

## **APPENDIX R**

### **Field and Laboratory Operations**

## FIELD AND LABORATORY OPERATIONS

### Sample Collection

The State Mussel Watch Program (SMWP) collects about 100 mussels at each station, which are randomly divided into two groups for trace element and synthetic organic chemical analysis. Based on recommendations by Goldberg (1980) and Risebrough *et al.* (1980), the SMWP samples 45 mussels, three replicates of 15 individuals each, for trace elements at each site. Trace element results in the SMWP represent a mean value for the three replicates. A single replicate of 45 composited individuals is analyzed for synthetic organic compounds.

Mussels of 55 to 65 mm in length are collected wherever possible in order to reduce size related effects. Mussels are collected from the highest tidal height where they occur in adequate numbers to reduce variability induced by habitat height. Stainless steel pry bars are used to collect mussels off rocks. The pry bars are cleaned and rinsed in the laboratory and rinsed again with seawater prior to use.

At locations where mussels are unavailable and sampling can be accomplished using scuba equipment, transplanted samples are used. The mussel transplant system used is one of the following three systems; 1) In an area of deep water and no structures, a bottom anchored submerged buoy system is used; 2) In areas with structures (ie. pilings, floating docks, etc.), a polypropylene line may be tied between two pilings or a line hung beneath a dock; 3) In areas of shallow water, samples may be placed on PVC or wooden stakes that are pounded into the substrate. Transplanted mussels are placed in polypropylene mesh bags and kept cool in ice chests for no more than 48 hours prior to deployment. To minimize the risk of contamination of the mussel from boat exhaust or surface film during deployment or retrieval, mussel samples are placed in polyethylene bags, where they remain until submerged and deployed. Upon retrieval from the subsurface buoy system, samples are again placed in polyethylene bags before being brought through the air-water interface. Once collected, the transplants are triple bagged. To minimize contamination caused by handling the mussel samples, polyethylene gloves are worn during collection, as well as processing, of mussel samples. A two month transplant period is adequate in most cases where pollutant uptake rates are expected to be high, but for trace elements in less contaminated environments, a six month interval may be necessary for an adequate sample (Stephenson *et al.* 1980). A four to six month transplant interval is used for organic chemicals to be consistent with transplant periods for trace elements.

Mussels to be analyzed for trace elements are placed in a ZIPLOCK<sup>®</sup> polyethylene bag of 4 mm thickness. The samples are placed inside two additional polyethylene ZIPLOCK<sup>®</sup> bags. Mussels to be analyzed for synthetic organic compounds are placed in a bag constructed of two layers of "heavy duty" aluminum foil. Prior to use, the foil is cleaned by heating to 500° C or by rinsing in hexane. Samples in the foil bags are placed in two polyethylene ZIPLOCK<sup>®</sup> bags. After bagging, all samples are placed in non-metallic ice chests and frozen using dry ice and stored at or below -20° C until processed.

### Laboratory Analysis

A detailed description of procedures and techniques discussed below can be found in the Department of Fish and Game's (DFG) *Laboratory Quality Assurance Program Plan* (DFG 1990). The following is a summary of the 1993-94 and 1994-95 Quality Assurance/Quality Control (QA/QC) results provided by the DFG's Water Pollution Control and Moss Landing Laboratories. Copies of the Laboratory Quality Assurance Program Plan and QA/QC results are available upon request.

## Trace Elements Analytical Techniques in Tissue and Sediment

The following procedures were employed for mussel dissection and homogenization for trace element analysis: Frozen mussels were removed individually from the bags, cleaned of epiphytic organisms and debris under running deionized water by personnel wearing polyethylene gloves, and allowed to thaw in clean polyethylene trays. Adductor muscles were severed and gonads removed with a MICRO<sup>R</sup>-cleaned stainless steel scalpel. Gonads were removed from mussels to reduce variability in trace element concentrations due to the sex of the organism (Stephenson *et al.* 1987). The remainder of the soft part was placed in a pre-weighted, acid-cleaned polypropylene 4 oz. jar and re-weighed. The shell lengths were also taken at this time. Samples were then homogenized to a paste-like consistency in the jars using a Brinkmann Polytron (Model PT10-35) equipped with a titanium generator (Model PTA 20). The homogenized samples were then refrozen at -20° C until analyzed.

