

1-3-dimethyl-5-ethylbenzene

*

1-2-diethylbenzene

*

1-2-dimethyl-4-ethylbenzene

379

1,2,3,5-tetramethylbenzene

30

Naphthalene

1360

Benzothiophene

62

2-methylnaphthalene

332

1-methylnaphthalene

223

Biphenyl

140

2-6-dimethylnaphthalene

427

2,3,6-trimethylnaphthalene

418

Fluorene

890

Dibenzothiophene

2175

Phenanthrene

2632

Anthracene

1893

1-methylphenanthrene

2911

Fluoranthene

6042

Pyrene

11693

Chrysene

5724

Benzo(e)pyrene

10721

Benzo(a)pyrene

10402

Perylene

11757

SUM(Naphthalene-Perylene)

69803

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E-9-Bottom

Wet weight extracted (grams)

50

Dry:Wet weight ratio

0.56

Concentration units

$\mu\text{g/kg dry}$

Ethylbenzene

*

M-xylene

*

O-xylene

*

Isopropylbenzene

*

1-ethyl-4-methylbenzene

*

1,2,4-trimethylbenzene

*

Secbutylbenzene

*

1-methyl-4-isopropylbenzene

28

1-3-dimethyl-5-ethylbenzene

*

1-2-diethylbenzene

*

1-2-dimethyl-4-ethylbenzene

*

1,2,3,5-tetramethylbenzene

*

Naphthalene

36

Benzothiophene

*

2-methylnaphthalene

19

1-methylnaphthalene

17

Biphenyl

5

2-6-dimethylnaphthalene

12

2,3,6-trimethylnaphthalene

7

Fluorene

23

Dibenzothiophene

79

Phenanthrene

118

Anthracene

61

1-methylphenanthrene

90

Fluoranthene

159

Pyrene

421

Chrysene

386

Benzo(e)pyrene

449

Benzo(a)pyrene

847

Perylene

*

SUM(Naphthalene-Perylene)

2727

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E-10-Top

Wet weight extracted (grams) 5
 Dry:Wet weight ratio 0.30
 Concentration units $\mu\text{g/kg dry}$

Ethylbenzene	735	
M-xylene	1167	
O-xylene	*	
Isopropylbenzene	*	
1-ethyl-4-methylbenzene	406	
1,2,4-trimethylbenzene	27	
Secbutylbenzene	932	
1-methyl-4-isopropylbenzene	13033	
1-3-dimethyl-5-ethylbenzene	*	
1-2-diethylbenzene	*	
1-2-dimethyl-4-ethylbenzene	*	
1,2,3,5-tetramethylbenzene	79	
Naphthalene	630
Benzothiophene	*	
2-methylnaphthalene	291
1-methylnaphthalene	129
Biphenyl	60
2-6-dimethylnaphthalene	197
2,3,6-trimethylnaphthalene	221
Fluorene	498
Dibenzothiophene	1398
Phenanthrene	883
Anthracene	359
1-methylphenanthrene	1235
Fluoranthene	2467
Pyrene	5764
Chrysene	1735
Benzo(e)pyrene	*	
Benzo(a)pyrene	1035
Perylene	759
SUM (Naphthalene-Perylene)	17981	

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E-10-Bottom

Wet weight extracted (grams) 50

Dry:Wet weight ratio 0.72

Concentration units $\mu\text{g/kg dry}$

Ethylbenzene	*	
M-xylene	*	
O-xylene	*	
Isopropylbenzene	*	
1-ethyl-4-methylbenzene	7	
1,2,4-trimethylbenzene	1	
Secbutylbenzene	*	
1-methyl-4-isopropylbenzene	6	
1-3-dimethyl-5-ethylbenzene	*	
1-2-diethylbenzene	*	
1-2-dimethyl-4-ethylbenzene	1	
1,2,3,5-tetramethylbenzene	*	
Naphthalene	1	
Benzothiophene	*	
2-methylnaphthalene	4	▨
1-methylnaphthalene	*	
Biphenyl	1	
2-6-dimethylnaphthalene	*	
2,3,6-trimethylnaphthalene	2	▨
Fluorene	*	
Dibenzothiophene	32	▨
Phenanthrene	*	
Anthracene	3	▨
1-methylphenanthrene	36	▨
Fluoranthene	1	
Pyrene	50	▨
Chrysene	*	
Benzo(e)pyrene	*	
Benzo(a)pyrene	*	
Perylene	*	
SUM(Naphthalene-Perylene)	132	

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E-11-Top

Wet weight extracted (grams)

5

Dry:Wet weight ratio

0.22

Concentration units

 $\mu\text{g/kg dry}$

Ethylbenzene 33854

M-xylene 40830

O-xylene 11324

Isopropylbenzene 493

1-ethyl-4-methylbenzene 1574

1,2,4-trimethylbenzene 3922

Secbutylbenzene 1745

1-methyl-4-isopropylbenzene 135924

1-3-dimethyl-5-ethylbenzene *

1-2-diethylbenzene *

1-2-dimethyl-4-ethylbenzene *

1,2,3,5-tetramethylbenzene 1174

Naphthalene 14535

Benzothiophene *

2-methylnaphthalene 5222

1-methylnaphthalene 5532

Biphenyl 3856

2-6-dimethylnaphthalene 6782

2,3,6-trimethylnaphthalene 5086

Fluorene 3936

Dibenzothiophene 2771

Phenanthrene 10305

Anthracene 1982

1-methylphenanthrene 2859

Fluoranthene *

Pyrene *

Chrysene 3173

Benzo(e)pyrene *

Benzo(a)pyrene *

Perylene 3366

SUM(Naphthalene-Perylene)

69408

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E-12-Top

Wet weight extracted (grams)

5

Dry:Wet weight ratio

0.30

Concentration units

 $\mu\text{g/kg dry}$

Ethylbenzene

*

M-xylene

610

O-xylene

*

Isopropylbenzene

*

1-ethyl-4-methylbenzene

366

1,2,4-trimethylbenzene

225

Secbutylbenzene

309

1-methyl-4-isopropylbenzene

6290

1-3-dimethyl-5-ethylbenzene

*

1-2-diethylbenzene

*

1-2-dimethyl-4-ethylbenzene

*

1,2,3,5-tetramethylbenzene

79

Naphthalene

2971

Benzothiophene

*

2-methylnaphthalene

566

1-methylnaphthalene

480

Biphenyl

166

2-6-dimethylnaphthalene

777

2,3,6-trimethylnaphthalene

514

Fluorene

790

Dibenzothiophene

*

Phenanthrene

3030

Anthracene

963

1-methylphenanthrene

1326

Fluoranthene

3097

Pyrene

6869

Chrysene

1854

Benzo(e)pyrene

*

Benzo(a)pyrene

897

Perylene

1232

SUM(Naphthalene-Perylene)

25532

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E-12-Bottom

Wet weight extracted (grams)

