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**Pacific Northwest  
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**Post-Remediation  
Biomonitoring of Pesticides  
and Other Contaminants in  
Marine Waters and Sediment  
Near the United Heckathorn  
Superfund Site, Richmond,  
California**

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July 2000

Prepared for the  
U.S. Environmental Protection Agency  
Region 9  
under a Related Services Agreement  
with the U.S. Department of Energy  
under Contract DE-AC06-76RLO 1830



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POST-REMEDIATION BIOMONITORING OF  
PESTICIDES AND OTHER CONTAMINANTS IN  
MARINE WATERS AND SEDIMENT NEAR THE  
UNITED HECKATHORN SUPERFUND SITE,  
RICHMOND, CALIFORNIA

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Sequim, Washington

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Pacific Northwest National Laboratory  
Richland, Washington

## **SUMMARY**

This report, PNNL-13059 Rev. 1, was published in July 2000 and replaces PNNL-13059 which is dated October 1999. The revision corrects tissue concentration units that were reported as dry weight but were actually wet weight, and updates conclusions based on the correct reporting units.

Marine sediment remediation at the United Heckathorn Superfund Site was completed in April 1997. Water and mussel tissues were sampled in February 1999 from four stations near Lauritzen Canal in Richmond, California, for Year 2 of post-remediation monitoring of marine areas near the United Heckathorn Site. Dieldrin and dichlorodiphenyl trichloroethane (DDT) were analyzed in water samples, tissue samples from resident mussels, and tissue samples from transplanted mussels deployed for 4 months. Concentrations of dieldrin and total DDT in water and total DDT in tissue were compared with Year 1 of post-remediation monitoring, and with preremediation data from the California State Mussel Watch program (tissues) and the Ecological Risk Assessment for the United Heckathorn Superfund Site (tissues and water). Mussel tissues were also analyzed for polychlorinated biphenyls (PCB), which were detected in sediment samples.

Chlorinated pesticide concentrations in water samples were similar to preremediation levels and did not meet remediation goals. Mean dieldrin concentrations in water ranged from 0.62 ng/L to 12.5 ng/L and were higher than the remediation goal (0.14 ng/L) at all stations. Mean total DDT concentrations in water ranged from 14.4 ng/L to 62.3 ng/L and exceeded the remediation goal (0.59 ng/L) at all stations. The highest concentrations of both DDT and dieldrin were found at the Lauritzen Canal/End station. Despite exceedence of the remediation goals, chlorinated pesticide concentrations in Lauritzen Canal water samples were notably lower in 1999 than in 1998. PCBs were not detected in water samples in 1999.

Tissue samples from biomonitoring organisms (mussels) provide an indication of the longer-term integrated exposure to contaminants in the water column, which overcomes the limitations of grab samples of water. Biomonitoring results indicated that the bioavailability of chlorinated pesticides has been reduced from preremediation levels both in the dredged area and throughout Richmond Harbor. Total DDT and dieldrin concentrations in mussel tissues were lower than measured levels from preremediation surveys and also lower than Year 1 levels from

post-remediation biomonitoring. The lowest levels were found at the Richmond Inner Harbor Channel station (37.6 µg/kg total DDT and 5.04 µg/kg dieldrin, wet weight; mean of resident and transplant mussels). Mean chlorinated pesticide concentrations were highest at Lauritzen Canal/End (794 µg/kg total DDT and 67.2 µg/kg dieldrin, wet weight), followed by Lauritzen Canal/Mouth (229 µg/kg total DDT and 16.7 µg/kg dieldrin, wet weight) and Santa Fe Channel/End (78 µg/kg total DDT and 6.25 µg/kg dieldrin, wet weight). These levels are 39% to 86% lower than those recorded by the California State Mussel Watch program prior to EPA's response actions. The levels of PCBs in transplanted mussel tissue were also reduced by 42% to 77% from preremediation levels (preremediation PCBs were not measured in resident mussels).

Surface sediment concentrations of dieldrin and DDT in November 1998 were highest in samples from the head or north end of Lauritzen Canal and progressively lower toward the mouth, or south end. Total DDT ranged from 130 ppm (dry weight) at the north end to 3 ppm at the south end. Dieldrin concentrations decreased from 3270 ppb (dry weight) at the north end to 52 ppb at the south end. These results confirmed elevated pesticide concentrations in sediments collected from Lauritzen Channel by Anderson et al. (1999). The pesticide concentrations were lower than maximum concentrations found in the 1993 Remedial Investigation but comparable to the median levels measured before remediation was completed. Sediment analyses also showed the presence of elevated PCB aroclor 1254, and very high levels of polynuclear aromatic hydrocarbons (PAH) in Lauritzen Channel.

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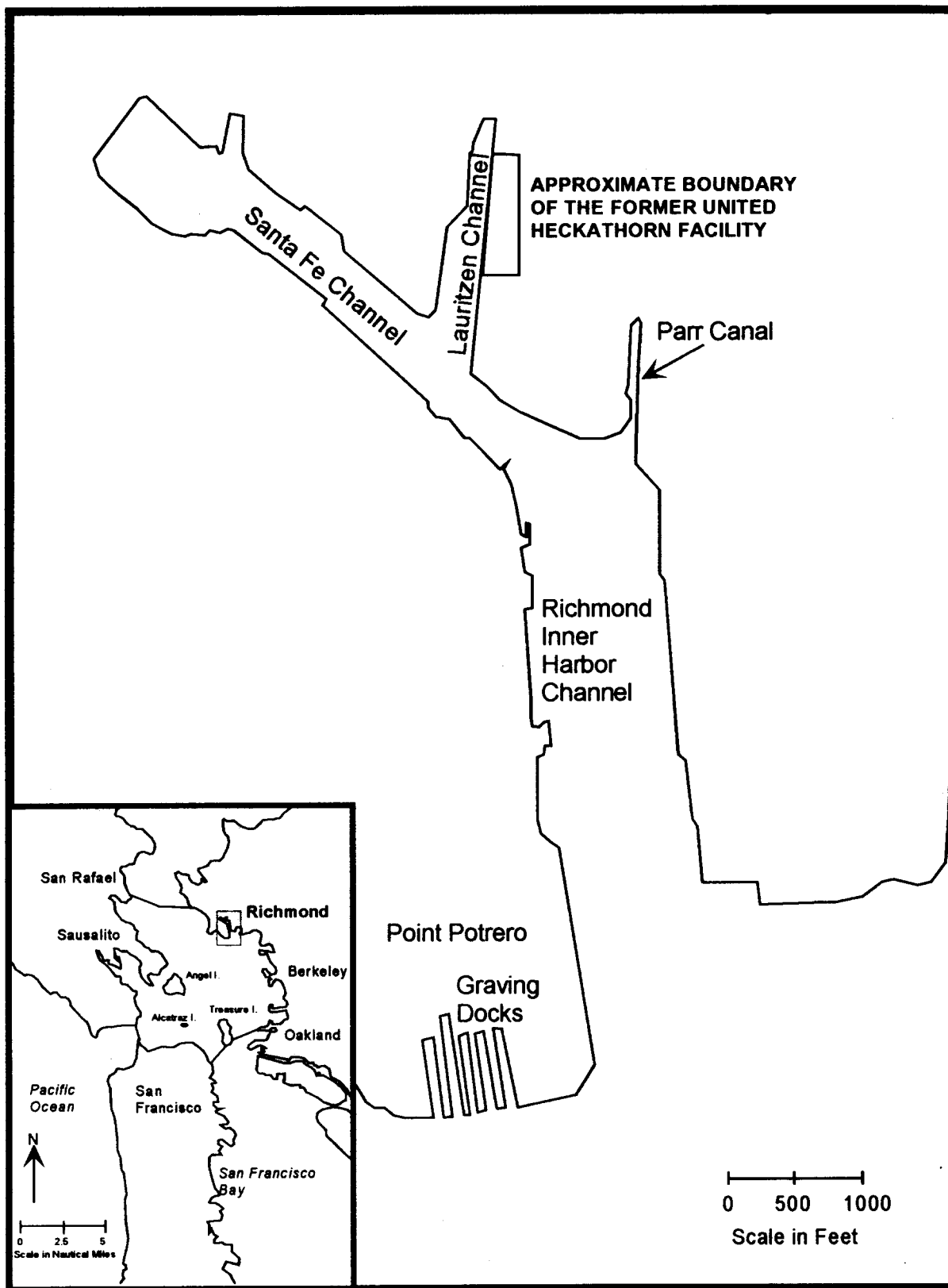


## **1.0 INTRODUCTION**

The United Heckathorn Site is located in Richmond Harbor, on the east side of San Francisco Bay in Contra Costa County, California (Figure 1.1). The site is an active marine shipping terminal operated by the Levin Richmond Terminal Corporation. The Site was listed by the U.S. Environmental Protection Agency (EPA) on its National Priorities List of Federal Superfund sites because of chemical contamination of upland and marine sediments and because the site had the highest levels of DDT contamination measured in the California State Mussel Watch program. A Remedial Investigation of adjacent marine areas revealed widespread sediment contamination with pesticides, particularly dichlorodiphenyl trichloroethan (DDT) and dieldrin (White et al. 1994). Significant pesticide contamination was limited to the soft, geologically recent deposits known as younger bay mud. Pesticide concentrations were highest in the Lauritzen Canal, and decreased with increasing distance from the former United Heckathorn Site, clearly indicating that Heckathorn was the source of contamination. An ecological risk assessment at the Heckathorn Site (Lee et al. 1994) reported data collected in 1991 and 1992 for contaminant concentrations in marine water, organisms, and sediments. This assessment revealed that DDT and dieldrin contamination originating from the United Heckathorn Site was actively transported to offsite areas via surface waters.

The final remedial actions at the Heckathorn Site outlined in the Record of Decision (ROD 1996) have the following major components:

- dredging of all soft bay mud from the Lauritzen Canal and Parr Canal, with offsite disposal of dredged material
- placement of clean sand cap material after dredging
- construction of a cap around the former Heckathorn facility to prevent erosion
- a deed restriction limiting use of the property at the former Heckathorn facility location to nonresidential uses
- marine monitoring to verify the effectiveness of the remedy.



**Figure 1.1.** Location of the United Heckathorn Superfund Site, Richmond, California

Remediation levels that would be protective of the environment and human health were established to provide benchmarks for determining the effectiveness of the remedial actions. The Feasibility Study (Lincoff et al. 1994) and the ROD reviewed federal and state environmental laws that contained Applicable or Relevant and Appropriate Requirements (ARARs) for the remedial actions. EPA marine chronic and human health water quality criteria (WQC) were identified as ARARs for surface water. Because the human health standards based on consumption of contaminated fish are lower than marine chronic criteria, these were used to establish remedial goals. No chemical-specific ARARs were identified as remedial goals for marine sediments or tissues at the site.

Sediment remediation by dredging, dewatering, and offsite disposal took place between July 1996 and March 1997. Extensive coring was conducted to verify that the younger bay (contaminated) mud was removed and that only older bay (less contaminated) mud remained. EPA collected and analyzed post-remedial samples of the remaining older bay mud for DDT, and found the average concentration to be 263 ug/kg dry weight, below the remedial goal of 590 ug/kg DDT dry weight. In April 1997, Lauritzen Canal was capped with 9100 cubic yards of clean sand, equivalent to an average depth of 1 ft over the dredged area, although cap thickness was probably variable because of the uneven, sloping channel bottom.

The purpose of marine monitoring is to demonstrate a reduction in flux of contaminants from the United Heckathorn Superfund Site following EPA response actions, which included soil removals, dredging, and cap placement at the former Heckathorn facility. The measurement endpoints for this long-term monitoring are mussels and surface waters. Remediation levels set forth in the ROD are provided in Table 1.1.

**Table 1.1.** Remediation Levels for Surface Water Specified in the Record of Decision for the United Heckathorn Superfund Site

Chemical	DDT (total) <sup>(a)</sup>	Dieldrin
Remediation Goal	0.59 ng/L	0.14 ng/L
(a) The sum of the 4,4'- and 2,4'-isomers of DDT, DDD (TDE), and DDE		

The first round of post-remedial biomonitoring was conducted 6 months after remediation (Antrim and Kohn 1998). Year 1 biomonitoring showed that pesticide concentrations in the tissues of mussels exposed at the site were somewhat elevated relative to those observed before remediation, and that tissue concentrations were considerably higher in Lauritzen Canal relative to those in the nearby Santa Fe and Richmond Harbor Channels. These results suggested that DDT was still present and bioavailable in Lauritzen Canal, especially near its head, relative to other waterways.

In October 1998, the Institute of Marine Sciences at the University of California, Santa Cruz (UCSC) reported finding 20 mg/kg total DDT (dry weight) in a Lauritzen Canal sediment sample (Anderson et al. 1999). Based on this observation, EPA collected four additional sediment samples in early November 1998 to verify the UCSC finding. Sediment analysis results are presented in this report along with Year 2 (1998-99) post-remedial biomonitoring results. Year 2 biomonitoring repeated the water, resident mussel, and transplanted mussel tissue sampling and analyses of Year 1 (1997-98). Year 2 results are compared with water and tissue pesticide data from two preremediation studies, as well as from the Year 1 monitoring study. The preremediation studies are the Ecological Risk Assessment conducted for the Heckathorn site by EPA (Lee et al. 1994) and the California State Mussel Watch Program. The four post-remedial water and tissue monitoring stations are the same as the State Mussel Watch Program stations in the project area.

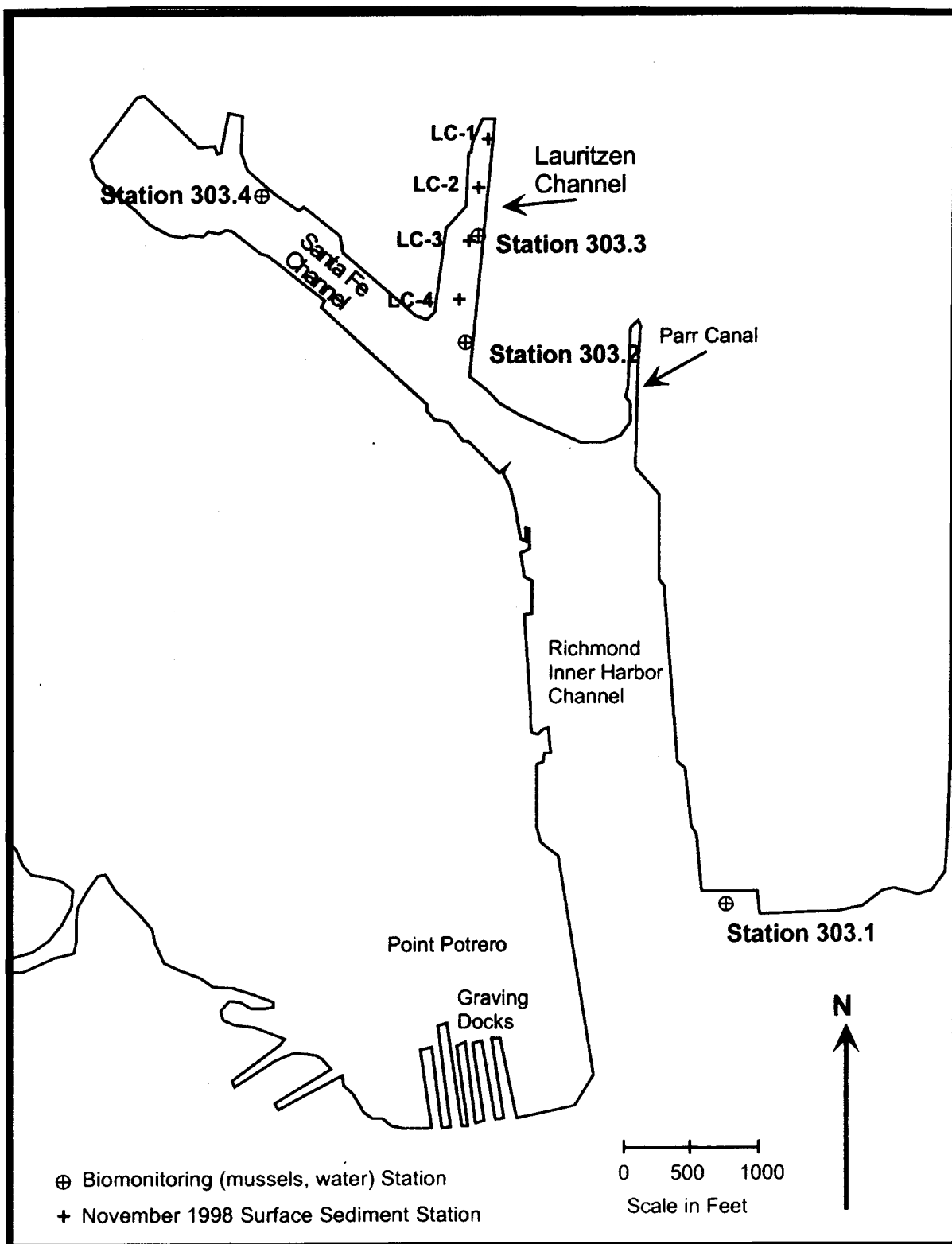
## **2.0 METHODS**

Methods for collection, processing, and analysis of tissue and water samples were outlined in the Field Sampling and Analysis Plan (Battelle 1997) and were the same as those used in Year 1 post-remediation monitoring. A brief review of these methods is provided here. All procedures for sampling, sample custody, and field/lab documentation, plus other aspects of documentation, quality assurance, and sample analysis were consistent with the Quality Assurance Project Plan (QAPjP) for Remedial Investigation and Feasibility Study of Marine Sediments at the United Heckathorn Superfund Site (Battelle 1992).

Four post-remediation monitoring stations were selected to duplicate stations sampled in the State Mussel Watch program (Figure 2.1). Three of the stations also approximate locations sampled during the Ecological Risk Assessment (Lee et al. 1994). The Lauritzen Canal/End Station (Mussel Watch Station 303.3) corresponds to the Ecological Risk Assessment-Lauritzen Canal Station; the Santa Fe Channel Station (Mussel Watch Station 303.4) corresponds to the Ecological Risk Assessment-Santa Fe Channel Station. The Richmond Inner Harbor Channel Station (Mussel Watch Station 303.1) is approximately 1200 ft inshore from the Ecological Risk Assessment-Richmond Inner Harbor station, which was at navigational nun buoy (No. 16). The Ecological Risk Assessment had no sampling station near the entrance to Lauritzen Canal (Mussel Watch Station 303.2, named Lauritzen Canal/Mouth). Mussel tissue samples were collected and analyzed in both preremediation studies, but water samples were analyzed only for the Ecological Risk Assessment. A more detailed description of sampling stations for 1998/1999 biomonitoring is provided in Table 2.1 and in Field Sampling Summary and Field Sampling Report memos (Appendix A; Lincoff 1998, 1999).

### **2.1 COLLECTION AND DEPLOYMENT OF TRANSPLANTED MUSSEL STOCK**

California mussels (*Mytilus californianus*) were collected on November 2, 1998, from Bodega Head, California, by the California Department of Fish and Game. This is the same area used for collection of transplant mussel stock by the California State Mussel Watch program (Gary Ichikawa, California Department of Fish and Game, personal communication).