A Perkin-Elmer Model 2280 spectrophotometer with deuterium arc background corrector and digital display was used for techniques employing conventional (flame) atomic absorption spectrophotometry (Al, Cd, Cu, Mn, Zn) and cold vapor technique for mercury. A Perkin-Elmer Model 3030 Zeeman atomic absorption spectrophotometer equipped with an HGA-600 graphite furnace and an AS-60 autosampler was used for techniques requiring a graphite furnace (Ag, As, Cr, Ni, Pb, Se). All analytical values were corrected using procedural blanks. Trace element detection limits are presented in Table R-1. The technique used for digesting samples was known as "teflon vessel digestion". Separate techniques were performed on sediments and tissues in the "teflon vessel digestion" technique.

The "teflon vessel digestion" technique for tissue and sediment were performed as follows: Samples were weighed into pre-cleaned 125 ml teflon digestion vessels. Three grams of tissue and one gram of sediment were used. Digestion of each tissue sample was accomplished by adding a 4:1 concentrated HNO<sub>3</sub>: 3 ml concentrated HClO<sub>4</sub> mixture and heating the sample on a warm (~75°) hotplate 2-3 hours. After the initial reaction, the teflon vessel was capped and heated in a 130° C oven for four hours. Once the digestate had cooled it was transferred to a clean polyethylene bottle and diluted up to 20 ml with Type II water. Sediment samples were digested using the same mixture as tissue samples except, instead of warming on a hotplate, sediment samples were heated in a 130° C oven for four hours. After the initial reaction, 3 ml of hydrofluoric acid was added to the sediment sample and the teflon vessel returned to a 130° C oven for 12 hours. Twenty ml of boric acid (2.5%) was added to each sediment sample before again returning to a 130° C oven for another 8 hours. Once the digestate was cool it was transferred to a clean polyethylene bottle and brought up to 20 ml with Type II water.

To protect sample integrity, all materials contacting samples during laboratory operations were analyzed for trace element content. To ensure accuracy, reference materials from the National Bureau of Standards (NBS) were analyzed (Table R-2).

## Synthetic Organic Compounds Analytical Techniques in Tissues

A 50 gram sample of tissue was spiked with a surrogate mixture of 4,4'-dibromo-octafluorobiphenyl, decachlorobiphenyl, and dibutylchlorodate (DBOB, DCB, DBCE) and extracted twice with acetonitrile by shaking for two hours. The sample extracts were combined, filtered, and partitioned with petroleum ether. An aliquot of the petroleum ether extract was eluted through a Florisil<sup>R</sup> column. The Florisil<sup>R</sup> columns were eluted

with petroleum ether (Fraction 1), six percent ethyl ether/petroleum ether (Fraction 2), and 15 percent ethyl ether/petroleum ether (Fraction 3). Fractions 2 and 3 were spiked with decachlorobiphenyl and all of the fractions were concentrated to an appropriate volume in a Zymark<sup>®</sup> Turbovap concentrator prior to analysis by gas chromatography. The DCB was used as a surrogate to determine analyte recovery of the F1 compounds and to determine relative retention times for all fractions. DBOB was used to check the analyte recovery of the F2 compounds but was found to elute with the F1 compounds. DBCE was used to check the analyte recovery of the F3 compounds. The percent recoveries for the surrogate compounds are listed in Table R-3 for 1994 and Table R-4 for 1995. A mixture of synthetic standards was eluted through the Florisil<sup>®</sup> column to determine the recovery and separation characteristics of the column. The distribution of synthetic organic compounds in the three fractions is listed in Table R-5. The detection limits for synthetic organics in mussels are presented in Table R-6.

At stations where the SMWP had previously detected endosulfan, samples were analyzed for endosulfan I, endosulfan II, and endosulfan sulfate. This required an additional elution through Florisil<sup>®</sup> with 50 percent ethyl ether/petroleum ether (Fraction 4, Table R-5). All other stations were analyzed for endosulfan I only. This fraction was also spiked with decachlorobiphenyl prior to the concentration step. Due to the high lipid content of the fraction all of the 50 percent extracts were diluted with iso-octane by a factor of ten prior to analysis by gas chromatography.

Two mussel samples were spiked with a solution containing known concentrations of target analytes to assess accuracy and matrix effects. Percent recoveries of the target analytes from the matrix spike are listed in Table R-7.