50

Dry:Wet weight ratio

0.72

Concentration units

 $\mu\text{g/kg dry}$

Ethylbenzene

*

M-xylene

*

O-xylene

*

Isopropylbenzene

*

1-ethyl-4-methylbenzene

*

1,2,4-trimethylbenzene

*

Secbutylbenzene

*

1-methyl-4-isopropylbenzene

5

1-3-dimethyl-5-ethylbenzene

*

1-2-diethylbenzene

*

1-2-dimethyl-4-ethylbenzene

2

1,2,3,5-tetramethylbenzene

*

Naphthalene

1

Benzothiophene

*

2-methylnaphthalene

2

1-methylnaphthalene

2

Biphenyl

1

2-6-dimethylnaphthalene

*

2,3,6-trimethylnaphthalene

*

Fluorene

*

Dibenzothiophene

34

Phenanthrene

4

Anthracene

1

1-methylphenanthrene

6

Fluoranthene

*

Pyrene

*

Chrysene

*

Benzo(e)pyrene

6

Benzo(a)pyrene

44

Perylene

*

SUM(Naphthalene-Perylene)

103

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E-13-Top

Wet weight extracted (grams) 5
 Dry:Wet weight ratio 0.24
 Concentration units $\mu\text{g/kg dry}$

Ethylbenzene	*	
M-xylene	*	
O-xylene	*	
Isopropylbenzene	*	
1-ethyl-4-methylbenzene	125	
1,2,4-trimethylbenzene	*	
Secbutylbenzene	*	
1-methyl-4-isopropylbenzene	467	
1-3-dimethyl-5-ethylbenzene	*	
1-2-diethylbenzene	*	
1-2-dimethyl-4-ethylbenzene	*	
1,2,3,5-tetramethylbenzene	*	
Naphthalene	476	
Benzothiophene	*	
2-methylnaphthalene	209	
1-methylnaphthalene	98	
Biphenyl	72	
2-6-dimethylnaphthalene	65	
2,3,6-trimethylnaphthalene	*	
Fluorene	342	
Dibenzothiophene	1990	
Phenanthrene	1038	
Anthracene	855	
1-methylphenanthrene	2116	
Fluoranthene	3876	
Pyrene	7856	
Chrysene	2811	
Benzo(e)pyrene	1559	
Benzo(a)pyrene	1639	
Perylene	2073	
SUM(Naphthalene-Perylene)	27096	

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E-13-Mid

Wet weight extracted (grams) 15

Dry:Wet weight ratio 0.52

Concentration units $\mu\text{g}/\text{kg}$ dry

Ethylbenzene	*	
M-xylene	*	
O-xylene	*	
Isopropylbenzene	*	
1-ethyl-4-methylbenzene	35	
1,2,4-trimethylbenzene	143	
Secbutylbenzene	56	
1-methyl-4-isopropylbenzene	248	
1-3-dimethyl-5-ethylbenzene	22	
1-2-diethylbenzene	*	
1-2-dimethyl-4-ethylbenzene	27	
1,2,3,5-tetramethylbenzene	43	
Naphthalene	1589
Benzothiophene	60
2-methylnaphthalene	445
1-methylnaphthalene	280
Biphenyl	125
2-6-dimethylnaphthalene	380
2,3,6-trimethylnaphthalene	267
Fluorene	1028
Dibenzothiophene	1341
Phenanthrene	2516
Anthracene	1484
1-methylphenanthrene	557
Fluoranthene	3205
Pyrene	5838
Chrysene	3616
Benzo(e)pyrene	3376
Benzo(a)pyrene	7542
Perylene	4601
SUM(Naphthalene-Perylene)	38252	

* < 1.0 $\mu\text{g}/\text{kg}$ dry weight

Sample I.D.

E-13-Bottom

Wet weight extracted (grams)

50

Dry:Wet weight ratio

0.72

Concentration units

$\mu\text{g/kg dry}$

Ethylbenzene	*	
M-xylene	*	
O-xylene	6	
Isopropylbenzene	2	
1-ethyl-4-methylbenzene	3	
1,2,4-trimethylbenzene	3	
Secbutylbenzene	*	
1-methyl-4-isopropylbenzene	7	
1-3-dimethyl-5-ethylbenzene	*	
1-2-diethylbenzene	*	
1-2-dimethyl-4-ethylbenzene	6	
1,2,3,5-tetramethylbenzene	*	
Naphthalene	7
Benzothiophene	*	
2-methylnaphthalene	6
1-methylnaphthalene	4
Biphenyl	1
2-6-dimethylnaphthalene	3
2,3,6-trimethylnaphthalene	*	
Fluorene	2
Dibenzothiophene	13
Phenanthrene	2
Anthracene	3
1-methylphenanthrene	*	
Fluoranthene	11
Pyrene	17
Chrysene	9
Benzo(e)pyrene	16
Benzo(a)pyrene	*	
Perylene	25
SUM(Naphthalene-Perylene)	119	

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E-14-Top

Wet weight extracted (grams) 5

Dry:Wet weight ratio 0.30

Concentration units $\mu\text{g/kg dry}$

Ethylbenzene	*	
M-xylene	2750	
O-xylene	*	
Isopropylbenzene	*	
1-ethyl-4-methylbenzene	*	
1,2,4-trimethylbenzene	231	
Secbutylbenzene	*	
1-methyl-4-isopropylbenzene	11225	
1-3-dimethyl-5-ethylbenzene	46	
1-2-diethylbenzene	*	
1-2-dimethyl-4-ethylbenzene	*	
1,2,3,5-tetramethylbenzene	71	
Naphthalene	2615
Benzo(b)thiophene	*	
2-methylnaphthalene	431
1-methylnaphthalene	164
Biphenyl	156
2-6-dimethylnaphthalene	446
2,3,6-trimethylnaphthalene	361	
Fluorene	543
Dibenzothiophene	*	
Phenanthrene	2262
Anthracene	635
1-methylphenanthrene	1428
Fluoranthene	2422
Pyrene	5626
Chrysene	1504
Benzo(e)pyrene	*	
Benzo(a)pyrene	*	
Perylene	1195
SUM(Naphthalene-Perylene)	20190	

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D. E-14-Bottom

Wet weight extracted (grams) 50
 Dry:Wet weight ratio 0.71
 Concentration units $\mu\text{g/kg dry}$

Ethylbenzene	*	
M-xylene	37	
O-xylene	17	
Isopropylbenzene	7	
1-ethyl-4-methylbenzene	8	
1,2,4-trimethylbenzene	2	
Secbutylbenzene	*	
1-methyl-4-isopropylbenzene	3	
1-3-dimethyl-5-ethylbenzene	1	
1-2-diethylbenzene	*	
1-2-dimethyl-4-ethylbenzene	8	
1,2,3,5-tetramethylbenzene	2	
Naphthalene	1
Benzothiophene	*	
2-methylnaphthalene	5
1-methylnaphthalene	3
Biphenyl	7
2-6-dimethylnaphthalene	4
2,3,6-trimethylnaphthalene	*	
Fluorene	1
Dibenzothiophene	56
Phenanthrene	8
Anthracene	5
1-methylphenanthrene	3
Fluoranthene	10
Pyrene	6
Chrysene	17
Benzo(e)pyrene	9
Benzo(a)pyrene	23
Perylene	*	
SUM(Naphthalene-Perylene)	157	

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E-15-Top

Wet weight extracted (grams) 5

Dry:Wet weight ratio 0.26

Concentration units $\mu\text{g}/\text{kg dry}$

Ethylbenzene 2055

M-xylene 3078

O-xylene *

Isopropylbenzene *

1-ethyl-4-methylbenzene 463

1,2,4-trimethylbenzene 134

Secbutylbenzene 266

1-methyl-4-isopropylbenzene 4479

1-3-dimethyl-5-ethylbenzene *

1-2-diethylbenzene *

1-2-dimethyl-4-ethylbenzene *

1,2,3,5-tetramethylbenzene 65

Naphthalene 1907

Benzothiophene *

2-methylnaphthalene 503

1-methylnaphthalene 309

Biphenyl 476

2-6-dimethylnaphthalene 1038

2,3,6-trimethylnaphthalene 1164

Fluorene 1334

Dibenzothiophene 2575

Phenanthrene 3426

Anthracene 2628

1-methylphenanthrene 2285

Fluoranthene 4762

Pyrene 9980

Chrysene 6041

Benzo(e)pyrene *

Benzo(a)pyrene 1314

Perylene 1550

SUM(Naphthalene-Perylene) 41293

* < 1.0 $\mu\text{g}/\text{kg dry weight}$

Sample I.D.

