PNNL-13632
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Year 4 Post-Remediation Biomonitoring of Pesticides and Other Contaminants in Marine Waters Near the United Heckathorn Superfund Site, Richmond, California

N.P. Kohn
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Sequim, Washington

December 2001

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

Pacific Northwest National Laboratory
Richland, Washington

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SUMMARY

Marine sediment remediation at the United Heckathorn Superfund Site in Richmond, California, was completed in April 1997. During January 2001, in Year 4 of post-remediation monitoring of marine areas near the United Heckathorn Site, water and mussel tissues were collected from four stations in and near Lauritzen Channel. Dieldrin and dichlorodiphenyl trichloroethane (DDT) were analyzed in water samples and in tissue samples from resident (i.e., naturally occurring) mussels. As in Year 3, no mussels were transplanted to the study area in Year 4. Year 4 concentrations of dieldrin and total DDT in water and total DDT in tissue were compared with those from Years 1, 2, and 3 of post-remediation monitoring (Antrim and Kohn 2000a,b[1], Kohn and Kropp 2000), and with preremediation data from the California State Mussel Watch Program (Rasmussen 1995) and the Ecological Risk Assessment for the United Heckathorn Superfund Site (Lee et al., 1994). Year 4 water samples and mussel tissues were also analyzed for polychlorinated biphenyls (PCB), which were detected in sediment samples during Year 2 monitoring. Contaminants of concern in Year 4 water samples were analyzed in both bulk (total) phase and dissolved phase, as were total suspended solids, to evaluate the contribution of particulates to the total contaminant concentration. This addition to the monitoring program was made because in previous years, suspended sediment was observed during water sampling and resulting replicate water concentrations were highly variable.

Mean chlorinated pesticide concentrations in some Year 4 water samples were lower than Year 3 levels, yet did not meet remediation goals. The exception was Richmond Inner Harbor Channel (Station 303.1), where concentrations of chlorinated pesticides in water were below the method detection limit (MDL). Mean total DDT concentrations in the total fraction of water samples collected at the other three stations ranged from 2.5 ng/L to 142.2 ng/L, exceeding the remediation goal (0.59 ng/L). Dieldrin concentrations in Year 4 water samples collected from Richmond Inner Harbor Channel were below the MDL. Mean dieldrin concentrations in the total fraction of water samples collected from the other three stations ranged from 0.46 ng/L to 8.49 ng/L, exceeding the remediation goal (0.14 ng/L). The highest concentrations of total DDT and dieldrin pesticides were found at Lauritzen Channel/End (Station 303.3). PCB Aroclor 1254 concentrations were below the MDL for all replicates collected from all four stations.

Tissue analyses indicated that the bioavailability of total DDT in Year 4 was generally similar to preremediation levels in the study area. Total DDT concentrations in mussel tissues measured in Year 4

1 Reports for Years 1 and 2 of post-remediation monitoring were revised and republished in July 2000, after discovery of a reporting unit error in the original documents published in 1998 and 1999. Revised documents were distributed to all names on the original distribution list; they are also available on the web by searching for "Heckathorn" at http://www.pnl.gov/main/publications.

were slightly higher than Year 3 values at all stations except Richmond Inner Harbor Channel. Total DDT (wet weight) concentrations were lower than preremediation levels at all three stations for which preremediation data were collected. Dieldrin concentrations measured in Year 4 were lower than Year 3 values at all stations. Year 4 dieldrin concentrations were lower than preremediation levels at those stations for which preremediation levels were determined. Mean chlorinated pesticide concentrations measured in Year 4 were highest in tissues from Lauritzen Channel/End (1,136 µg/kg total DDT and 32.1 µg/kg dieldrin wet weight), whereas the lowest mean total DDT and dieldrin levels were from tissues collected from Richmond Inner Harbor Channel (25 µg/kg and 0.7 µg/kg wet weight, respectively). Aroclor 1254 concentrations measured in tissue collected in Year 4 were much lower than Year 3 values at all stations except Lauritzen Channel/End where the values were similar. Aroclor 1254 concentration in mussel tissue collected in Year 4 was highest at Lauritzen Channel/End (158 µg/kg wet weight) and lowest at Richmond Inner Harbor Channel (53 µg/kg wet weight).

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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 U.S. Environmental Protection Agency (EPA) listed the site 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 dichlorodiphenyl trichloroethane (DDT) contamination measured during the California State Mussel Watch program (Rasmussen 1995). A remediation investigation of adjacent marine areas revealed widespread contamination of sediment by pesticides, particularly 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 Lauritzen Channel 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 sediment. This assessment revealed that DDT and dieldrin contamination originating from the United Heckathorn Site had been actively transported to offsite areas via surface waters.

Major components of the final remediation actions at the Heckathorn Site outlined in the Record of Decision (ROD 1996) are

- dredging of all younger bay mud from Lauritzen Channel and Parr Canal, with offsite disposal of the dredged material
- placement of clean sand after dredging
- construction of a cap around the former Heckathorn facility to prevent erosion
- enactment of 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 remediation.

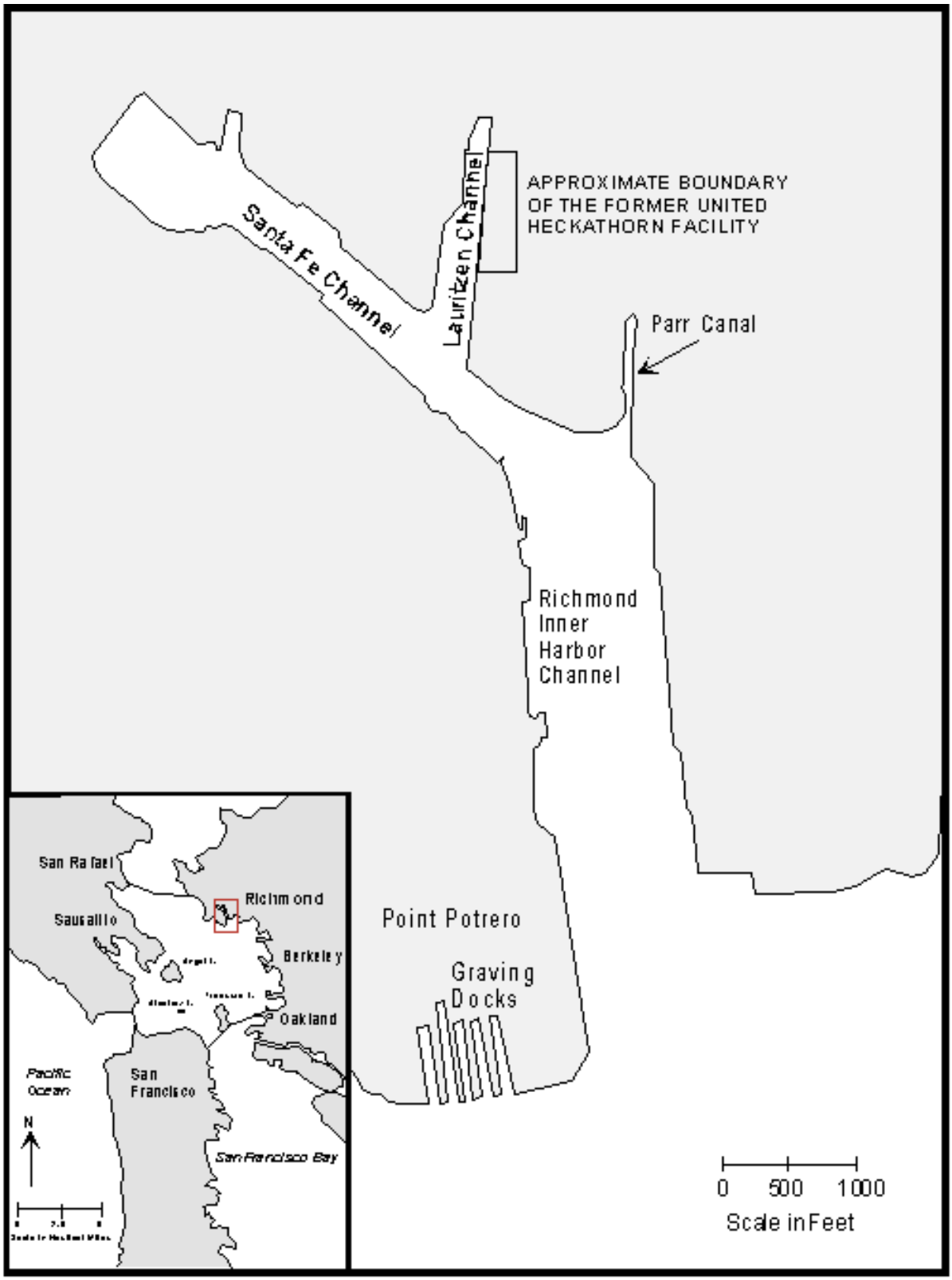

Figure 1.1. Location of the United Heckathorn Superfund Site, Richmond, California.

Remediation levels protective of the environment and human health were established to provide benchmarks for determining the effectiveness of the remediation 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 remediation actions. EPA marine chronic and human health water quality criteria were identified as ARARs for surface water. Human health standards based on consumption of contaminated fish were used to establish remediation goals because they are lower than marine chronic criteria. No chemical-specific ARARs were identified as remediation 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 core sampling was conducted to verify that the younger-bay (contaminated) mud was removed and that only older-bay (less contaminated) mud remained. EPA collected post-remediation samples of the remaining older-bay mud, and analyses determined the average concentration of DDT to be 263 µg/kg dry weight (Lincoff 1997), below the remediation goal of 590 µg/kg DDT dry weight specified in the ROD. In April 1997, 9100 cubic yards of clean sand were placed in Lauritzen Channel to improve the older-bay mud surface for colonization by benthic invertebrates. The volume of sand was equivalent to an average depth of 1 ft over the dredged area, although the exact layer thickness undoubtedly varied because of the uneven, sloping channel bottom. Since remediation and sand placement in 1997, Lauritzen Channel has returned to industrial use by Levin Richmond Terminals and Manson Construction, resulting in frequent vessel traffic throughout the channel.

The purpose of the marine monitoring study is to document the expected reduction in flux of contaminants from the United Heckathorn Superfund Site following EPA response actions. The measurement endpoints for this long-term monitoring are mussels and surface waters. The remediation levels for waters set forth in the ROD are 0.59 ng/L for total DDT (the sum of the 4,4'- and 2,4'-isomers of DDT, DDD, and DDE) and 0.14 ng/L for dieldrin.

Year 1 of post-remediation biomonitoring was conducted 6 months after remediation (Antrim and Kohn 2000a). Year 1 biomonitoring showed that pesticide concentrations in the tissues of mussels exposed at the site were higher than those observed before remediation. Year 2 monitoring, conducted about 18 months after remediation, showed tissue levels that were much reduced from Year 1 and that only exceeded preremediation levels at Richmond Inner Harbor Channel (Antrim and Kohn 2000b). During both years, the concentrations were higher at Lauritzen Channel stations than at the Richmond Inner Harbor Channel or Santa Fe Channel stations. These results suggested that DDT was still present and bioavailable in Lauritzen Channel, especially near its head.

This report focuses on the Year 4 (2001) post-remediation biomonitoring results. Year 4 biomonitoring repeated the water and resident mussel tissue sampling and analyses of Years 1, 2, and 3 (1997–2000). As in Year 3, EPA decided not to measure transplanted mussels for post-remediation monitoring in Year 4 (Appendix A). Year 4 results are compared with water and tissue pesticide data from two preremediation studies (Lee et al., 1994; Rasmussen 1995) and the Years 1, 2, and 3 monitoring studies (Antrim and Kohn 2000a, 2000b; Kohn and Kropp 2000). Comparisons with Years 1 and 2 were done using the revised data for those years, published in 2000; the reports published in 1998 and 1999 reported tissue data with incorrect units (dry weight instead of wet weight) and therefore required correction. Corrected copies of the Year 1 and Year 2 monitoring reports are available on the web at http://www.pnl.gov/main/publications. Mussel tissue samples were collected and analyzed in both preremediation studies, but water samples were analyzed only for the ecological risk assessment (Lee et al., 1994). The four post-remediation water and tissue-monitoring stations are the same as those of the State Mussel Watch Program in the project area.

2.0 METHODS

Detailed methods for the collection, processing, and analysis of tissue and water samples in Year 4 were outlined in the Field Sampling and Analysis Plan (Battelle 1997). Methods were the same as those used in previous years of post-remediation monitoring, with the exception of adding total suspended solids and dissolved contaminants to the water analyses. A brief review of methods is provided here. All procedures for sampling, sample custody, field and lab documentation, other aspects of documentation, quality assurance, and sample analysis were consistent with the more general procedures described in the Quality Assurance Project Plan (QAPP) for Remediation Investigation and Feasibility Study of Marine Sediments at the United Heckathorn Superfund Site (Battelle 1992). All samples were collected by EPA and analyzed at Battelle Marine Sciences Laboratory (MSL).