**Figure 2.1.** Sampling Stations for Long-Term Post-Remediation Monitoring of the United Heckathorn Site

**Table 2.1.** Sampling Stations for Year 2 Post-Remediation Monitoring (1998-1999) of the United Heckathorn Site

Station Number	Station Name	Location <sup>(a)</sup>	Remarks
303.1	Richmond Inner Harbor Channel	37°54' 32.74" N 122°21' 33.91" W	On western most wooden dolphin, near abandoned Ford automotive plant, southeast of public fishing pier
303.2	Lauritzen Canal/Mouth (South)	37°55' 12.53" N 122°22' 01.02" W	On east side of canal, on pilings beneath the Levin Dock near the northern end of a large wooden fender structure
303.3	Lauritzen Canal/End (North)	37°55'22.54" N 122°21' 59.99" W	On east side of canal, southern end of small wooden pier that extends out into the channel
303.4	Santa Fe Channel/End	37°55' 20.61" N 122°21' 16.80" W	At northwest corner of floating boat shed, east of small boat fuel dock

(a) Data from November 1998.

At the EPA Region 9 laboratory in Richmond, California, mussels were cleaned to remove epiphytes, and sorted to select individuals at approximately 40-mm to 60-mm shell length. Selected mussels were placed in tubular plastic mesh bags, divided into three groups of approximately 20 mussels each, and kept separate using plastic cable ties. Mussels were held moist overnight at 12°C. Mesh bags with transplanted mussels were tied to nylon rope and suspended subtidally at four sampling stations. Deployment of transplanted mussels in the field was completed on November 3, the day following their collection. Nylon ropes were placed inconspicuously to avoid vandalism.

## 2.2 TISSUE AND WATER SAMPLE COLLECTION AND ANALYSIS

A background mussel tissue sample was prepared from the transplant mussel stock on the day of initial deployment (November 3, 1998). Fifty whole mussels were placed in two layers of ashed aluminum foil, labeled, and packed in a sealed Ziploc bag. The sample was stored at the

EPA Region 9 laboratory at -20°C until being shipped and processed with other tissue samples in February 1999.

After transplanted mussels had been deployed for approximately 4 months, seawater, transplanted California mussels (*M. californianus*), and resident bay mussels (*M. edulis*) were collected for analysis. Samples were collected at all four stations on February 23, 1999 (Figure 2.1). Resident bay mussels could have been one of several subspecies or hybrids in the *M. edulis* complex that cannot be easily distinguished by the shells alone (Harbo 1997).

Location coordinates presented in Table 2.1 were recorded for each station using a Global Positioning System with differential correction (dGPS). Samples were collected at near low tide on a calm, sunny day. Ambient water temperature was 12°C. A field sampling report prepared by EPA Region 9 staff is provided in Appendix A (Lincoff 1999).

Surface water samples were collected approximately 0.3 m below the water surface. To collect a sample, a bottle was submerged, the cap was removed under water to allow water in, and the cap replaced before the bottle was lifted from the water. At each station, three 2-L water samples were collected for analysis by Battelle Marine Sciences Laboratory (MSL). Additional water samples were collected for quality control (i.e., matrix spike, matrix spike duplicate, and blind duplicate samples). Water samples were chilled to and held at 4°C until extracted. Salinity of water samples was not measured in the field or in the laboratory.

Resident mussels were collected from approximately +0.4 ft mean lower low water (MLLW) at Richmond Inner Harbor Channel, Lauritzen Canal/Mouth, and Lauritzen Canal/End.

Transplanted mussels had been deployed at approximately -2 ft MLLW at Richmond Inner Harbor Channel, Lauritzen Canal/Mouth, and Lauritzen Canal/End Stations. Resident and transplanted mussels at these stations were from a fixed height in the intertidal zone. At the Santa Fe Channel/End Station, resident mussels were collected from just below the water surface at a floating dock on which transplanted mussels had been deployed at 1 ft below the water surface. Thus, mussels at the Santa Fe Channel/End station were at a fixed height relative to the water surface.

Mussels were cleaned gently in the field to remove external growth and packaged whole in ashed foil and plastic bags, as described above for the background tissue sample. Mussel samples were frozen at -20°C, shipped to the analytical laboratory in coolers, and held at -20°C until soft tissue samples were processed for analysis. To prepare tissue samples, mussels were



partially thawed, the valve or shell length was measured, byssus threads were cut from the tissue, and soft tissues were transferred to a sample jar. Sand and mud on the soft tissue were rinsed off with deionized water. Each tissue sample was composed of between 35 and 45 individual mussels. The total wet weight of each tissue sample was recorded. Tissue samples were refrozen at -20°C until extracted.

Chemical analyses followed methods described in the QAPjP (Battelle 1992). Water and tissues samples were analyzed for chlorinated pesticides. Tissue samples were also analyzed for total lipids and PCB aroclors. Total DDT was calculated as the sum of detected concentrations for six DDT compounds: 2,4-DDE, 4,4-DDE, 2,4-DDD, 4,4-DDD, 2,4-DDT, and 4,4-DDT. The detection limit was not used in calculation of total DDT. The California State Mussel Watch program (Rasmussen 1995) and the Ecological Risk Assessment for the United Heckathorn Superfund Site (Lee et al. 1994) calculated total DDT or sum of DDTs in the same manner.

### **2.3 SEDIMENT SAMPLE COLLECTION AND ANALYSIS**

To verify levels of DDT found in surface sediment samples from October 1998 (Anderson et al. 1999), sediment was sampled by EPA personnel from four stations in the Lauritzen Canal on November 3, 1998 (Table 2.2). Samples were collected midchannel, with stations progressing from the north end (LC-1) at the head of the canal to the south end or mouth/entrance of the canal (LC-4) (Figure 2.1). Station coordinates were determined using dGPS. Sediment was collected using an Eckman dredge that collects an intact sample from the top 10 cm of sediment. Samples were removed from the dredge using station-dedicated trowels and placed in precleaned glass jars with Teflon lined lids. A duplicate sediment sample was collected from one station for quality control (QC) purposes.

Sediment sample analyses followed methods described in the QAPjP (Battelle 1992). Sediment samples were analyzed for total solids, total organic carbon (TOC), grain size, polynuclear aromatic hydrocarbons (PAHs), pesticides, and polychlorinated biphenyls (PCBs or aroclors).

**Table 2.2.** Sediment Sampling Stations from November 3, 1998, at the United Heckathorn Superfund Site

Station Number	Station Name	Location <sup>(a)</sup>	Time
LC-1	Lauritzen Canal North	37°55' 27.65" N 122°21' 59.86" W	1455
LC-2	Lauritzen Canal North/Center	37°55' 23.74" N 122°22' 00.19" W	1445
LC-3	Lauritzen Canal South/Center	37°55' 19.59" N 122°22' 01.31" W	1440
LC-4	Lauritzen Canal South	37°55' 20.61" N 122°21' 16.80" W	1427

### **3.0 RESULTS AND DISCUSSION**

This section presents the results of physical measurements to assess the size and health of transplanted and resident mussels, as well as the results of chemical analyses of water, mussel tissue, and sediment samples. All extractions and analyses were conducted within target holding times. Complete data tables, including QC data, are provided in Appendix B for water and tissue analyses and in Appendix C for sediment analyses. In the following discussion, the current water monitoring data are compared with preremediation data from the Ecological Risk Assessment, post-remediation data from 1998, and the remedial goals for the site. The current tissue monitoring data are compared with preremediation tissue concentrations from the State Mussel Watch Program and the Ecological Risk Assessment, and post-remediation data from 1998. The sediment data are used to evaluate the current distribution of DDT in Lauritzen Channel.

#### **3.1 MUSSEL SIZE AND HEALTH**

Raw data for shell length measurements and mean wet weight per mussel are provided in Table 3.1. Mussels collected for tissue samples were of similar size, although a few individuals (<3% of the total) exceeded the preferred size range of 4.0 to 6.5 cm, the combined preference ranges from Rasmussen (1995) and Lee et al. (1994). Shell length of transplanted California mussels in the background sample ranged from 3.6 cm to 6.5 cm (mean = 4.7 cm). Four months later, California mussels transplanted to the study site were between 4.2 cm and 7.1 cm long (mean = 5.4 cm). Resident mussels collected in February 1999 ranged from 4.0 cm to 6.6 cm shell length (mean = 5.3 cm). The overall mean wet weight of individual mussels was calculated as the total wet weight of the tissue sample divided by the number of individuals per sample. Mean wet weight per mussel of soft tissues was 3.54 g for the background sample, and 7.16 g and 4.01 g for transplanted and resident mussels in February 1999, respectively.

**Table 3.1.** Length and Weight Data from Mussels Collected for Tissue Samples in February 1999 for Post-Remediation Monitoring of the United Heckathorn Superfund Site

Mussel #	Shell Length (cm)								Background
	Station								
	303.1		303.2		303.3		303.4		
	Transplant	Resident	Transplant	Resident	Transplant	Resident	Transplant	Resident	
1	5.30	5.88	6.05	5.51	6.03	5.95	6.00	4.56	5.11
2	5.58	5.44	5.20	5.87	5.74	5.17	6.00	5.18	4.80
3	5.31	5.78	5.09	5.63	5.02	4.54	6.06	5.38	4.18
4	6.11	5.87	6.06	5.44	5.07	5.30	5.36	5.38	4.32
5	5.34	5.37	5.20	6.13	5.34	4.63	6.03	6.14	5.14
6	4.77	5.30	5.70	5.86	6.05	5.32	5.80	4.90	4.70
7	6.33	4.83	6.09	5.84	5.07	5.23	5.17	5.10	4.61
8	5.88	5.96	5.90	5.90	6.02	5.24	5.02	6.10	6.10
9	5.79	4.62	5.95	4.89	4.60	6.30	4.35	5.19	5.30
10	6.10	4.86	5.02	5.53	5.10	5.16	6.30	5.49	5.43
11	5.58	5.24	6.24	5.53	5.75	6.12	6.56	5.81	6.10
12	5.68	5.06	5.40	5.75	5.70	5.23	6.08	4.41	4.40
13	5.12	5.40	5.37	5.31	5.44	5.65	5.48	6.35	6.46
14	5.74	5.33	5.26	5.08	6.21	5.96	4.70	5.10	4.92
15	4.59	4.94	5.18	6.60	5.05	6.17	5.53	4.52	4.43
16	5.14	4.65	5.11	5.63	5.02	4.84	5.50	5.58	4.26
17	5.80	5.95	6.32	6.25	5.94	5.84	5.35	4.69	4.52
18	5.70	5.86	6.45	5.55	5.30	5.83	5.74	5.75	4.30
19	7.08	5.51	6.37	5.65	5.83	4.05	5.36	5.00	4.50
20	5.09	5.73	5.90	6.03	5.76	5.75	5.50	5.00	4.21
21	5.10	5.38	5.54	5.67	5.43	5.16	5.02	6.24	5.43
22	5.49	5.08	4.99	4.90	4.86	5.10	5.60	4.80	6.05
23	4.82	4.84	4.79	5.30	5.39	4.70	5.11	6.08	4.84
24	5.05	5.36	5.39	5.00	4.83	4.25	5.09	5.77	4.33
25	6.36	5.55	5.30	4.92	4.64	5.20	5.14	5.00	4.00
26	5.95	4.70	5.77	4.77	4.63	5.74	5.48	4.62	4.90
27	5.33	5.36	5.13	4.83	6.03	4.40	5.83	4.88	4.50
28	5.53	4.69	4.90	4.70	4.95	5.17	6.30	4.12	3.85
29	4.16	4.42	5.35	4.43	4.95	5.41	5.80	5.71	4.20

Table 3.1. (contd)

Shell Length (cm)									
Mussel #	Station								Background
	303.1		303.2		303.3		303.4		
	Transplant	Resident	Transplant	Resident	Transplant	Resident	Transplant	Resident	
30	4.74	4.43	5.50	4.75	6.46	5.55	6.39	4.55	4.89
31	5.33	5.43	5.37	5.17	5.05	6.54	5.40	5.00	4.50
32	5.86	5.22	5.03	6.16	6.00	6.31	4.53	5.50	4.73
33	5.43	5.70	5.14	5.40	4.90	5.15	5.18	5.16	4.63
34	5.53	4.88	4.80	5.32	5.53	5.98	5.60	4.63	3.80
35	4.68	5.50	6.30	5.33	4.88	6.04	5.50	5.35	4.72
36	5.24	5.10	5.56	5.02	5.17	5.37	5.60	5.52	4.61
37	6.22	4.66	4.88	5.63	5.30	5.05	5.50	4.92	4.00
38	6.80	4.95	5.82	5.09	4.79	5.49	4.46	5.55	4.63
39	5.23	5.44	4.84	5.20	4.23	5.20	5.00	5.32	5.43
40	5.66	5.26	5.95	5.33	4.31	5.49	4.78	4.85	4.17
41	5.11	4.95	5.60	5.26	4.50	5.74	5.00	4.92	5.39
42	5.91	4.98	4.80	5.30	4.67	5.39	5.05	4.14	4.54
43	5.42	4.84	5.55	5.95	4.91	4.85	4.48	4.15	4.98
44	4.60	5.04	6.04	5.73	4.38	4.90		4.93	4.50
45	4.34	4.21	5.31		4.96				4.45
46			5.36						5.59
47									4.63
48									3.69
49									3.63
50									4.65
mean	5.47	5.17	5.49	5.42	5.21	5.36	5.41	5.18	4.71
min	4.16	4.21	4.79	4.43	4.23	4.05	4.35	4.12	3.63
max	7.08	5.96	6.45	6.60	6.46	6.54	6.56	6.35	6.46
mean length	transplants	5.39	background	4.71	resident	5.28			
mean wt. per mussel (g wet)	6.46	3.32	8.54	4.95	5.44	4.74	8.19	3.04	3.54
mean weight (g wet)	transplants	7.16	background	3.54	resident	4.01			

Transplanted California mussels grew in both length and weight during the 4-month deployment period. The lipid content was similar for the background tissue sample (8.13% dry weight) and transplanted mussel samples collected in February 1999 (range of 7.50% to 8.21% dry weight, mean of 7.98%). These data indicate that the transplanted mussels were in good health after 4 months of deployment, and that bioaccumulation of contaminants was not likely to have been compromised by poor health or limited food availability for the transplanted organisms. Lipid content of resident mussels was similar to but slightly more variable than that of transplanted mussels, ranging from 7.57% to 9.82% dry weight (mean of 8.40%). It should be noted that tissue lipid content is not a definitive indicator of organism health, because lipid content in bivalves can vary significantly depending on the availability of food and the bivalve's reproductive cycle.

### **3.2 WATER**

Triplicate water samples were collected on the same day at each site. These grab samples provide instantaneous data for water column concentrations of DDT compounds and dieldrin. Such data, however, provide no information about the temporal variability or vertical stratification of these contaminants in the water column, information that could be useful for interpretation of biomonitoring results. The inability to evaluate temporal or spatial variability of water chemistry should be considered when these data are compared with results from earlier studies. It should be noted that differences between two sampling events do not necessarily verify trends, and grab samples are not necessarily representative of normal conditions. Water grab samples also were collected and analyzed for Year 1 of post-remediation monitoring in January 1998. Preremediation water samples collected for the Ecological Risk Assessment (Lee et al. 1994) provided data for evaluation of temporal variability because samples were taken over three successive days at two different sampling periods, approximately 4 months apart.

Water samples collected in February 1999 for Year 2 of post-remediation monitoring were extracted with solvent, and solvent extracts were concentrated to 0.2-mL volume for an overall enhancement factor of approximately 10,000 in an attempt to achieve detection levels below the remediation goals. The achieved detection limit in water samples was 0.11 ng/L for dieldrin and ranged from 0.01 ng/L to 0.05 ng/L for the six DDT compounds. Recoveries of surrogate compounds ranged from 57.1% to 134% and exceeded the target range (40%-120%) in only one replicate sample. All data were corrected using the PCB 198 surrogate recovery. Blank

spike recoveries were within the target range of 40%-120% for the two spiked analytes, dieldrin and 4,4'-DDT. In the method blank, two analytes were detected, 4,4'-DDE (0.04 ng/L ) and 4,4'-DDT (1.66 ng/L); samples with less than five times the blank concentration are flagged with a "B." Matrix spike recoveries were variable and exceeded the target range of 40%-120% in two of four instances. High native levels of spiked compounds, as well as other chlorinated pesticides, in the sample probably caused this poor recovery of matrix spike compounds. Loss of replicate samples during shipment and analysis resulted in data for three replicates of Sample 303.4 and two replicates of Samples 303.1, 303.2, and 303.3. Replicate precision was poor, which is not uncommon for field collected samples. Surrogate compound and blank spike recoveries indicated acceptable laboratory precision of the laboratory analyses, which indicates that poor replicate precision was largely attributable to variability in replicate field samples.

Concentrations of DDT and dieldrin measured Year 2 post-remediation water samples are shown in Table 3.2. The mean of replicate water samples from each station is presented in Table 3.3 along with data from Year 1 post-remediation monitoring in 1998, preremediation monitoring in 1991/1992, and remedial goals. Water column concentrations of dieldrin were lower at all four stations in 1999 than in 1998 (Table 3.3). The largest difference was found at Lauritzen Canal Mouth (Station 303.2), where dieldrin in water samples was 8.18 ng/L in 1998 and 0.48 ng/L in 1999. Water concentrations of total DDT at all stations ranged from about 3 ng/L to 83 ng/L in replicate water samples (Table 3.2). The highest mean concentration of total DDT in 1999 was from Lauritzen Canal/End (Station 303.3; 62.3 ng/L), and the lowest mean concentration was from the Lauritzen Canal/Mouth (Station 303.2; 4.61 ng/L). Station 303.2 also had the lowest mean concentration of dieldrin. Total DDT concentrations in Lauritzen Canal water were notably lower than concentrations measured in 1998 (Table 3.3). An anomalous finding was the increase in total DDT in water from Station 303.1, Richmond Inner Harbor Channel, between 1998 and 1999. This station is relatively open to water exchange with Richmond Harbor and San Francisco Bay. The increase in the mean concentration of total DDT at Station 303.4 (Santa Fe Channel/End) is due to high levels of 4,4'-DDD and 4,4'-DDT in one replicate sample. As stated above, post-remediation water samples represent a "snapshot" of contaminant concentrations taken at a single point in time.

**Table 3.2. Concentrations of DDT and Dieldrin in Water Samples Collected in February 1999 for Post-Remediation Monitoring of the United Heckathorn Superfund Site**

			Concentration in Water (ng/L)								Total	
Water Sample ID	Replicate	Location	Dieldrin	2,4'-DDE	4,4'-DDE	2,4'-DDD	4,4'-DDD	2,4'-DDT	4,4'-DDT	DDT		
303.1	1	Richmond	0.57	0.07		1.81	1.41	5.70	0.92	9.96		19.9
303.1	2	Inner Harbor	0.67	0.01	U <sup>(a)</sup>	2.38	1.52	2.06	0.22	2.68	B <sup>(b)</sup>	8.86
303.2	1	Lauritzen	0.43	0.01	U	0.37	0.34	1.18	0.17	1.08	B	3.14
303.2	2	Canal Mouth	0.52	0.45		0.49	0.62	1.75	0.28	2.49	B	6.08
303.5	1	(c)	0.90	0.01	U	0.41	0.48	1.25	0.21	0.52	B	2.87
303.3	1	Lauritzen	6.28	0.30		2.96	5.82	13.5	4.86	13.8		41.2
303.3	2	Canal End	18.8	0.43		3.81	8.16	21.4	8.15	41.4		83.4
303.4	1	Santa Fe	0.23	0.01	U	1.69	2.40	15.0	1.51	30.7		51.3
303.4	2	Channel End	0.66	0.74		0.52	0.38	0.94	0.19	0.05	U	2.77
303.4	3		0.23	0.12		0.25	0.21	0.72	0.16	2.20	B	3.66

(a) U Not detected at or above given concentration.

(b) B Concentration is less than 5x blank value.

(c) Blind duplicate sample from station 303.2.