E-15-Bottom

Wet weight extracted (grams)

50

Dry:Wet weight ratio

0.73

Concentration units

µg/kg dry

Ethylbenzene

*

M-xylene

*

O-xylene

*

Isopropylbenzene

*

1-ethyl-4-methylbenzene

5

1,2,4-trimethylbenzene

*

Secbutylbenzene

1

1-methyl-4-isopropylbenzene

17

1-3-dimethyl-5-ethylbenzene

*

1-2-diethylbenzene

*

1-2-dimethyl-4-ethylbenzene

3

1,2,3,5-tetramethylbenzene

4

Naphthalene

18

Benzothiophene

*

2-methylnaphthalene

9

1-methylnaphthalene

7

Biphenyl

4

2-6-dimethylnaphthalene

5

2,3,6-trimethylnaphthalene

*

Fluorene

3

Dibenzothiophene

49

Phenanthrene

19

Anthracene

*

1-methylphenanthrene

23

Fluoranthene

17

Pyrene

61

Chrysene

12

Benzo(e)pyrene

*

Benzo(a)pyrene

*

Perylene

*

SUM(Naphthalene-Perylene)

227

* < 1.0 µg/kg dry weight

Sample I.D.

E-16-Bottom

Wet weight extracted (grams) 50

Dry:Wet weight ratio 0.71

Concentration units $\mu\text{g/kg dry}$

Ethylbenzene	*	
M-xylene	*	
O-xylene	325
Isopropylbenzene	7
1-ethyl-4-methylbenzene	7
1,2,4-trimethylbenzene	1	
Secbutylbenzene	*	
1-methyl-4-isopropylbenzene	3
1-3-dimethyl-5-ethylbenzene	2
1-2-diethylbenzene	*	
1-2-dimethyl-4-ethylbenzene	*	
1,2,3,5-tetramethylbenzene	1	
Naphthalene	1	
Benzothiophene	*	
2-methylnaphthalene	5
1-methylnaphthalene	3
Biphenyl	7
2-6-dimethylnaphthalene	4
2,3,6-trimethylnaphthalene	*	
Fluorene	2	
Dibenzothiophene	91
Phenanthrene	7
Anthracene	7
1-methylphenanthrene	8
Fluoranthene	10
Pyrene	6
Chrysene	12
Benzo(e)pyrene	15
Benzo(a)pyrene	35
Perylene	*	
SUM(Naphthalene-Perylene)	212	

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E-17-Top

Wet weight extracted (grams) 5
 Dry:Wet weight ratio 0.28
 Concentration units $\mu\text{g/kg dry}$

Ethylbenzene	8842	
M-xylene	9081	
O-xylene	989	
Isopropylbenzene	*	
1-ethyl-4-methylbenzene	474	
1,2,4-trimethylbenzene	121	
Secbutylbenzene	*	
1-methyl-4-isopropylbenzene	1785	
1-3-dimethyl-5-ethylbenzene	*	
1-2-diethylbenzene	*	
1-2-dimethyl-4-ethylbenzene	*	
1,2,3,5-tetramethylbenzene	*	
Naphthalene	348
Benzothiophene	*	
2-methylnaphthalene	193
1-methylnaphthalene	119
Biphenyl	66
2-6-dimethylnaphthalene	96
2,3,6-trimethylnaphthalene	251
Fluorene	143
Dibenzothiophene	763
Phenanthrene	387
Anthracene	256
1-methylphenanthrene	571
Fluoranthene	1124
Pyrene	2342
Chrysene	559
Benzo(e)pyrene	*	
Benzo(a)pyrene	*	
Perylene	3495
SUM(Naphthalene-Perylene)	10714	

* < 1.0 $\mu\text{g/kg dry weight}$

Sample L.D.

E-17-Bottom

Wet weight extracted (grams) 50

Dry:Wet weight ratio 0.72

Concentration units $\mu\text{g/kg dry}$

Ethylbenzene	*	
M-xylene	*	
O-xylene	*	
Isopropylbenzene	11	
1-ethyl-4-methylbenzene	7	
1,2,4-trimethylbenzene	*	
Secbutylbenzene	*	
1-methyl-4-isopropylbenzene	14	
1-3-dimethyl-5-ethylbenzene	2	
1-2-diethylbenzene	*	
1-2-dimethyl-4-ethylbenzene	2	
1,2,3,5-tetramethylbenzene	1	
Naphthalene	34	
Benzothiophene	*	
2-methylnaphthalene	11	
1-methylnaphthalene	7	
Biphenyl	5	
2-6-dimethylnaphthalene	4	
2,3,6-trimethylnaphthalene	*	
Fluorene	7	
Dibenzothiophene	42	
Phenanthrene	33	
Anthracene	13	
1-methylphenanthrene	5	
Fluoranthene	25	
Pyrene	51	
Chrysene	22	
Benzo(e)pyrene	79	
Benzo(a)pyrene	112	
Perylene	339	
SUM(Naphthalene-Perylene)	790	

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E-18-Top

Wet weight extracted (grams) 25

Dry:Wet weight ratio 0.76

Concentration units $\mu\text{g/kg dry}$

Ethylbenzene 102

M-xylene 141

O-xylene *

Isopropylbenzene *

1-ethyl-4-methylbenzene *

1,2,4-trimethylbenzene *

Secbutylbenzene 8

1-methyl-4-isopropylbenzene 7

1-3-dimethyl-5-ethylbenzene *

1-2-diethylbenzene *

1-2-dimethyl-4-ethylbenzene 2

1,2,3,5-tetramethylbenzene *

Naphthalene 1

Benzothiophene *

2-methylnaphthalene 3

1-methylnaphthalene 3

Biphenyl 1

2-6-dimethylnaphthalene *

2,3,6-trimethylnaphthalene *

Fluorene *

Dibenzothiophene 2

Phenanthrene 4

Anthracene *

1-methylphenanthrene 2

Fluoranthene *

Pyrene *

Chrysene *

Benzo(e)pyrene *

Benzo(a)pyrene *

Perylene *

SUM(Naphthalene-Perylene) 16

* < 1.0 $\mu\text{g/kg dry weight}$

Sample I.D.