The four post-remediation monitoring stations selected are those stations in the project area that were sampled during 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 Channel/End Station (Mussel Watch Station 303.3) corresponds to the Ecological Risk Assessment-Lauritzen Channel 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 Channel (Mussel Watch Station 303.2, Lauritzen Channel/Mouth). A more detailed description of sampling stations for the Year 3 biomonitoring is provided in Table 2.1 and in the Field Sampling Summary and Field Sampling Report memorandum (Appendix A).

2.1 TISSUE AND WATER SAMPLE COLLECTION

Approximately 45 resident blue mussels (*Mytilus edulis*) were collected from each of the four stations on January 17, 2001 (Figure 2.1). Resident mussels could have been one of several subspecies or hybrids in the *M. edulis* complex that cannot easily be distinguished by the shells alone (Harbo 1997). The coordinates presented in Table 2.1 for each station were determined in 1998 by using a Global Positioning System (GPS) with differential correction. In Year 4, stations were revisited by using the visual landmarks listed in Table 2.1.

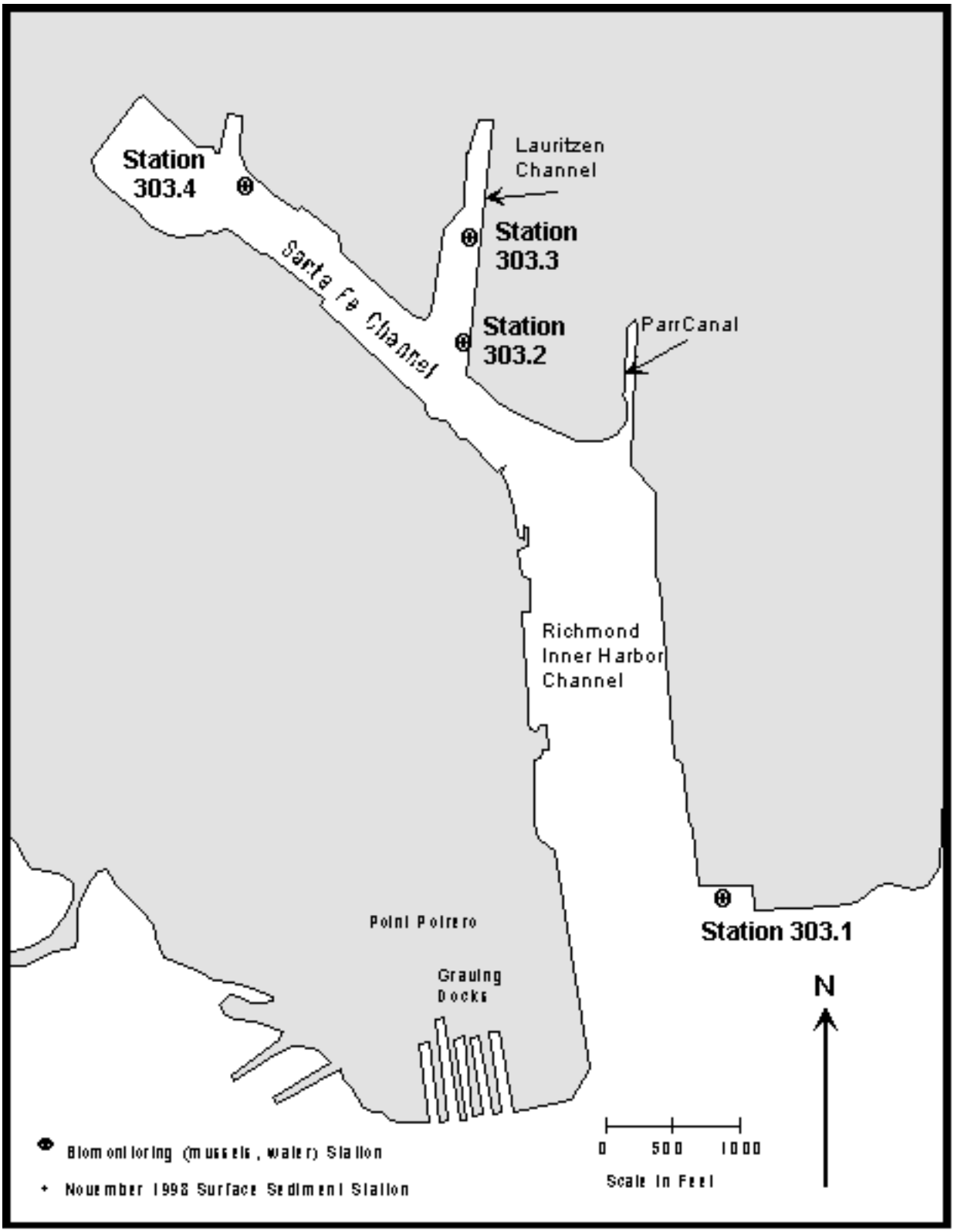

Figure 2.1. Sampling stations for long-term post-remediation monitoring of the United Heckathorn Site.

Table 2.1. Sampling Stations for Year 4 Post-remediation Monitoring (2000-2001) of the United Heckathorn Site

Station Number	Station Name	Location[(a)]	Remarks
303.1	Richmond Inner Harbor Channel	37º54' 32.8" N 122º21' 34.5" W	On western most wooden dolphin, near abandoned Ford automotive plant, southeast of public fishing pier. Blind duplicate seawater sample labeled 303.5.
303.2	Lauritzen Channel/Mouth (South)	37º55' 12.6" N 122º22' 01.2" 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 Channel/End (North)	37º55' 22.5" N 122º21' 59.9" W	On east side of canal, southern end of small wooden pier that extends out into the channel. Collected extra water for quality control (matrix spike and matrix spike duplicate)
303.4	Santa Fe Channel/End	37º55' 21.53" N 122º21' 18.37" W	At northwest corner of floating boat shed, east of small boat fuel dock

[(a)] Data from January 6, 1998.

Mussels were collected near the surface of the water, at about mean lower low water (MLLW) at Richmond Inner Harbor Channel (Station 303.1), and at -0.4 ft MLLW at Lauritzen Channel/Mouth and Lauritzen Channel/End (Stations 303.2 and 303.3, respectively). At Santa Fe Channel/End (Station 303.4), mussels were collected near the surface from a floating dock. Thus, mussels at the Santa Fe Channel/End station were collected at a fixed depth relative to the water surface. Weather at the time of collection was sunny and calm. Ambient water temperature was 10ºC to 12ºC.

Mussels were cleaned gently in the field to remove external growth and packaged whole in ashed foil and plastic bags. Mussels were frozen at -20ºC, shipped to the analytical laboratory in coolers, and held at -20ºC until they were prepared for analysis. To prepare tissue samples, mussels were partially thawed, the valve or shell length was measured, and byssal threads were cut from the tissue. Sand and mud on the soft tissue were rinsed off with deionized water and soft tissues were transferred to a sample jar. Each tissue sample consisted of from 42 to 46 mussels. The total wet weight of each tissue sample was recorded. Tissue samples were refrozen and stored at -20ºC until extracted.

On January 17, 2001, surface water samples were collected approximately 1 ft (0.3 m) below the water surface. To collect a sample, a bottle was submerged, the cap was removed underwater to allow water in, and the cap replaced before the bottle was lifted from the water. At each station, three 3.8-L (1 gal) water samples were collected for analysis. Additional water samples were collected for quality control (QC) analyses (i.e., matrix spike, matrix spike duplicate [MS/MSD], and blind duplicate samples) (Table 2.1). Water samples were chilled to and held at 4ºC until extracted.

2.2 TISSUE AND WATER SAMPLE ANALYSIS

Chemical analyses followed methods described in the QAPP (Battelle 1992). The water samples collected on January 17, 2001, were split upon receipt for total suspended solids, total pesticide, and dissolved pesticide analysis. Total suspended solids were analyzed in bulk water samples according to Standard Method 2540-D, Solids (APHA 1998) on January 23, 2001. To create the water sample for dissolved pesticide analysis, an aliquot of the bulk water sample was filtered through a 0.45-µm glass fiber filter. Bulk and filtered water samples (for total and dissolved pesticides) were extracted on January 20 through January 23, 2001, and analyzed for chlorinated pesticides and polychlorinated biphenyl (PCB) aroclors January 22 through January 27, 2001, within acceptable holding times. Sample-specific detection limits (Appendix B) were calculated using the sample volume and achieved detection limits for water samples determined in a previous study at MSL.

The mussel tissue samples collected on January 17, 2001, were extracted February 5, 2001, and analyzed for chlorinated pesticides and PCB aroclors on February 12, 2001, within acceptable holding times. Tissue samples were also analyzed for percentage of lipids. Sample-specific detection limits (Appendix B) were calculated using the sample weight and an achieved detection limits for tissue samples determined in a previous study at MSL. 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), following the methods used in the California State Mussel Watch Program (Rasmussen 1995) and in the Ecological Risk Assessment of Marine Sediments at the United Heckathorn Superfund Site (Lee et al. 1994). Undetected analytes were not included in the total DDT calculation.

3.0 RESULTS AND DISCUSSION

This section presents the results of physical measurements to assess the size and condition of the resident mussels, and the results of chemical analyses of the water and mussel tissue samples. All extractions and analyses were conducted within the target holding times specified in the QAPP. Complete chemistry data tables, including associated QC data, are provided in Appendix B. In the following discussion, the Year 4 water data are compared with preremediation data from the Ecological Risk Assessment (Lee et al., 1994), post-remediation data from 1998, 1999, and 2000 (Antrim and Kohn 2000a, 2000b; Kohn and Kropp 2000), and the remediation goals for the site. The Year 4 tissue data are compared with preremediation tissue concentrations from the California State Mussel Watch Program (Rasmussen 1995) and the Ecological Risk Assessment (Lee et al., 1994), and with postremediation data from 1998, 1999, and 2000 (Antrim and Kohn 2000a, 2000b; Kohn and Kropp 2000).

3.1 MUSSEL SIZE AND CONDITION

Raw data for shell-length measurements and mean wet weight per mussel are provided in Appendix C. Only resident (i.e., naturally-occurring) mussels were analyzed. Mussels collected for tissue samples ranged from 4.1 cm to 7.2 cm in shell length (Table 3.1). Shell lengths of 5 mussels (~3% of the total) were not within the preferred size range of 4.0 cm to 6.5 cm, which is a combination of the preference ranges cited by Rasmussen (1995) and Lee et al. (1994). There were no differences in the mean shell length among stations (Table 3.1). The grand mean shell length (all stations) was 5.32 cm (standard deviation 0.06) in Year 4, which is similar to the mean shell length of resident mussels analyzed in previous monitoring years (5.61 cm, 5.28 cm, and 5.34 cm in Years 1, 2, and 3, respectively). The station mean wet weight per mussel, which was calculated as the total wet weight of the station tissue sample divided by the number of individuals per sample, ranged from 4.5 g to 7.1 g (Table 3 1). The overall mean wet weight per mussel (calculated as the mean of the station means) was 5.75 g (standard deviation 1.10).

Lipid content of resident mussels ranged from 6.7% to 8.0% dry weight (Table 3.1; grand mean of 7.6%; standard deviation of 0.61%). Note 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. However, because nonpolar organic contaminants tend to accumulate in fatty tissues, normalizing contaminant data to tissue lipid content permits more equitable comparisons among samples to be made.

Table 3.1. Summary of Length and Weight Data from Mussels Collected for Tissue Samples in January 2001 for Post-remediation Monitoring of the United Heckathorn Superfund Site

	Station			
	303.1 Richmond Inner Harbor Channel	303.2 Lauritzen Channel/Mouth	303.3 Lauritzen Channel/End	303.4 Santa Fe Channel/End
Shell Length (cm)				
n	46	48	46	46
min	4.11	4.35	4.09	4.09
max	6.55	6.59	6.45	7.21
mean	5.32	5.40	5.29	5.28
standard deviation	0.68	0.58	0.55	0.65
n outside range[a]	2	2	0	1
grand mean[b]	5.32			
standard deviation	0.06			
Tissue Wet Weight (g)				
sample weight	272.82	341.91	251.95	205.96
mean wt/mussel	5.93	7.12	5.48	4.48
grand mean	5.75			
standard deviation	1.10			
Lipid Content (% dry weight)				
	7.81	7.80	8.00	6.66
grand mean	7.57			
standard deviation	0.61			

(a) number of individuals outside preferred size range of 4.0-6.5 cm.
(b) mean of all stations combined.

3.2 WATER

The triplicate water samples that were collected at each site provide a snapshot of water-column concentrations of DDT compounds and dieldrin. Such samples provide no information about the temporal variability or vertical stratification of these contaminants in the water column, information that would be useful in the interpretation of the biomonitoring results. The inability to evaluate temporal or spatial variability of water chemistry should be considered when these data are compared with the results of earlier studies. The differences between two such sampling events do not necessarily verify trends; nor are individual samples necessarily representative of typical conditions.

In Year 4, a larger volume of water sample was collected from each monitoring station to evaluate dissolved pesticides and total suspended solids as well as total pesticides. In previous years, only total pesticides were measured in bulk water samples, and results were highly variable. Suspended particulates in the water column were considered to contribute to the variability in pesticide concentrations between replicate samples; hence, the modification to the program in Year 4 to evaluate suspended particulates and associated pesticides. Total pesticide and total suspended solids concentrations in water samples are provided in Table 3.2; dissolved pesticide concentrations in water samples are provided in Table 3.3.