Ten percent of the samples were analyzed in duplicate. Table R- 8 lists duplicate sample results. A method blank representative of all materials and solutions contacting the sample was analyzed for contamination. To preclude errors due to contamination, a vertical solvent was blank analyzed for each set of glassware before introducing a new sample.

### **Synthetic Organic Compounds Analytical Techniques in Sediment**

In 1994, approximately 30 grams of each sediment sample was spiked with a surrogate mixture of DBOB, DCB and DBCE. After adding approximately 200 ml of a 1:1 solution of acetone:dichloromethane, the sample was extracted with an orbital shaker for two hours at 300 rpm. These steps were repeated after the sample was filtered. After evaporating and exchanging solvents, the sample extract was eluted through a Florisil<sup>®</sup> column using the four solvent mixtures (F1, F2, F3 and F4).

In 1995, the method for analyzing synthetic organics in sediments was modified. Twenty grams of sediment was dried by mixing with sodium sulfate. After adding 200 ml of 1:1 solution of hexane/acetone, each sample was spiked with 1 ml of the DBOB, DCB and DBCE solution. The samples were then placed on an orbital shaker for two hours at 300 rpm. The sample was filtered, re-extracted with fresh solvent, and the extracts were combined. After evaporating and exchanging solvents, the sample extract was eluted through a Florisil<sup>®</sup> column with petroleum ether (Fraction 1) and a solution of 50% ethyl ether/petroleum ether (Fraction 2). Sediment detection limits are listed in Table R-6. Duplicate sample analysis results are listed in Table R-9.

## Instrument and Analytical Conditions for Chlorinated Hydrocarbons

Chlorinated hydrocarbons were determined with a Varian Model 3500 gas chromatograph equipped with a Model 8035 autosampler, temperature programmable on-column injector, and dual Ni<sup>63</sup> electron capture detectors. A 5 meter J&W DB5 fused silica capillary pre-column is connected to the temperature programmable injector, the column effluent is split using a press-fit "Y" connector to a 60 meter J&W DB5 and a 60 meter J&W DB17 column. The DB5 and DB17 columns are connected to the electron capture detectors. All three columns have a 0.25 mm ID and a 25 µm liquid phase thickness. Helium was used as the carrier gas at a linear velocity of 35 cm/sec and nitrogen was used as the detector makeup gas at a flow of 25 ml/min. Chromatographic data was acquired and processed with a Hewlett-Packard Chem-Station, version A.03.02.

All samples were analyzed using a single injection for each extract under the following conditions:

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Injector temperature program:

Initial temperature - 70 °C  
Program rate - 300 °C/min  
Final temperature - 280°C  
Final temperature hold time - 70 min

Column temperature program:

Initial temperature - 70°C  
Program rate 1 - 15°C/min to 210°C  
Program 1 hold time - 10 min  
Program rate 2 - 2°C/min to 280°C

Final temperature hold time - 11 min

Detector temperature: 330°C

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## Analytical Techniques for Polynuclear Aromatic Hydrocarbon Compounds (PAHs) in Flesh

A 20 gram tissue sample was dried with sodium sulfate, spiked with a surrogate mixture of deuterated PAH compounds and extracted with dichloromethane. Sample extracts were cleaned up using gel permeation chromatography followed by alumina and silica gel chromatography.

Sample extracts were analyzed using a Varian Saturn II Ion Trap GC-MS. One microliter of sample extract was injected into a J&W Scientific DB-5MS, 30 meter x 0.25 mm I.D. fused silica capillary column with a 0.25 µm film thickness. The GC oven temperature was initially held at 70°C for two minutes. The temperature ramp was 15°C per minute until the oven reached 150°C. The second temperature ramp was 2°C per minute to a final temperature of 280°C and held for 5 minutes. Initial injector temperature was 70° and was programmed to 280° at 300°/min immediately after injection. The GC carrier gas was helium at a linear velocity of 37 cm/sec. Detection limits of the PAHs are reported in Table R-10. Results of duplicate analyses for PAHs in mussel and sediment are listed in Tables R-11, R-12, and R-13. Matrix spike recoveries for mussel tissue and sediment are listed in Table R-14.