**Table 3.3** Comparison of Post-Remediation Concentration of Total DDT and Dieldrin in Water Samples with Preremediation Levels and Remedial Goal Concentrations (all concentrations are ng/L)

Water Sample ID Location	<u>Remedial Goals</u>		<u>Pre-Remediation<sup>(a)</sup></u>		<u>1998 Post-Remediation</u>		<u>1999 Post-Remediation</u>	
	Total DDT	Dieldrin	Total DDT	Dieldrin	Total DDT	Dieldrin	Total DDT	Dieldrin
303.1 Richmond Inner Harbor Channel	0.59	0.14	1	<1	0.65	0.65	14.4	0.62
303.2 Lauritzen Canal/Mouth	0.59	0.14	no sample	no sample	42.6	8.18	4.61	0.48
303.3 Lauritzen Canal/End	0.59	0.14	50	18	103	18.1	62.3	12.5
303.4 Santa Fe Channel/End	0.59	0.14	8.6	1.8	11	2.47	19.2	0.37

(a) Pre-remediation water concentration is average of samples collected in October 1991 and February 1992 for the Ecological Risk Assessment (Lee et al. 1994)

The relatively high variability in replicate samples indicates that these contaminants could be inconsistently distributed in the water, perhaps in association with organic or particulate materials.

Water concentrations of dieldrin and total DDT were well above remediation goals in all water samples and at all sampling stations (Table 3.3). The most elevated contaminant concentrations were found in Lauritzen Canal/End water (Station 303.3), where total DDT and dieldrin levels were 106 and 89 times greater, respectively, than remedial goals. PCBs were undetected in all water samples.

### **3.3 TISSUES**

Tissue samples from biomonitoring organisms provide a time-integrated indication of contaminant concentrations in the water column. These values therefore are not susceptible to small-scale temporal or spatial variability in contaminant concentrations as are grab samples of water. For tissue sample analysis, all quality control requirements were met. Achieved detection limits ranged from 0.27 µg/kg to 13 µg/kg (wet weight). The background tissue sample had 8.73 µg/kg total DDT, 1.34 µg/kg dieldrin, and <2.2 µg/kg Aroclor 1254 (wet weight). Results of tissue analyses (in wet weight, also converted to dry weight) from transplanted and resident mussels are provided in Table 3.4.

The post-remediation data are summarized (mean values in wet weight) and compared with preremediation data in Table 3.5. Evaluation of wet weight data is appropriate for ecological risk assessment because wet weight data represent concentrations of contaminants available to consumers of the tissues. All tissue data discussed below are either wet weight or lipid weight tissue concentrations. Year 2 post-remediation levels of total DDT were highest at the Lauritzen Canal/End (Station 303.3) and decreased at sites more distant from Station 303.3 or with increased exposure to water exchange. Total DDT concentrations (wet weight) in mussels from Lauritzen Canal/End were 606 µg/kg in resident and 981 µg/kg in transplanted mussels. At the Lauritzen Canal/Mouth, total DDT levels in mussels were 176 µg/kg (resident) and 282 µg/kg (transplanted). At the Santa Fe Channel/End station, total DDT levels were 75.6 µg/kg in resident mussels and 79.7 µg/kg in transplanted mussels. The lowest concentrations were found at the Richmond Inner Harbor Channel station, where total DDT in tissues was 29.7 µg/kg in resident and 45.5 µg/kg in transplanted mussels. The trend for dieldrin in mussel tissues was

**Table 3.4.** Concentrations of DDT, Dieldrin, and PCB Aroclor 1254 in Tissue Samples Collected in February 1999 for Post-Remediation Monitoring of the United Heckathorn Site

Analyte	Sample ID and Concentration (µg/kg wet wt)								
	Background <sup>(a)</sup>	303.1 Richmond Inner Harbor Channel		303.2 Lauritzen Canal Mouth		303.3 Lauritzen Canal End		303.4 Santa Fe Channel End	
		Transplant	Resident	Transplant	Resident	Transplant	Resident	Transplant	Resident
2,4 DDD	0.35 U <sup>(b)</sup>	6.26	2.45	40.7	16.1	119	75.6	10.3	6.58
2,4 DDE	5.68	1.17	1.42	2.78	1.88	7.80	4.65	0.80	0.55
2,4 DDT	0.49 U	4.17	3.37	43.1	32.0	167	113	7.64	10.5
4,4 DDD	0.68	18.7	7.18	101	37.7	311	143	32.1	18.9
4,4 DDE	2.37	8.17	8.21	32.7	31.6	87.5	71.5	12.8	17.5
4,4 DDT	0.34 U	7.07	7.08	61.9	56.6	289	198	16.1	21.6
<b>DIELDRIN</b>	<b>1.34</b>	<b>8.22</b>	<b>1.86</b>	<b>26.9</b>	<b>6.50</b>	<b>106</b>	<b>28.4</b>	<b>9.73</b>	<b>2.77</b>
<b>Total DDT (wet wt)<sup>(c)</sup></b>	<b>8.73</b>	<b>45.5</b>	<b>29.7</b>	<b>282</b>	<b>176</b>	<b>981</b>	<b>606</b>	<b>79.7</b>	<b>75.6</b>
Percent Dry Wt	16.2	12.3	8.4	10.3	7.70	10.9	9.2	9.9	9.4
Total DDT (dry wt)	54	370	354	2740	2284	9003	6584	805	805
Dieldrin (dry wt)	8.3	67	22	261	84	972	309	98.3	29.5
Lipids (% dry wt)	8.13	7.50	7.57	8.21	9.19	8.00	7.00	8.20	9.82
DDT (ppb <sup>(d)</sup> lipid)	663	4937	4672	33369	24855	112534	94061	9823	8193
Dieldrin (ppb lipid)	102	891	293	3181	919	12156	4410	1199	300
Aroclor 1254 (wet wt)	13.5 U	40.9	51.0	48.9	75.0	79.7	124	36.7	67.4
Aroclor 1254 (dry wt)	83 U	333	607	475	974	731	1348	371	717
Aroclor 1254 (ppb lipid)	1025 U	4434	8020	5783	10599	9140	19255	4521	7302

(a) Background tissue concentration is from coastal *M. californianus* prior to deployment (transplanting) in Richmond Harbor.

(b) U Not detected at or above given concentration.

(c) Total DDT is sum of detected 2,4- and 4,4- DDD, DDE, and DDT.

(d) ppb parts per billion (µg contaminant/kg lipid).

**Table 3.5.** Comparison of Post-Remediation Total DDT, Dieldrin, and PCBs in Tissues with Preremediation Concentrations ( $\mu\text{g/kg}$  wet weight)

Station Number	Station Name	State Mussel Watch <sup>(a)</sup> Transplant	Ecological Risk Assessment <sup>(b)</sup> Resident	1998 (Year 1) Post-Remediation Transplant	1998 (Year 1) Post-Remediation Resident	1999 (Year 2) Post-Remediation Transplant	1999 (Year 2) Post-Remediation Resident
<u>Total DDT (<math>\mu\text{g/kg}</math> wet weight)</u>							
303.1	Richmond Inner Harbor Channel	47.0 <sup>(c)</sup>	40	113	127	45.5	29.7
303.2	Lauritzen Canal/Mouth	629 <sup>(d)</sup>	---	1448	1222	282	176
303.3	Lauritzen Canal/End	5074 <sup>(d)</sup> 1369 <sup>(c)</sup>	2900	3502	4504	981	606
303.4	Santa Fe Channel/End	369 <sup>(c)</sup>	350	613	256	79.7	75.6
<u>Dieldrin (<math>\mu\text{g/kg}</math> wet weight)</u>							
303.1	Richmond Inner Harbor Channel	7.7 <sup>(c)</sup>	4	11.2	5.43	8.22	1.86
303.2	Lauritzen Canal/Mouth	87.0 <sup>(d)</sup>	---	165	40.3	26.9	6.50
303.3	Lauritzen Canal/End	602 <sup>(d)</sup> 100 <sup>(c)</sup>	97	279	184	106	28.4
303.4	Santa Fe Channel/End	32.5 <sup>(c)</sup>	19	83.1	8.18	9.73	2.77
<u>Total PCBs (<math>\mu\text{g/kg}</math> wet weight)</u>							
303.1	Richmond Inner Harbor Channel	176 <sup>(c)</sup>	not measured	not measured	not measured	40.9	51.0
303.2	Lauritzen Canal/Mouth	120 <sup>(d)</sup>	not measured	not measured	not measured	48.9	75.0
303.3	Lauritzen Canal/End	196 <sup>(d)</sup> 137 <sup>(c)</sup>	not measured	not measured	not measured	79.7	124
303.4	Santa Fe Channel/End	138 <sup>(c)</sup>	not measured	not measured	not measured	36.7	67.4

(a) Most recent data available from State Mussel Watch program, transplanted California mussels (Rasmussen 1995).

(b) Average concentration in resident mussel tissue from samples collected in October 1991 and February 1992 (Lee et al., 1994).

(c) State Mussel Watch program sample from March 1991 (Rasmussen 1995).

(d) State Mussel Watch program sample from January 1988 (Rasmussen 1995).

similar, with the highest levels at Lauritzen Canal/End (mean of 67.2 µg/kg dieldrin in resident and transplanted mussels) and the lowest levels at the Richmond Inner Harbor Channel station (mean of 5.04 µg/kg dieldrin in resident and transplanted mussels). PCB Aroclor 1254 was detected in both resident and transplanted mussels collected from post-remedial monitoring stations in 1999. Wet weight PCB concentrations were highest in Lauritzen Canal/End (102 µg/kg mean, transplant and resident), about twice that of the other stations (46 µg/kg to 62 µg/kg mean, transplant and resident) (Table 3.4).

Tissue burdens from Year 2 of post-remediation biomonitoring were reduced from pre-remediation levels and Year 1 post-remediation levels (Table 3.5). EPA response actions began at the site in 1989 with the removal of shoreline pesticide deposits containing up to 100% DDT. California Mussel Watch samples from both 1988 and 1991 were available from only one station, but these data suggest that significant reductions in contaminant bioavailability occurred at Station 303.3 near the end of Lauritzen Canal following removal of shoreline deposits (Table 3.5). However, the first year of biomonitoring indicated slightly increased bioavailability of pesticides in Lauritzen Canal. Total DDT and dieldrin levels in Year 1 (1998) post-remediation resident mussel tissue samples were approximately twice pre-remediation levels measured in 1992 (Lee et al., 1994). Year 2 post-remediation biomonitoring showed these compounds reduced from 1992 pre-remediation levels by an average of 66% in resident mussel tissue samples (mean of three stations), with a 71%-85% reduction in Lauritzen and Santa Fe Channels and a 26% to 54% reduction in Richmond Harbor Channel. These data showed an area-wide reduction in bioavailability of these pesticides.

The reduction in tissue burdens of PCBs was also notable. Year 2 post-remediation biomonitoring showed Aroclor 1254 reduced in transplanted mussels by 42% to 77% (average 63%) from 1992 preremediation levels. Preremediation PCB data were only available from the State Mussel Watch Program.

A direct comparison of contaminant concentrations expressed as tissue wet weight from different sampling dates is confounded by differences in lipid content of tissues. To correct for differences in lipid content of tissue samples, dry weight tissue data were divided by the lipid content (% dry weight). Lipid-normalized values for total DDT and dieldrin, expressed as micrograms pesticide/kilogram lipid weight (µg/kg lipid), are provided in Table 3.4. Year 2 lipid-normalized data are summarized and compared with previous data in Table 3.6.

**Table 3.6. Comparison of Lipid-Normalized Post-Remediation Total DDT, Dieldrin, and PCBs in Tissues with Preremediation Concentrations (ug/kg lipid weight)**

Station Number	Station Name	State Mussel Watch <sup>(a)</sup> Transplant	Ecological Risk Assessment <sup>(b)</sup> Resident	1998 (Year 1) Post-Remediation Transplant	1998 (Year 1) Post-Remediation Resident	1999 (Year 2) Post-Remediation Transplant	1999 (Year 2) Post-Remediation Resident
<u>Total DDT (ug/kg lipid weight)</u>							
303.1	Richmond Inner Harbor Channel	9,215 <sup>(c)</sup>	3,275	9,961	12,313	4,937	4,672
303.2	Lauritzen Channel/Mouth	78,481 <sup>(d)</sup>	---	134,247	134,633	33,369	24,855
303.3	Lauritzen Channel/End	583,819 <sup>(d)</sup> 380,361 <sup>(c)</sup>	250,411	368,816	427,423	112,534	94,061
303.4	Santa Fe Channel/End	47,283 <sup>(c)</sup>	21,919	51,016	45,695	9,823	8,193
<u>Dieldrin (ug/kg lipid weight)</u>							
303.1	Richmond Inner Harbor Channel	1,507 <sup>(c)</sup>	322	988	525	891	293
303.2	Lauritzen Canal/Mouth	10,861 <sup>(d)</sup>	---	15,293	4439	3,181	919
303.3	Lauritzen Canal/End	69,272 <sup>(d)</sup> 27,778 <sup>(c)</sup>	8,590	29,387	17,463	12,156	4,410
303.4	Santa Fe Channel/End	4,167 <sup>(c)</sup>	1,126	6914	1462	1,199	300
<u>Total PCBs (ug/kg lipid weight)</u>							
303.1	Richmond Inner Harbor Channel	34,440 <sup>(c)</sup>	not measured	not measured	not measured	4,434	8,020
303.2	Lauritzen Canal/Mouth	14,981 <sup>(d)</sup>	not measured	not measured	not measured	5,783	10,599
303.3	Lauritzen Canal/End	22,554 <sup>(d)</sup> 38,056 <sup>(c)</sup>	not measured	not measured	not measured	9,140	19,255
303.4	Santa Fe Channel/End	17,667 <sup>(c)</sup>	not measured	not measured	not measured	4,521	7,302

(a) Most recent data available from State Mussel Watch program, transplanted California mussels (Rasmussen 1995).

(b) Average concentration in resident mussel tissue from samples collected in October 1991 and February 1992 (Lee et al., 1994).

(c) State Mussel Watch program sample from March 1991 (Rasmussen 1995).

(d) State Mussel Watch program sample from January 1988 (Rasmussen 1995).

Although lipid-normalized values from Year 1 biomonitoring in 1998 showed an increase in DDT and dieldrin in mussel tissues relative to concentrations measured in 1991/1992 for the Ecological Risk Assessment (Lee et al., 1994), a reduction in bioavailability of total DDT was demonstrated by Year 2 biomonitoring. Year 2 resident mussels in Lauritzen Channel had total lipid-normalized DDT levels 62% lower than in 1991/1992 and lipid-normalized dieldrin levels 49% lower than in 1991/1992. Biomonitoring with transplanted mussels revealed the same pattern, with lipid-normalized total DDT reduced by 46% to 79% throughout the harbor, relative to 1991/1992. Lipid-normalized dieldrin concentrations in transplanted mussels were 41% to 71% lower than in 1991/1992.

Either transplanted or resident mussels appear to be acceptable for biomonitoring at the study site, but continued monitoring with both species could increase understanding of differences found between the species. Interspecies differences in total body burdens could have arisen from a variety of factors, including differences in feeding, growth rate during exposure, lipid content of tissues, duration of exposure, and height in the water column. Transplanted mussels, species *M. californianus*, had negligible initial DDT and dieldrin contamination, and were exposed for a known time period at the study site (i.e., 4 months). Resident mussels were adult *M. edulis*, which occur naturally at the study site. Although their age is undetermined, they were selected at approximately 40 mm to 60 mm shell length. It is possible that some of these individuals were present at sample stations before remediation was completed in April 1997. Resident and transplanted mussels collected for tissue samples were similar in length (Table 3.1). Although the mean weight per mussel and weight:length ratio were similar for resident mussels and the background sample (transplanted mussels not deployed at the study site) in 1999, transplanted mussels collected after 4 months deployment had significantly greater weight and weight:length ratio than resident mussels collected for tissue samples. Data from 1998 show the opposite, a higher weight:length ratio in resident mussels than in transplanted mussels. Transplanted mussels had consistently higher dry weight than did resident mussels in 1998 and 1999. Neither resident nor transplanted mussels had consistently higher lipid content in 1998 or 1999.

The comparability of resident and transplanted mussels was further evaluated by calculating the relative percent difference (RPD; difference/mean X 100) between resident and transplant tissue concentrations. On a wet weight concentration basis, the RPD between resident and transplant total DDT burden was 42% to 47% for both Lauritzen Canal stations and the Richmond Inner

Harbor Channel station, whereas the RPD for the Santa Fe Channel/End station was 5%. Transplanted mussel tissue DDT concentrations were consistently higher than those of residents, at least on a wet weight basis. When the tissue concentrations were lipid-normalized, the RPDs between resident and transplant total DDT concentrations were all <30% (range 6% to 29%), indicating that differences in lipid content could account for some of the observed differences between residents and transplants. A much larger difference between transplants and residents was noted in dieldrin concentrations, which were 2.8 to 4 times higher in transplants than in residents even on a lipid-normalized basis (RPDs of 94% to 120%). A similar pattern was noted during the first year of post-remedial monitoring, when lipid-normalized concentrations were 1.7 to 4.7 times higher in transplants than in residents. In Year 2 biomonitoring, transplanted mussels were consistently higher for both total DDT and dieldrin in dry weight, wet weight, and lipid weight values (Table 3.5).

Observed differences between transplanted and resident mussels also may have been attributable, in part, to height in the water column. At all stations except Santa Fe Channel/End (Station 303.4), resident mussels were collected from approximately +0.4 ft MLLW, and transplanted mussels were held at approximately -2 ft MLLW. At the Santa Fe Channel/End station, resident and transplanted mussels were attached to a floating dock and were consistently 0.4 ft and 1.0 ft below the water surface, respectively. This station, where resident and transplanted mussels were consistently submerged and at a similar distance from the water surface, had the lowest RPD for the difference between total DDT but not dieldrin in resident and transplanted mussels. At all other stations, resident mussels were exposed to surface waters and the air more frequently than were transplanted mussels. Transplanted mussels were exposed to water slightly lower (~1.6 ft) in the water column than were resident mussels.

PCB Aroclor 1254 was detected in both resident and transplanted mussels collected from post-remedial monitoring stations. A difference was observed between transplant and resident mussels, but Aroclor 1254 was seen at consistently higher concentrations in resident mussels than in transplanted mussels (Table 3.4). Possible reasons are that the resident mussels carry a persistent background body burden (no PCBs were detected in background transplants from Bodega Head), the transplants are less efficient at accumulating PCBs, or that PCBs accumulate more slowly than pesticides. The difference in height in the water column does not appear to be a factor. The station with the greatest difference in concentration (Santa Fe End,



Harbor Channel station, whereas the RPD for the Santa Fe Channel/End station was 5%. Transplanted mussel tissue DDT concentrations were consistently higher than those of residents, at least on a wet weight basis. When the tissue concentrations were lipid-normalized, the RPDs between resident and transplant total DDT concentrations were all <30% (range 6% to 29%), indicating that differences in lipid content could account for some of the observed differences between residents and transplants. A much larger difference between transplants and residents was noted in dieldrin concentrations, which were 2.8 to 4 times higher in transplants than in residents even on a lipid-normalized basis (RPDs of 94% to 120%). A similar pattern was noted during the first year of post-remedial monitoring, when lipid-normalized concentrations were 1.7 to 4.7 times higher in transplants than in residents. In Year 2 biomonitoring, transplanted mussels were consistently higher for both total DDT and dieldrin in dry weight, wet weight, and lipid weight values (Table 3.5).