Complete water chemistry and QC data are provided in Appendix B. In the method blank for the total fraction, all analytes were below the MDL. However, for the dissolved fraction, two analytes were detected in the method blank, 4,4'-DDD (0.36 ng/L) and 2,4'-DDT (0.82 ng/L). Associated sample concentrations that are less than five times the blank concentration are flagged with a "B" in Table 3.3. Recoveries of spiked surrogate compounds (PCB 103 and PCB 198) in Year 4 water samples ranged from 61% to 146%. Surrogate recoveries for seven replicates (Appendix B) were outside the target range (40% to 120%). Blank spike recoveries of dieldrin, 4,4'-DDT, and aroclor 1254 were within the target range (40% to 120%), except for 4,4'-DDT in one blank spike (129%). MS/MSD recoveries for dieldrin and Aroclor 1254 were within the target range (40% to 120%) in both the total and dissolved fraction MS/MSD samples. MS/MSD recoveries for 4,4'-DDT were also within the target range for the dissolved fraction; however, MS/MSD recoveries for 4,4'-DDT could not be calculated for the total fraction because the spiking levels were too low relative to the analyte concentration occurring in the field samples.

Total DDT concentrations in bulk water samples ranged from undetected at Richmond Inner Harbor Channel Station 303.1, to 294 ng/L in one of the replicates from Lauritzen Channel/End Station 303.3 (Table 3.2). Results were fairly consistent between replicates except at Station 303.3, at which all three replicates differed considerably, ranging from about 40 ng/L to 294 ng/L. The high variability in replicate samples at Station 303.3 indicates that contaminants could be inconsistently distributed in the water column, perhaps in association with organic or particulate materials. Total suspended solids were also variable at Station 303.3. When suspended solids were removed and only the dissolved fraction of pesticides analyzed, total DDT concentrations were much lower and very similar between replicates at Station 303.3, suggesting that DDT compounds were associated with the particles (Table 3.3). At the other stations, total DDT concentrations were much lower initially, and were not noticeably lower in the dissolved fraction (once particles were removed).

Table 3.2. Concentrations of DDT, Dieldrin, and Total Suspended Solids (TSS) in the Total Fraction of Water Samples Collected in January 2001 for Post-remediation Monitoring of the United Heckathorn Superfund Site

Station	Location	TSS (mg/L)	Concentration (ng/L) Dieldrin	2,4'-DDE	4,4'-DDE	2,4'-DDD	4,4'-DDD	2,4'-DDT	4,4'-DDT	Total DDT	Aroclor 1254
303.1 a	Richmond Inner Harbor Channel	0.003	0.06 U[a]	0.11 U	0.07 U	0.12 U	0.07 U	0.05 U	0.08 U	ND[b]	16.6 U
303.1 b		0.006	0.06 U	0.11 U	0.07 U	0.13 U	0.07 U	0.05 U	0.08 U	ND	17.0 U
303.1 c		0.000	0.06 U	0.12 U	0.07 U	0.13 U	0.07 U	0.06 U	0.08 U	ND	17.3 U
	Mean	0.003	NA[e]	NA	NA	NA	NA	NA	NA	NA	NA
	standard deviation	0.003	NA	NA	NA	NA	NA	NA	NA	NA	NA
303.2 a	Lauritzen Channel/ Mouth	0.000	0.48	0.11 U	0.12	0.42	1.10	0.05 U	1.07	2.71	16.0 U
303.2 b		0.001	0.51	0.11 U	0.06 U	0.36	1.16	0.05 U	1.20	2.72	16.0 U
303.2 c		0.001	0.40	0.11 U	0.48	0.54	1.23	0.05 U	0.96	3.21	16.3 U
	Mean	0.001	0.46	NA	0.30	0.44	1.16	NA	1.08	2.88	NA
	standard deviation	0.001	0.06	NA	0.13	0.09	0.07	NA	0.12	0.29	NA
303.3 a	Lauritzen Channel/ End	0.007	15.0	0.20	4.66	6.86	12.5	69.9	200 D[e]	294	16.3 U
303.3 b		0.006	6.09	0.08 U	1.37	2.82	6.37	8.84	72.9 D	92.3	16.6 U
303.3 c		0.001	4.38	0.09 U	1.15	2.35	5.36	5.61	25.5	40.0	13.6 U
	Mean	0.005	8.49	NA	2.39	4.01	8.08	28.1	99.5	142	NA
	standard deviation	0.003	5.70	NA	1.97	2.48	3.86	36.2	90.2	134	NA
303.4 a	Santa Fe Channel End	0.002	0.48	0.08 U	0.06	0.09 U	1.79	0.04 U	0.29	2.14	12.3 U
303.4 b		0.006	0.39	0.08 U	0.22	0.58	1.48	0.04	0.26	2.58	12.5 U
303.4 c		0.000	0.51	0.09 U	0.19	0.75	1.61	0.14	0.13	2.82	12.5 U
	Mean	0.003	0.46	NA	0.16	0.67	1.63	0.09	0.23	2.51	NA
	standard deviation	0.003	0.06	NA	0.09	0.12	0.16	0.07	0.09	0.34	NA

(a) U Not detected at or above concentration shown.
(b) ND None detected.
(c) Mean calculated using detected values only.
(d) NA Not applicable
(e) D Diluted 10 X

Table 3.3. Concentrations of DDT and Dieldrin in the Dissolved Fraction of Water Samples Collected in January 2001 for Post-remediation Monitoring of the United Heckathorn Superfund Site

		Concentration (ng/L)								
Station	Location	Dieldrin	2,4'-DDE	4,4'-DDE	2,4'-DDD	4,4'-DDD	2,4'-DDT	4,4'-DDT	Total DDT	Aroclor 1254
303.1 a	Richmond Inner Harbor Channel	0.04 U[a]	0.08 U	0.04 U	0.09 U	0.16 B[b]	0.04 U	0.05 U	0.16	11.5 U
303.1 b		0.04 U	0.07 U	0.04 U	0.08 U	0.04 U	0.03 U	0.05 U	ND[c]	10.3 U
303.1 c		0.34	0.07 U	0.04 U	0.08 U	0.15 B	0.03 U	0.05 U	0.49	10.3 U
	mean[d]	0.34	NA[e]	NA	NA	0.16	NA	NA	0.33	NA
	standard deviation	NA	NA	NA	NA	0.01	NA	NA	0.23	NA
303.2 a	Lauritzen Channel/ Mouth	0.40	0.07 U	0.32	0.46	1.20 B	0.04 U	0.61	2.59	11.0 U
303.2 b		0.56	0.07 U	0.30	0.39	1.07 B	0.18 B	0.62	2.56	11.1 U
303.2 c		0.42	0.08 U	0.38	0.48	1.08 B	0.22 B	0.41	2.57	11.4 U
	mean	0.46	NA	0.33	0.44	1.12	0.20	0.55	2.57	NA
	standard deviation	0.09	NA	0.04	0.05	0.07	0.03	0.12	0.02	NA
303.3 a (QC1)	Lauritzen Channel/ End	4.96	0.08 U	0.75	2.22	4.54	1.81 B	2.79	12.11	11.2 U
303.3 b (QC2)		3.90	0.07 U	0.66	1.97	4.59	1.34 B	2.24	10.80	10.7 U
303.3 c		3.82	0.07 U	0.58	1.62	3.34	1.22 B	1.49	8.25	11.0 U
	mean	4.23	NA	0.66	1.94	4.16	1.46	2.17	10.4	NA
	standard deviation	0.64	NA	0.09	0.30	0.71	0.31	0.65	1.96	NA
303.4 a	Santa Fe Channel End	0.52	0.07 U	0.18	0.51	1.37 B	0.07 U	0.23	2.29	11.1 U
303.4 b		0.40	0.08 U	0.18	0.52	1.23 B	0.04 U	0.26	2.19	11.2 U
303.4 c		0.48	0.07 U	0.16	0.48	1.17 B	0.11 B	0.22	2.14	11.1 U
	mean	0.47	NA	0.17	0.50	1.26	0.11	0.24	2.21	NA
	standard deviation	0.06	NA	0.01	0.02	0.10	NA	0.02	0.08	NA

(a) U Undetected above given concentration.
(b) B Analyte detected in blank; sample concentration is <5 times the blank concentration.
(c) ND None detected.
(d) Mean calculated using only detected values.
(e) NA Not applicable.

As in previous years, Lauritzen Channel/End (Station 303.3) had the highest mean concentration of total DDT in 2001 (Table 3.4); the lowest mean detected concentration was from Santa Fe Channel/End (Station 303.4). All target analytes were undetected at Richmond Inner Harbor Channel (Station 303.1). Total DDT concentrations in the total fraction of water samples collected from Lauritzen Channel in 2001 were lower than those measured in 2000 (Figure 3.1; Table 3.4), if all replicates of the 2000 data are considered. One replicate sampled in 2000 had much higher concentrations of contaminants than the other two (Kohn and Kropp 2000). Figure 3.1 shows water concentrations for all years at all stations, with Year 3 (2000) data for Station 303.3 plotted with and without the anomalous replicate. Concentrations of total DDT in the dissolved fraction of water samples from Richmond Inner Harbor Channel (Station 303.1) and Santa Fe Channel/End (Station 303.4) were lower in 2001 than in 2000 (Figure 3.1; Table 3.4). For the first time during the monitoring program, the total DDT concentration measured at a station (Richmond Inner Harbor Channel; Station 303.1) was lower than the remediation goal of 0.59 ng/L.

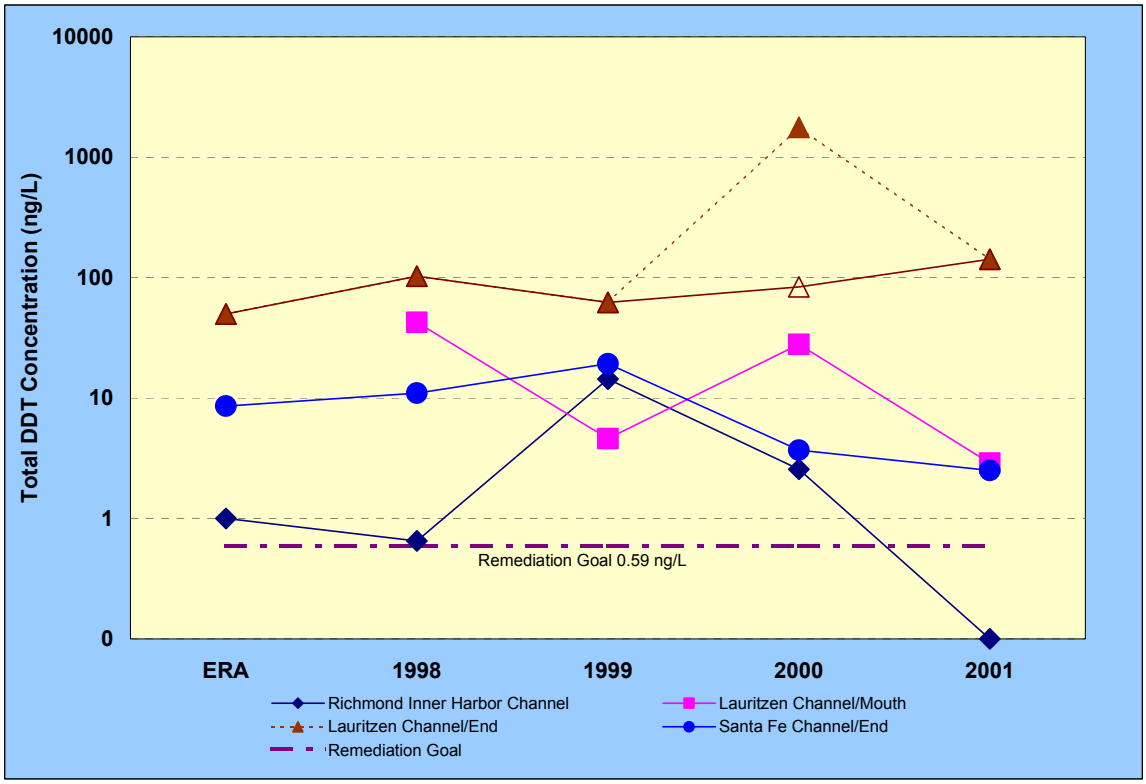

Figure 3.1. Comparison of preremediation (Ecological Risk Assessment) and post-remediation total DDT concentrations in water samples (total fraction) collected at the United Heckathorn Site. The open triangle for Station 303.3 sampled in 2000 is the mean value of only two replicates.