## **Analytical Techniques for Tributyltin (TBT)**

Tributyltin was extracted from tissues by mixing 10 g of tissue, 10 ml of 50% HCL, and 25 ml of methylene chloride for 15 hours. The mix was then centrifuged for five minutes. The methylene chloride was removed and evaporated under a stream of air and the residue was dissolved in hexane. The hexane was washed in a 3% NaOH solution to remove all monobutyl- and dibutyl-tins, mixed for 10 seconds, centrifuged for 5 minutes, and re-evaporated to dryness. The residue was digested with 1 ml of concentrated nitric acid and diluted to 5 ml with Type II water. The solution was analyzed on a Perkin Elmer Model 3030 Zeeman Atomic Absorption Spectrophotometer equipped with a Model 500 Graphite Furnace and an AS60 Autosampler. Ten  $\mu\text{l}$  sample was co-injected with 10  $\mu\text{l}$  of matrix modifier consisting of 100  $\mu\text{g}$  phosphate and 10  $\mu\text{g}$  magnesium nitrate per injection. Tributyltin detection limit is provided in Table R-6. A PACS sample, marine sediment reference material from the National Research Council of Canada, was used as a reference material for tributyltin. In 1993-94, the laboratory result was 1.09  $\mu\text{g/g}$  dry weight with a certified value of 1.27 $\pm$ 0.22. In 1994-95, the laboratory result was 1.57 $\pm$ 0.02  $\mu\text{g/g}$  dry weight with the same certified value. Duplicate tributyltin analysis was not performed in either 1993-94 or 1994-95.

## **Procedure for Lipid Determination**

As synthetic organic concentrations in organisms may vary with lipid content, it is customary to provide lipid data when reporting tissue concentrations. A thoroughly homogenized sample weighing approximately 5 g (wet weight) is macerated and dried with anhydrous granular  $\text{Na}_2\text{SO}_4$ . The dried sample is transferred to a blender with 150 ml of petroleum ether and blended for two minutes at high speed. The liquid is vacuum-filtered into a 250 ml filter flask through a 10 cm Buchner funnel containing Whatman #1 filter paper. The sample is blended once more with an additional 150 ml of petroleum ether and filtered. The filtrate is concentrated to approximately 25 ml with heat (steam bath) and nitrogen steam. The remaining filtrate is then quantitatively transferred into a 50 ml pre-weighed planchet. The petroleum ether is evaporated, the planchet containing the residue is reweighed, and the percent lipid is calculated.

## TABLE R-1

State Mussel Watch Program  
Trace Element Detection Limits

### Tissue and Sediment

Element	Detection Limit	
	( $\mu\text{g/g}$ , ppm dry weight)	( $\mu\text{g/g}$ , ppm wet weight)
Aluminum	1.0	0.2
Arsenic	0.25	0.04
Cadmium	0.002	0.0003
Chromium	0.02	0.003
Copper	0.003	0.0005
Mercury	0.03	0.005
Manganese	0.05	0.008
Nickel	0.1	0.02
Lead	0.03	0.005
Selenium	0.1	0.02
Silver	0.002	0.0003
Titanium	0.5	0.08
Zinc	0.02	0.003

**TABLE R-2**

State Mussel Watch Program  
Trace Element Analysis of Reference Materials ( $\mu\text{g/g}$ , dry weight)\*

Error! Bookmark not defined.	1993-94**		1994-95**	
	NBS Oyster	NBS Dolt2	NBS Oyster	NBS Dolt2
Ag	1.72±0.16 (1.68±0.15)	NA	1.43±0.11 (1.68±0.15)	NA
Al	174±5 (202.5±14.1)	NA	194±14 (202.5±14.1)	NA
As	13.4±1.0 (14.0±1.2)	NA	11.8±0.6 (14.0±1.2)	NA
Cd	4.6±0.4 (4.15±0.38)	21.2±1.5 (20.8±0.5)	4.31±0.37 (4.15±0.38)	NA
Cr	0.94±0.08 (1.43±0.46)	NA	1.15±0.06 (1.43±0.46)	NA
Cu	64.2±2.5 (66.3±4.3)	27.0 (25.8±1.1)	61.9±1.3 (66.3±4.3)	NA
Hg	0.066±0.012 (0.064±0.007)	NA	0.073±0.005 (0.064±0.007)	2.09±0.11 (1.99±0.10)
Mn	12.6±0.5 (12.3±1.5)	6.68±0.36 (6.88±0.56)	11.9±0.4 (12.3±1.5)	NA
Ni	2.09±0.34 (2.25±0.44)	NA	2.70±0.10 (2.25±0.44)	NA
Pb	0.34±0.04 (0.371±0.014)	NA	0.33±0.01 (0.371±0.014)	NA
Se	NA	NA	1.98±0.24 (2.21±0.24)	NA
Zn	821±9.2 (830±57)	96.5±3.5 (85.8±2.5)	897±10 (830±57)	NA

\* Sample values are given first, followed by reference values in parentheses, both values include 95% confidence interval where appropriate.