Observed differences between transplanted and resident mussels also may have been attributable, in part, to height in the water column. At all stations except Santa Fe Channel/End (Station 303.4), resident mussels were collected from approximately +0.4 ft MLLW, and transplanted mussels were held at approximately -2 ft MLLW. At the Santa Fe Channel/End station, resident and transplanted mussels were attached to a floating dock and were consistently 0.4 ft and 1.0 ft below the water surface, respectively. This station, where resident and transplanted mussels were consistently submerged and at a similar distance from the water surface, had the lowest RPD for the difference between total DDT but not dieldrin in resident and transplanted mussels. At all other stations, resident mussels were exposed to surface waters and the air more frequently than were transplanted mussels. Transplanted mussels were exposed to water slightly lower (~1.6 ft) in the water column than were resident mussels.

PCB Aroclor 1254 was detected in both resident and transplanted mussels collected from post-remedial monitoring stations. A difference was observed between transplant and resident mussels, but Aroclor 1254 was seen at consistently higher concentrations in resident mussels than in transplanted mussels (Table 3.4). Possible reasons are that the resident mussels carry a persistent background body burden (no PCBs were detected in background transplants from Bodega Head), the transplants are not more efficient at accumulating PCBs, or that PCBs accumulate more slowly than pesticides. The difference in height in the water column does not appear to be a factor. The station with the greatest difference in concentration (Santa Fe End,

Station 303.4) is the one with no difference in water column height, lending further credence to the possibility of a background body burden of Aroclor 1254.

### 3.4 SEDIMENTS

Surface sediment samples were collected in November 1998 along the length of Lauritzen Canal at four stations in the approximate center of the channel (Figure 1.1). These samples were taken primarily to evaluate the distribution of DDT contamination in the canal but were also analyzed for other pesticides, PAHs, and PCB aroclors to evaluate potential input of contaminants from other sources. For pesticide and PCB analyses, all QC requirements were met, which indicated acceptable accuracy and precision of these data. Achieved detection limits ranged from 21.2 µg/kg to 81.7 µg/kg (dry weight) for pesticides and was 23.3 µg/kg (dry weight) for PCB aroclor 1254. Quality control limits for agreement between duplicate sediment samples (RPD) were exceeded for four of the six pesticides detected, which indicates that sediment at the site was not homogeneous. For PAH analyses, recoveries of internal spikes were below the quality control limits of 40%-120% for low molecular weight PAHs (LPAH; naphthalene and acenaphthene). For the standard reference material, detected values were within acceptable limits for LPAHs but high for three high molecular weight PAHs (HPAH). Recoveries of matrix spike compounds exceeded QC limits for most PAHs because the spike levels were inappropriate (generally an order of magnitude below concentrations in the sample). Recovery of matrix spike compounds added at concentrations within an order of magnitude of sediment levels were within QC limits. Analysis of a duplicate sediment samples indicated acceptable analytical precision. All QC requirements were met for conventional parameters.

Results of sediment analyses for conventional parameters and chemical contaminants are presented in Table 3.7. Sediment from the inner end of Lauritzen Canal (Station LC-1/Lauritzen Canal North) was oily and produced a sheen on the water surface when the dredge was retrieved. This sediment was predominantly silt and clay (68%) and sand (32%), with a relatively high TOC content (3.11%) and low percentage of total solids (19%). At Station LC-2 (Lauritzen Canal North/Center) sediment was primarily sand (67%) that was high in total solids (64%) and low in TOC content (0.89%). This sediment seems to be dominated by sand cap material. Sediment samples from the south end of the canal (Stations LC-3 and LC-4) were similar, a very soft gray to black mud mixed with chunks of clay. At Station LC-3, sediment was 91% silt and

**Table 3.7. Results of Analyses of Sediment Samples Collected on November 3, 1998, for Post-Remediation Monitoring of the United Heckathorn Superfund Site**

	<u>LC-1</u>	<u>LC-2</u>	<u>LC-3</u>	<u>LC-4</u>
	Lauritzen Canal North	Lauritzen Canal North/Center	Lauritzen Canal South/Center	Lauritzen Canal South
<u>Conventional Measurements (Percent dry weight)</u>				
Gravel	0.10	0.68	0.00	0.00
Sand	31.67	67.14	9.03	14.04
Silt	43.05	10.61	25.26	23.93
Clay	25.19	21.57	65.71	62.03
TOC	3.11	0.89	1.67	1.53
Total Solids	19.39	64.04	36.37	36.79
<u>Chlorinated Pesticides (ug/kg dry weight)</u>				
A-BHC	204 U <sup>(a)</sup>	60.6 U	55.9 U	25.8 U
B-BHC	204 U	60.7 U	55.9 U	25.8 U
G-BHC	122 U	36.5 U	33.7 U	15.5 U
D-BHC	204 U	60.7 U	55.9 U	25.8 U
Heptachlor	77.0 U	40.0	21.1 U	9.73 U
Aldrin	790	60.5	43.1	15.8 U
Heptachlor Epoxide	250 U	74.2 U	68.4 U	31.6 U
g-Chlordane	1660	60.7 U	55.9 U	25.8 U
Endosulfan I	3240	60.7 U	55.9 U	25.8 U
a-Chlordane	1000	59.5	17.7 U	8.18 U
Dieldrin	3270	382	171	51.5
4,4'-DDE	84400	383	323	93.8
Endrin	671	507	55.9 U	25.8 U
Endosulfan II	204 U	60.7 U	55.9 U	25.8 U
4,4'-DDD	15700	3150	4080	1190
Endrin Aldehyde	204 U	60.7 U	55.9 U	25.8 U
Endosulfan Sulfate	204 U	60.7 U	55.9 U	25.8 U
4,4'-DDT	30100	10400	5850	1450
Toxaphene	16.1 U	4.79 U	9.06 U	8.11 U
<u>Total DDT (ppm dry weight)</u>	<u>130</u>	<u>13.9</u>	<u>10.3</u>	<u>2.7</u>

Table 3.7. (contd.)

	LC-1	LC-2	LC-3	LC-4
	Lauritzen Canal North	Lauritzen Canal North/Center	Lauritzen Canal South/Center	Lauritzen Canal South
<u>PCB Aroclors (ug/kg dry weight)</u>				
1242	16.1 U	4.79 U	9.06 U	8.11 U
1248	16.1 U	4.79 U	9.06 U	8.11 U
1254	981	245	150	89.9
1260	16.1 U	4.79 U	9.06 U	8.11 U
<u>PAHs (ug/kg dry weight)</u>				
naphthalene	1960	112	178	134
Acenaphthalene	102	212	704	473
Acenaphthene	1830	73.3	303	125
Fluorene	3490	162	394	199
phenanthrene	9120	676	1250	728
anthracene	1760	696	2810	1070
<b>Total LPAH</b>	<b>18262</b>	<b>1931</b>	<b>5639</b>	<b>2729</b>
fluoranthene	5100	2140	5700	4510
pyrene	3870	1340	3170	2700
benzo[a] anthracene	1170	1150	3080	1970
chrysene	1710	1560	4580	2580
benzo[b] fluoranthene	1230	1740	3720	2220
benzo[k] fluoranthene	425	626	1420	822
benzo[a] pyrene	655	1080	2320	1360
indeno [1,2,3-c,d] pyrene	278	396	789	463
dibenzo [a,h] anthracene	93.9	124	234	142
benzo [g,h,i] perylene	288	338	633	407
<b>Total HPAH</b>	<b>14820</b>	<b>10494</b>	<b>25646</b>	<b>17174</b>
<b>TOTAL PAH (ppm)</b>	<b>33.1</b>	<b>12.4</b>	<b>31.3</b>	<b>19.9</b>

(a) U Undetected above given concentration.

clay, with 36% total solids and 1.67% TOC. At Station LC-4, sediment was approximately 86% silt and clay, with 37% total solids and 1.53% TOC.

Concentrations of dieldrin and DDT were highest in sediment from the inner end of Lauritzen Canal (Station LC-1) and progressively lower toward the mouth, or southern end, of the canal. Total DDT ranged from 130 ppm (mg/kg dry wt.) at station LC-1 to 3 ppm at Station LC-4 (Table 3.7). Dieldrin concentrations decreased from 3270 ppb ( $\mu\text{g/kg}$  dry wt.) to 52 ppb at Stations LC-1 and LC-4, respectively. The trend in sediment concentration of these two contaminants was remarkably similar (Figure 3.1). Relative to Station LC-1, dieldrin and total DDT concentrations were lower by approximately 89%, 93%, and 98% at Stations LC-2, LC-3, and LC-4, respectively.

The median total DDT levels measured for the Remedial Investigation in 1993 were 47 ppm and 1.5 ppm for the northern and southern portions of Lauritzen Canal, respectively (White et al. 1994). Maximum measured levels of total DDT in 1993 were significantly higher (121 to 633 ppm). Sediment collected for this study had total DDT levels between the median and maximum levels measured before remediation activities (i.e., dredging and capping).

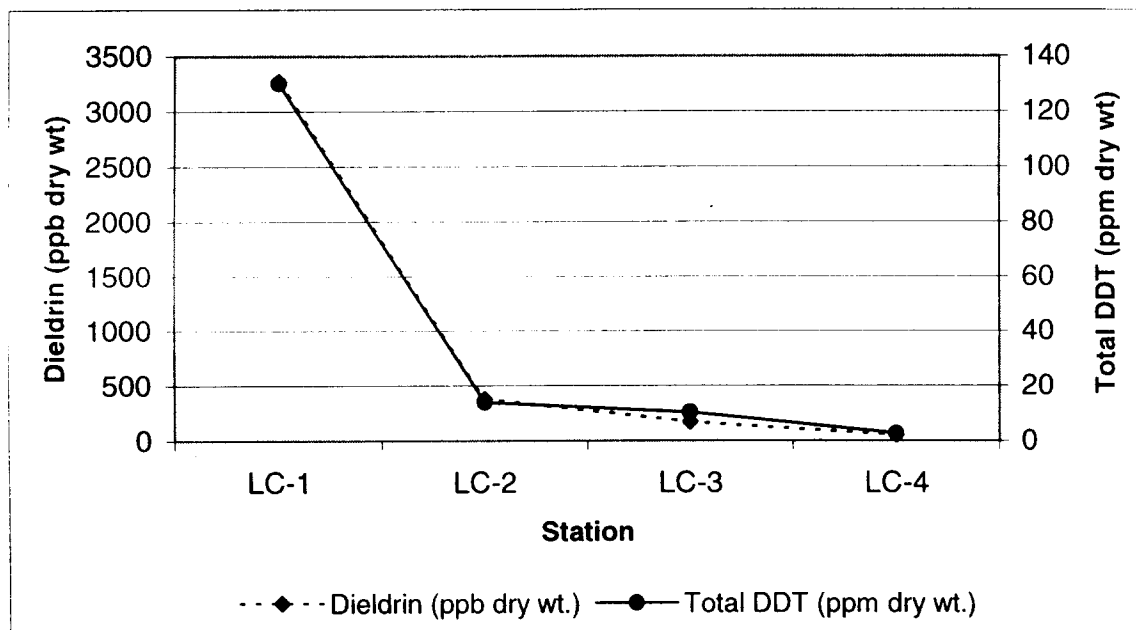
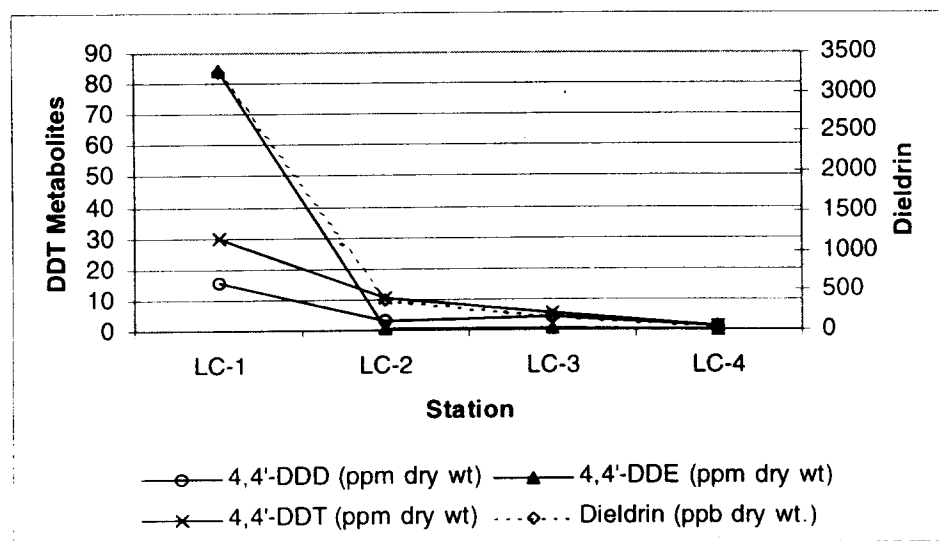


Figure 3.1. Sediment Concentration of Total DDT and Dieldrin in Sediment Samples from Lauritzen Canal, November 1998

Total DDT concentrations in Lauritzen Canal surface sediment samples from November 1998 were at least an order of magnitude higher than the median levels measured in the adjacent Federal Santa Fe Channel in 1993 for the Remedial Investigation. Total DDT levels from Stations LC-1, LC-2, and LC-3 in 1998 were one to two orders of magnitude higher than the maximum level measured in the Federal Santa Fe Channel in 1993. The maximum dieldrin concentrations measured for the Remedial Investigation were 16,000 ppb at the north end of Lauritzen Canal, 500 ppb at the south end of the canal, and 40 ppb in the Federal Santa Fe Channel (White et al. 1994). Sediment samples collected for this study had dieldrin concentrations comparable to maximum levels measured in 1993.

The relative contribution to total DDT of different DDT metabolites (i.e., DDT, DDE and DDD) differed between LC-1 and other sediment stations. For example, DDE was found at a notably higher concentration at station LC-1 compared with other sediment stations (84,400 ppb vs. <400 ppb) (Figure 3.2). Thus, DDE constituted 65% of the total DDT value at Station LC-1, versus 3% at other stations (Table 3.7). White et al. (1994) presented the relative contribution of DDT metabolites from sediment collected in Lauritzen Canal, Santa Fe Channel, and Inner Richmond Harbor. The DDT metabolite distribution in sediment from the south end of Lauritzen Canal in 1998 is similar to that of Lauritzen Canal sediment from 1993.



**Figure 3.2.** Sediment Concentration of DDT, DDE, DDD, and Dieldrin in Sediment Samples from Lauritzen Canal, November 1998

Elevated sediment concentrations of DDT and dieldrin in Lauritzen Canal were likely to have contributed to elevated contaminant levels found in the water column and biomonitoring organisms in February 1999. Station 303.3 at Lauritzen Canal/End (northern end) had the highest levels of both total DDT and dieldrin of the water and tissue sampling stations. Water concentrations of both contaminants were approximately 95% lower at the mouth of Lauritzen Canal (Station 303.2) than at the end of the canal (Station 303.3) in February 1999. Mussel tissue levels from both resident and transplanted organisms were about 75% lower at the canal mouth than in comparison with the canal end.

Concentrations of other analytes in sediment samples (i.e., pesticides, aroclors, and PAHs) were consistently highest at the end of Lauritzen Canal (Table 3.7). In general, these analytes were lowest in the sandy sediment sample collected at LC-2 (Lauritzen Canal North/Center). Only one PCB was detected. The sediment concentration of Aroclor 1254 declined progressively from 981  $\mu\text{g/kg}$  (dry weight) at the north end to 89.9  $\mu\text{g/kg}$  (dry weight) at the southern end (or mouth) of Lauritzen canal. Thus, the spatial trend of sediment contamination was similar for dieldrin, DDT, and PCB, but not for PAHs.

#### **4.0 CONCLUSIONS**

Results from the first post-remediation monitoring (1997-1998) indicated that chlorinated pesticides remained in the Lauritzen Canal and in the semi-enclosed waters nearby. Grab samples of water collected in February 1999 indicate that the total DDT and dieldrin concentrations in the water are similar to preremediation levels. Thus, remediation goals for total DDT and dieldrin in water have not yet been achieved for the study site. However, biomonitoring confirmed that the bioavailability of total DDT and dieldrin demonstrated by resident and transplanted bivalves is dramatically lower at all study stations relative to preremediation data. Bioavailability of these two pesticides also decreased between Year 1 and Year 2 of biomonitoring. Further biomonitoring will be important to determine whether these data are representative of long-term bioavailability of pesticides from the Lauritzen Canal sediment.

Surface sediment collected in November 1998 from the Lauritzen Canal showed significant contamination of DDT, dieldrin, and other compounds. Levels of DDT and dieldrin were lower than but comparable to preremediation concentrations in the Lauritzen Canal.

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**APPENDIX A**  
**FIELD SUMMARY REPORTS**




UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
REGION IX LABORATORY  
1337 S. 46TH STREET  
BLDG 201  
RICHMOND, CA 94804-4698

January 13, 1998

MEMORANDUM

SUBJECT: Summary of United Heckathorn Post-Remedial  
Mussel and Surface Water Sampling

FROM: Andrew Lincoff, PMD-2   
Regional Laboratory

TO: Dick Vesperman, SFD-7-3  
Remedial Project Manager

Attached is the Field Sampling Summary for the post-remedial mussel and surface water sampling at the United Heckathorn Superfund Site in Richmond, California. Transplanted California mussels were deployed at four locations in Richmond Harbor in September, 1997. On January 6 and 7, 1998, seawater samples, resident mussels and the transplanted mussels were collected. Samples were shipped to the Battelle Marine Sciences Laboratory in Sequim, Washington for analysis. Replicate samples were taken for analysis at the Regional Laboratory. Results are expected to be available in approximately two months and will be forwarded to you in separate reports.

If you have any questions, please call me at (510) 412-2330.

Attachment

✓ CC: LIAM ANTRIM

Field Sampling Summary for Mussels and Surface Water  
at the United Heckathorn Site in  
Richmond, California, conducted 1/6 - 1/7/98.

Andrew Lincoff  
EPA Region 9 Laboratory  
PMD-2  
January 13, 1998

## INTRODUCTION

This sampling event involved the collection of mussels and surface water samples from the Lauritzen Channel at the United Heckathorn Superfund Site and at other locations in Richmond Harbor in Richmond, California.

Sampling was performed by Andrew Lincoff and Amy Wagner of the EPA Region 9 Laboratory. Some of the mussels retrieved had been transplanted in September, 1997 with the assistance of Liam Antrim, of the Battelle Marine Sciences Laboratory, EPA's Superfund Program contractor.

Sampling was performed in accordance with Battelle's "United Heckathorn Post-Remediation Field Monitoring Plan" (FSP), dated February 5, 1997, with minor deviations discussed herein. The most significant change was that additional replicate samples were taken for analysis by the EPA Regional Laboratory in order to perform an inter-laboratory comparison to provide additional information regarding the accuracy of the results.

## OBJECTIVE

EPA conducted this field sampling as part of the oversight of a final Remedial Action under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA or Superfund) at the United Heckathorn Site in Richmond, California. The sampling effort involved collecting physical environmental samples to analyze for the presence of hazardous substances.

The United Heckathorn Site was used to formulate pesticides from approximately 1947 to 1966. Soils at the Site and sediments in Richmond Harbor were contaminated with various chlorinated pesticides, primarily DDT, as a result of these pesticide formulation activities. The final remedy contained in EPA's October, 1994 Record of Decision addressed remaining hazardous substances, primarily in the marine environment. The major marine components of the selected remedy included:

- Dredging of all soft bay mud from the Lauritzen Channel and Parr Canal, with offsite disposal of dredged material.

decided to take additional sample volumes for analysis by the EPA Regional Lab in Richmond, California. These samples were taken at the same locations and at the same time as the Battelle samples.

2. The FSP called for ambient salinity measurements to be made during sampling. These were mistakenly not performed in the field, but will be performed by Battelle in the laboratory.