Table 3.4. Comparison of Post-Remediation Concentration of Total DDT and Dieldrin in Water Samples with Preremediation Levels and Remedial Goal Concentrations

Water		Water Concentration (ng/L)					
Sample ID	Location	Remediation Goal	Preremediation[a]	1998 Postremediation	1999 Postremediation	2000 Postremediation	2001 Postremediation
Total DDT							
303.1	Richmond Inner Harbor Channel	0.59	1	0.65	14.4	2.56	ND [b]
303.2	Lauritzen Channel/Mouth	0.59	no sample	42.6	4.61	27.9	2.88
303.3	Lauritzen Channel/End	0.59	50	103	62.3	83.7 (w/o rep b) 1773 (all reps)	142
303.4	Santa Fe Channel/ End	0.59	8.6	11	19.2	3.70	2.51
Dieldrin							
303.1	Richmond Inner Harbor Channel	0.14	<1	0.65	0.62	1.57	ND
303.2	Lauritzen Channel/Mouth	0.14	no sample	8.18	0.48	8.96	0.46
303.3	Lauritzen Channel/End	0.14	18	18.1	12.5	83 (w/o rep b) 625 (all reps)	8.49
303.4	Santa Fe Channel/ End	0.14	1.8	2.47	0.37	2.11	0.46

(a) Preremediation water concentration is the average of samples collected in October 1991 and February 1992 for the Ecological Risk Assessment (Lee et al. 1994)
(b) ND None detected.

Concentrations of dieldrin in replicates of the total fraction of water samples collected at Richmond Inner Harbor Channel (Station 303.1) in Year 4 were below the MDL. Dieldrin concentrations among replicate samples collected at the remaining three stations ranged from about 0.4 ng/L to 15.0 ng/L (Table 3.2). Mean concentrations of dissolved dieldrin ranged from 0.46 ng/L to 8.49 ng/L (Table 3.3). Concentrations of dieldrin at all four stations were lower in 2001 than in 2000 (Figure 3.2, Table 3.4).

Water concentrations of total DDT and dieldrin were above remediation goals in all water samples and at all stations except Richmond Inner Harbor Channel (Table 3.4, Figures 3.1 and 3.2). The most elevated contaminant concentrations were still found in water samples collected from Lauritzen Channel/End (Station 303.3), where contaminated sediment remains and may be periodically resuspended by vessel traffic.

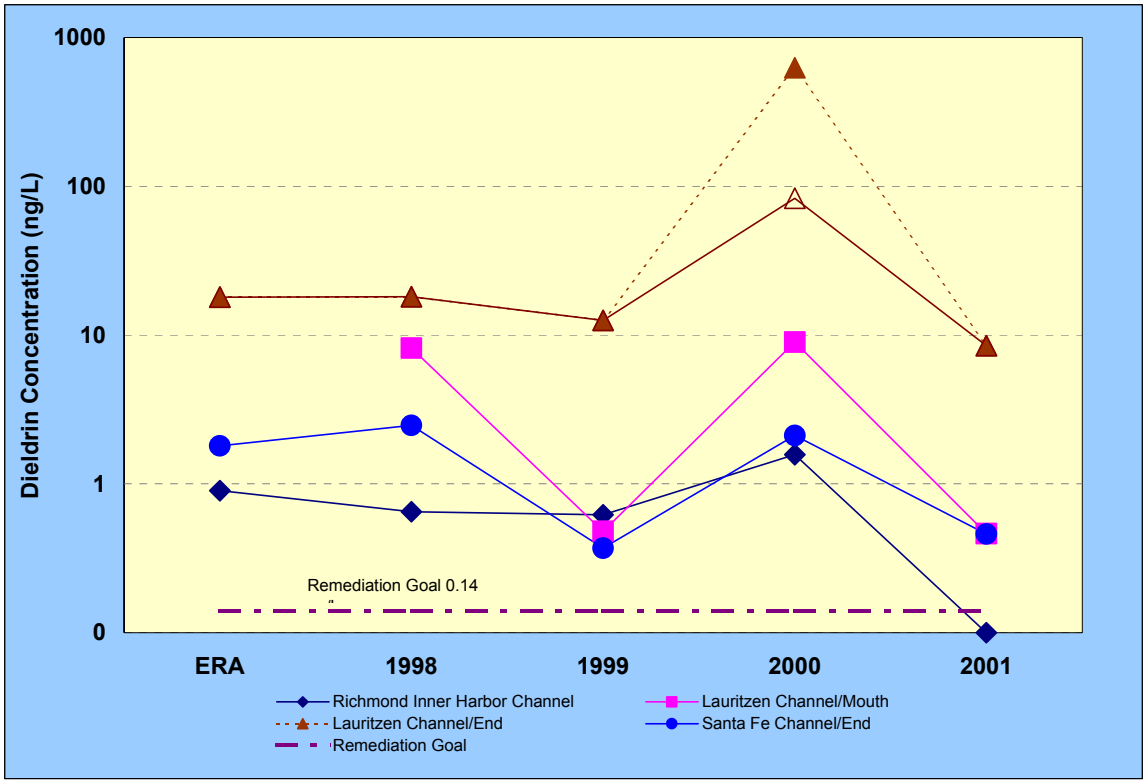

Figure 3.2. Comparison of preremediation (Ecological Risk Assessment) and post-remediation dieldrin concentrations in water samples (total fraction) collected at the United Heckathorn Site. The open triangle for station 303.3 sampled in 2000 is the mean value of only two replicates.

Concentrations of both total and dissolved PCB Aroclor 1254 in water samples collected from all stations in 2001 were below the MDL (Table 3.2). Aroclor 1254 concentrations at two stations, Lauritzen

Channel/Mouth (Station 303.2) and Lauritzen Channel/End (Station 303.3), were considerably lower than they were in 2000.

An attempt to address replicate variability and suspended sediment influence was made by analyzing total suspended solids and dissolved and total pesticides and PCBs in water samples. At most stations, there was little difference between concentrations of analytes found in the total and dissolved fractions of the water samples (Tables 3.2 and 3.3). However, there were substantial differences in analyte concentrations in the two fractions at Lauritzen Channel/End (Station 303.3). For example, the concentrations of total DDT and dieldrin in the dissolved fraction were much lower and much less variable than it was in the total fraction (Figure 3.3).

3.3 TISSUES

Tissue samples from biomonitoring organisms provide a time-integrated indication of contaminant concentrations in the water column and are not as susceptible to small-scale temporal or spatial variability in contaminant concentrations as are water samples. For tissue analyses, all QC requirements, except the precision of the MS/MSD analysis for 4,4'-DDT (72% relative percent difference), were met.

The post-remediation tissue data are summarized in Table 3.5 and compared with preremediation data in Tables 3.6 (wet-weight basis) and 3.7 (lipid-normalized basis). 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. Lipid-normalization removes differences attributable to tissue moisture and lipid content, allowing a better assessment of bioavailability between years and stations (Figures 3.4 and 3.5).

As in previous years, Year 4 post-remediation levels of total DDT were highest at the Lauritzen Channel/End (Station 303.3) and decreased at sites more distant from Station 303.3 or at sites with increased exposure to water exchange. Total DDT concentrations (wet weight) in resident mussels were 1136 µg/kg at Lauritzen Channel/End and 340 µg/kg at the Lauritzen Channel/Mouth (Station 303.2). At Santa Fe Channel/End (Station 303.4), total DDT levels were 149 µg/kg. The lowest concentrations were found at Richmond Inner Harbor Channel (Station 303.1), where total DDT in tissues was 25 µg/kg. The trend for dieldrin in mussel tissues was similar, with the highest levels occurring at Lauritzen Channel/End (32.1 µg/kg) and the lowest levels found at Richmond Inner Harbor Channel (0.71 µg/kg).

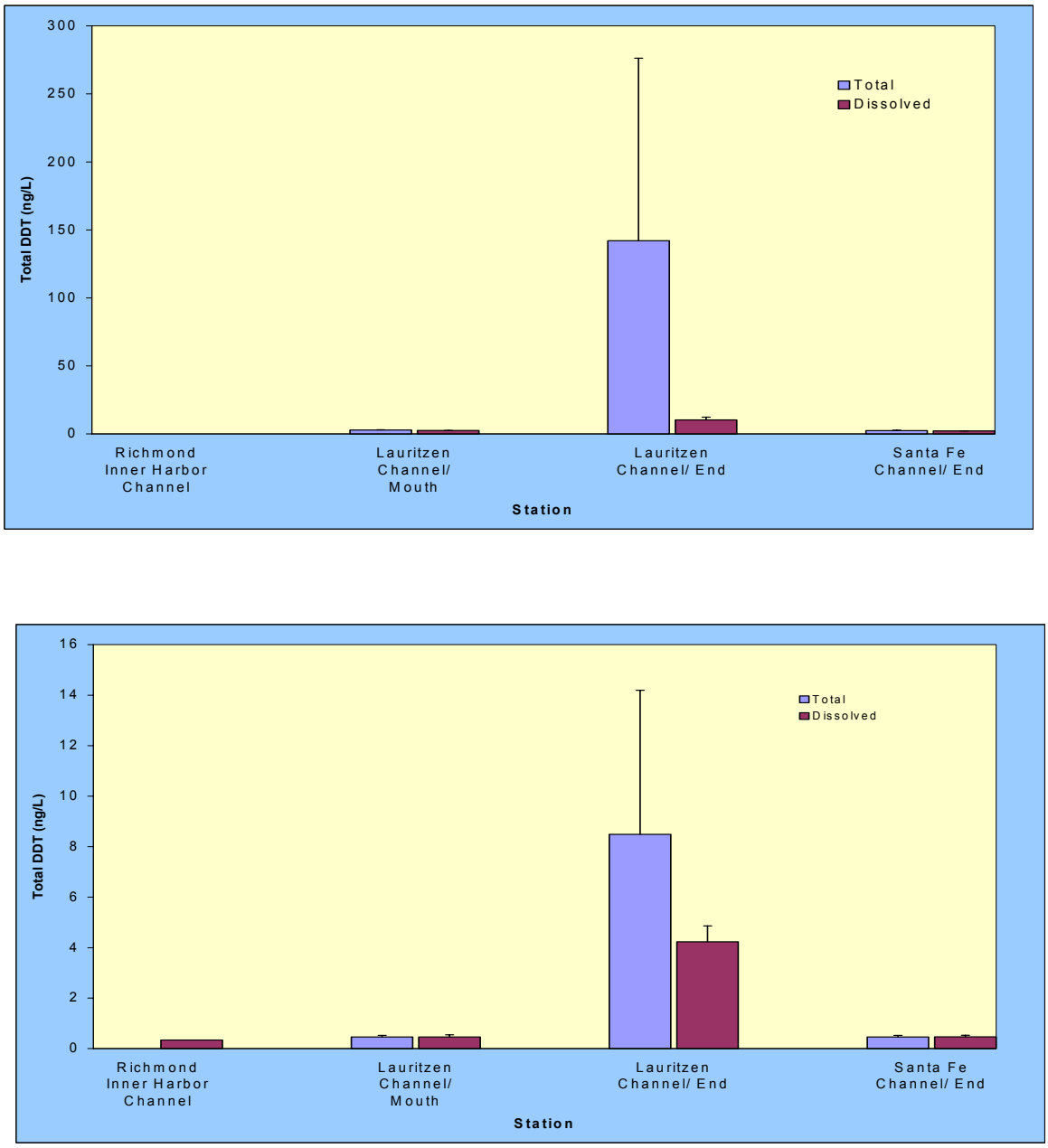

Figure 3.3. Total DDT (top) and dieldrin (bottom) concentrations in total and dissolved water fractions from samples collected in January 2001.

Table 3.5. Concentrations of DDT, Dieldrin, and PCB Aroclor 1254 in Tissue Samples Collected in January 2001 for Post-Remediation Monitoring of the United Heckathorn Site

	Sample ID and Concentration (µg/kg)			
Station Location	303.1 Richmond Inner	303.2 Lauritzen	303.3 Lauritzen	303.4 Santa Fe
Analyte	Harbor Channel	Channel Mouth	Channel End	Channel End
2,4'-DDD	0.33	1.92	4.71	0.67 U
2,4'-DDE	6.45	57.4 D	160 D	30.3
2,4'-DDT	3.35	47.8 D	144 D	20.2
4,4'-DDD	8.67	119 D	357 D	58.6
4,4'-DDE	2.23	42.9	169 D	12.4
4,4'-DDT	3.83	71.3 D	301 D	27.9
Total DDT (wet wt)	**24.9**	**340.3**	**1135.7**	**149.4**
Dieldrin (wet wt)	**0.71**	**6.27**	**32.1**	**3.32**
Percent Dry Wt	8.8	8.04	10.9	10.3
Total DDT (dry wt)	283	4233	10429	1464
Dieldrin (dry wt)	8	78	295	32
Lipids (% dry wt)	7.81	7.79	8.00	6.66
DDT (ppb[(b)] lipid)	3623	54337	130360	21885
Dieldrin (ppb lipid)	103	1001	3685	486
Aroclor 1254 (wet wt)	53	92	158	99
Aroclor 1254 (dry wt)	603	1143	1451	969
Aroclor 1254 (ppb lipid)	7726	14673	18136	14546

(a) Total DDT is sum of detected 2,4- and 4,4- DDD, DDE, and DDT.
(b) ppb parts per billion (µg contaminant/kg lipid).
D Sample diluted 10 X (station 303.2) or 20 X (station 303.3)
U Not detected at or above given value.