E -19-Bottom

Wet weight extracted (grams) 50

Dry:Wet weight ratio 0.71

Concentration units $\mu\text{g/kg dry}$

Ethylbenzene	*	
M-xylene	*	
O-xylene	*	
Isopropylbenzene	*	
1-ethyl-4-methylbenzene	8	
1,2,4-trimethylbenzene	*	
Secbutylbenzene	3	
1-methyl-4-isopropylbenzene	*	
1-3-dimethyl-5-ethylbenzene	*	
1-2-diethylbenzene	*	
1-2-dimethyl-4-ethylbenzene	*	
1,2,3,5-tetramethylbenzene	*	
Naphthalene	5
Benzothiophene	*	
2-methylnaphthalene	4
1-methylnaphthalene	4
Biphenyl	1
2-8-dimethylnaphthalene	*	
2,3,6-trimethylnaphthalene	*	
Fluorene	*	
Dibenzothiophene	18
Phenanthrene	3
Anthracene	2
1-methylphenanthrene	*	
Fluoranthene	5
Pyrene	15
Chrysene	*	
Benzo(e)pyrene	*	
Benzo(a)pyrene	*	
Perylene	150
SUM(Naphthalene-Perylene)	207	

* < 1.0 $\mu\text{g/kg dry weight}$

Table A8 Samples containing a significant unresolved complex mixture (UCM) using Capillary Gas Chromatography and an Electron-Capture Detector

E-18-T	U- 1-B
E-16-T	A- 1-Grab
E- 3-B	
E- 2-B	E- 1-Top
E-17-B	E- 3-Top
	E- 5-Top
U- 2-B	E- 4-Top
U- 1-B	E- 8-Top
B- 9-B	E- 7-Top
	E-15-Top
	E- 4-M*
	E-13-M
	E-11-T

*Sample with largest UCM shown in figure 3.

TABLE A9.
SAMPLE I.D. DUWAMISH #2 REFERENCE SAMPLE (NOAA)

AROMATIC COMPONENT	MICROGRAMS/ KILOGRAM OF DRY SEDIMENT		

SAMPLE WEIGHT DRY WT./WET RATIO=.46			
14 GRAMS, DRY	924A REP 2	922A REP 1	926A REP 3

NAPHTHALENE	146	62	160
BENZOTHIOPHENE	0	0	0
2-METHYLNAPHTHALENE	92	57	96
1-METHYLNAPHTHALENE	57	33	60
BIPHENYL	35	26	31
2-6-DIMETHYLNAPHTHALENE	119	84	125
ACENAPHTHYLENE	0	16	10
ACENAPHTHENE	168	149	187
2,3,6-TRIMETHNAPHTHALENE	123	150	72
FLUORENE	201	387	178
DIBENZOTHIOPHENE	34	210	20
PHENANTHRENE	1111	873	925
ANTHRACENE	353	285	268
5,6-BENZOQUINOLINE	134	342	125
1-METHYLPHENANTHRENE	117	121	101
FLUORANTHENE	2491	2508	2377
PYRENE	2035	2090	1966
BENZO(A)ANTHRACENE	1262	1244	1169
CHRYSENE	2157	2038	1825
BENZO(B)FLUORANTHENE	2205	1999	1689
BENZO(K)FLUORANTHENE	260	366	304
B(E)PYRENE	999	1044	791
B(A)PYRENE	1847	2010	1531
PERYLENE	513	675	597
INDENO(1,2,3-C,D)PYRENE	897	560	932
BENZO(G,H,I)PERYLENE	710	582	722

Mac Leod et al. (1982) published ranges for each of the compounds derived from intercomparison studies. Our values are at or slightly above the high end of these reported concentrations.

QA PROJECT PLAN

for

Everett Harbor Sampling Program of
the U. S. Army Corps of Engineers

COE Interagency Agreement No.
TD 1819 Revision 1

Pacific Northwest Laboratory
Richland, WA 99352

Approvals:

9-20-84
J. W. Anderson, PNL Project Manager Date

9-20-84
J. A. Strand, PNL Environmental Sciences Date
Section Manager

9-21-84
B. E. Vaughan, PNL Environmental Sciences Date
Department Manager

9-24-84
J. E. Ruffin, PNL Quality Assurance Date
Office Manager

10-4-84
John Malek, COE Project Officer Date

TABLE OF CONTENTS

Introduction

This Quality Assurance (QA) Project Plan is for the U.S. Army Corps of Engineers (COE) sponsored project, "Everett Harbor Sampling Program" that is contracted to Pacific Northwest Laboratory (PNL) in COE Interagency Agreement No. (TD 1819). This plan includes the appropriate elements from the EPA document QAMS-005/80, "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans," and elements from PNL-MA-65, "Quality Assurance Manual." Revisions to this plan will be made by reissuing the sections with changes and the Title and Table of Contents pages or by reissuing the QA Plan. The Plan follows closely the EPA Region X QA Project Plan for "Water Quality Management Program for Puget Sound" contracted to PNL under Interagency Agreement No. (TD 1789).

<u>Section</u>	<u>Pages</u>	<u>Revision</u>	<u>Date</u>
1. Title Page	1	0	
2. Table of Contents	2	0	
3. Project Description	1	0	
4. Project Organization & Responsibility	2	0	
5. Quality Assurance Objectives	1	0	
6. Sampling Procedures	1	0	
7. Sample Custody	1	0	
8. Analytical Procedures	11	0	
9. Data Reduction, Validation and Reporting	1	0	
10. Internal Quality Control Checks	1	0	
11. Performances and System Audits	1	0	
12. Preventive Maintenance	1	0	
13. Specific Routine Procedures Used to Assess Data Precision, Accuracy and Completeness	1	0	

14. Corrective Action	1	0
15. Quality Assurance Reports to Management	1	0

Appendix

A. Applicable sections of PNL Quality Assurance Manual PNL-MA-65

Distribution

PNL

J. W. Anderson
E. A. Crecelius
S. L. English
J. E. Ruffin/QA File
D. E. Ryder
— J. A. Strand
W. L. Templeton
B. E. Vaughan

COE

Burt Hamner

PROJECT DESCRIPTION

It is the objective of this program to conduct chemical analyses of sediment from Everett Harbor. Two upland sites will be cored and cores will be taken by boat from 19 stations in the harbor. Four grab samples of sediment will be taken at near shore sites.

Locations for sampling, the methods and depth of sampling and the type of analyses to be conducted has all been specified by the sponsor (see Work Plan). The COE will be responsible for obtaining the sediment cores from 2 upland and 19 aquatic sites. Pacific Northwest Laboratory will provide one or more individuals to be present during sampling to catalog the time, location and details of the samples taken. These PNL staff will also be responsible for removing the sediment samples from the selected positions in the cores, placing them in precleaned containers, providing labeling and cataloging each in the project data book. The PNL staff will then transport samples on ice to the Marine Research Laboratory (MRL) where analysis of grain size, oil and grease, total solids, total volatile solids, trace metals (As, Cu, Cd, Hg, Pb and Zn), aromatic hydrocarbons and polychlorinated biphenyls (PCB as 1254) will take place. During analysis of PCB and PAH, the appearance of unknown peaks of significant magnitude or unresolved envelopes will be noted and brought to the attention of the COE. Subsamples of each sediment will be sent to subcontractors for analysis of total organic carbon and sulfides. At six selection stations one or two sections will be sampled for analysis of all priority pollutants which will be determined by a subcontractor.

PROJECT ORGANIZATION AND RESPONSIBILITY

All sediment sampling and most analyses will be performed by PNL personnel. Additional subcontractor will be responsible for analysis of sulfides and total organic carbon. Program management review will be provided by B. E. Vaughan, Department Manager, Ecological Sciences Department or his representative. Key project personnel, project responsibilities and line authority within PNL are shown in Figure 1.