**NBS** refers to the National Bureau of Standards.

**DOLT2** refers to dogfish liver from the National Research Council of Canada.

\*\* Sample Year = State Fiscal Year (July 1 - June 30).

NA = Not Analyzed.



**TABLE R-3**

State Mussel Watch Program  
 Percent Recovery of Surrogate Compounds for 1994

Station Number	Station Name	DBOB	DCB	DBCE
10.0	Trinidad Head	51	68	73
100.0	Mad River Slough	58	81	84
103.0	Eureka Channel	51	71	82
202.0	Bodega Head	55	81	66
404.0	Sandholdt Bridge	55	70	82
414.0	Pacific Grove	52	78	90
414.0 Dup	Pacific Grove	57	79	105
420.0	Monterey Harbor/Coast Guard Jetty	53	60	77
507.3	Mugu Lagoon/Calleguas Creek	57	76	78
601.0	LA Harbor/National Steel	53	82	74
602.0	LA Harbor/West Basin	55	78	70
605.0	LA Harbor/Cabrillo Pier	56	72	81
616.0	LA Harbor/Consolidated Slip	55	80	33
618.0	LA Harbor/Angels Gate	57	51	78
648.0	Malibu	52	50	78
650.0	Santa Monica	54	53	70
662.0	Royal Palms	58	85	84
681.0	Catalina Island/West	51	82	96
713.0	Huntington Harbour/Edinger Street	60	84	77
713.0 Dup	Huntington Harbour/Edinger Street	57	83	67
715.0	Huntington Harbour/Warner Ave Brdg	54	77	74
715.0 Dup	Huntington Harbour/Warner Ave Brdg	58	83	81
724.0	Newport Bay/Highway 1 Bridge	54	81	72
725.0	Newport Bay/Crows Nest	55	83	81
726.4	Newport Bay/Rhine Channel/End	56	81	84
883.4	San Diego Bay/Continental Maritime	52	77	64
894.0	SD Bay/Harbor Is/E Basin/Storm Dr	53	80	69

DBOB = 4,4'-dibromo-octafluorobiphenyl  
 DCB = decachlorobiphenyl  
 DBCE = dibutylchloroendate

Dup = Duplicate analysis.

**TABLE R-4**

State Mussel Watch Program  
Percent Recovery of Surrogate Compounds for 1995

Station Number	Station Name	DBOB	DCB	DBCE
10.0	Trinidad Head	66	88	81
202.0	Bodega Head	54	93	42
404.0	Sandholdt Bridge	64	110	56
404.0 Dup	Sandholdt Bridge	62	110	61
601.0	LA Harbor/National Steel	80	130	120
605.0	Cabrillo Pier	75	120	77
616.0	LA Harbor/Consolidated Slip	75	140	81
618.0	LA Harbor/Angels Gate	76	120	78
648.0	Malibu	80	95	73
650.0	Santa Monica	92	130	91
662.0	Royal Palms	87	130	86
664.0	Cabrillo Beach	78	110	77
713.0	Huntington Harbour/Edinger Street	73	110	69
715.0	Huntington Harbour/Warner Ave Brdg	70	110	55
723.4	Newport Bay/Turning Bas.	66	120	56
724.0	Newport Bay/Highway 1 Bridge	73	120	57
725.0	Newport Bay/Crows Nest	74	120	61
726.4	Newport Bay/Rhine Channel/End	68	120	66
750.0	Oceanside	77	110	68
882.0	24th St. Maritime Terminal/South	72	120	62
894.0	SD Bay/Harbor Is/E Basin/Storm Dr	46	78	46
894.0 Dup	SD Bay/Harbor Is/E Basin/Storm Dr	69	110	50
899.0	San Diego Bay/Shelter Is/Fshg Pier	66	99	44

DBOB = 4,4'-dibromo-octafluorobiphenyl  
DCB = decachlorobiphenyl  
DBCE = dibutylchloroendate

Dup = Duplicate analysis.