Tissue burdens of total DDT from Year 4 of post-remediation biomonitoring were similar to Year 3 post-remediation levels at Richmond Inner Harbor Channel (303.1) and Lauritzen Channel/Mouth (303.2), but were about two times higher than Year 3 at Lauritzen Channel/End (303.3) and Santa Fe Channel/End (303.4) (Table 3.6, Table 3.7, Figure 3.4). Tissue burdens of dieldrin were all slightly lower in Year 4 than in Year 3, but were very similar to Year 2 (Table 3.6). Annual tissue analyses have shown very similar patterns of DDT and dieldrin fluctuation over the years of post-remediation monitoring (Figures 3.4 and 3.5). In Year 1, total DDT (wet weight) concentrations were up to 3 times greater than the preremediation levels (Figure 3.4), but in Year 2 they were substantially reduced from the 1992 preremediation levels.

Table 3.6. Comparison of Post-Remediation Total DDT, Dieldrin, and PCBs in Tissues with Preremediation Concentrations (µg/kg wet weight)

Station Number	Station Name	State Mussel Watch[a] Transplant	Ecological Risk Assessment[b] Resident	1998 (Year 1) Post-remediation Resident	1999 (Year 2) Post-remediation Resident	2000 (Year 3) Post-remediation Resident	2001 (Year 4) Post-remediation Resident
Total DDT							
303.1	Richmond Inner Harbor Channel	47.0[c]	40	127	30	52	25
303.2	Lauritzen Channel/Mouth	629[d]	---	1222	176	310	340
303.3	Lauritzen Channel/End	5074[d] 1369[c]	2900	4504	606	522	1,136
303.4	Santa Fe Channel/End	369[c]	350	256	76	75	150
Dieldrin							
303.1	Richmond Inner Harbor Channel	7.7[c]	4.0	5.43	1.9	5.4	0.7
303.2	Lauritzen Channel/Mouth	87.0[d]	---	40.3	6.5	27.7	6.3
303.3	Lauritzen Channel/End	602[d] 100[c]	97.0	184	28.4	42.7	32.1
303.4	Santa Fe Channel/End	32.5[c]	19.0	8.18	2.8	6.4	3.3
Total PCBs							
303.1	Richmond Inner Harbor Channel	176[c]	not measured	not measured	51	150	53
303.2	Lauritzen Channel/Mouth	120[d]	not measured	not measured	75	187	92
303.3	Lauritzen Channel/End	196[d] 137[c]	not measured	not measured	124	169	158
303.4	Santa Fe Channel/End	138[c]	not measured	not measured	67	123	99

(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).

Table 3.7. Comparison of Lipid-Normalized Post-remediation Total DDT, Dieldrin, and PCBs in Tissues with Lipid-Normalized Preremediation Concentrations (μg/kg lipid)

Station Number	Station Name	State Mussel Watch[a] Transplant	Ecological Risk Assessment[b] Resident	1998 (Year 1) Post-remediation Resident	1999 (Year 2) Post-remediation Resident	2000 (Year 3) Post-remediation Resident	2001 (Year 4) Post-remediation Resident
Total DDT							
303.1	Richmond Inner Harbor Channel	9,215[c]	3,275	12,313	4,672	4,423	3,623
303.2	Lauritzen Channel/Mouth	78,481[d]		134,633	24,855	31,281	54,337
303.3	Lauritzen Channel/End	583,819[d] 380,361[c]	250,411	427,423	94,061	80,657	130,360
303.4	Santa Fe Channel/End	47,283[c]	21,919	45,695	8,193	9,182	21,885
Dieldrin							
303.1	Richmond Inner Harbor Channel	1,507[c]	322	525	293	457	103
303.2	Lauritzen Channel /Mouth	10,861[d]		4,439	919	2,791	1,001
303.3	Lauritzen Channel /End	69,272[d] 27,778[c]	8,590	17,463	4,410	6,598	3,685
303.4	Santa Fe Channel/End	4,167[c]	1,126	1462	300	779	486
Total PCBs							
303.1	Richmond Inner Harbor Channel	34,440[c]	not measured	not measured	8,020	12,752	7,726
303.2	Lauritzen Channel /Mouth	14,981[d]	not measured	not measured	10,599	18,842	14,673
303.3	Lauritzen Channel /End	22,554[d] 38,056[c]	not measured	not measured	19,255	26,112	18,136
303.4	Santa Fe Channel/End	17,667[c]	not measured	not measured	7,302	15,028	14,546

(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).

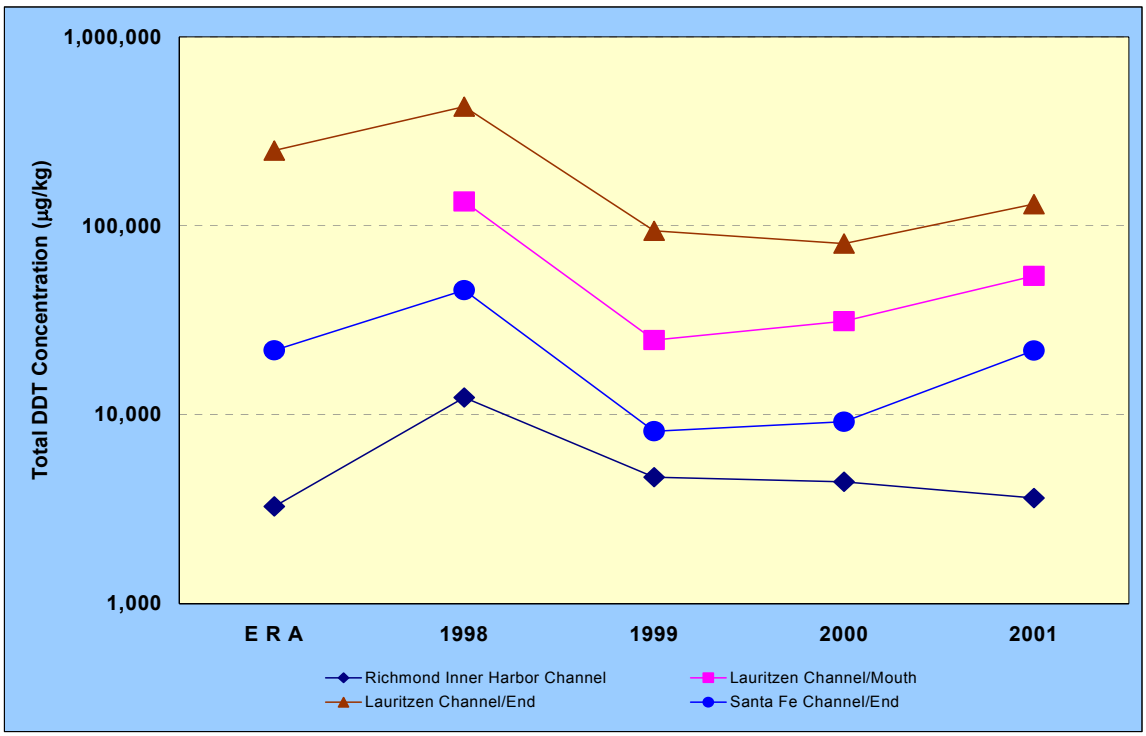

Figure 3.4. Comparison of preremediation (Ecological Risk Assessment) and post-remediation total DDT concentrations in mussel tissue samples collected at the United Heckathorn Site.

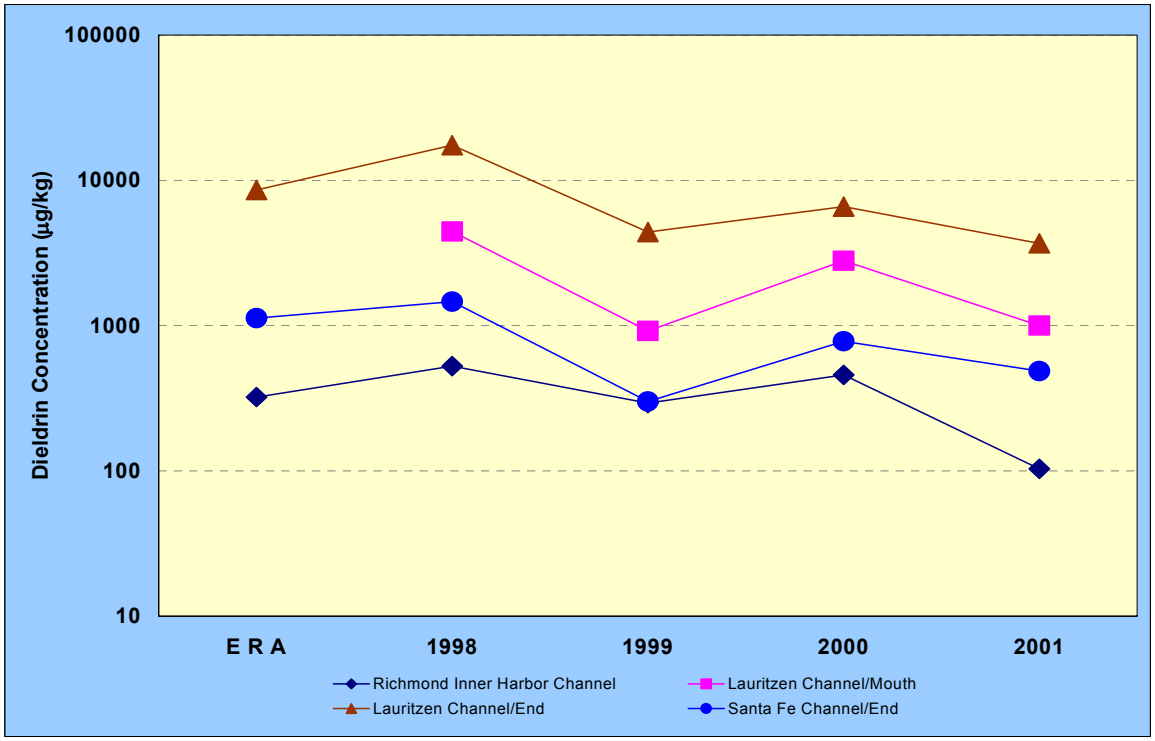

Figure 3.5. Comparison of preremediation (Ecological Risk Assessment) and post-remediation dieldrin concentrations in mussel tissue samples collected at the United Heckathorn Site.

Year 3 (2000) values were similar to but slightly less than (Stations 303.1 and 303.3) or slightly greater than (Stations 303.2 and 303.4) Year 2 levels. The pattern for dieldrin was similar, as Year 1 (1998) post-remediation resident mussel tissue levels were greater than preremediation levels measured in 1992 (Lee et al., 1994), and Year 2 levels showed a substantive reduction from Year 1 levels (Figure 3.5). However, levels found in Year 3 were 1.5 to 3 times higher than Year 2 levels (Figure 3.5), and in one case (Station 303.1) were about the same as Year 1 levels. As noted above, the present Year 4 dieldrin results are lower than those of Year 3, but very similar to those of Year 2.

Aroclor 1254 was the only PCB detected in mussels collected from post-remediation monitoring stations in 2001. Wet-weight PCB concentrations were highest in Lauritzen Channel/Mouth (158 µg/kg), and lowest at Richmond Harbor Inner Channel (53 µg /kg) (Table 3.5). These Year 4 PCB concentrations were lower than Year 3 concentrations but similar or slightly higher than Year 2 concentrations (PCBs were not measured in Year 1). PCBs in Year 4 resident mussels were still lower (5% to 70%; average 31% on a wet weight basis) than 1988 or 1991 (Mussel Watch) preremediation levels for transplanted mussels.

4.0 CONCLUSIONS

Results from the fourth post-remediation monitoring survey indicated that chlorinated pesticides remain bioavailable in the Lauritzen Channel and in the semi-enclosed waters nearby. Discrete water samples collected in January 2001 indicated that the total DDT and dieldrin concentrations in the water at Lauritzen Channel/End were similar to preremediation levels. However, concentrations of DDT and dieldrin at Richmond Inner Harbor Channel were lower than preremediation levels and were lower than the remediation goals for the first time since remediation occurred. Concentrations at Lauritzen Channel/Mouth and Santa Fe Channel/End were lower than preremediation levels, but were still above the remediation goals. Thus, remediation goals for total DDT and dieldrin in water have not yet been fully achieved for the study site. Monitoring of DDT and dieldrin in water samples should continue to include analysis of both total and dissolved phases and total suspended solids to assess the contribution of particulate matter to the pesticide concentrations in water.

Year 4 biomonitoring showed total DDT and dieldrin are still bioavailable to resident mussels in Lauritzen, Santa Fe, and Richmond Harbor Channels to varying degrees from previous years. Total DDT concentrations in mussels from all stations were approximately half the preremediation concentrations, while dieldrin concentrations were 30% or less of the preremediation concentrations. However, total DDT concentrations in mussels from Lauritzen and Santa Fe Channels were higher in 2001 than in 2000. This was not the case for dieldrin and PCBs, which were lower in 2001 than in 2000 at all stations. Biomonitoring using mussel tissues will continue to provide documentation of changes in the long-term bioavailability of pesticides from the Lauritzen Channel sediment that cannot be assessed through water sample analyses alone.

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APPENDIX A

FIELD SUMMARY REPORTS

Field Sampling Summary for Mussels and Surface Water
at the United Heckathorn Site in
Richmond, California, conducted 1/17/2001.