The Project Manager is responsible for the scientific content of the program and the design and execution of methodologies and procedures in a manner that is consistent with good scientific practices and meets the QA/QC requirements set forth in this document.

The Project Manager shall be responsible for project quality assurance and the preparation and implementation of the project quality assurance plan.

Figure 1. Project Organization and Line Authority

QUALITY ASSURANCE OBJECTIVES

In general terms, the Quality Assurance objective of this research program is to collect representative samples, analyze them accurately and report the results in standard comparable formats. The sampling plan and means of obtaining sediment samples has already been determined by COE.

Accuracy and Precision

For trace metal and organic pollutant analysis, the objective is that the total error be no greater than 10% of the accepted standard value. The objective for precision is 20% relative standard deviation assuming a 95% confidence level and a normal distribution.

Overall accuracy will be affected by error in sampling. Strict adherence to the sampling protocol will help assure the representativeness of the samples. Duplicate samples from the same sampling location will be analyzed to provide data on with-in laboratory and sampling error.

Completeness

The target for completeness of data in order to meet the project needs is 80% and the measure to be used is the percent of total reported data classified as valid in the data validation process. All laboratory data will be reviewed for completeness and checked for correct procedures, instrument performance, calibration, and reasonableness.

Comparability

Toxicant concentrations will be expressed in units consistent with other organizations reporting similar data, e.g., mg/l, µg/l, mg/g, µg/g, parts per million, parts per billion.

SAMPLING PROCEDURES

The sampling grid to be used in site selection has been designated by the sponsor (COE) and is shown in the Work Plan. The method of sampling on upland sites (2) will be a drilling device which together with the personnel will be supplied by the COE. Pacific Northwest Laboratory will coordinate sample taking from selected depths in these cores and provide labelled sample containers. On board ship PNL staff will be present to handle the cores taken by COE. The field coordinator (E. A. Crecelius) will be present on the boat or will provide a trained staff member to section the cores at the proper depth and transfer samples of sediment from the cores to precleaned, labelled containers. All aquatic samples will be kept cold (on ice or in a refrigerator) until receipt at MRL in Sequim. It is anticipated that at 2- or 3-day intervals shipments of samples back to MRL will take place.

SAMPLE CUSTODY

At the beginning of each cruise day, all cleaned subsample containers anticipated for use on that day will be placed aboard the vessel. The containers will be marked with a serial number representing a station code number, a replicate number and coordinates. During the course of the cruise day, the appropriate size aliquots will be taken from the samples and the containers holding aliquots will be sealed. During the cruise day the samples will be in the custody of the PNL staff member on board the vessel. At the close of the cruise day, the collection date will be inscribed on each aliquot container, the subsamples will be signed by the collector and a list of samples and subsamples taken will be logged in the PNL official Laboratory Record Book(s) assigned to the project. The Laboratory Record Book will also provide a record of the disposition of the subsample. A copy of the sample manifest will be given to subcontractors at the time of sample transfer. The custody of the remaining samples will remain with PNL. The remaining subsamples will be delivered to PNL's Marine Research Laboratory, Sequim, Washington. There they will be given to the appropriate analytical staff who will log the receipt of these samples into their appropriate PNL Record Books. As analyses are completed, the data pertaining to those samples will also be logged into the analytical Laboratory Record Books.

Laboratory Record Books shall be uniquely numbered, bound and paginated. Data entries shall be:

- o dedicated solely to this project
- o in ink
- o complete enough to provide who, what, when, where, how and what happened
- o with no erasures or snopake. Errant data will be crossed out with a single line and initiated and dated. If more than a transcription error, reason shall be noted.
- o signed by person entering the data
- o co-signed by Project Manager to indicate review and acceptance.

ANALYTICAL PROCEDURES

Total Extractable Hydrocarbons (Oil and Grease)

Sampling

Twenty grams of sediment, 20 g of anhydrous sodium sulfate and 25 ml of carbon tetrachloride (CCl_4) will be placed into a labeled 250-ml bottle with ground glass stopper. The bottle will be placed on a shaker and agitated overnight (16 h). The CCl_4 containing the extractable organic material will receive infrared analysis.

Analytical Procedures

Infrared analysis of total extractable hydrocarbons will be done using a modification of the procedure described by Simard (A. G. Simard et al., Anal. Chem. 23:1384 [1951]).

Analysis will be done on a Beckman® Acculab 4 infrared spectrophotometer in accordance with the instrument's operating manual.

Calibration Procedures and Frequency

IR spectrophotometer shall be calibrated weekly when used by means of a Beckman® polystyrene wavelength/wavenumber calibrator for IR spectrophotometers, part No. 80785; Polystyrene film vs. air (absorbance and frequency after APAC 1961).

Grain-Size Analysis

Grain-size analysis will be to estimate percent fractions of gravel, sand, silt and clay. An approximate 100 g wet weight sample will be wet sieved through 2-mm and 62-micron screens. Material retained on the screens will be dried (100°C for 24 h) and weighed to give total sample contributions

for gravel and sand respectively. Material washed into the pan will be transferred to a 1-liter graduated cylinder and distilled water will be added to bring the total volume to 1 liter. The graduated cylinder will be stoppered and inverted three times for mixing and then allowed to stand for 7 minutes. Ten-ml pipette samples will be taken in the upper quarter and lower quarter of the graduated cylinder to determine concentrations of silt and clay respectively. Contents of the pipettes will be dried (100°C for 24 h) and the residue weighed. Weight of residue times 100 will be used to calculate total weight of silt and clay in sample. Percent contribution of each fraction will be based on the aggregate weight of all fractions.

Total Solids and Total Volatile Solids

Total Residue and Moisture (Preparation of Evaporating Dish)

Evaporating dishes will be ignited for at least 60 min at a temperature of at least 550°C, then cooled in a desiccator, weighed to the nearest 10 mg, and kept in the desiccator until ready for use.

Fluid Samples

If the (25-50 g) sample contains enough moisture to flow more or less readily, it will be stirred to homogenize, then placed in a prepared evaporating dish and weighed to the nearest 10 mg. It will be evaporated to dryness on a water bath, dried at 103°C for 1 hr, cooled in an individual desiccator containing fresh desiccant, and weighed.

Solid Samples

If the sample consists of discrete pieces of solid material (dewatered sludge, for example) cores will be taken from each piece with a No. 7 cork borer, or pulverized on a clean surface by hand, using rubber gloves. After 25-50 g is placed in a prepared evaporating dish and weighed to the nearest 10 mg, it will be placed in an oven at 103°C overnight. It will ultimately

be cooled in an individual desiccator containing fresh desiccant and weighed. Heating for this prolonged period may result in a loss of volatile organic matter and ammonium carbonate, but it is usually necessary in order to realize thorough drying of samples of this kind.

Volatile Residue

Volatile residue which includes organic matter and volatile inorganic salts will be determined as for total residue above by igniting it in an electric muffle furnace at 550°C for 60 minutes, avoiding loss of solids by decrepitation. It will then be cooled in a desiccator and reweighed. Results will be reported as percent ash and volatile solids.

Calculations.

$$\text{Total residue (\%)} = \frac{A \times 100}{B}$$

$$\text{Volatile residue (\%)} = \frac{A - C}{A} \times 100$$

$$\text{Fixed residue (\%)} = \frac{C \times 100}{A}$$

where A = weight of dried solids
B = weight of sample
C = weight of ash.