**TABLE R-5**

State Mussel Watch Program  
Distribution of Synthetic Organic Compounds Among  
Four Fractions of a Standard Florisil<sup>®</sup> Column

(0%) Fraction 1	(6%) Fraction 2	(15%) Fraction 3
HCH, alpha*	HCH, alpha*	dacthal
aldrin	HCH, beta	diazinon
chlordene, alpha	HCH, gamma	dichlorobenzophenone, p,p'
chlordene, gamma	HCH, delta	dieldrin
DDE, o,p'	cis-chlordane	endosulfan I
DDE, p,p'	trans-chlordane	endrin
DDMU, p,p**	chlorpyrifos	malathion
DDT, o,p'	DDD, o,p'	oxadiazon
DDT, p,p**	DDD, p,p'	parathion, ethyl
heptachlor	DDMU p,p**	parathion, methyl
hexachlorobenzene	DDT, p,p**	tetradifon (tedion)
trans-nonachlor	dicofol (kelthane)	
PCB 1248	ethion	
PCB 1254	heptachlor epoxide	
PCB 1260	methoxychlor	
	cis-nonachlor	
	oxychlordane	
	toxaphene	
		<u>(50%) Fraction 4</u>
		endosulfan II
		endosulfan sulfate

\* Found in both 0% and 6% fractions.

**TABLE R-6**

State Mussel Watch Program  
Synthetic Organic Compounds Analyzed  
and Their Detection Limits in Mussel and Sediment

Compound	Detection Limit (ng/g, ppb dry weight)
aldrin	1
cis-chlordane	1
trans-chlordane	1
chlordene, alpha	1
chlordene, gamma	1
chlorpyrifos	4
dacthal	2
DDD, o,p'	5
DDD, p,p'	3
DDE, o,p'	3
DDE, p,p'	3
DDMU,p,p'	5
DDT, o,p'	4
DDT, p,p'	4
diazinon	50
dichlorobenzophenone-p,p'	3
dicofol (Kelthane)	10
dieldrin	1
endosulfan I	1
endosulfan II	10
endosulfan sulfate	50
endrin	6
ethion	20
HCH, alpha	1
HCH, beta	3
HCH, gamma	0.8
HCH, delta	2
heptachlor	1
heptachlor epoxide	1
HCB	1
methoxychlor	15
cis-nonachlor	1
trans-nonachlor	1
oxadiazon	2
oxychlordane	1
parathion, ethyl	10
parathion, methyl	10
PCB 1248	50
PCB 1254	10
PCB 1260	10
tetradifon (Tedion)	10
toxaphene	100
tributyltin	20

**TABLE R-7**

State Mussel Watch Program  
Results of Matrix Spike Analyses: 1993-95 Synthetic Organic Compounds  
Mussel Tissue

Station Name	1994 Bodega Head	1995 Pacific Grove
Station Number	202.0	414.0
Species	RCM	RCM
<u>Compound</u>	Percent Recovery	Percent Recovery
aldrin	59	58
cis-chlordane	92	94
trans-chlordane	95	84
oxychlordane	87	77
cis-nonachlor	100	88
trans-nonachlor	94	65
alpha chlordene	65	61
gamma chlordene	64	64
chlorpyrifos	86	59
dicofol	not spiked	72
dichlorobenzophenone	not spiked	39
dacthal	91	67
diazinon	97	70
dieldrin	110	75
endosulfan I	110	72
endosulfan II	110	83
endosulfan sulfate	89	77
endrin	110	69
ethion	100	71
alpha HCH	91	58
beta HCH	76	65
gamma HCH	77	60
delta HCH	81	47
o,p'-DDD	100	95
p,p'-DDD	120	97
o,p'-DDE	84	74
p,p'-DDE	80	50
p,p'-DDMU	66	70
o,p'-DDT	84	62
p,p'-DDT	110	94
heptachlor	58	46
heptachlor epoxide	96	88
hexachlorobenzene	61	62
methoxychlor	120	82
oxadiazon	71	50
ethyl parathion	95	58
methyl parathion	45	42
PCB 1248	not spiked	not spiked
PCB 1254	not spiked	not spiked
PCB 1260	not spiked	not spiked
tetradifon	110	68
toxaphene	not spiked	not spiked

RCM = Resident California Mussel.