Andrew Lincoff
EPA Region 9 Laboratory
PMD-2
February 2, 2001

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 on January 17, 2001 by Andrew Lincoff, Amy Wagner and Peter Husby 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. Subsequent monitoring has found some remaining contamination of surface sediment.

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 round occurred in March, 1999. This is the fourth round of sampling. The seasonal timing was 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. In the first two rounds, both transplanted and resident mussels are analyzed to determine any difference. Based on the results of the first two rounds and discussions with California State Mussel Watch Program personnel, only resident mussels were collected in the third and fourth rounds.

Laboratory results are expected from Battelle in approximately one month.

FIELD NOTES AND OBSERVATIONS

1. Samples were collected on January 17, 2001 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. Station 303.2 is on the eastern side of the Laurtizen near its mouth, 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 collected 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

Station	Date	Time	Location	Remarks
303.1	1/17/01	1334	37 54' 32.8" N 122 21' 34.5" W	Richmond Channel Blind dup. seawater labeled 303.5
303.2	1/17/01	1445	37 55' 12.6" N 122 22' 01.2" W	Lauritzen South
303.3	1/17/01	1430	37 55' 22.5" N 122 21' 59.9" W	Lauritzen North MS/MSD Seawater
303.4	1/17/01	1410	37 55' 21.53" N 122 21' 18.37" W	Santa Fe

Seawater and resident Bay mussels were collected at each station for analysis by Battelle. At each station three 1 gallon replicate seawater samples were collected. At station 303.3, two additional 1 gallon seawater samples were collected for Battelle QA/QC. An additional single blind duplicate of seawater sample 303.1 was collected and shipped to the Battelle Lab with the fictitious station number 303.5.

At each station, approximately 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 at 1 ft above Mean Lower Low Water (MLLW) for the mussels collected from pilings at station 303.1, 303.2, and 303.3. At station 303.4, the mussels were collected near the surface from a floating dock.

3. The water temperature at each station was 50 EF, with the exception of station 303.3 where the temperature was 51 EF.

APPENDIX B

ANALYTICAL RESULTS FROM WATER AND TISSUE SAMPLES

QA/QC SUMMARY

PROJECT: Heckathorn Biomonitoring Year 4
PARAMETER: Pesticides, Total Suspended Solids (TSS)
LABORATORY: Battelle/Marine Sciences Laboratory, Sequim, Washington
MATRIX: Water

SAMPLE CUSTODY: Five water samples (multiple containers of each) were received on 1/19/01. All containers were received in good condition. Cooler temperatures upon arrival ranged from 2.9 to 5.9°C. Samples were assigned a Battelle Central File (CF) identification number (1611) and were entered into Battelle's log-in system.

QA/QC DATA QUALITY OBJECTIVES:

Analyte	Extraction Method	Analytical Method	Range of Recovery	Relative Precision	Detection Limits Target (ng/L)	Detection Limits Achieved (ng/L)
2,4'-DDE	$MeCl_2$	GC-ECD	40-120%	±30%	0.05	0.01
Dieldrin	$MeCl_2$	GC-ECD	40-120%	±30%	0.05	0.12
4,4'-DDE	$MeCl_2$	GC-ECD	40-120%	±30%	0.05	0.03
2,4'-DDD	$MeCl_2$	GC-ECD	40-120%	±30%	0.05	0.03
4,4'-DDD	$MeCl_2$	GC-ECD	40-120%	±30%	0.05	0.05
2,4'-DDT	$MeCl_2$	GC-ECD	40-120%	±30%	0.05	0.05
4,4'-DDT	$MeCl_2$	GC-ECD	40-120%	±30%	0.05	0.05
PCB Aroclor 1254	$MeCl_2$	GC-ECD	40-120%	±30%	20	14.2
Total Suspended Solids				≤20%	10 mg/L	0.001 mg/L

METHOD: Water samples for analysis of chlorinated pesticides and PCBs were processed according to Battelle SOP MSL-O-010, *Extraction and Clean-Up of Water for Surrogate Internal Standard Method*. Water samples were split to produce a total and dissolved sample for each sample received. 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 (DB-1701) gas chromatography with electron-capture detection (GC/ECD) according to SOP MSL-O-004, *Analysis of Polychlorinated Biphenyls and Chlorinated Pesticides by Gas Chromatography with Electron Capture Detection*, which is based on EPA Method 8081 (EPA 1986).

TSS samples were determined according to Standard Methods 2540-D, Solids (APHA 1998).

HOLDING TIMES: All pesticide extractions and analyses were conducted within target holding times: 14 days to extraction, and 40 days to analysis after extraction. Samples were received on 1/19/01 and held at 4°C. Samples were extracted on 1/20/01 through 1/23/01 and analyzed between 1/22/01 to 1/27/01.

Total suspended solids were analyzed within target holding time of 7 days. Samples were received 1/19/01 and processed 1/23/01.

QA/QC SUMMARY

DETECTION LIMITS:

Detection limits for organics 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.

Sample detection limits are calculated using the achieved detection limit and the sample volume.

BLANKS/BLANK SPIKES:

One procedural blank and two blank spikes were analyzed for the total samples. Three analytes of interest, dieldrin, 4,4'-DDT, and Aroclor 1254, were spiked into the samples at concentrations of 44.6 ng/L dieldrin and 4,4'-DDT in blank spike A and 48.1 ng/L dieldrin and 4,4'-DDT in blank spike B. Aroclor 1254 was spiked into the blank spikes A and B at 446 ng/L and 481 ng/L, respectively.

One procedural blank and two blank spikes were analyzed for the dissolved samples. Three analytes of interest, dieldrin, 4,4'-DDT, and Aroclor 1254, were spiked into the samples at concentrations of 48.5 ng/L dieldrin and 4,4'-DDT in blank spike A and 47.6 ng/L dieldrin and 4,4'-DDT in blank spike B. Aroclor 1254 was spiked into the blank spikes A and B at 485 ng/L and 476 ng/L, respectively.

All analytes were undetected except 4,4'-DDD and 2,4'-DDT in the dissolved blank. Samples with 4,4'-DDE and 4,4'-DDT detected at concentrations less than 5 times their blank values were flagged with a "B".

Blank spike recoveries were within of the target range of 40%-120% for dieldrin and Aroclor 1254 in both blank spikes A and B). Recovery of 4,4'-DDT was slightly outside the recovery limits in total blank spike B (129%) and within recovery limits in blank spike A.

Precision of the blank spikes replicate analysis, expressed as the RPD between the two replicates, were within the QC limits of ±30% for dieldrin, 4,4'-DDT, and Aroclor 1254 in both total and dissolved spikes.

MATRIX SPIKES AND MATRIX SPIKE DUPLICATES:

A matrix spike and matrix spike duplicate (MS/MSD) were prepared for total and dissolved phase and analyzed using sample 303.3. Three analytes of interest, dieldrin, 4,4'-DDT, and Aroclor 1254, were spiked into the total samples at concentrations of 26.0 ng/L dieldrin and 4,4'-DDT in the MS and 27.2 ng/L dieldrin and 4,4'-DDT in the MSD. Aroclor 1254 was spiked into the samples at 260 ng/L in the MS and 272 ng/L in the MSD. Recovery of 4,4'-DDT could not be calculated in the total MS/MSD because the spike concentration selected was too low relative to the native concentration of 4,4'-DDT in the sample. Concentrations of 4,4'-DDT were 8 times higher in the sample than the spike level chosen for these analytes; therefore, calculation of recovery was not feasible. Recoveries of 4,4'-DDT in the dissolved MS/MSD met the recovery criteria.

REPLICATES:

Three field replicate samples were provided for four samples. Relative standard deviation (RSD) between the three field replicates is reported in the data summary table. This information is not used to assess precision.

Sample 303.3 was analyzed in duplicate to assess laboratory precision. RPDs for all analytes in the total phase met QC criteria of ±30%. RPDs for all analytes except Dieldrin in the dissolved phase did not meet QC criteria for precision. No corrective action, other than verifying GC-ECD results, was taken. The presence of particulates was noted in the sample.

QA/QC SUMMARY

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 were within the target range of 40%-120% with the exception of surrogate PCB 198 in sample 1611-1D and 2D (303.1) at 121 and 128% respectively, sample 1611-5D (303.2) at 126%, 1611-8T, 9T, 9D and 14D (303.3) at 121, 146, 124, and 133% respectively.

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.

American Public Health Association. 1998. *Standard Method for the Examination of Water and Wastewater.* APHA, Washington, D.C.

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

UNITED HECKATHORN
Pesticides in Water
Samples Received 1/19/01

LOCATION:	**Richmond Inner Harbor**							
MSL Code	1611-1T	1611-2T	1611-3T		1611-1D	1611-2D	1611-3D	
STATION NO	303.1A	303.1B	303.1C	**TOTAL RSD**	303.1A	303.1B	303.1C	**DISS RSD**
Matrix	Water	Water	Water		Water	Water	Water	
TSS (mg/L)	0.003	0.006	0.000		NA	NA	NA	
Extraction Date	01/20/01	01/20/01	01/20/01		01/22/01	01/22/01	01/22/01	
Dilution	1X	1X	1X		1X	1X	1X	
Analytical Batch	1	1	1		1	1	1	
Unit	ng/L	ng/L	ng/L		ng/L	ng/L	ng/L	
2,4'-DDE	0.11 U	0.11 U	0.12 U	NA	0.08 U	0.07 U	0.07 U	NA
Dieldrin	0.06 U	0.06 U	0.06 U	NA	0.04 U	0.04 U	0.34	NA
4,4'-DDE	0.07 U	0.07 U	0.07 U	NA	0.04 U	0.04 U	0.04 U	NA
2,4'-DDD	0.12 U	0.13 U	0.13 U	NA	0.09 U	0.08 U	0.08 U	NA
4,4'-DDD	0.07 U	0.07 U	0.07 U	NA	0.16 B	0.04 U	0.15 B	NA
2,4'-DDT	0.05 U	0.05 U	0.06 U	NA	0.04 U	0.03 U	0.03 U	NA
4,4'-DDT	0.08 U	0.08 U	0.08 U	NA	0.05 U	0.05 U	0.05 U	NA
AROCLORS								
1254	16.6 U	17.0 U	17.3 U		11.5 U	10.3 U	10.3 U	
SURROGATE RECOVERIES (%)								
PCB103	61.0	81.7	82.2		78.1	82.1	76.4	
PCB198	76.9	108	106		121 #	128 #	113	

U Not detected at or above DL shown
NA Not available/applicable
B Concentration is less than 5x blank value
Outside QAQC recovery limits (40-120% recovery for BS, MS/MSD and surrogate recovery; ≤30% RPD for lab reps)

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360/681-3643

UNITED HECKATHORN
Pesticides in Water
Samples Received 1/19/01

LOCATION:	Lauritzen - South							
MSL Code	1611-4T	1611-5T	1611-6T		1611-4D	1611-5D	1611-6D	
STATION NO	303.2A	303.2B	303.2C	**TOTAL RSD**	303.2A	303.2B	303.2C	**DISS RSD**
Matrix	Water	Water	Water		Water	Water	Water	
TSS (mg/L)	0.000	0.001	0.001		NA	NA	NA	
Extraction Date	01/20/01	01/20/01	01/20/01		01/22/01	01/22/01	01/22/01	
Dilution	1X	1X	1X		1X	1X	1X	
Analytical Batch	1	1	1		1	1	1	
Unit	ng/L	ng/L	ng/L		ng/L	ng/L	ng/L	
2,4'-DDE	0.11 U	0.11 U	0.11 U	NA	0.07 U	0.07 U	0.08 U	NA
Dieldrin	0.48	0.51	0.40	**12%**	0.40	0.56	0.42	**19%**
4,4'-DDE	0.12	0.06 U	0.48	NA	0.32	0.30	0.38	**12%**
2,4'-DDD	0.42	0.36	0.54	**21%**	0.46	0.39	0.48	**11%**
4,4'-DDD	1.10	1.16	1.23	**6%**	1.20 B	1.07 B	1.08 B	**6%**
2,4'-DDT	0.05 U	0.05 U	0.05 U	NA	0.04 U	0.18 B	0.22 B	NA
4,4'-DDT	1.07	1.20	0.96	**11%**	0.61	0.62	0.41	**22%**
AROCLORS								
1254	16.0 U	16.0 U	16.3 U		11.0 U	11.1 U	11.4 U	
SURROGATE RECOVERIES (%)								
PCB103	78.5	77.7	78.0		74.6	83.9	74.6	
PCB198	106	103	104		111	126 #	115	

U Not detected at or above DL shown
NA Not available/applicable
B Concentration is less than 5x blank value
Outside QAQC recovery limits (40-120% recovery for BS, MS/MSD and surrogate recovery; ≤30% RPD for lab reps)