Precision and accuracy. On digested sludge (42% ash) the allowable standard deviation shall be 0.2% ash (n = 1; 13 x 10). On elutriated sludge (29% ash) the standard deviation shall to be 0.2 percent ash (n = 1; 4 x 10).

Analysis of Trace Metals

Sediments

Sample Handling. Sediment samples to be analyzed for trace metals will be 30-g aliquots taken from well-homogenized primary samples. Aliquots will be taken with a Teflon-coated[®] spatula and placed into acid-cleaned polystyrene jars.

Prior to analysis, the samples will be freeze-dried to constant weight with a Vitris[®] freeze-mobile 12, and ultimately homogenized on a Spex 8000[®] mixer-mill. All handling of the samples will be carried out in the container of collection so no tool ever touches the sample. This procedure has been shown to yield no contamination for any trace metal measured.

Analytical Procedure

Sediment Digestion Procedure. Prior to analysis by atomic absorption spectroscopy, the samples will be digested in a 5:2:5 mixture of HNO_3 , H_2SO_4 , and H_2O (Bloom, 1983). This procedure oxidizes all organic matter and releases all anthropogenic Pb, Cd, Zn, Cu, Hg, As, Tl, Ag and Be. All digestates will be made up to a dilution of 1.0 g of sediment (dry weight) to 100 ml of solution. This insures greater reproducibility in graphite furnace analysis and ease of data reduction.

Atomic Absorption Procedures: Lead, Copper, Cadmium, Thallium, Silver, Beryllium and Zinc. Lead, Cu, Cd, Tl, Ag, Be, and Zn will be measured using graphite furnace Zeeman atomic Absorption Spectroscopy (GFZAAS). Through the use of state-of-the-art Perkin-Elmer[®] Zeeman background corrector and Model 5000 spectrophotometer, very accurate measurements are provided even in such complex matrices as digested sediments. The detection limits will be: cadmium and zinc, 0.005 mg g^{-1} dry weight, and lead and copper, 0.05 mg g^{-1} dry weight. These limits are at least 10 times lower than any values that have been encountered in the Puget Sound environment.

Atomic Absorption Procedure: Mercury. Mercury will be measured using the cold-vapor atomic absorption technique, in conjunction with dual gold traps (Bloom and Crecelius, Marine Chemistry, 1983). A Laboratory Data Control U.V. monitor with 30-cm cell will be used to perform the analysis. With this technique, a detection limit of 0.005 ug g^{-1} dry weight will be attained. This limit is 10 times lower than any value previously measured from Puget Sound sediments.

Calibration Procedures and Frequency

All atomic absorption calibration is done by the method of standard additions according to manufacturer's instructions. A calibration curve is prepared each day and all samples are analyzed under conditions which give a response in the linear range. A standard in the linear range is run every tenth sample. Standards are prepared from commercially available (1000 mg l^{-1}) atomic absorption standards.

Reference Materials (Accuracy)

For every 20 samples analyzed, a standard material will be analyzed to assure the accuracy of the method. These materials will include: National Bureau of Standards (NBS) estuarine sediment and National Research Council of Canada (NRCC) estuarine sediment. Measured values will deviate from the established value by more than one standard deviation no more than 10 percent of the time. When this does occur, the parameters will be reexamined and the standards rerun. A repeat of the deviant value prompts an indepth search of a cause.

Note: For lead, analysis may consistently be reported 20 to 30 percent low on standard estuarine sediments due to the high crustal/anthropogenic ratio in these clean sediments. We will therefore also intercalibrate 5% of the more enriched samples collected by nondestructive X-ray fluorescence spectroscopy. These values shall be within $\pm 10\%$ of the atomic absorption measurements.

Blanks

Reagent percent blanks will be run for every 20 samples. These values will be reported.

Replicate Analysis (Precision)

For every 20 samples one triplicate analysis will be done to determine precision. Spike and analyses will not be performed for metals because recoveries are determined by analysis of standard reference sediments.

Precision, Accuracy, and Completeness

The precision and accuracy of the laboratory methods will be determined in accordance with COE guidelines for assessing and reporting quality for environmental measurements. All quality control data will be recorded and maintained at the PNL and will be available to the Project Manager upon request.

Hydride Generation Procedures: Arsenic

A 1.00-g aliquot of freeze-dried and homogenized sediment is placed into a 100-ml snap-cap volumetric flask. Five milliliters of deionized water is added to form a slurry and then 7 ml of the acid digestion mixture is added. After 5 minutes, the caps are removed and the flasks heated at 80 to 90°C for 2 hours. Upon cooling the samples are diluted to the mark with deionized water, shaken, and allowed to settle overnight. An appropriate-sized aliquot of the supernatant liquid (25-100 μ l) is added to 20 ml of deionized water and run as for total arsenic using the hydride generation technique described by Andrae (1983). Detection limit is 0.3 ppm.

X-ray Fluorescence Procedure

Sediments in Task 3 will be analyzed for a suite of metals (Al, Si, K, Ca, Cr, Cu, Ti, Mn, Fe, Ni, Zn, As, Pb) by nondestructive X-ray fluorescence spectroscopy. This work will be performed by Mr. Ron Sanders PNL Richland, Washington. The equipment will be checked for accuracy by analyzing one geological standard for each 15 samples. Accuracy and precision will be determined using the NBS and NRCC standard marine sediments or rocks.

Analysis of Aromatic Petroleum Hydrocarbons and Polychlorinated Biphenyls in Sediment

Only glass, teflon, or metals are allowed to come in contact with samples or reagents. All labware is washed in hot soapy water, rinsed three times with hot tap water and finally rinsed three times with glass distilled or MILLIQUE® pure deionized water. Some items are precleaned in an ultrasonic bath. Labware is dried in an oven at 90°C. All labware is rinsed prior to use with high purity methylene chloride or carbon tetrachloride. Distilled-in-glassgrade solvents (from Burdick and Jackson Laboratories, Inc.) are used throughout the work. Procedural system blanks are analyzed to check for contamination. Ethyl ether is distilled prior to use to avoid preservatives and the buildup of peroxides. Tissue and sediment samples are stored at -75°C until used.

Soxhlet Extraction of Sediments

Extraction of hydrocarbons associated with suspended particulates and sediments is facilitated by the use of a Soxhlet Extraction Apparatus (Kontes, K585000-0023). The sample, contained in an extraction thimble (Kontes, K-586500-0266), is exposed for 12 hours to a hot, freshly distilled benzene/methanol mixture. The extracted components percolate through the bottom of a coarse-porosity, fritted glass extraction thimble and are siphoned off via a self-priming device to the solvent boiling flask. The components are concentrated in the boiling flask which is concurrently furnishing fresh benzene/methanol to the sample.

A 50-gram wet sample is initially processed in 150 ml Corex centrifuge tubes having Teflon[®] lined screw caps. The wet weight is recorded. Water is removed from sample by mixing for 30 minutes with 50 ml methanol. Centrifuge the sample/methanol mixture at 1000 rpm for 10 minutes. Decant the methanol/water into a 125-ml Erlenmyer with Teflon[®] sealed screw cap. Repeat the methanol extraction procedure using an additional 50 ml methanol. Refrigerate the water/methanol mix until a later step.