**TABLE R-8**  
 State Mussel Watch Program  
 Results of Duplicate Sample Analysis: 1994 Synthetic Organic Compounds Quality Control - Mussel Tissue  
 (ng/g dry weight)

Station Name	Hunting Harbor/ Edinger Street 713.0 TCM		Hunting Harbor/ Warner Ave. Bridge 715.0 TCM		Pacific Grove 414.0 RCM	
	1	2	1	2	1	2
<b>COMPOUNDS</b>						
aldrin			ND	1.1		
cis-chlordane	22	24	39	40	2.4	2.0
cis-nonachlor	11	11	23	24		
alpha-chlordene	1.8	1.6	2.4	2.7		
gamma-chlordene			ND	1.0		
oxychlordane	1.0	1.0	1.8	1.6		
trans-chlordane	20	20	35	35	1.9	1.5
trans-nonachlor	20	21	36	36	1.2	1.5
chlorpyrifos	11	8.8	11	14		
dacthal	19	19	3.0	2.2		
DDD, o,p'	11	11	14	14		
DDD, p,p'	34	34	47	49		
DDE, o,p'	12	13	12	12		
DDE, p,p'	220	230	330	340	24	26
DDT, o,p'						
DDT, p,p'	15	14	14	15	5.0	4.3
DDMU,p,p'	15	14	16	18		
diazinon						
dieldrin	13	13	14	10	5.9	7.5
endosulfan I						
endosulfan II						
endosulfan sulfate						
hexachlorobenzene						
alpha-HCH					1.3	1.3
gamma-HCH						
heptachlor epoxide						
oxadiazon			12	14		
PCB 1248						
PCB 1254	100	100	140	170		
PCB 1260						
toxaphene						
percent moisture	88.2	88.5	87.8	88.2	84.9	85.6
percent lipid	0.443	0.484	0.554	0.555	0.525	0.430

TCM = Transplanted California Mussel.

RCM = Resident California Mussel.

ND = Not Detected.

R-14

**TABLE R-8 (continued)**

State Mussel Watch Program

Results of Duplicate Sample Analysis: 1994 Synthetic Organic Compounds Quality Control - Sediment  
(ng/g dry weight)

Station Name Station No.	L.A. Harbor/ Consolidated Slip 616.0 SED	
	1	2
<u>REPLICATE</u>		
<u>COMPOUNDS</u>		
aldrin	1.4	1.1
cis-chlordane	24	20
cis-nonachlor	10	11
alpha-chlordene	3.6	3.4
gamma-chlordene	2.2	2.0
oxychlordane	1.7	1.9
trans-chlordane	29	21
trans-nonachlor	21	18
chlorpyrifos	18	13
dacthal	2.1	2.2
DDD, o,p'	30	33
DDD, p,p'	130	130
DDE, o,p'	11	9.8
DDE, p,p'	220	210
DDT, o,p'	12	13
DDT, p,p'	79	320
DDMU,p,p'	12	12
diazinon		
dieldrin	13	7.9
endrin	9.4	12
endosulfan I		
endosulfan II		
endosulfan sulfate		
heptachlor	ND	1.0
heptachlor epoxide	7.3	3.9
hexachlorobenzene	1.0	1.0
alpha-HCH		
beta-HCH	ND	5.7
gamma-HCH		
heptachlor epoxide		
oxadiazon	12	11
PCB 1248	130	100
PCB 1254	170	190
PCB 1260	390	360
toxaphene	580	600
percent moisture	54.2	NA

SED = Sediment.

ND = Not Detected.

**TABLE R-9**

State Mussel Watch Program

Results of Duplicate Sample Analysis: 1995 Synthetic Organic Compounds Quality Control - Mussel Tissue (ng/g dry weight)