3. When the transplanted mussels were deployed in September 1997, a second set was hung beneath the Ford automotive plant for duplication in case of vandalism at Station 303.1. As none of the mussels were disturbed, the additional set (called 303.1X in the field log) was discarded.

### FIELD NOTES AND OBSERVATIONS

1. Samples were taken on January 6 and 7, 1997 at low tide. The weather during the sampling was calm with clouds and occasional light rain. The ambient water temperature was 12 C at all sample locations.

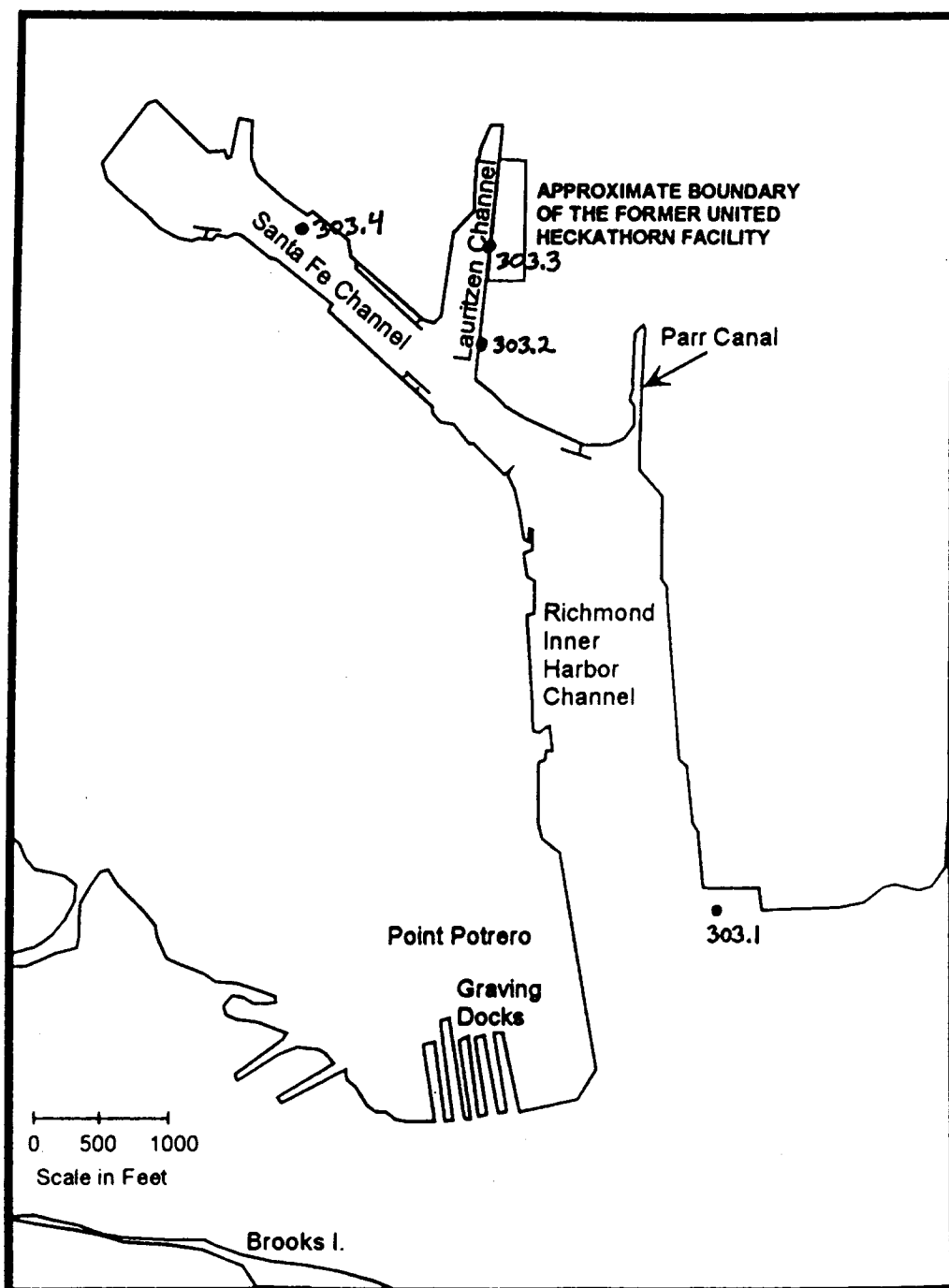
2. Factors which may influence the results included ongoing dredging in Richmond Harbor and pier maintenance at the Levin Terminal in the Lauritzen Channel. The Richmond Harbor deepening project has been ongoing since the fall of 1997. The dredging started in the upper Santa Fe Channel, near site 303.4, and was near Brooks Island and Point Potrero when the samples were retrieved. The effect of the dredging during the mussel deployment is uncertain. The dredging probably resuspended sediment containing some DDT and dieldrin which could raise values. On the other hand, the dredging removed most of the remaining 2% of the mass of DDT from Richmond Harbor not removed by the Superfund Remedy. Thus the results could be lower than they would have been without the deepening project.

Another less likely potential influence was the replacement of piles at the Levin Pier during the retrieval of samples. Conceivably, the pile driving could have resuspended sediment beneath the pier and increased the pesticide load in mussels and seawater samples.

3. The sample station numbers, locations, date and times, and other information are shown in Figure 1 and listed in Table 1, below. Location coordinates were determined using GPS with differential correction. As discussed in the FSP, the station numbers are those used by the California Mussel Watch Program. Station 303.1 is at the entrance to the Richmond Inner Harbor Channel near the old Ford automotive plant. Mussels were deployed and collected from the western-most of the large dolphins near the plant. Station 303.2 is on the eastern side of the Lauritzen near its mouth. Mussels were deployed from pilings beneath the Levin Dock near the northern end of a large wooden fender structure. Station 303.3 is approximately 2/3 of the way up the Lauritzen Channel, on the eastern side. Mussels were hung from the southern end of a small wooden pier which extends out into the channel. This location is very close to where the highest levels of pesticide residues were removed from the Heckathorn Site. Station 303.4 is in

Figure 1

Sample Locations 1/6 - 1/7/98



Field Sampling Summary for Mussels and Surface Water  
at the United Heckathorn Site in  
Richmond, California, conducted 2/23/99.

Andrew Lincoff  
EPA Region 9 Laboratory  
PMD-2  
May 13, 1999

## INTRODUCTION

This sampling event involved the collection of mussels and surface water samples from the Lauritzen Channel at the United Heckathorn Superfund Site and at other locations in Richmond Harbor in Richmond, California. This report concludes the sampling event begun with the deployment of mussels on November 3, 1998, as discussed in the November 19, 1998 Field Sampling Report.

Sampling was performed by Andrew Lincoff and Peter Husby of the EPA Region 9 Laboratory with the assistance of Dick Vesperman, United Heckathorn RPM. Some of the mussels retrieved had been transplanted to Richmond Harbor in November, 1998 with the assistance of Amy Wagner of the EPA Region 9 Laboratory.

Sampling was performed in accordance with Battelle's "United Heckathorn Post-Remediation Field Monitoring Plan" (FSP), dated February 5, 1997.

## OBJECTIVE

EPA conducted this field sampling as part of the oversight of a final Remedial Action under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA or Superfund) at the United Heckathorn Site in Richmond, California. The sampling effort involved collecting physical environmental samples to analyze for the presence of hazardous substances.

The United Heckathorn Site was used to formulate pesticides from approximately 1947 to 1966. Soils at the Site and sediments in Richmond Harbor were contaminated with various chlorinated pesticides, primarily DDT, as a result of these pesticide formulation activities. The final remedy contained in EPA's October, 1994 Record of Decision addressed remaining hazardous substances, primarily in the marine environment. The major marine components of the selected remedy included:

- Dredging of all soft bay mud from the Lauritzen Channel and Parr Canal, with offsite disposal of dredged material.

- Marine monitoring to verify the effectiveness of the remedy.

The first component of the remedy selected in the ROD called for dredging all "young bay mud" from those channels in Richmond Harbor which contained average DDT concentrations greater than 590 ppb (dry wt.). The dredging was completed in April, 1997. The short-term monitoring, performed according to EPA's September 5, 1996 FSP, consisted of sediment chemistry monitoring to ensure that the average sediment concentration after dredging was below the cleanup level selected in the ROD. This monitoring was completed shortly prior to the placement of the sand cap in April, 1997.

Long-term monitoring is addressed by Battelle's February 5, 1997 FSP. The purpose of the long-term monitoring is to demonstrate the effectiveness of the remedy. Prior to the remediation, mussels in the Lauritzen Channel contained the highest levels of DDT and dieldrin in the State, and surface water exceeded EPA's Ambient Water Quality Criteria for DDT by a factor of 50. Lower but still elevated levels were found in mussels and surface water in the Santa Fe Channel. It was concluded in EPA's Remedial Investigation that these elevated levels were the result of continuous flux from contaminated sediments. Approximately 98% of the mass of DDT in sediments in Richmond Harbor was removed by the remedial dredging. The long-term monitoring will demonstrate whether this action has succeeded in reducing the levels of DDT in mussels and surface waters.

Battelle's FSP included monitoring using both transplanted California mussels and resident Bay mussels. The first round of the long-term sampling occurred in January, 1998. The second year's transplanted mussels were deployed in November, 1998 and retrieved after approximately four months of exposure. The length of the deployment and seasonal timing were chosen to match the protocol used by the California State Mussel Watch Program, in order to permit comparison with the State's results over the past 15 years. Both transplanted and resident mussels are analyzed to determine any difference.

Laboratory results are expected from Battelle in approximately one month.

## FIELD NOTES AND OBSERVATIONS

1. Samples were collected on February 23, 1999 at low tide. The weather during the sampling was sunny and calm.

2. The sample station numbers, locations, date and times, and other information are listed in Table 1, below. Location coordinates were determined using GPS with differential correction on 1/6/98. As discussed in the FSP, the station numbers are those used by the California Mussel Watch Program. Station 303.1 is at the entrance to the Richmond Inner Harbor Channel near the old Ford automotive plant. Mussels were deployed and collected from the western-most of the large dolphins near the plant. Station 303.2 is on the eastern side of the Lauritzen near its mouth. Mussels were deployed from pilings beneath the Levin Dock near the northern end of a large wooden fender structure. Station 303.3 is approximately 2/3 of the way up the Lauritzen



Channel, on the eastern side. Mussels were hung from the southern end of a small wooden pier which extends out into the channel. This location is very close to where the highest levels of pesticide residues were removed from the Heckathorn Site. Station 303.4 is in the upper Santa Fe Channel at the far western end of a large covered floating marina on the northern side.

Table 1  
Mussel and Seawater Sample Locations

<u>Station</u>	<u>Date</u>	<u>Time</u>	<u>Location</u>	<u>Remarks</u>
303.1	2/23/99	1341	37 54' 32.8" N 122 21' 34.5" W	Richmond Channel
303.2	2/23/99	1312	37 55' 12.6" N 122 22' 01.2" W	Lauritzen South Blind Dup. Seawater labeled 303.5
303.3	2/23/99	1254	37 55' 22.5" N 122 21' 59.9" W	Lauritzen North MS/MSD Seawater
303.4	2/23/99	1222	37 55' 21.53" N 122 21' 18.37" W	Santa Fe

Seawater, transplanted California Mussels, and resident Bay mussels were collected at each station for analysis by Battelle. At each station three 2 liter replicate seawater samples were collected for analysis by Battelle. At station 303.3, two additional 2 liter seawater samples were collected for Battelle QA/QC. An additional single 2 liter blind duplicate of seawater sample 303.2 was collected and shipped to the Battelle Lab with the fictitious station number 303.5.

At each station, approximately 45 transplanted mussels and 45 resident mussels were collected. The 45 mussels per sample sent to Battelle is large enough for any sample to be selected by Battelle for laboratory QA/QC.

The ~~resident mussels~~ were all collected near the surface, which at the collection times and dates was approximately 0.4 foot above Mean Lower Low Water for the samples collected from pilings at stations 303.1, 303.2, and 303.3. At station 303.4, the mussels were collected near the surface from a floating dock. The transplanted mussels were deployed at the following approximate depths: 303.1, -2 ft MLLW; 303.2, -2 ft. MLLW, 303.3, -2 ft MLLW. At station 303.4 the transplanted mussels were hung from a floating dock, and were always approximately 1 ft. below sea level.

## **APPENDIX B**

### **ANALYTICAL RESULTS FROM WATER AND TISSUE SAMPLES**

# Analytical Chemistry Data Package

## Project: Heckathorn Biomonitoring Year 2 1999 Sample Collection

Battelle Project No. 20212

CF No. 1321

### Contents:

- **Analysis of Pesticides in Tissues**
  - Data Table
  - QA/QC Narrative
  - Custody Forms
- **Analysis of Pesticides in Water**
  - Data table
  - QA/QC Narrative
  - Custody Forms

Approvals:

  
Project Manager

5/12/99  
Date

  
QA/QC Officer

5/13/99  
Date

**BATTELLE MARINE SCIENCES LABORATORY**

1529 West Sequim Bay Road

Sequim, WA 98382-9099

360/681-3643

**UNITED HECKATHORN**

Pesticides in Tissues

Samples Received 2/25/99

MSL Code	1321-6	1321-7	1321-8	1321-9	1321-10	1321-11	1321-12	1321-13	1321-14
STATION NO	303.3	303.3	303.1	303.1	303.2	303.2	303.4	303.4	202
LOCATION	LC-N-RES	LC-N-TRANS	RH-RES	RH-TRANS	LC-S-RES	LC-S-TRANS	SFC-RES	SFC-TRANS	BODEGA HEAD
Matrix	Tissue	Tissue	Tissue	Tissue	Tissue	Tissue	Tissue	Tissue	Tissue
Wet Wt (g)	10.6	10.0	10.0	10.1	10.2	10.0	10.0	10.2	10.6
Percent Wet Wt	90.8	89.1	91.6	87.7	92.3	89.7	90.6	90.1	83.8
Extraction Date	03/04/99	03/04/99	03/04/99	03/04/99	03/04/99	03/04/99	03/04/99	03/04/99	03/04/99
Percent Lipids (DW)	7.00	8.00	7.57	7.50	9.19	8.21	9.82	8.20	8.13
Dilution	5X	5X			2X	2X			
Analytical Batch	1	1	1	1	1	1	1	1	1
Unit (wet wt)	ng/g	ng/g	ng/g	ng/g	ng/g	ng/g	ng/g	ng/g	ng/g
2,4'-DDE	4.65	7.80	1.42	1.17	1.88	2.78	0.55	0.80	5.68
Dieldrin	28.4	106	1.86	8.22	6.50	26.9	2.77	9.73	1.34
4,4'-DDE	71.5	87.5	8.21	8.17	31.6	32.7	17.5	12.8	2.37
2,4'-DDD	75.6	119	2.45	6.26	16.1	40.7	6.58	10.3	0.35 U
4,4'-DDD	143	311	7.18	18.7	37.7	101	18.9	32.1	0.68
2,4'-DDT	113	167	3.37	4.17	32.0	43.1	10.5	7.64	0.49 U
4,4'-DDT	198	289	7.08	7.07	56.6	61.9	21.6	16.1	0.34 U

**SURROGATE RECOVERIES (%)**

PCB103	97.3	99.2	76.5	79.9	87.0	71.5	74.3	73.8	73.7
PCB198	88.8	86.1	80.3	82.8	82.0	65.9	76.2	75.3	74.6

M Mean used to calculate QC

U Not detected at or above DL shown

ND Analyte not detected

**BATTELLE MARINE SCIENCES LABORATORY**

1529 West Sequim Bay Road

Sequim, WA 98382-9099

360/681-3643

**UNITED HECKATHORN**

Pesticides in Tissues

Samples Received 2/25/99

MSL Code	Blank	BSA Blank	Spike	Percent	BSB Blank	Spike	Percent	1321-13	1321-13 dup	
STATION NO		Spike A	Amount	Recovery	Spike B	Amount	Recovery	303.4	303.4	
LOCATION								SFC-TRANS	SFC-TRANS	RPD
Matrix	Tissue	Tissue			Tissue			Tissue	Tissue	
Wet Wt (g)	NA	NA			NA			10.2	10.4	
Percent Wet Wt	NA	NA			NA			90.1	90.1	
Extraction Date	03/04/99	03/04/99			03/04/99			03/04/99	03/04/99	
Percent Lipids (DW)	0.08							8.20		
Dilution										
Analytical Batch	1	1			1			1	1	
Unit (wet wt)	ng/g	ng/g	ng/g	%	ng/g	ng/g	%	ng/g	ng/g	%
2,4'-DDE	0.27 U	1.05	NS	NA	0.70	NS	NA	0.80	0.81	1%
Dieldrin	0.29 U	9.56	10.0	96%	9.68	10.0	97%	9.73	10.0	3%
4,4'-DDE	1.03 U	1.03 U	NS	NA	1.03 U	NS	NA	12.8	13.2	3%
2,4'-DDD	0.38 U	0.38 U	NS	NA	0.38 U	NS	NA	10.3	10.9	6%
4,4'-DDD	0.36 U	0.36 U	NS	NA	0.36 U	NS	NA	32.1	30.6	5%
2,4'-DDT	0.52 U	0.52 U	NS	NA	0.52 U	NS	NA	7.64	8.22	7%
4,4'-DDT	0.36 U	12.0	10.0	120%	11.3	10.0	113%	16.1	15.8	2%
<b><u>SURROGATE RECOVERIES (%)</u></b>										
PCB103	88.2	82.0			70.0			73.8	89.0	
PCB198	91.1	86.1			77.6			75.3	86.9	

U Not detected at or above DL shown

**BATTELLE MARINE SCIENCES LABORATORY**

1529 West Sequim Bay Road

Sequim, WA 98382-9099

360/681-3643

**UNITED HECKATHORN**

Pesticides in Tissues

Samples Received 2/25/99

		MSA			MSB			
MSL Code	1321-9	1321-9	Spike	Percent	1321-9	Spike	Percent	
STATION NO	303.1	303.1			303.1			
LOCATION	RH-TRANS	Spike A	Amount	Recovery	Spike B	Amount	Recovery	RPD
Matrix	Tissue	Tissue			Tissue			
Wet Wt (g)	10.1	10.2			10.1			
Percent Wet Wt	87.7	87.7			87.7			
Extraction Date	03/04/99	03/04/99			03/04/99			
Percent Lipids (DW)	7.50							
Dilution								
Analytical Batch	1	1			1			
Unit (wet wt)	ng/g	ng/g	ng/g	%	ng/g	ng/g	%	%
2,4'-DDE	1.17	2.27	NS	NA	2.00	NS	NA	
Dieldrin	8.22	15.6	9.77	76%	16.4	9.91	83%	9%
4,4'-DDE	8.17	9.19	NS	NA	8.97	NS	NA	
2,4'-DDD	6.26	7.26	NS	NA	7.19	NS	NA	
4,4'-DDD	18.7	20.3	NS	NA	20.3	NS	NA	
2,4'-DDT	4.17	4.74	NS	NA	4.65	NS	NA	
4,4'-DDT	7.07	18.0	9.77	112%	18.9	9.91	119%	6%