The methanol extracted sediment is then transferred to the coarse-porosity extraction thimble along with 50 ml of methanol. To the 50-ml boiling flask, 150 ml of benzene are added. The temperature is maintained using a hemispherical heating mantle (Kontes, Series O, K-721000, 270 w-115v), variac setting 48. A Friedrich condenser is connected to the top of the extraction apparatus. Wrap the apparatus with aluminum foil up to the condenser to conserve heat.

The extract from the soxhlet apparatus is combined with the methanol/water extract in a 500 ml separatory funnel. Rinse the soxhlet apparatus with 10 ml hexane and add to the separatory funnel. Rinse the Erlenmyer flask with 10 ml methanol. Add to the separatory funnel. Add 25 ml of distilled water to the separatory funnel. Shake the separatory funnel for ~2 minutes. Allow layers to separate with benzene/hexane on top and methanol/water on the bottom. The methanol/water is drained off into a 250-ml separatory funnel. Further extract this with 10 ml hexane. Shake, allow to separate, drain water/methanol to waste and add remaining hexane layer to the 500 ml separatory funnel containing the benzene/hexane mixture. The benzene/hexane mixture is passed through 15 grams of dry sodium sulfate contained in a 125 ml Chromaflex[®] chromatographic column and collected in a 500-ml round bottomed flask. The column is rinsed with 15 ml of hexane. Using a roto-evaporator (water bath temp. 50°C) the extract is reduced quickly in volume to ~4 ml. The concentrated extract is transferred to a 50 ml centrifuge tube. The evaporator flask is rinsed with heptane and this is added to the extract. This is blown down under nitrogen to ~1 ml. The sample is now ready for silica gel chromatography.

In order to get the wet:dry ratio, the extracted sediment is placed on aluminum foil and dried in a hood overnight. It is then placed into an oven at 120°C for another 24 hours. After cooling the dried sediment is weighed.

Silica Gel Chromatographic Analysis

Assemble a 25-cm long glass chromatography column, integral 125 ml reservoir, Teflon® stopcock and (-ring and a coarse glass frit (Kontes, K422250). A small wad of silanized glass wool (Supelco, 2-0411) is packed into the bottom of the column using a stainless steel rod. Silica gel (Davison 923, 100-200 mesh, available from Supelco) is activated in a drying oven overnight at 150°C and stored in a dessicator. Fifteen grams of silica gel are weighed out into a tared 50 ml beaker. Thirty milliliters of hexane are poured into the beaker containing the silica gel forming a slurry. The slurry is poured into the chromatography column with the aid of an Eby type glass wash bottle (Kontes, K-331000). Elute the hexane, do not allow it to drop below the top of the silica gel. Pass 30 ml methylene chloride through the silica gel to wash away any organic contaminants. This is accomplished by adding about 1 ml via a Pasteur pipette to the top of the column and allowing it to pass into the silica gel before adding the remainder of the methylene chloride. In order to minimize disturbance of the column bed, the solvent can be carefully allowed to run down the sides of the reservoir. When the last of the methylene chloride reaches the top of the silica gel, the column is rinsed with 40 ml hexane. In order to insure that all traces of methylene chloride are washed away, several 1-2 ml aliquots of hexane can be used to wash down the sides of the reservoir before the bulk of the 40 ml is passed through the column. When the hexane reaches the silica gel bed top, the sample may be carefully layered on top of the silica gel and allowed to pass into the column bed. This is eluted first with 40 ml hexane which is caught in a 40 ml graduated conical tube. This fraction contains the saturated hydrocarbons. The hexane elution is followed by an elution of the aromatic components by 86 ml of 20% by volume methylene chloride in hexane. This fraction must be collected in 2 conical tubes and recombined as evaporation allows. When both aromatic hydrocarbons and PCB are to be extracted and analyzed, a 50% methylene chloride and hexane elution is

utilized. Evaporate samples under a stream of nitrogen to less than 4 ml. The concentrated silica gel extracts can be stored prior to GC analysis in a low temperature freezer.

Gas Chromatographic Analysis

A silica gel column extract is subsampled to gain information about the concentration of the sample. This step is necessary if the concentrations of hydrocarbons and/or interfering components are high. Samples derived from, for example, bulk oil, microlayer, and contaminated sediments are usually subsampled and a preliminary GC analysis completed before diluting the analyte to a suitable concentration. When samples are derived from slightly to unpolluted waters, tissues, and other uncontaminated sources, a further concentration step may be required prior to analysis. To do this the 4 ml silica gel extract is split into two equal portions in case of the accidental loss of a sample during the evaporations step. One portion is set aside. The other is evaporated to 0.5 ml and to it 0.5 ml of an internal standard is added. This one ml is evaporated to 200 μ l in a 1 ml crimp seal reaction vial (Supelco, *-3123) and finally transferred to a 100 ml crimp seal reaction vial (Supelco, 3-3208) just before analysis. Teflon[®]-faced neoprene, 11 mm crimp seals (Supelco, 3-3233) are used to cap the vials. After being punctured by the needle, the crimp seal is replaced.

The internal standard method of calculating the absolute amount of a component in a sample is used. Internal standards are made up of 37 ppm 2,6,10 trimethyldodecane and 50 ppm of hexamethylbenzene in hexane for the analysis of saturate and aromatic hydrocarbons respectively. Standard saturate and aromatic hydrocarbon solutions are made up from analytical reference standards. Saturates are quantified using standards having 7-31 carbons including phytane and pristane. The concentrations of each are about 20 ppm. Thirty selected aromatic hydrocarbons, having 1-4 aromatic rings, can be quantified. Standard solutions for instrument calibration are prepared by mixing 1:1 the appropriate internal standard with the standards solution.

The Hewlett-Packard 7671A automatic liquid sampler delivers a 2 μ l injection to the Hewlett-Packard 5840A gas chromatograph. A flame ionization detector responds to hydrocarbon components eluted from the fused silica capillary column (DB-1, J&W Scientific®). The sample injection is split 10:1, column flow 1 ml/min. Helium is the carrier gas; nitrogen is used as a make-up gas. The injector and detector temperature is maintained at 300°C. Temperature programming is from 60°C (4 min hold) to 290°C at 4°C/min, final hold 15 minutes. Prior to injection the injector needle is rinsed six times with hexane and three times with the next sample.

DATA REDUCTION, VALIDATION AND REPORTING

For chemical measurement parameters, data will be reduced and reported as concentrations expressed as milligrams or micrograms per gram dry weight of sediment or other standard units.

Bioassay results will be reported as percent mortality of amphipods in different sediments and percent mortality and percent abnormal development for oyster larvae.

The Field Coordinator (J. S. Young) is responsible for reviewing all raw data input and generated reports. Traceability of data shall be established by official Laboratory Record Books. All Laboratory Record Books shall be reviewed and signed off by the responsible Coordinator and the Project Manager.

INTERNAL QUALITY CONTROL CHECKS

Applicable internal quality control checks are identified in individual chemical or biological protocols. Each protocol provides for internal QC checks applicable to that procedure. Data entered into log books are reviewed (as indicated by counter signature) by the Task Leader to verify that appropriate procedures were followed.