B.5

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

UNITED HECKATHORN
Pesticides in Water
Samples Received 1/19/01

LOCATION:	**Lauritzen - North**							
MSL Code	1611-7T	1611-8T	1611-9T		1611-9D	1611-14D	1611-15D	
STATION NO	303.3A	303.3B	303.3C	**TOTAL RSD**	303.3C	303.3 QC1	303.3 QC2	**DISS RSD**
Matrix	Water	Water	Water		Water	Water	Water	
TSS (mg/L)	0.007	0.006	0.001		NA	NA	NA	
Extraction Date	01/20/01	01/20/01	01/20/01		01/22/01	01/23/01	01/23/01	
Dilution	10x	10x	1x		1x	1x	1x	
Analytical Batch	1	1	1		1	1	1	
Unit	ng/L	ng/L	ng/L		ng/L	ng/L	ng/L	
2,4'-DDE	0.20	0.08 U	0.09 U	NA	0.07 U	0.08 U	0.07 U	NA
Dieldrin	15.0	6.09	4.38	**67%**	3.82	4.96	3.90	**15%**
4,4'-DDE	4.66	1.37	1.15	**82%**	0.58	0.75	0.66	**13%**
2,4'-DDD	6.86	2.82	2.35	**62%**	1.62	2.22	1.97	**16%**
4,4'-DDD	12.5	6.37	5.36	**48%**	3.34	4.54	4.59	**17%**
2,4'-DDT	69.9	8.84	5.61	**129%**	1.22 B	1.81 B	1.34 B	**21%**
4,4'-DDT	200 D	72.9 D	25.5	**91%**	1.49	2.79	2.24	**30%**
AROCLORS								
1254	16.3 U	16.6 U	13.6 U		11.0 U	11.2 U	10.7 U	
SURROGATE RECOVERIES (%)								
PCB103	74.1	89.9	98.8		74.2	83.2	94.9	
PCB198	97.5	121 #	146 #		111	124 #	133 #	
PCB103 - Dilution	85.3 D	82.9 D						
PCB198 - Dilution	83.8 D	80.3 D						

U Not detected at or above DL shown
NA Not available/applicable
B Concentration is less than 5x blank value
D Dilution reported (see header)
Outside QAQC recovery limits (40-120% recovery for BS, MS/MSD and surrogate recovery; ≤30% RPD for lab reps)

B.6

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

UNITED HECKATHORN
Pesticides in Water
Samples Received 1/19/01

LOCATION:	**Santa Fe Channel**							
MSL Code	1611-10T	1611-11T	1611-12T		1611-10D	1611-11D	1611-12D	
STATION NO	303.4A	303.4B	303.4C	**TOTAL RSD**	303.4A	303.4B	303.4C	**DISS RSD**
Matrix	Water	Water	Water		Water	Water	Water	
TSS (mg/L)	0.002	0.006	0.000		NA	NA	NA	
Extraction Date	01/20/01	01/20/01	01/20/01		01/22/01	01/22/01	01/22/01	
Dilution	1X	1X	1X		1X	1X	1X	
Analytical Batch	1	1	1		1	1	1	
Unit	ng/L	ng/L	ng/L		ng/L	ng/L	ng/L	
2,4'-DDE	0.08 U	0.08 U	0.09 U	NA	0.07 U	0.08 U	0.07 U	NA
Dieldrin	0.48	0.39	0.51	**14%**	0.52	0.40	0.48	**13%**
4,4'-DDE	0.06	0.22	0.19	**54%**	0.18	0.18	0.16	**7%**
2,4'-DDD	0.09 U	0.58	0.75	NA	0.51	0.52	0.48	**4%**
4,4'-DDD	1.79	1.48	1.61	**10%**	1.37 B	1.23 B	1.17 B	**8%**
2,4'-DDT	0.04 U	0.04	0.14	NA	0.07 U	0.04 U	0.11 B	NA
4,4'-DDT	0.29	0.26	0.13	**38%**	0.23	0.26	0.22	**9%**
AROCLORS								
1254	12.3 U	12.5 U	12.5 U		11.1 U	11.2 U	11.1 U	
SURROGATE RECOVERIES (%)								
PCB103	83.6	70.4	68.2		76.8	76.0	72.4	
PCB198	86.3	94.5	83.0		116	113	117	

U Not detected at or above DL shown
NA Not available/applicable
B Concentration is less than 5x blank value

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360/681-3643

UNITED HECKATHORN
Pesticides in Water
Samples Received 1/19/01

LOCATION:	United Heckathorn	
MSL Code	1611-13T	1611-13D
STATION NO	303.5	303.5
Matrix	Water	Water
TSS (mg/L)	0.005	NA
Extraction Date	01/20/01	01/23/01
Dilution	1X	1X
Analytical Batch	1	1
Unit	ng/L	ng/L
2,4'-DDE	0.08 U	0.08 U
Dieldrin	0.13	0.11
4,4'-DDE	0.05 U	0.15
2,4'-DDD	0.09 U	0.22
4,4'-DDD	0.05 U	0.56 B
2,4'-DDT	0.04 U	0.15 B
4,4'-DDT	0.11	0.81
AROCLORS		
1254	11.6 U	11.2 U
SURROGATE RECOVERIES (%)		
PCB103	75.0	71.6
PCB198	107	105

U Not detected at or above DL shown
NA Not available/applicable
B Concentration is less than 5x blank value

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1529 West Sequim Bay Road
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360/681-3643

UNITED HECKATHORN
Pesticides in Water
Samples Received 1/19/01

		BSA			*BSB*					*BSA*			*BSB*			
MSL Code	Blank 1	Blank	Spike	Percent	Blank	Spike	Percent		Blank 2	Blank	Spike	Percent	Blank	Spike	Percent	
STATION NO		Spike A	Amount	Recovery	Spike B	Amount	Recovery	RPD		Spike A	Amount	Recovery	Spike B	Amount	Recovery	RPD
Matrix	Water	Water			Water				Water	Water			Water			
TSS (mg/L)	0.000	NA			NA				NA				NA			
Extraction Date	01/20/01	01/20/01			01/20/01				01/22/01				01/22/01			
Dilution	1x	1x			1x				1x				1x			
Analytical Batch	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	0.20 U	0.20 U	NS	NA	0.21 U	NS	NA		0.21 U	0.21 U	NS	NA	0.21 U	NS	NA	
Dieldrin	0.11 U	34.3	44.6	77%	38.4	48.1	80%	4%	0.11 U	35.9	48.5	74%	41.0	47.6	86%	15%
4,4'-DDE	0.12 U	0.11 U	NS	NA	2.30	NS	NA		0.12 U	1.95	NS	NA	0.12 U	NS	NA	
2,4'-DDD	0.22 U	0.22 U	NS	NA	0.23 U	NS	NA		0.23 U	0.24 U	NS	NA	0.23 U	NS	NA	
4,4'-DDD	0.12 U	0.12 U	NS	NA	0.13 U	NS	NA		0.36	0.13 U	NS	NA	0.13 U	NS	NA	
2,4'-DDT	0.10 U	0.09 U	NS	NA	0.10 U	NS	NA		0.82	0.10 U	NS	NA	0.10 U	NS	NA	
4,4'-DDT	0.14 U	51.5	44.6	115%	62.1	48.1	129% #	11%	0.15 U	57.1	48.5	117%	53.7	47.6	113%	4%
AROCLORS																
1254	29.9 U	448	446	100%	571	481	119%	17%	31.3 U	484	485	100%	429	476	90%	10%
SURROGATE RECOVERIES (%)																
PCB103	60.5	70.7			84.7				70.8	76.6			75.9			
PCB198	76.5	94.8			93.4				112	111			118			

B.9

U Not detected at or above DL shown
\# Outside QAQC recovery limits (40-120% recovery for BS, MS/MSD and surrogate recovery; ≤30% RPD)

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

UNITED HECKATHORN
Pesticides In Water
Samples Received 1/19/01

		MSA			*MSB*					*MSA*			*MSB*			
MSL Code	1611-7T	1611-7T	Spike	Percent	1611-7T	Spike	Percent		1611-15D	1611-15D	Spike	Percent	1611-15D	Spike	Percent	
STATION NO	303.3A								303.3 QC2							
		Spike A	Amount	Recovery	Spike B	Amount	Recovery	RPD		Spike A	Amount	Recovery	Spike B	Amount	Recovery	RPD
Matrix	Water	Water			Water				Water	Water			Water			
TSS (mg/L)	0.007	NA			NA				NA	NA			NA			
Extraction Date	01/20/01	01/20/01			01/20/01				01/23/01	01/23/01			01/23/01			
Dilution	10x	10x			10x				1x	1x			1x			
Analytical Batch	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	0.20	0.11 U	NS	NA	0.12 U	NS	NA		0.07 U	0.09 U	NS	NA	0.09 U	NS	NA	
Dieldrin	15.0	34.5	26.0	75%	33.2	27.2	67%	11%	3.90	21.5	20.7	85%	19.8	20.8	76%	11%
4,4'-DDE	4.66	3.86	NS	NA	0.07 U	NS	NA		0.66	1.21	NS	NA	1.15	NS	NA	
2,4'-DDD	6.86	4.43	NS	NA	0.13 U	NS	NA		1.97	1.33	NS	NA	1.15	NS	NA	
4,4'-DDD	12.5	10.2	NS	NA	9.93	NS	NA		4.59	5.42	NS	NA	4.97	NS	NA	
2,4'-DDT	69.9	43.3	NS	NA	31.1	NS	NA		1.34 B	0.96 B	NS	NA	0.95 B	NS	NA	
4,4'-DDT	200 D	111 D	26.0	NC	143 D	27.2	NC	NA	2.24	26.4	20.7	117%	24.6	20.8	108%	8%
AROCLORS																
1254	16.3 U	297	260	108%	286	272	99%	9%	10.7 U	208	207	100%	195	208	94%	7%
SURROGATE RECOVERIES (%)																
PCB103	74.1	78.7			77.2				94.9	76.6			82.0			
PCB198	97.5	95.2			88.1				133 #	118			120			
PCB103 - Dilution	85.3 D	87.8 D			88.5 D											
PCB198 - Dilution	83.8 D	81.9 D			89.3 D											

U Not detected at or above DL shown
NC Not calculable
Outside QAQC recovery limits (40-120% recovery for BS, MS/MSD and surrogate recovery; ≤30% RPD for lab reps)
B Concentration is less than 5x blank value
D Dilution reported (see header)

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1529 West Sequim Bay Road
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360/681-3643

UNITED HECKATHORN
Pesticides in Water
Samples Received 1/19/01

		Dup			*Dup*	
MSL Code	1611-8T	1611-8r2		1611-14D	1611-14Dr2	
STATION NO	303.3B	303.3B		303.3 QC1	303.3 QC1	
			RPD			RPD
Matrix	Water	Water		Water	Water	
TSS (mg/L)	0.006	NA		NA	NA	
Extraction Date	01/20/01	01/20/01		01/23/01	01/23/01	
Dilution	10x	10x		1x	10x	
Analytical Batch	1	1		1	1	
Unit	ng/L	ng/L	%	ng/L	ng/L	%
2,4'-DDE	0.08 U	0.08 U	NA	0.08 U	0.07 U	NA
Dieldrin	6.09	5.54	9%	4.96	5.73	14%
4,4'-DDE	1.37	1.62	17%	0.75	1.09	37% #
2,4'-DDD	2.82	2.81	0%	2.22	3.16	35% #
4,4'-DDD	6.37	5.92	7%	4.54	6.25	32% #
2,4'-DDT	8.84	9.42	6%	1.81 B	33.2	179% #
4,4'-DDT	72.9 D	57.3 D	24%	2.79	64.7 D	183% #
AROCLORS						
1254	16.6 U	11.8 U		11.2 U	10.9 U	
SURROGATE RECOVERIES (%)						
PCB103	89.9	87.0		83.2	75.5	
PCB198	121 #	124 #		124 #	107	
PCB103 - Dilution	82.9 D	83.6 D			83.1 D	
PCB198 - Dilution	80.3 D	86.8 D			87.3 D	

U Not detected at or above DL shown
\# Outside QAQC recovery limits (40-120% recovery for BS, MS/MSD and surrogate recovery; ≤30% RPD for lab reps)
B Concentration is less than 5x blank value
D Dilution reported (see header)

B.11

QA/QC SUMMARY

PROJECT: Heckathorn Biomonitoring Year 4
PARAMETER: Pesticides, PCBs, and Total Lipids
LABORATORY: Battelle/Marine Sciences Laboratory, Sequim, Washington
MATRIX: Tissues

SAMPLE CUSTODY: Four mussel tissue samples were received on 1/19/01. All samples were received in good condition. The cooler temperature on arrival was 2.9°C. Mussels were shucked in the wet laboratory, placed in clean glass jars, and returned to the chemistry laboratory for analysis on 1/23/01. The temperature was not recorded; samples were hand-delivered. Mussel samples were then assigned a Battelle Central File (CF) identification number (1611) and were entered into Battelle's log-in system.