**SURROGATE RECOVERIES (%)**

PCB103	79.9	81.5	82.2
PCB198	82.8	84.6	82.6

U Not detected at or above DL shown

**BATTELLE MARINE SCIENCE LABORATORIES**

1529 West Sequim Bay Road  
Sequim, Washington 98382-9099  
360/681-3643

**UNITED HECKATHORN**

PCBs in Tissues  
Samples Received 3/2/99

MSL Code	1321-6	1321-7	1321-8	1321-9	1321-10	1321-11	1321-12	1321-13	1321-14
STATION NO	303.3	303.3	303.1	303.1	303.2	303.2	303.4	303.4	202
LOCATION	LC-N-RES	LC-N-TRANS	RH-RES	RH-TRANS	LC-S-RES	LC-S-TRANS	SFC-RES	SFC-TRANS	BODEGA HEAD
Matrix	Tissue	Tissue	Tissue	Tissue	Tissue	Tissue	Tissue	Tissue	Tissue
Extract Date	3/4/99	3/4/99	3/4/99	3/4/99	3/4/99	3/4/99	3/4/99	3/4/99	3/4/99
Analysis Date	8/10/99	8/10/99	8/10/99	8/10/99	8/10/99	8/10/99	8/10/99	8/10/99	8/10/99
Wet Wt (g)	10.6	10.0	10.0	10.1	10.2	10.0	10.0	10.2	10.6
Percent wet wt	90.8	89.1	91.6	87.7	92.3	89.6	90.6	90.1	83.8
Analytical Rep	1	1	1	1	1	1	1	1	1
Units (wet wt)	ng/g	ng/g	ng/g	ng/g	ng/g	ng/g	ng/g	ng/g	ng/g
Aroclor 1242	ND	ND	ND	ND	ND	ND	ND	ND	ND
Aroclor 1248	ND	ND	ND	ND	ND	ND	ND	ND	ND
Aroclor 1254	124	79.7	51.0	40.9	75.0	48.9	67.4	36.7	13.5 U
Aroclor 1260	ND	ND	ND	ND	ND	ND	ND	ND	ND

U	Not detected at or above DL shown
NA	Not applicable/available
ND	Not detected
NS	Not spiked

**BATTELLE MARINE SCIENCE LABORATORIES**

1529 West Sequim Bay Road  
 Sequim, Washington 98382-9099  
 360/681-3643

**UNITED HECKATHORN**

PCBs in Tissues  
 Samples Received 3/2/99

MSL Code STATION NO LOCATION	BSA				BSB				MSA				MSB		
	Blank	Blank	SPK	Percent	Blank	SPK	Percent	1321-9	1321-9	SPK	Percent	1321-9	SPK	Percent	
		Spike A	AMT	Recovery	Spike B	AMT	Recovery	303.1	Spike A	AMT	Recovery	Spike B	AMT	Recovery	
	RH-TRANS														
Matrix	Tissue	Tissue			Tissue			Tissue	Tissue			Tissue			
Extract Date	3/4/99	3/4/99			3/4/99			3/4/99	3/4/99			3/4/99			
Analysis Date	8/10/99	8/10/99			8/10/99			8/10/99	8/10/99			8/10/99			
Wet Wt (g)								10.1	10.2			10.1			
Percent wet wt								87.7	87.7			87.7			
Analytical Rep	1	1			1			1	1			2			
Units (wet wt)	ng/g	ng/g	ng/g	%	ng/g	ng/g	%	ng/g	ng/g	ng/g	%	ng/g	ng/g	%	
Aroclor 1242	ND	ND	NS	NA	ND	NS	NA	ND	ND	NS	NA	ND	NS	NA	
Aroclor 1248	ND	ND	NS	NA	ND	NS	NA	ND	ND	NS	NA	ND	NS	NA	
Aroclor 1254	14.3 U	107	100	107%	109	100	109%	40.9	138	97.7	99%	138	99.1	98%	
Aroclor 1260	ND	ND	NS	NA	ND	NS	NA	ND	ND	NS	NA	ND	NS	NA	

U Not detected at or above DL shown  
 NA Not applicable/available  
 ND Not detected  
 NS Not spiked



**BATTELLE MARINE SCIENCE LABORATORIES**

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<i>DUP</i>			
MSL Code	1321-13	1321-13	
STATION NO	303.4	303.4	
LOCATION	SFC-TRANS	SFC-TRANS	RPD
Matrix	Tissue	Tissue	
Extract Date	3/4/99	3/4/99	
Analysis Date	8/10/99	8/10/99	
Wet Wt (g)	10.2	10.4	
Percent wet wt	90.1	90.1	
Analytical Rep	1	2	
Units (wet wt)	ng/g	ng/g	
Aroclor 1242	ND	ND	
Aroclor 1248	ND	ND	
Aroclor 1254	36.7	40.5	9%
Aroclor 1260	ND	ND	

U	Not detected at or above DL shown
NA	Not applicable/available
ND	Not detected
NS	Not spiked

## QA/QC SUMMARY

**PROJECT:** Heckathorn Biomonitoring Year 2  
**PARAMETER:** Pesticides and Total Lipids  
**LABORATORY:** Battelle/Marine Sciences Laboratory, Sequim, Washington  
**MATRIX:** Tissues

**SAMPLE CUSTODY:** Nine mussel tissue samples were received on 2/25/99. All samples were received in good condition. The cooler temperature on arrival was 5.1 °C. Samples were assigned a Battelle Central File (CF) identification number (1321) and were entered into Battelle's log-in system.

### QA/QC DATA QUALITY OBJECTIVES:

<u>Analyte</u>	<u>Extraction Method</u>	<u>Analytical Method</u>	<u>Range of Recovery</u>	<u>Relative Precision</u>	<u>Achieved Detection Limit (ng/g)</u>
2,4'-DDE	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	0.27
Dieldrin	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	0.29
4,4'-DDE	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	1.03
2,4'-DDD	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	0.38
4,4'-DDD	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	0.36
2,4'-DDT	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	0.52
4,4'-DDT	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	0.36
Total Lipids	CHCl <sub>3</sub>	Gravimetric	NA	±30%	NA

**METHOD:** Chlorinated pesticides were analyzed according to a Battelle SOP based on EPA Method 8081 (EPA 1986) with modifications based on Krahn et al. (1988). Tissue samples were macerated and extracted with methylene chloride. Interferences were removed by aluminum/silicon column chromatography followed by high-performance liquid chromatography (HPLC) clean-up. Sample extracts were then transferred to cyclohexane and analyzed by capillary-column (DB-1701) gas chromatography with electron-capture detection (GC/ECD). Total lipids were determined according to the Bligh et al. (1959) method, modified to accommodate a smaller sample size. Lipids were extracted from separate aliquots of tissue samples using chloroform and the lipid weight obtained gravimetrically.

**HOLDING TIMES:** All extractions and analyses were conducted within target holding times: 14 days to extraction (refrigerated, not frozen), and 40 days to analysis after extraction. Samples were received on 2/25/99 and held at 4°C. Samples were extracted on 3/4/99 and analyzed on 3/18/99. Lipid extractions were conducted on 3/10/99.

## QA/QC SUMMARY

<b>DETECTION LIMITS:</b>	Detection limits were determined by a previously conducted MDL study where replicates were analyzed and the standard deviation was multiplied by the Student's-t value for the number of replicates.
<b>BLANKS/BLANK SPIKES:</b>	One procedural blank and two blank spikes were analyzed. All analytes were undetected in the blank. Blank spike recoveries of the two spiked analytes of interest, dieldrin and 4,4'-DDT, were within the target range of 40%-120%.
<b>REPLICATES:</b>	One tissue sample (303.4 SFC-TRANS) was analyzed in duplicate. Precision for duplicate analysis is reported by calculating the relative percent difference (RPD) of replicate results. RPDs for all analytes of interest ranged from 1% to 7%, and were all within the QC limits of $\pm 30\%$ .
<b>MATRIX SPIKES:</b>	A matrix spike and matrix spike duplicate were analyzed using sample 303.1 RH-TRANS. Recoveries of the two spiked analytes of interest, dieldrin and 4,4'-DDT, were within the target range of 40%-120% in both the MS and MSD. The RPD between the MS and MSD was $<30\%$ for both dieldrin and 4,4'-DDT.
<b>SURROGATE RECOVERIES:</b>	Chlorinated compounds PCBs 103 and 198 were added to each sample during the preparation step as surrogates to assess the efficiency of the extraction procedure. Surrogate recoveries ranged from 65.9% to 99.2%.
<b>REFERENCES:</b>	<p>Bligh, E.G., and W.J. Dyer. 1959. A Rapid Method of Total Lipid Extraction and Purification. <i>Canadian Journal of Biochemistry and Physiology</i>. 37:8 911-917.</p> <p>Krahn, M.M, CA Wigren, R.W. Pearce, S.K. Moore, R.G. Bogar, W. D. McLeod, Jr., S.L. Chan, and D.W. Brown. 1988. <i>New HPLC Cleanup and Revised Extraction Procedures for Organic Contaminants</i>. NOAA Technical Memorandum MNFS F/NWC-153. Standard Analytical Procedures of the NOAA National Facility, 1988. National Oceanic and Atmospheric Administration, National Marine Fisheries Service, Seattle, WA.</p> <p>U.S. EPA. 1986 (Revised 1990). <i>Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846</i>. 3rd ed. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C.</p>

cc: Project Manager/Central File  
Login File

1321

## SAMPLE LOGIN

Project Manager: BARROWS  
Date Received: 3/2/99  
Batch: 2

PROJECT: UNITED HECKATHOR

SPONSOR CODE	BATTELLE CODE	MATRIX	STORAGE LOCATION	PARAMETERS REQUESTED	COLLECTION DATE	INITIALS
303.3 LC-N-RES	1321*6	TISSUE	ORG LAB 215	PEST, LIPIDS	2/26/99	MLFM
303.3 LC-N-TRANS	1321*7	TISSUE	ORG LAB 215	PEST, LIPIDS	2/26/99	MLFM
303.1 RH-RES	1321*8	TISSUE	ORG LAB 215	PEST, LIPIDS	2/26/99	MLFM
303.1 RH-TRANS	1321*9	TISSUE	ORG LAB 215	PEST, LIPIDS	2/26/99	MLFM
303.2 LC-S-RES	1321*10	TISSUE	ORG LAB 215	PEST, LIPIDS	2/26/99	MLFM
303.2 LC-S-TRANS	1321*11	TISSUE	ORG LAB 215	PEST, LIPIDS	2/26/99	MLFM
303.4 SFC-RES	1321*12	TISSUE	ORG LAB 215	PEST, LIPIDS	2/26/99	MLFM
303.4 SFC-TRANS	1321*13	TISSUE	ORG LAB 215	PEST, LIPIDS	2/26/99	MLFM
202.00 BODEGA HEAD	1321*14	TISSUE	ORG LAB 215	PEST, LIPIDS	2/26/99	MLFM

COMMENTS:



Pacific Northwest Division  
Marine Sciences Laboratory  
1529 West Sequim Bay Road  
Sequim, Washington 98382

Date 3.2.99

Page 1 of 1

1321 Bat 2 EAC BB Bat 2

BC-1800-192 (07/94)

**REGION 9**  
**75 Hawthorne Street**  
**San Francisco, California 94105**

Q 27511

**BATTELLE MARINE SCIENCES LABORATORY**  
 1529 West Sequim Bay Road  
 Sequim, WA 98382-9099  
 360/681-3643

**UNITED HECKATHORN**  
 Pesticides in Water  
 Samples Received 2/25/99

MSL Code	1321-1A	1321-1B	1321-1C	1321-2B	1321-2C	1321-3A	1321-3C	1321-4	1321-5A	1321-5B
STATION NO	303.4	303.4	303.4	303.3	303.3	303.2	303.2	303.5	303.1	303.1
LOCATION	SFC	SFC	SFC	L-N	L-N	L-S	L-S	L sample	RHC	RHC
Matrix	Water	Water	Water	Water	Water	Water	Water	Water	Water	Water
Extraction Date	3/1/99	3/1/99	3/1/99	3/2/99	3/2/99	3/1/99	3/1/99	3/2/99	3/2/99	3/2/99
Dilution					2X					
Analytical Batch	1	1	1	1	1	1	1	1	1	1
Unit	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L
2,4'-DDE	0.01 U	0.74	0.12	0.30	0.43	0.01 U	0.45	0.01 U	0.07	0.01 U
Dieldrin	0.23	0.66	0.23	6.28	18.8	0.43	0.52	0.90	0.57	0.67
4,4'-DDE	1.69	0.52	0.25	2.96	3.81	0.37	0.49	0.41	1.81	2.38
2,4'-DDD	2.40	0.38	0.21	5.82	8.16	0.34	0.62	0.48	1.41	1.52
4,4'-DDD	15.0	0.94	0.72	13.5	21.4	1.18	1.75	1.25	5.70	2.06
2,4'-DDT	1.51	0.19	0.16	4.86	8.15	0.17	0.28	0.21	0.92	0.22
4,4'-DDT	30.7	0.05 U	2.20 B	13.8	41.4	1.08 B	2.49 B	0.52 B	9.96	2.68 B

**SURROGATE RECOVERIES (%)**

PCB103	68.9	75.1	134	75.8	81.3	80.1	101	68.7	79.5	61.3
PCB198	80.5	67.6	124	86.8	82.6	85.7	81.1	71.9	82.8	76.1

U Not detected at or above DL shown

B Concentration is less than 5x blank value

**BATTELLE MARINE SCIENCES LABORATORY**  
 1529 West Sequim Bay Road  
 Sequim, WA 98382-9099  
 360/681-3643

**UNITED HECKATHORN**  
 Pesticides in Water  
 Samples Received 2/25/99

MSL Code STATION NO LOCATION	BSA				BSB			MSA				MSB			RPD
	Blank	Blank	Spike	Percent	Blank	Spike	Percent	1321-2C 303.3 L-N	1321-2	Spike	Percent	1321-2	Spike	Percent	
Matrix	Water	Water			Water			Water	Water			Water			
Extraction Date	3/2/99	3/1/99			3/1/99			3/2/99	3/2/99			3/2/99			
Dilution								2X	2X			5X			
Analytical Batch	1	1			1			1	1			1			
Unit (dry wt)	ng/L	ng/L	ng/L	%	ng/L	ng/L	%	ng/L	ng/L	ng/L	%	ng/L	ng/L	%	%
2,4'-DDE	0.01 U	0.01 U	NS	NA	0.01 U	NS	NA	0.43	0.01 U	NS	NA	0.78	NS	NA	
Dieldrin	0.11 U	4.56	5.00	91%	4.17	5.00	83%	18.8	27.8	5.46	165% #	21.6	5.46	51% #	105%
4,4'-DDE	0.04	0.43	NS	NA	0.50	NS	NA	3.81	3.74	NS	NA	3.30	NS	NA	
2,4'-DDD	0.03 U	2.98	NS	NA	2.79	NS	NA	8.16	8.08	NS	NA	7.24	NS	NA	
4,4'-DDD	0.05 U	0.05 U	NS	NA	0.05 U	NS	NA	21.4	17.9	NS	NA	14.8	NS	NA	
2,4'-DDT	0.05 U	0.05 U	NS	NA	0.05 U	NS	NA	8.15	7.87	NS	NA	10.4	NS	NA	
4,4'-DDT	1.66	6.20	5.00	91%	6.08	5.00	88%	41.4	43.8	5.46	44% #	35.5	5.46	-108% #	NC

**SURROGATE RECOVERIES (%)**

PCB103	57.1	118	72.4	81.3	75.8	78.9
PCB198	81.6	87.7	81.7	82.6	82.5	86.0

U Not detected at or above DL shown  
 NC Not calculable  
 # Outside QAQC recovery limits



**BATTELLE MARINE SCIENCES LABORATORY**

1529 West Sequim Bay Road

Sequim, WA 98382-9099

360/681-3643

**UNITED HECKATHORN****Pesticides in Water: Precision of Field Replicates**

MSL Code	1321-1A	1321-1B	1321-1C		1321-2B	1321-2C		1321-3A	1321-3C		1321-5A	1321-5B	
STATION NO	303.4	303.4	303.4		303.3	303.3		303.2	303.2		303.1	303.1	
LOCATION	SFC	SFC	SFC	RSD	L-N	L-N	RPD	L-S	L-S	RPD	RHC	RHC	RPD
Matrix	Water	Water	Water		Water	Water		Water	Water		Water	Water	
Extraction Date	3/1/99	3/1/99	3/1/99		3/2/99	3/2/99		3/1/99	3/1/99		3/2/99	3/2/99	
Dilution						2X							
Analytical Batch	1	1	1		1	1		1	1		1	1	
Unit	ng/L	ng/L	ng/L		ng/L	ng/L		ng/L	ng/L		ng/L	ng/L	
2,4'-DDE	0.01 U	0.74	0.12	NA	0.30	0.43	36% &	0.01 U	0.45	NA	0.07	0.01 U	NA
Dieldrin	0.23	0.66	0.23	66% &	6.28	18.8	100% &	0.43	0.52	19%	0.57	0.67	16%
4,4'-DDE	1.69	0.52	0.25	93% &	2.96	3.81	25%	0.37	0.49	28%	1.81	2.38	27%
2,4'-DDD	2.40	0.38	0.21	122% &	5.82	8.16	33% &	0.34	0.62	58% &	1.41	1.52	8%
4,4'-DDD	15.0	0.94	0.72	147% &	13.5	21.4	46% &	1.18	1.75	39% &	5.70	2.06	94% &
2,4'-DDT	1.51	0.19	0.16	124% &	4.86	8.15	51% &	0.17	0.28	49% &	0.92	0.22	123% &
4,4'-DDT	30.7	0.05 U	2.20 B	NA	13.8	41.4	100% &	1.08 B	2.49 B	79% &	9.96	2.68 B	115% &

**SURROGATE RECOVERIES (%)**

PCB103	68.9	75.1	134		75.8	81.3		80.1	101		79.5	61.3
PCB198	80.5	67.6	124		86.8	82.6		85.7	81.1		82.8	76.1

U Not detected at or above DL shown

B Concentration is less than 5x blank value

&amp; Exceeds QC limits for precision (30%).

NA Not applicable (RSD/RPD only calculated when analyte concentration is a detectable value in all replicates).

**BATTELLE MARINE SCIENCE LABORATORIES**

1529 West Sequim Bay Road  
Sequim, Washington 98382-9099  
360/681-3643

**UNITED HECKATHORN**

PCBs in Water  
Samples Received 2/25/99

MSL Code	1321-1A	1321-1B	1321-1C	1321-2B	1321-2C	1321-3A	1321-3C	1321-4	1321-5A	1321-5B
STATION NO	303.4	303.4	303.4	303.3	303.3	303.2	303.2	303.5	303.1	303.1
LOCATION	SFC	SFC	SFC	L-N	L-N	L-S	L-S	L sample	RHC	RHC
Matrix	Water	Water	Water	Water	Water	Water	Water	Water	Water	Water
Extract Date	3/1/99	3/1/99	3/1/99	3/2/99	3/2/99	3/1/99	3/1/99	3/2/99	3/2/99	3/2/99
Analysis Date	8/10/99	8/10/99	8/10/99	8/10/99	8/10/99	8/10/99	8/10/99	8/10/99	8/10/99	8/10/99
Analytical Rep	1	1	1	1	1	1	1	1	1	1
Units	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L
Aroclor 1242	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Aroclor 1248	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Aroclor 1254	12.7 U	25.9	13.4 U	14.4 U	20.5	13.8 U	13.9 U	14.1 U	14.6 U	14.5 U
Aroclor 1260	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND

U	Not detected at or above DL shown
NA	Not applicable/available
ND	Not detected
NS	Not spiked

**BATTELLE MARINE SCIENCE LABORATORIES**

1529 West Sequim Bay Road  
 Sequim, Washington 98382-9099  
 360/681-3643

**UNITED HECKATHORN**

PCBs in Water  
 Samples Received 2/25/99

MSL Code STATION NO LOCATION	BSA				BSB			MSA				MSB		
	Blank	Blank Spike A	SPK AMT	Percent Recovery	Blank Spike B	SPK AMT	Percent Recovery	1321-2* 303.3 L-N	1321-2 Spike A	SPK AMT	Percent Recovery	1321-2 Spike B	SPK AMT	Percent Recovery
Matrix	Water	Water			Water			Water	Water			Water		
Extract Date	3/2/99	3/1/99			3/1/99			3/2/99	3/2/99			3/2/99		
Analysis Date	8/10/99	8/10/99			8/10/99			8/10/99	8/10/99			8/10/99		
Analytical Rep	1	1			2			1	1			2		
Units	ng/L	ng/L	ng/L	%	ng/L	ng/L	%	ng/L	ng/L	ng/L	%	ng/L	ng/L	%
Aroclor 1242	ND	ND	NS	NA	ND	NS	NA	ND	ND	NS	NA	ND	NS	NA
Aroclor 1248	ND	ND	NS	NA	ND	NS	NA	ND	ND	NS	NA	ND	NS	NA
Aroclor 1254	13.3 U	45.6	50.0	91%	49.5	50.0	99%	16.3	72.5	54.6	103%	59.0	54.6	78%
Aroclor 1260	ND	ND	NS	NA	ND	NS	NA	ND	ND	NS	NA	ND	NS	NA

U Not detected at or above DL shown

NA Not applicable/available

ND Not detected

NS Not spiked

\* Average of column A used to calculate spike recoveries

## QA/QC SUMMARY

**PROJECT:** Heckathorn Biomonitoring Year 2  
**PARAMETER:** Pesticides  
**LABORATORY:** Battelle/Marine Sciences Laboratory, Sequim, Washington  
**MATRIX:** Water

**SAMPLE CUSTODY:** Fifteen water samples in three coolers were received on 2/25/99. All containers were received in good condition except one replicate of sample 303.1 (Richmond Harbor), which had broken in transit. Cooler temperatures upon arrival were 5.0°C in two of the coolers and 4.2°C in the third. Samples were assigned a Battelle Central File (CF) identification number (1321) and were entered into Battelle's log-in system.