Example of Quality Control efforts are:

1. Replicates of sediment subsamples from the same core
2. Replicates of samples for chemical analysis from the same sediment
3. Checks on instrument calibrations
4. Internal standardization for chemical analyses
5. Transfer and transport blanks

PERFORMANCE AND SYSTEM AUDITS

System Audit

This project will be subject to possible inclusion in the PNL audit schedule. Due to the short time frame of scheduled work it may or may not be reviewed by PNL auditing personnel. It is anticipated that a COE representative will visit our laboratory for the purpose of evaluating laboratory operations and quality control procedures. Also, communication between COE and the PNL QA office can take place throughout the project.

PREVENTATIVE MAINTENANCE

Preventative maintenance and spare part inventory on all project test or measuring apparatus are conducted in accordance with manufacturers' specifications. In addition, PNL maintains extensive craft services and instrument repair facilities. Finally, all critical measurement equipment has back-up units which can be used during unforeseen downtime.

CORRECTIVE ACTION

This is a Research and Development project and collection of routine monitoring data is not a part of this project. Variances in system performance are brought to the attention of the Project Manager by the analytical staff or the Coordinator. The Project Manager is responsible for implementing corrective action. The corrective action taken will be based on the judgment of the Coordinator upon consultation with the Project Manager.

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QUALITY ASSURANCE REPORTS TO MANAGEMENT

Variances and non-conformances of major significance shall be reported to line management immediately. Other QA reports will be included in the program manager's report to management.

Any audit reports will be submitted to both the Project Manager and Section Manager.

APPENDIX A

APPLICABLE SECTIONS OF PNL QUALITY ASSURANCE MANUAL, PNL-MA-65

The following parts of the PNL Quality Assurance Manual, PNL-MA-65, shall be applied to this project in addition to other activities described in this QAPP. The sections or selected paragraphs applicable to this project are identified by the QA Manual section or paragraph number. Additional information is also provided for clarification or interpretation.

4.1 PROCUREMENT - This section applies when a purchase requisition is processed. The applicable parts of Sections 9.1 and 12.1 apply if receiving inspection is to be applied to a specific procurement.

5.1 INSTRUCTIONS, PROCEDURES AND DRAWINGS

5.1.1 These subsections apply to written protocols and procedures that & are applicable to this project (see the main body of this QAPP 5.1.3 for protocol and procedure requirements). Procedures for bioassays are referenced in the Work Plan and are from published papers.

5.1.2 This subsection applies if any part of this project is performed by any PNL group other than the Environmental Sciences Department.

6.1 DOCUMENT CONTROL - This section applies to all written protocols and procedures and is applicable to procedures common to more than one working group.

6.1.1(1) The Project Manager shall maintain a list of all protocols and procedures and other documents that are to be handled as controlled documents.

6.1.1(2) All controlled documents shall be approved by the Project Manager or a designated alternate.

- 10.1 CONTROL OF MEASURING AND TEST EQUIPMENT - This section of PNL-MA-65 applies to the measuring and test equipment used to perform the analyses described in this QAPP (see also Sections 8 and 9 of this QAPP).
- 13.1 NONCONFORMANCE AND CORRECTIVE ACTION - This section applies when one of the described conditions occurs. (See also Section 15 of this QAPP for corrective action associated with project data.)
- 14.1 QA RECORDS - The Project Manager shall assure that a file is maintained which contains or references the location of the QA records. These records shall include but are not limited to:
- o project financial author contract
 - o proposal (scope statement) and QAPP
 - o formal periodic and final reports (copies or by reference)
 - o publications, speeches, and formal reviews (copies or by reference)
 - o description of experimental design and setup, operating conditions and data collection procedures
 - o a list of the investigators/project organization
 - o basic data, original or by reference to storage locations
 - o analysis methods, techniques, models, computer codes (may be by reference)
 - o calculations, data analysis and assumptions to support each finding
 - o identification and location of all relevant information not contained in project file, e.g., pertinent literature references, etc.
 - o Records Plan and a list (index) of relevant project records that include the custodian for each and the storage location.
 - o Laboratory Record Books

SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY AND COMPLETENESS

Analyses of single samples reported without statistical inferences may be made as indications of relative or comparative concentrations.

When replicate samples are taken for chemical analysis, concentrations are reported as the mean $\pm$ SD. Data precision and accuracy on the chemical analysis will be evaluated by review of data by the Coordinator and comparison to the appropriate quality control checks and standards.

Table A-11. Interlaboratory Comparisons

	Battelle μg/kg dry	NYT μg/kg dry	% of Battelle's value
A-4-Grab			
Anthracene	809	3691	456
Benzo(a)pyrene	nd	nd	-
Chrysene	977	nd	0
Fluoranthene	2599	2962	114
Fluorene	1061	nd	0
Naphthalene	1333	nd	0
Phenanthrene	3135	1237	39
Pyrene	6692	nd	0
PCB 1254	255	21	8
E-22-Top			
Anthracene	599	201	34
Benzo(a)pyrene	1778	nd	0
Chrysene	1781	nd	0
Fluoranthene	2883	2742	95
Fluorene	94	115	123
Naphthalene	135	83	62
Phenanthrene	1125	900	80
Pyrene	1475	3004	204
PCB 1254	1200	783	65

* unresolved GC peak

nd-not detectable, less than 10ppb

Table A-11. Interlaboratory Comparisons

Performance evaluation of New York Testing Laboratories, Inc with regard to the recovery of selected PAH's and the PCB mixture Arochlor 1254 added to an uncontaminated sediment from Sequim Bay, Washington. To the wet sample 100 μ L were added from a mixture of PAH and PCB components. The spike amended sediment was stirred and then frozen at -80°C . The dry to wet ratio for the sample was 0.46.

COMPONENT	AMOUNT added μg	NOMINAL $\mu\text{g/kg dry}$	MEASURED $\mu\text{g/kg dry}$	% RECOVERED
Anthracene	11.35	285	N.D.	0
Benzo(a)pyrene	11.0	256	N.D.	0
Chrysene	12.2	284	N.D.	0
Fluoranthene	6.7	156	N.D.	0
Flourene	13.6	316	N.D.	0
Naphthalene	10.75	250	N.D.	0
Phenanthrene	11.35	264	107	41
Pyrene	12.75	296	N.D.	0
PCB 1254	100.0	2174	716	33

Table A-11. Interlaboratory Comparisons

The following tables intercompare the results of independent analysis of a select group of Priority pollutants reported by separate laboratories. A common sample was stirred, divided and distributed to Battelle at Sequim and New York Testing, Inc. (NYT). Battelle used a method involving a benzene-methanol soxhlet extraction, silica gel clean-up and cap GC analysis while NYT used E.P.A. methods 608 and 625.

E-11-Top	Battelle µg/kg dry	NYT µg/kg dry	% of Battelle's value
Anthracene	1982	nd	0
Benzo(a)pyrene	nd	nd	-
Chrysene	3173	nd	0
Fluoranthene	*	1518	-
Fluorene	3938	nd	0
Naphthalene	14535	nd	0
Phenanthrene	10305	1454	14
Pyrene	*	1859	-
PCB 1254	717	86	12
E-1-Top			
Anthracene	6481	907	14
Benzo(a)pyrene	4733	nd	0
Chrysene	12775	3146	25
Fluoranthene	9828	3596	37
Fluorene	1992	418	21
Naphthalene	1980	771	39
Phenanthrene	9103	2093	23
Pyrene	21390	3685	17
PCB 1254	585	260	44

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