QA/QC DATA QUALITY OBJECTIVES:

Analyte	Extraction Method	Analytical Method	Range of Recovery	Relative Precision	Detection Limits: Target (ng/g wet)	Detection Limits: Achieved (ng/g)
2,4'-DDE	$MeCl_2$	GC-ECD	40-120%	±30%	2	0.27
Dieldrin	$MeCl_2$	GC-ECD	40-120%	±30%	2	0.29
4,4'-DDE	$MeCl_2$	GC-ECD	40-120%	±30%	2	1.03
2,4'-DDD	$MeCl_2$	GC-ECD	40-120%	±30%	2	0.38
4,4'-DDD	$MeCl_2$	GC-ECD	40-120%	±30%	2	0.36
2,4'-DDT	$MeCl_2$	GC-ECD	40-120%	±30%	2	0.52
4,4'-DDT	$MeCl_2$	GC-ECD	40-120%	±30%	2	0.36
PCB Aroclor 1254	$MeCl_2$	GC-ECD	40-120%	±30%	20	14.3
Total Lipids	$CHCl_3$/ CH_3OH	Gravimetric	NA	±30%	NA	NA

METHOD: Tissue samples for analysis of chlorinated pesticides and PCBs were processed according to Battelle SOP MSL-O-009, *Extraction and Clean-Up of Sediments and Tissues for Semivolatile Organics Following the Surrogate Internal Standard Method*, which is derived from NOAA NS&T and EPA methods with modifications from Krahn et al. (1988). Tissue samples were macerated and extracted with methylene chloride. Interferences were removed using an aluminum/silicon column chromatography step followed by a high-performance liquid chromatography (HPLC) clean-up according to SOP MSL-O-006, *HPLC Cleanup of Sediment and Tissue Extracts for Semivolatile Pollutants*. Sample extracts were then transferred to cyclohexane and analyzed by capillary-column (DB-1701) gas chromatography with electron-capture detection (GC/ECD) according to SOP MSL-O-004, *Analysis of Polychlorinated Biphenyls and Chlorinated Pesticides by Gas Chromatography with Electron Capture Detection*, which is based on EPA Method 8081 (EPA 1986). Total lipids were determined according to the Bligh et al. (1959) method modified to use a smaller sample size. Lipids were extracted from separate aliquots of tissue samples using chloroform and methanol, 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 1/23/01 and held at 4°C. Samples were extracted on 2/5/01 and analyzed on 2/12/01. Lipid extractions were conducted on 2/5/01.

QA/QC SUMMARY

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.

Sample detection limits are calculated using the achieved detection limit and the sample weight.

BLANKS/BLANK SPIKES: One procedural blank and two blank spikes were analyzed. All spiked analytes (dieldrin, 4,4'-DDT, and PCB Aroclor 1254) were undetected in the blank. Blank spike recoveries of dieldrin, 4,4'-DDT, and Aroclor 1254 were within the target range of 40%-120%.

REPLICATES: One tissue sample [1611-17 (303.2)] was analyzed in duplicate for chlorinated compounds and lipids. Precision for duplicate analysis is reported by calculating the relative percent difference (RPD) of replicate results. RPDs for all analytes of interest ranged from 3% to 18%, and were all within the QC limits of ±30%.

MATRIX SPIKES: A matrix spike and matrix spike duplicate pair was analyzed using sample 303.4. Recoveries of the three spiked analytes of interest, dieldrin, 4,4'-DDT, and Aroclor 1254, were within the target range of 40%-120% in both the MS and MSD.

Replicate precision of the MS/MSD analysis, expressed as the RPD between the MS and MSD, was within the QC criteria of ±30% for dieldrin (14%) and Aroclor 1254 (5%). Precision of the MS/MSD analysis for 4,4'-DDT (72% RPD) exceeded QC criteria. No corrective action was taken.

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 were within the target range of 40%-120%, ranging from 66.0% to 94.1%.

REFERENCES:

Bligh, E.G., and W.J. Dyer. 1959. A Rapid Method of Total Lipid Extraction and Purification. *Canadian Journal of Biochemistry and Physiology.* 37:8 911-917.

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. *New HPLC Cleanup and Revised Extraction Procedures for Organic Contaminants.* 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.

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.

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

UNITED HECKATHORN
Pesticides in Tissue (Dry Weight)
Samples Received 1/19/01

			Dup		
MSL Code	1611-16	1611-17	1611-17	1611-18	1611-19
STATION NO	303.1	303.2	303.2	303.3	303.4
LOCATION	**Richmond Inner Harbor**	**Lauritzen - South**	**Lauritzen - South**	**Lauritzen - North**	**Santa Fe Channel**
Matrix	Tissue	Tissue	Tissue	Tissue	Tissue
Extraction Wet Wt (g)	10.09	10.08	10.10	10.18	10.11
Percent Wet Wt	91.20	91.96	91.96	89.11	89.75
Percent Dry Wt	8.80	8.04	8.04	10.89	10.25
Extraction Date	02/05/2001	02/05/2001	02/05/2001	02/05/2001	02/05/2001
Percent Lipids (DW)	7.81	7.79	7.81	8.00	6.66
Dilution	1x	10X	10x	20X	1x
Analytical Batch	1	1	1	1	1
Unit (dry wt)	ng/g	ng/g	ng/g	ng/g	ng/g
2,4'-DDE	3.75	23.9	19.9	43.3	6.54
4,4'-DDE	73.3	714 D	766 D	1469.2 D	296
2,4'-DDD	38.1	595 D	611 D	1322 D	197
4,4'-DDD	98.5	1480 D	1604 D	3278 D	572
2,4'-DDT	25.3	534	506 D	1552 D	121
4,4'-DDT	43.5	887 D	980 D	2764 D	272
Total (dw)	282.5	4232.8	4487.6	10428.926	1464.1
Dieldrin	8.07	78.0	75.2	295	32.4
	3617.2			130361.6	
AROCLORS					
1254	603.4	1143.0	0.0	1450.9	968.8

U Not detected at or above DL shown
D Dilution reported (see header)

DRY WEIGHT RESULTS CALCULATED FROM WET WEIGHT ANALYSIS

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

UNITED HECKATHORN
Pesticides in Tissue (Wet Weight)
Samples Received 1/19/01

		BSA			*BSB*				*DUP*	
MSL Code	Blank	Blank	Spike	Percent	Blank	Spike	Percent	1611-17	1611-17	
STATION NO								303.2	303.2	
		Spike A	Amount	Recovery	Spike B	Amount	Recovery			RPD
Matrix	Tissue	Tissue			Tissue			Tissue	Tissue	
Extraction Wet Wt (g)	NA	NA			NA			10.08	10.10	
Percent Wet Wt	NA	NA			NA			91.96	91.96	
Extraction Date								02/05/01	02/05/01	
Percent Lipids (DW)	NA	NA			NA			7.79	7.81	0%
Dilution								10x	10x	
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	0.27 U	NS	NA	0.27 U	NS	NA	1.92	1.60	18%
Dieldrin	0.29 U	6.91	10.0	69%	7.71	10.0	77%	6.27	6.05	4%
4,4'-DDE	1.03 U	1.03 U	NS	NA	1.03 U	NS	NA	57.4 D	61.6 D	7%
2,4'-DDD	0.38 U	0.38 U	NS	NA	0.38 U	NS	NA	47.8 D	49.1 D	3%
4,4'-DDD	0.36 U	0.36 U	NS	NA	0.36 U	NS	NA	119 D	129 D	8%
2,4'-DDT	0.52 U	0.52 U	NS	NA	0.52 U	NS	NA	42.9	40.7 D	5%
4,4'-DDT	0.36 U	10.5	10.0	105%	10.4	10.0	104%	71.3 D	78.8 D	10%
AROCLORS										
1254	14.3 U	112	100	112%	108	100	108%	91.9	94.4	3%
SURROGATE RECOVERIES (%)										
PCB103	76.8	82.1			80.7			66.0	67.0	
PCB198	80.1	85.8			85.5			69.0	69.0	
PCB103 - Dilution	NA	NA			NA			84.3 D	81.7 D	
PCB198 - Dilution	NA	NA			NA			75.3 D	74.3 D	
PCB103 - Aroclor 1254	77.2	101			82.7			66.1	67.1	
PCB198 - Aroclor 1254	80.3	86.4			86.4			69.0	69.1	

U Not detected at or above DL shown
D Dilution reported (see header)
NA Not applicable
NS Not spiked

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

UNITED HECKATHORN
Pesticides in Tissue (Wet Weight)
Samples Received 1/19/01

		MSA			*MSB*			
MSL Code	1611-19	1611-19	Spike	Percent	1611-19	Spike	Percent	
STATION NO	303.4	Spike A	Amount	Recovery	Spike B	Amount	Recovery	RPD
Matrix	Tissue	Tissue			Tissue			
Extraction Wet Wt (g)	10.11	10.11			10.11			
Percent Wet Wt	89.75	89.75			89.75			
Extraction Date	02/05/01	02/05/01			02/05/01			
Percent Lipids (DW)	6.66	NA			NA			
Dilution	1x	1x			1x			
Analytical Batch	1	1			1			
Unit (wet wt)	ng/g	ng/g	ng/g	%	ng/g	ng/g	%	%
2,4'-DDE	0.26 U	0.97	NS	NA	0.63	NS	NA	
Dieldrin	3.32	9.41	9.79	62%	10.4	9.89	72%	15%
4,4'-DDE	30.3	28.3	NS	NA	22.4	NS	NA	
2,4'-DDD	20.2	17.6	NS	NA	13.5	NS	NA	
4,4'-DDD	58.6	54.0	NS	NA	42.7	NS	NA	
2,4'-DDT	12.4	11.4	NS	NA	8.96	NS	NA	
4,4'-DDT	27.9	38.2	9.79	105%	32.8	9.89	50%	71% #
AROCLORS								
1254	99.3	170	97.9	72%	167	98.9	68%	6%
SURROGATE RECOVERIES (%)								
PCB103	70.2	74.4			79.4			
PCB198	77.2	82.0			79.2			
PCB103 - Dilution	NA	NA			NA			
PCB198 - Dilution	NA	NA			NA			
PCB103 - Aroclor 1254	70.2	74.7			79.5			
PCB198 - Aroclor 1254	77.2	82.3			79.3			

U Not detected at or above DL shown
Outside QAQC recovery limits (40-120% recovery for BS, MS/MSD and surrogate recovery; ≤30% RPD for lab reps)
NA Not applicable
NS Not spiked

APPENDIX C

MUSSEL SHELL LENGTH
RAW DATA

Resident Mussels Only, Year 4, 2001

Sample ID	Shell Length (cm to nearest 0.01 cm) 303.10	303.40	303.20	303.30
1	4.11	4.35	4.09	4.09
2	4.21	4.45	4.45	4.23
3	4.32	4.48	4.47	4.33
4	4.33	4.51	4.49	4.38
5	4.35	4.66	4.50	4.43
6	4.42	4.70	4.52	4.46
7	4.42	4.72	4.73	4.49
8	4.47	4.73	4.79	4.61
9	4.51	4.78	4.84	4.63
10	4.62	4.80	4.85	4.71
11	4.78	4.87	4.86	4.78
12	4.79	4.93	4.88	4.81
13	4.84	4.93	5.02	4.81
14	4.91	5.03	5.05	4.81
15	4.99	5.06	5.06	4.83
16	4.99	5.07	5.07	4.83
17	5.05	5.12	5.08	5.05
18	5.18	5.17	5.14	5.07
19	5.20	5.22	5.18	5.10
20	5.23	5.27	5.18	5.16
21	5.23	5.27	5.19	5.17
22	5.25	5.30	5.19	5.24
23	5.28	5.36	5.20	5.24
24	5.36	5.38	5.22	5.25
25	5.40	5.45	5.22	5.29
26	5.43	5.49	5.23	5.33
27	5.47	5.53	5.23	5.40
28	5.47	5.56	5.23	5.54
29	5.52	5.57	5.24	5.59
30	5.58	5.57	5.43	5.60
31	5.63	5.58	5.61	5.66
32	5.66	5.62	5.63	5.72
33	5.68	5.63	5.65	5.75
34	5.74	5.75	5.68	5.76
35	5.77	5.77	5.68	5.78
36	5.86	5.82	5.70	5.79
37	5.89	5.88	5.81	5.82
38	5.92	5.96	5.82	5.82
39	5.92	5.97	5.83	5.88
40	6.20	6.01	5.88	5.92
41	6.29	6.05	5.89	6.03
42	6.34	6.05	6.03	6.04
43	6.41	6.05	6.19	6.05
44	6.46	6.08	6.27	6.09
45	6.52	6.26	6.42	6.26
46	6.55	6.40	6.45	7.21
47		6.52		
48		6.59		

Resident Mussels Only, Year 4, 2001

Sample ID	Shell Length (cm to nearest 0.01 cm) 303.10	303.40	303.20	303.30
n	46	48	46	46
mean length (mm)	5.32	5.40	5.29	5.28
min length (mm)	4.11	4.35	4.09	4.09
max length (mm)	6.55	6.59	6.45	7.21
ratio min:max	0.63	0.66	0.63	0.57
Grand mean	5.32			
Standard deviation	0.06			
wet weight (g)				
Jar + sample	574.35	641.80	554.97	505.65
jar	301.53	299.89	303.02	299.69
sample only	272.82	341.91	251.95	205.96
n	46	48	46	46
mean wt (g)/mussel	5.93	7.12	5.48	4.48
mean wt/mean size (g/mm)	1.12	1.32	1.04	0.85
Grand mean	5.75			
Standard deviation	0.61			

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