### QA/QC DATA QUALITY OBJECTIVES:

<u>Analyte</u>	<u>Extraction Method</u>	<u>Analytical Method</u>	<u>Range of Recovery</u>	<u>Relative Precision</u>	<u>Achieved Detection Limit (ng/L)</u>
2,4'-DDE	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	0.01
Dieldrin	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	0.11
4,4'-DDE	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	0.03
2,4'-DDD	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	0.03
4,4'-DDD	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	0.05
2,4'-DDT	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	0.05
4,4'-DDT	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	0.05

**METHOD:** Chlorinated pesticides were analyzed according to a Battelle SOP based on EPA Method 8081 (EPA 1986). Water samples were extracted with methylene chloride. Interferences were removed by aluminum/silicon column chromatography. Sample extracts were then transferred to cyclohexane and analyzed by capillary-column gas chromatography with electron-capture detection (GC/ECD).

**HOLDING TIMES:** All extractions and analyses were conducted within target holding times: 14 days to extraction, and 40 days to analysis after extraction. Samples were received on 2/25/99 and held at 4°C. Samples were extracted on 3/1/99 and analyzed on 3/19/99.

**DETECTION LIMITS:** Detection limits were determined by a previously conducted MDL study where replicates were analyzed and the standard deviation was multiplied by the Student's-t value for the number of replicates.

**BLANKS/BLANK SPIKES:** One procedural blank and two blank spikes were analyzed. All analytes except 4,4'-DDE and 4,4'-DDT were undetected in the blank. Samples with 4,4'-DDT concentrations less than 5 times the blank value (1.66 ng/L) were flagged with a "B".

Blank spike recoveries were within of the target range of 40%-120% for the two spiked analytes of interest, dieldrin (91% and 83%) and 4,4'-DDT (91% and 88%).

**MATRIX SPIKES:** A matrix spike and matrix spike duplicate were prepared and analyzed using two additional samples of sample 303.3 (Lauritzen North). Two

## QA/QC SUMMARY

analytes of interest, dieldrin and 4,4'-DDT, were, spiked into the sample at 5.46 ng/L. Recovery of dieldrin was outside of the target range of 40%-120% in the MS (165%) and within QC criteria in the MSD (51%). Recovery of 4,4'-DDT was within QC criteria in the MS (44%) but outside QC criteria in the MSD. The poor recovery results can likely be attributed to the high native levels of dieldrin and 4,4'-DDT, as well as other chlorinated pesticides, in the sample. Concentrations of dieldrin and 4,4'-DDT were almost 4 to 8 times higher in the sample than the spike level chosen for these analytes; therefore, calculation of recovery was not feasible.

### REPLICATES:

Three field replicate samples were provided for four of the samples: 303.4 (Santa Fe Channel), 303.3 (Lauritzen North), 303.2 (Lauritzen South), and 303.1 (Richmond Harbor). However, one replicate of 303.1 was broken during shipping, and one replicate from each of samples 303.3 and 303.2 were lost during the extraction procedure when the concentrator tubes separated from the evaporator flasks. Three replicates of sample 303.4 and two replicates of samples 303.3, 303.2, and 303.1 were available for determining precision.

Replication between field samples was poor. Precision of triplicate analyses is reported by calculating the relative standard deviation (RSD) of replicate results. RSDs for all analytes of interest detected in all three replicates of sample 303.4 ranged from 66% to 147%, and exceeded the data quality criteria for precision,  $\leq 30\%$ . Precision of duplicate analyses is expressed as the relative percent difference (RPD) between the two analyses. RPDs for all analytes of interest detected in both replicates of samples 303.3, 303.2, and 303.1 ranged from 8% to 123%.

### SURROGATE RECOVERIES:

Chlorinated compounds PCBs 103 and 198 were added to each sample during the preparation step as surrogates to assess the efficiency of the extraction procedure. Surrogate recoveries ranged from 57.1% to 134%.

### REFERENCES:

U.S. EPA. 1986 (Revised 1990). *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846. 3rd ed. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C.

## SAMPLE LOGIN

Project Manager: BARROWS  
Date: 2/25/99  
Batch: 1

PROJECT: UNITED HECKATHORN

SPONSOR CODE	BATTELLE CODE	MATRIX	STORAGE LOCATION	PARAMETERS REQUESTED		COLLECTION DATE	INITIALS
303.4	1321*1	WATER	ORG. LAB	PEST/ PCB	3 CONTAINERS	2/23/99	MLFM
303.3	1321*2	WATER	ORG. LAB	PEST/ PCB	5 CONTAINERS	2/23/99	MLFM
303.2	1321*3	WATER	ORG. LAB	PEST/ PCB	3 CONTAINERS	2/23/99	MLFM
303.5	1321*4	WATER	ORG. LAB	PEST/ PCB	1 CONTAINERS	2/23/99	MLFM
303.1	1321*5	WATER	ORG. LAB	PEST/ PCB	3 CONTAINERS (ONE BROKEN)	2/23/99	MLFM

[illegible]

## **APPENDIX C**

### **ANALYTICAL RESULTS FROM SEDIMENT SAMPLES**





# Applied Marine Sciences, Inc.

502 N. Highway 3, Suite B • League City, TX 77573 • (281) 554-7272 • Fax (281) 554-6356

## Summary Table

Project Number: PO # SEQ-24538-ESB  
Project Title: Heckathorn  
Client: BattelleMarine Sciences Lab  
AMS Project Number: 9902-01

Date Sampled: NA  
Date Received: 2/3/99  
Matrix: Soil  
Methods: Grain Size-PSEP, 1986  
TOC-PSEP, 1986  
Total Solids, EPA 160.3

Client Sample ID	AMS Sample ID	Gravel (%)	Sand (%)	Silt (%)	Clay (%)	TOC (%)	Total Solids (%)
1286-1	3745	0.00	14.04	23.93	62.03	1.53	36.79
1286-2	3746	0.00	9.03	25.26	65.71	1.67	36.37
1286-3	3747	0.68	67.14	10.61	21.57	0.89	64.04
1286-4	3748	0.10	31.67	43.05	25.19	3.11	19.39

Quality Assurance: These analyses performed in accordance with EPA guidelines for quality assurance.

AMS, Inc. Project Manager



# Applied Marine Sciences, Inc.

502 N. Highway 3, Suite B • League City, TX 77573 • (281) 554-7272 • Fax (281) 554-6356

Project Number: PO# SEQ-24538-ESB  
Project Title: Heckathorn  
Client: Battelle-MSL  
Client Sample ID: 1286-1  
AMS Sample ID: 3745

AMS Project Number: 9902-01

Date Sampled: NA

Date Received: 2/3/99

Matrix: Soil

## Total Solids (EPA 160.3)

Result	Unit	MDL	Date Analyzed
36.79	%	0.01%	2/5/99

## Total Organic Carbon (PSEP, 1986)

Result	Unit	MDL	Date Analyzed
1.53	%	0.01%	2/10/99

## Grain Size (PSEP, 1986)

Size Class	Particle Diameter (mm)	Result (%)	Date Analyzed
Gravel	> 2	0.00%	2/8/99
Sand	<2 to 0.0625	14.04%	2/8/99
Silt	<0.0625 to 0.0039	23.93%	2/8/99
Clay	<0.0039	62.03%	2/8/99

Quality Assurance: These analyses were performed in accordance with EPA guidelines for quality assurance.

AMS, Inc. Project Manager



# Applied Marine Sciences, Inc.

502 N. Highway 3, Suite B • League City, TX 77573 • (281) 554-7272 • Fax (281) 554-6356

Project Number: PO# SEQ-24538-ESB  
Project Title: Heckathorn  
Client: Battelle-MSL  
Client Sample ID: 1286-2  
AMS Sample ID: 3746

AMS Project Number: 9902-01

Date Sampled: NA

Date Received: 2/3/99

Matrix: Soil

## Total Solids (EPA 160.3)

Result	Unit	MDL	Date Analyzed
36.37	%	0.01%	2/5/99

## Total Organic Carbon (PSEP, 1986)

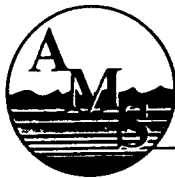
Result	Unit	MDL	Date Analyzed
1.67	%	0.01%	2/10/99

## Grain Size (PSEP, 1986)

Size Class	Particle Diameter (mm)	Result (%)	Date Analyzed
Gravel	> 2	0.00%	2/8/99
Sand	<2 to 0.0625	9.03%	2/8/99
Silt	<0.0625 to 0.0039	25.26%	2/8/99
Clay	<0.0039	65.71%	2/8/99

Quality Assurance: These analyses were performed in accordance with EPA guidelines for quality assurance.

AMS, Inc. Project Manager



# Applied Marine Sciences, Inc.

502 N. Highway 3, Suite B • League City, TX 77573 • (281) 554-7272 • Fax (281) 554-6356

Project Number: PO# SEQ-24538-ESB  
Project Title: Heckathorn  
Client: Battelle-MSL  
Client Sample ID: 1286-3  
AMS Sample ID: 3747

AMS Project Number: 9902-01

Date Sampled: NA

Date Received: 2/3/99

Matrix: Soil

## Total Solids (EPA 160.3)

Result	Unit	MDL	Date Analyzed
64.04	%	0.01%	2/5/99

## Total Organic Carbon (PSEP, 1986)

Result	Unit	MDL	Date Analyzed
0.89	%	0.01%	2/10/99

## Grain Size (PSEP, 1986)

Size Class	Particle Diameter (mm)	Result (%)	Date Analyzed
Gravel	> 2	0.68%	2/8/99
Sand	<2 to 0.0625	67.14%	2/8/99
Silt	<0.0625 to 0.0039	10.61%	2/8/99
Clay	<0.0039	21.57%	2/8/99

Quality Assurance: These analyses were performed in accordance with EPA guidelines for quality assurance

AMS, Inc. Project Manager



# Applied Marine Sciences, Inc.

502 N. Highway 3, Suite B • League City, TX 77573 • (281) 554-7272 • Fax (281) 554-6356

Project Number: PO# SEQ-24538-ESB  
Project Title: Heckathorn  
Client: Battelle-MSL  
Client Sample ID: 1286-4  
AMS Sample ID: 3748

AMS Project Number: 9902-01

Date Sampled: NA

Date Received: 2/3/99

Matrix: Soil

## Total Solids (EPA 160.3)

Result	Unit	MDL	Date Analyzed
19.39	%	0.01%	2/5/99

## Total Organic Carbon (PSEP, 1986)

Result	Unit	MDL	Date Analyzed
3.11	%	0.01%	2/10/99

## Grain Size (PSEP, 1986)

Size Class	Particle Diameter (mm)	Result (%)	Date Analyzed
Gravel	> 2	0.10%	2/8/99
Sand	<2 to 0.0625	31.67%	2/8/99
Silt	<0.0625 to 0.0039	43.05%	2/8/99
Clay	<0.0039	25.19%	2/8/99

Quality Assurance: These analyses were performed in accordance with EPA guidelines for quality assurance

AMS, Inc. Project Manager

# ***QUALITY CONTROL DOCUMENTATION***



# Applied Marine Sciences, Inc.

502 N. Highway 3, Suite B • League City, TX 77573 • (281) 554-7272 • Fax (281) 554-6356

## AMS QUALITY CONTROL REPORT

Project Number: P.O. # SEQ-24538-ESB  
Project Title: Heckathorn  
Client: Battelle Marine Sciences  
Client Sample ID: 1286-1  
AMS Sample ID: 3745

AMS Project #: 9902-01  
Date Sampled: NA  
Date Received: 2/3/99  
Matrix: Soil

### Total Solids (EPA 160.3)

Sample	Replicate	RPD	QC Limits	Date
Result %	Result %	%	% RPD	Analyzed
36.79	35.77	2.81	<25	2/5/99

Samples in Batch (AMS ID):      3745      3747  
   3746      3748

Quality Assurance: These analyses performed in accordance with EPA guidelines for quality assurance.

AMS, Inc. Project Manager



# Applied Marine Sciences, Inc.

502 N. Highway 3, Suite B • League City, TX 77573 • (281) 554-7272 • Fax (281) 554-6356

## Quality Control Report

Project Number: P.O. # SEQ-24538-ESB  
Project Title: Heackathorn  
Client: Battelle Marine Sciences  
Client Sample ID: 1286-1

AMS Project #: 9902-01  
Date Sampled: NA  
Date Received: 2/3/99  
Date Analyzed: 2/10/99  
Matrix: Soil  
Method: PSEP, 1986

### Continuing Calibration Data

AMS Sample ID	Parameter	SRM Result %	SRM Theoretical %	RPD %	QC Limits % RPD
Std1	TOC	4.87	4.80	1.45	<15

### TOC Method Blank

AMS Sample ID	Weight (g)	Result (ug CO <sub>2</sub> )	TOC (%)	TDL (%)
Blank	0.4960	20.7	ND	0.01

### Replicate Analysis

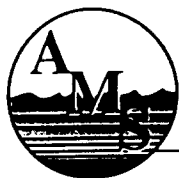
AMS Sample ID	Parameter	Sample Result %	Replicate Result %	RPD %	QC Limits % RPD
3745	TOC	1.53	1.49	2.65	<25

Samples in Batch (AMS ID):    3745    3747  
   3746    3748

Quality Assurance: These analyses are performed in accordance with EPA guidelines for quality assurance.

AMS, Inc. Project Manager





# Applied Marine Sciences, Inc.

502 N. Highway 3, Suite B • League City, TX 77573 • (281) 554-7272 • Fax (281) 554-6356

## QUALITY CONTROL REPORT

Project No.: PO#SEQ-24538-ESB  
Project Title: Heckathorn  
Client: Battelle Marine Science Lab  
Client Sample ID: 1286-1 *LR 4*  
AMS Sample ID: 3745

AMS Project Number: 9902-01  
Date Sampled: NA  
Date Received: 2/3/99  
Date Analyzed: 2/8/99  
Matrix: Soil  
Method: PSEP, 1986

Size Class	U.S. Standard Sieve Size	Diameter (mm)	Sample Result %	Duplicate Result %	RPD %	#	QC Limits % RPD
Gravel	No. 10	>2	0.00	0.00	0.00		<25
Sand	No. 230	<2 to 0.0625	14.04	13.72	2.31		<25
Silt		<0.0625 to 0.0039	23.93	24.14	0.71		<25
Clay		<0.0039	62.03	62.14	0.18		<25

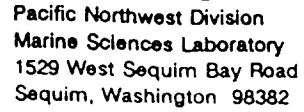
# Column to be used to flag RPD values with an asterisk  
\* Values outside of QC Limits

RPD: 0 out of 4 outside limits

Samples in Batch (AMS ID):    3745    3747  
   3746    3748

AMS, Inc. Project Manager

## ***CHAIN OF CUSTODY***



Date 2/2/99 Page 1 of 1

BC-1800-192 (07/9)

**BATTELLE MARINE SCIENCES LABORATORY**  
 1529 West Sequim Bay Road  
 Sequim, WA 98382-9099  
 360/681-3643

**HECKATHORN**  
 PAHs in Sediments  
 Samples Received 11/6/98

MSL Code	1286-1	1286-2	1286-3	1286-4
Sponsor ID	LC-4	LC-3	LC-2	LC-1
Matrix	Sed	Sed	Sed	Sed
Wet Wt (g)	10.2	10.1	10.0	10.1
Percent Dry Wt	38.4	34.7	65.8	19.5
Extraction Date	12/9/98	12/9/98	12/9/98	12/9/98
Analytical Batch	1	1	1	1
Unit (dry wt)	ng/g	ng/g	ng/g	ng/g
naphthalene	134	178	112	1960
1 methyl naphthalene	52.0	61.1	48.3	2790
Acenaphthalene	473	704	212	102
Acenaphthene	125	303	73.3	1830
Fluorene	199	394	162	3490
phenanthrene	728	1250	676	9120
anthracene	1070	2810	696	1760
fluoranthene	4510	5700	2140	5100
pyrene	2700	3170	1340	3870
benzo[a] anthracene	1970	3080	1150	1170
chrysene	2580	4580	1560	1710
benzo[b] fluoranthene	2220	3720	1740	1230
benzo[k] fluoranthene	822	1420	626	425
benzo[a] pyrene	1360	2320	1080	655
indeno [1,2,3-c,d] pyrene	463	789	396	278
dibenzo [a,h] anthracene	142	234	124	93.9
benzo [g,h,i] perylene	407	633	338	288
<b><u>SURROGATE RECOVERIES (%)</u></b>				
d8 naphthalene	18.4 #	35.3 #	26.5 #	26.6 #
d10 Acenaphthene	27.6 #	48.5	39.7 #	48.1
d10 phenanthrene	47.0	69.0	59.6	71.9
d12 chrysene	64.7	91.5	78.6	81.1
d12 perylene	63.3	90.9	77.2	77.6
d14 dibenzo[a,h] anthracene	80.0	110	92.8	112

U Not detected at or above DL shown  
 # Outside Surrogate Recovery limits of 40-120%

**BATTELLE MARINE SCIENCES LABORATORY**  
 1529 West Sequim Bay Road  
 Sequim, WA 98382-9099  
 360/681-3643

**HECKATHORN**  
 PAHs in Sediment  
 Samples Received 11/6/98

	BSA				BSB		
MSL Code	Blank	Blank	Spike	Percent	Blank	Spike	Percent
Sponsor ID		Spike A	Amount	Recovery	Spike B	Amount	Recovery
Matrix	Sed	Sed			Sed		
Wet Wt (g)	NA	NA			NA		
Percent Dry Wt	NA	NA			NA		
Extraction Date							
Analytical Batch	1	1			1		
Unit (dry wt)	ng/g	ng/g	ng/g	%	ng/g	ng/g	%
naphthalene	5.45 U	97.6	96.5	101%	96.1	96.5	100%
1 methyl naphthalene	5.45 U	5.45 U	NS	NA	5.45 U	NS	NA
Acenaphthalene	5.79 U	85.2	96.5	88%	88.2	96.5	91%
Acenaphthene	5.19 U	99.1	96.5	103%	106	96.5	110%
Fluorene	10.3 U	90.3	96.5	94%	94.5	96.5	98%
phenanthrene	12.2 U	86.3	96.5	89%	90.6	96.5	94%
anthracene	14.9 U	81.8	96.5	85%	87.1	96.5	90%
fluoranthene	6.19	65.0	96.5	61%	64.8	96.5	61%
pyrene	7.49	69.7	96.5	64%	69.2	96.5	64%
benzo[a] anthracene	12.3	93.3	96.5	84%	102	96.5	93%
chrysene	9.62	84.9	96.5	78%	91.4	96.5	85%
benzo[b] fluoranthene	11.8	96.4	96.5	88%	104	96.5	96%
benzo[k] fluoranthene	11.3	91.5	96.5	83%	97.2	96.5	89%
benzo[a] pyrene	10.3	82.9	96.5	75%	93.0	96.5	86%
indeno [1,2,3-c,d] pyrene	6.90	67.3	96.5	63%	71.9	96.5	67%
dibenzo [a,h] anthracene	8.32	67.5	96.5	61%	72.8	96.5	67%
benzo [g,h,i] perylene	8.20	64.3	96.5	58%	71.0	96.5	65%

#### **SURROGATE RECOVERIES (%)**

d8 naphthalene	54.6	55.4	47.5
d10 Acenaphthene	67.0	59.5	52.6
d10 phenanthrene	51.1	48.9	46.9
d12 chrysene	107	84.6	80.3
d12 perylene	66.2	73.2	62.6
d14 dibenzo[a,h] anthracene	80.9	86.0	77.2

U Not detected at or above DL shown

(1) Concentrations is the sum of chrysens and triphenylene

(2) Concentrations is the sum of benzo [b] fluoranthene and benzo[j]fluoranthene

(3) Concentration is the sum of of dibenz(a,c)anthracene and dibenz(a,h)anthracene

@ Outside RPD limits of  $\pm 30\%$

# Outside Surrogate Recovery limits of 40-120%

& Outside SRM recovery limits of 70-130%

SL Inappropriate spike level

**BATTELLE MARINE SCIENCES LABORATORY**  
 1529 West Sequim Bay Road  
 Sequim, WA 98382-9099  
 360/681-3643

**HECKATHORN**  
 PAHs in Sediment  
 Samples Received 11/6/98

MSL Code	DUP		RPD	SRM		Percent Recovery
	1286-3	1286-3		1941a	cert value range	
Sponsor ID	LC-2	LC-2				
Matrix	Sed	Sed		Sed		
Wet Wt (g)	10.0	10.1		2.49		
Percent Dry Wt	65.8	65.8		100		
Extraction Date	12/9/98	12/9/98		12/9/98		
Analytical Batch	1	1		1		
Unit (dry wt)	ng/g	ng/g	%	ng/g	ng/g	%
naphthalene	112	105	6%	1050	1010	±140 104%
1 methyl naphthalene	48.3	37.5	25%	238	NA	NA NA
Acenaphthalene	212	191	10%	138	NA	NA NA
Acenaphthene	73.3	65.5	11%	66.4	NA	NA NA
Fluorene	162	139	15%	87.4	97.3	±8.6 90%
phenanthrene	676	518	26%	499	489	±23 102%
anthracene	696	695	0%	229	184	±14 124%
fluoranthene	2140	2390	11%	958	981	±78 98%
pyrene	1340	1520	13%	728	811	±24 90%
benzo[a] anthracene	1150	1190	3%	494	427	±25 116%
chrysene	1560	1560	0%	623 <sup>(1)</sup>	380	±24 164% &
benzo[b] fluoranthene	1740	1600	8%	1170 <sup>(2)</sup>	740	±110 158% &
benzo[k] fluoranthene	626	593	5%	393	361	±18 109%
benzo[a] pyrene	1080	995	8%	542	628	±52 86%
indeno [1,2,3-c,d] pyrene	396	387	2%	422	501	±58 84%
dibenzo [a,h] anthracene	124	119	4%	104 <sup>(3)</sup>	73.9	±9.7 141% &
benzo [g,h,i] perylene	338	330	2%	392	525	±67 75%

#### SURROGATE RECOVERIES (%)

d8 naphthalene	26.5 #	36.4 #	20.7 #
d10 Acenaphthene	39.7 #	56.9	32.2 #
d10 phenanthrene	59.6	76.0	51.2
d12 chrysene	78.6	96.4	70.7
d12 perylene	77.2	95.5	64.9
d14 dibenzo[a,h] anthracene	92.8	116	78.9

U Not detected at or above DL shown

(1) Concentrations is the sum of chrysens and triphenylene

(2) Concentrations is the sum of benzo [b] fluoranthene and benzo[j]fluoranthene

(3) Concentration is the sum of of dibenz(a,c)anthracene and dibenz(a,h)anthracene

@ Outside RPD limits of ±30%

# Outside Surrogate Recovery limits of 40-120%

& Outside SRM recovery limits of 70-130%

SL Inappropriate spike level

**BATTELLE MARINE SCIENCES LABORATORY**

1529 West Sequim Bay Road

Sequim, WA 98382-9099

360/681-3643

**HECKATHORN**

Pesticides in Tissue

Samples Received 1/9/98

MSL Code	MSA		Spike Amount	Percent Recovery	MSB		Percent Recovery	RPD
	1286-3	1286-3			1286-3	Spike		
Sponsor ID	LC-2	Spike A			Spike B	Amount		
Matrix	Sed	Sed			Sed			
Wet Wt (g)	10.0	10.3			10.6			
Percent Dry Wt	65.8	65.8			65.8			
Extraction Date	12/9/98	12/9/98			12/9/98			
Analytical Batch	1	1			1			
Unit (dry wt)	ng/g	ng/g	ng/g	%	ng/g	ng/g	%	%
naphthalene	112	184	74.0	97%	203	74.0	123%	23%
1 methyl naphthalene	48.3	39.7	NS	NA	37.9	NS	NA	NA
Acenaphthalene	212	230	74.0	24% # SL	328	74.0	157% # SL	NA
Acenaphthene	73.3	155	74.0	110%	139	74.0	89%	22%
Fluorene	162	227	74.0	88%	241	74.0	107%	19%
phenanthrene	676	607	74.0	-93% # SL	613	74.0	-85% # SL	NA
anthracene	696	649	74.0	-64% # SL	1210	74.0	695% # SL	NA
fluoranthene	2140	2090	74.0	-68% # SL	2030	74.0	-149% # SL	NA
pyrene	1340	1350	74.0	14% # SL	1320	74.0	-27% # SL	NA
benzo[a] anthracene	1150	1090	74.0	-81% # SL	1360	74.0	284% # SL	NA
chrysene	1560	1400	74.0	-216% # SL	2230	74.0	905% # SL	NA
benzo[b] fluoranthene	1740	1490	74.0	-338% # SL	2110	74.0	500% # SL	NA
benzo[k] fluoranthene	626	592	74.0	-46% # SL	801	74.0	236% # SL	NA
benzo[a] pyrene	1080	956	74.0	-168% # SL	1390	74.0	419% # SL	NA
indeno [1,2,3-c,d] pyrene	396	405	74.0	12% # SL	554	74.0	214% # SL	NA
dibenzo [a,h] anthracene	124	164	74.0	54%	211	74.0	118%	74% @
benzo [g,h,i] perylene	338	348	74.0	14% # SL	482	74.0	195% # SL	NA

**SURROGATE RECOVERIES (%)**

d8 naphthalene	26.5 #	33.3 #	32.1 #
d10 Acenaphthene	39.7 #	48.5	46.1
d10 phenanthrene	59.6	68.7	70.4
d12 chrysene	78.6	81.7	103
d12 perylene	77.2	80.7	89.1
d14 dibenzo[a,h] anthracene	92.8	97.6	110

U Not detected at or above DL shown

(1) Concentrations is the sum of chrysens and triphenylene

(2) Concentrations is the sum of benzo [b] fluoranthene and benzo[j]fluoranthene

(3) Concentration is the sum of of dibenz(a,c)anthracene and dibenz(a,h)anthracene

@ Outside RPD limits of  $\pm 30\%$ 

# Outside Surrogate Recovery limits of 40-120%

&amp; Outside SRM recovery limits of 70-130%

SL Inappropriate spike level

**BATTELLE MARINE SCIENCES LABORATORY**  
 1529 West Sequim Bay Road  
 Sequim, WA 98382-9099  
 360/681-3643

**HECKATHORN**  
 Pesticides in Sediment  
 Samples Received 11/6/98

MSL Code	1286-1	1286-2	1286-3	1286-4
Sponsor ID	LC-4	LC-3	LC-2	LC-1
Matrix	Sed	Sed	Sed	Sed
Wet Wt (g)	10.2	10.1	10.0	10.1
Percent Dry Wt	38.4	34.7	65.8	19.5
Extraction Date	2/3/99	2/3/99	2/3/99	2/3/99
Dilution	1x	2x	5x	10x
Analytical Batch	1	1	1	1
Unit (dry wt)	ng/g	ng/g	ng/g	ng/g
A-BHC	25.8 U	55.9 U	60.6 U	204 U
B-BHC	25.8 U	55.9 U	60.7 U	204 U
G-BHC	15.5 U	33.7 U	36.5 U	122 U
D-BHC	25.8 U	55.9 U	60.7 U	204 U
Heptachlor	9.73 U	21.1 U	40.0	77.0 U
Aldrin	15.8 U	43.1	60.5	790
Heptachlor Epoxide	31.6 U	68.4 U	74.2 U	250 U
g-Chlordane	25.8 U	55.9 U	60.7 U	1660
Endosulfan I	25.8 U	55.9 U	60.7 U	3240
α-Chlordane	8.18 U	17.7 U	59.5	1000
Dieldrin	51.5	171	382	3270
4,4'-DDE	93.8	323	383	84400
Endrin	25.8 U	55.9 U	507	671
Endosulfan II	25.8 U	55.9 U	60.7 U	204 U
4,4'-DDD	1190	4080	3150	15700
Endrin Aldy.	25.8 U	55.9 U	60.7 U	204 U
Endosulfan Sulfate	25.8 U	55.9 U	60.7 U	204 U
4,4'-DDT	1450	5850	10400	30100
Toxaphene	8.11 U	9.06 U	4.79 U	16.1 U
<b><u>AROCLORS</u></b>				
1242	8.11 U	9.06 U	4.79 U	16.1 U
1248	8.11 U	9.06 U	4.79 U	16.1 U
1254	89.9	150	245	981
1260	8.11 U	9.06 U	4.79 U	16.1 U
<b><u>SURROGATE RECOVERIES (%)</u></b>				
PCB103	95.0	85.5	85.0	83.3
PCB198	75.2	68.1	69.6	64.3

M Mean used to calculate QC  
 U Not detected at or above DL shown  
 ND Analyte not detected



**BATTELLE MARINE SCIENCES LABORATORY**  
 1529 West Sequim Bay Road  
 Sequim, WA 98382-9099  
 360/681-3643

**HECKATHORN**  
 Pesticides in Sediment  
 Samples Received 11/6/98

MSL Code Sponsor ID	BSA				BSB		
	Blank	Blank Spike A	Spike Amount	Percent Recovery	Blank Spike B	Spike Amount	Percent Recovery
Matrix	Sed	Sed			Sed		
Wet Wt (g)	NA	NA			NA		
Percent Dry Wt	NA	NA			NA		
Extraction Date	2/3/99	2/3/99			2/3/99		
Dilution	1x	5x			5x		
Analytical Batch	1	1			1		
Unit (dry wt)	ng/g	ng/g	ng/g	%	ng/g	ng/g	%
A-BHC	66.7 U	66.7 U	NS	NA	66.7 U	NS	NA
B-BHC	66.7 U	66.7 U	NS	NA	66.7 U	NS	NA
G-BHC	40.2 U	3010	4170	72%	3170	4170	76%
D-BHC	66.7 U	66.7 U	NS	NA	66.7 U	NS	NA
Heptachlor	25.2 U	2740	4170	66%	3030	4170	73%
Aldrin	30.3 U	3270	4170	78%	3480	4170	83%
Heptachlor Epoxide	81.7 U	81.7 U	NS	NA	81.7 U	NS	NA
g-Chlordane	66.7 U	66.7 U	NS	NA	66.7 U	NS	NA
Endosulfan I	66.7 U	66.7 U	NS	NA	66.7 U	NS	NA
a-Chlordane	21.2 U	21.2 U	NS	NA	21.2 U	NS	NA
Dieldrin	53.0 U	7270	8330	87%	8010	8330	96%
4,4'-DDE	23.2 U	23.2 U	NS	NA	23.2 U	NS	NA
Endrin	66.7 U	8130	8330	98%	8770	8330	105%
Endosulfan II	66.7 U	66.7 U	NS	NA	66.7 U	NS	NA
4,4'-DDD	67.3 U	357	NS	NA	381	NS	NA
Endrin Aldy.	66.7 U	226	NS	NA	228	NS	NA
Endosulfan Sulfate	66.7 U	66.7 U	NS	NA	66.7 U	NS	NA
4,4'-DDT	59.3 U	7720	8330	93%	7900	8330	95%
Toxaphene	23.3 U	23.3 U	NS	NA	23.3 U	NS	NA

**AROCLORS**

1242	23.3 U	23.3 U	NS	NA	23.3 U	NS	NA
1248	23.3 U	23.3 U	NS	NA	23.3 U	NS	NA
1254	23.3 U	171	250	68%	23.3 U	NS	NA
1260	23.3 U	23.3 U	NS	NA	23.3 U	NS	NA

**SURROGATE RECOVERIES (%)**

PCB103	75.8	88.1			89.5		
PCB198	61.5	70.4			70.8		

M Mean used to calculate QC  
 U Not detected at or above DL shown  
 ND Analyte not detected  
 @ Outside RPD limits of  $\pm 30\%$

**BATTELLE MARINE SCIENCES**  
 1529 West Sequim Bay Road  
 Sequim, WA 98382-9099  
 360/681-3643

**HECKATHORN**  
 Pesticides in Sediment  
 Samples Received 11/6/98

MSL Code	DUP		RPD	SRM		Percent Recovery
	1286-3	1286-3		1941a	certified value	
Sponsor ID	LC-2					
Matrix	Sed	Sed		Sed		
Wet Wt (g)	10.0	10.1		2.49		
Percent Dry Wt	65.8	65.8		100		
Extraction Date	2/3/99	2/3/99		2/3/99		
Dilution	5x	2x		1x		
Analytical Batch	1	1		1		
Unit (dry wt)	ng/g	ng/g	%	ng/g	ng/g	%
A-BHC	60.6 U	60.2 U	NA	0.40 U	NA	
B-BHC	60.7 U	60.3 U	NA	0.40 U	NA	
G-BHC	36.5 U	36.3 U	NA	0.24 U	NA	
D-BHC	60.7 U	60.3 U	NA	0.40 U	NA	
Heptachlor	40.0	22.7 U	NA	0.29 U	NA	
Aldrin	60.5	37.8	46% @	0.18 U	NA	
Heptachlor Epoxide	74.2 U	73.8 U	NA	0.49 U	NA	
g-Chlordane	60.7 U	60.3 U	NA	0.40 U	NA	
Endosulfan I	60.7 U	60.3 U	NA	0.40 U	NA	
a-Chlordane	59.5	25.0	82% @	1.74	2.33	25%
Dieldrin	382	305	22%	0.69 U	NA	
4,4'-DDE	383	393	3%	5.00	6.59	24%
Endrin	507	60.3 U	NA	0.40 U	NA	
Endosulfan II	60.7 U	60.3 U	NA	0.40 U	NA	
4,4'-DDD	3150	5780	59% @	6.01	5.06	19%
Endrin Aldy.	60.7 U	85.2	NA	0.40 U	NA	
Endosulfan Sulfate	60.7 U	60.3 U	NA	0.78 U	NA	
4,4'-DDT	10400	5550	61% @	680	NA	
Toxaphene	4.79 U	4.74 U	NA	12.7 U	NA	

**AROCLORS**

1242	4.79 U	4.74 U	NA	12.7 U	
1248	4.79 U	4.74 U	NA	12.7 U	
1254	245	262	7%	12.7 U	
1260	4.79 U	4.74 U	NA	12.7 U	

**SURROGATE RECOVERIES (%)**

PCB103	85.0	80.9	
PCB198	69.6	62.0	

M Mean used to calculate QC  
 U Not detected at or above DL  
 ND Analyte not detected  
 @ Outside RPD limits of  $\pm 30\%$

**BATTELLE MARINE SCIENCES**  
 1529 West Sequim Bay Road  
 Sequim, WA 98382-9099  
 360/681-3643

**HECKATHORN**  
 Pesticides in Sediment  
 Samples Received 11/6/98

MSL Code Sponsor ID	MSA				MSB			
	1286-3 LC-2	1286-3 Spike A	Spike Amount	Percent Recovery	Spike B	Spike Amount	Percent Recovery	RPD
Matrix	Sed	Sed			Sed			
Wet Wt (g)	10.0	10.3			10.6			
Percent Dry Wt	65.8	65.8			65.8			
Extraction Date	2/3/99	2/3/99			2/3/99			
Dilution	5x	5x			5x			
Analytical Batch	1	1			1			
Unit (dry wt)	ng/g	ng/g	ng/g	%	ng/g	ng/g	%	%
A-BHC	60.6 U	59.9 U	NS	NA	58.7 U	NS	NA	
B-BHC	60.7 U	59.9 U	NS	NA	58.7 U	NS	NA	
G-BHC	36.5 U	2830	3740	76%	2870	3670	78%	3%
D-BHC	60.7 U	59.9 U	NS	NA	58.7 U	NS	NA	
Heptachlor	40.0	2830	3740	75%	2780	3670	75%	0%
Aldrin	49.2 M	2910	3740	76%	2800	3670	75%	2%
Heptachlor Epoxide	74.2 U	73.4 U	NS	NA	71.9 U	NS	NA	
g-Chlordane	60.7 U	59.9 U	NS	NA	58.7 U	NS	NA	
Endosulfan I	60.7 U	59.9 U	NS	NA	58.7 U	NS	NA	
a-Chlordane	59.5	19.0 U	NS	NA	18.6 U	NS	NA	
Dieldrin	344 M	6630	7490	84%	6590	7330	85%	2%
4,4'-DDE	383	453	NS	NA	442	NS	NA	
Endrin	507	7320	7490	91%	7240	7330	92%	1%
Endosulfan II	60.7 U	59.9 U	NS	NA	58.7 U	NS	NA	
4,4'-DDD	3150	3730	NS	NA	4120	NS	NA	
Endrin Aldy.	60.7 U	59.9 U	NS	NA	58.7 U	NS	NA	
Endosulfan Sulfate	60.7 U	59.9 U	NS	NA	58.7 U	NS	NA	
4,4'-DDT	7975 M	12100	7490	55%	12800	7330	66%	18%
Toxaphene	4.79 U	4.69 U	NS	NA	4.55 U	NS	NA	
AROCLORS								
1242	4.79 U	4.69 U	NS	NA	4.55 U	NS	NA	
1248	4.79 U	4.69 U	NS	NA	4.55 U	NS	NA	
1254	254 M	385	148	89%	399	144	101%	13%
1260	4.79 U	4.69 U	NS	NA	4.55 U	NS	NA	
SURROGATE RECOVERIES (%)								
PCB103	85.0	88.7			88.7			
PCB198	69.6	67.7			68.8			

M Mean used to calculate QC  
 U Not detected at or above DL  
 ND Analyte not detected  
 @ Outside RPD limits of  $\pm 30\%$

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