Station Name	SD Bay/Harbor Is/ E Basin/Storm Dr		Sandholt Bridge	
Station No.	894.0		404.0	
Species	TCM		TCM	
REPLICATE	1	2	1	2
<u>COMPOUNDS</u>				
aldrin				
cis-chlordane	25	27	31	33
cis-nonachlor	8.4	8.3	15	16
gamma-chlordene	2.2	3.2	1.2	1.0
alpha-chlordene	2.1	3.2	1.4	1.1
oxychlordane	5.2	5.7	1.3	4.2
trans-chlordane	25	26	25	27
trans-nonachlor	14	20	36	34
chlorpyrifos	ND	4.1	17	18
dacthal			140	140
DDD, o,p'	47	51	100	100
DDD, p,p'	140	150	400	420
DDE, o,p'			42	41
DDE, p,p'	30	39	1700	1600
DDT, o,p'	8.6	8.9	200	200
DDT, p,p'	32	41	680	730
DDMU,p,p'	23	27	55	54
diazinon				
dieldrin	6.0	6.4	300	300
endrin			22	23
endosulfan I			6.7	6.5
endosulfan II			22	20
endosulfan sulfate			82	95
heptachlor	1.9	ND		
hexachlorobenzene				
alpha-HCH				
gamma-HCH			0.91	0.87
heptachlor epoxide	1.3	ND	3.8	2.9
oxadiazon	2.0	2.0	7.3	7.6
PCB 1248	11,800	15,000		
PCB 1254	6,900	9,400	260	250
PCB 1260	210	250	18	16
toxaphene			870	930
percent moisture	87.4	87.2	85.9	86.3
percent lipid	0.441	0.412	0.844	0.838

TCM = Transplanted California Mussel.

ND = Not Detected.



**TABLE R-9 (continued)**

State Mussel Watch Program

Results of Duplicate Sample Analysis: 1995 Synthetic Organic Compounds Quality Control - Sediment  
(ng/g dry weight)

Station Name	Mugu Drainage 1	
Station No.	508.0 SED	
REPLICATE	1	2
COMPOUNDS		
aldrin		
cis-chlordane	6.7	6.7
cis-nonachlor	ND	3.4
gamma-chlordene		
alpha-chlordene	0.75	0.74
oxychlordane		
trans-chlordane	4.7	5.1
trans-nonachlor	5.5	5.8
chlorpyrifos	10	10
dacthal	53	52
DDD, o,p'	10	13
DDD, p,p'	44	44
DDE, o,p'	5.4	5.8
DDE, p,p'	340	300
DDT, o,p'	16	21
DDT, p,p'	71	77
DDMU,p,p'		
diazinon		
dieldrin	4.1	4.1
ethion	26	20
endrin	8.2	7.6
endosulfan I		
endosulfan II	3.1	2.9
endosulfan sulfate		
hexachlorobenzene	ND	0.43
alpha-HCH		
gamma-HCH		
heptachlor epoxide		
oxadiazon	13	13
PCB 1248		
PCB 1254		
PCB 1260		
toxaphene 320	320	
percent moisture	37.9	37.7

SED = Sediment.

ND = Not Detected.

**TABLE R-10**

State Mussel Watch Program  
Polynuclear Aromatic Hydrocarbons (PAHs) Analyzed  
and Their Detection Limits in Mussel and Sediment

Compound	Detection Limit (ng/g, ppb dry weight)
naphthalene	10
1-methylnaphthalene	10
2-methylnaphthalene	10
biphenyl	10
2,6-dimethylnaphthalene	10
acenaphthylene	10
acenaphthene	10
2,3,5-trimethylnaphthalene	10
fluorene	10
phenanthrene	10
anthracene	10
1-methylphenanthrene	10
fluoranthene	10
pyrene	10
benz[a]anthracene	10
chrysene	10
benzo[b]fluoranthene	10
benzo[k]fluoranthene	10
benzo[e]pyrene	10
benzo[a]pyrene	10
perylene	10
indeno[1,2,3-cd]pyrene	10
dibenz[a,h]anthracene	10
benzo[ghi]perylene	10

**TABLE R-11**

State Mussel Watch Program  
 Results of Duplicate Sample Analysis: 1994 Polynuclear Aromatic Hydrocarbons Quality Control  
 Mussel Tissue  
 (ng/g dry weight)

Station Name	Huntington Harbor/ Edinger Street 713.0 TCM		Blind 4 713.0 TCM	
Station No.				
Species				
REPLICATE	1	2	1	2
<u>COMPOUNDS</u>				
naphthalene	33	34	31	37
1-methylnaphthalene				
2-methylnaphthalene	33	35	24	19
biphenyl				
2,6-dimethylnaphthalene				
acenaphthylene				
acenaphthene				
2,3,5-trimethylnaphthalene				
fluorene				
phenanthrene	36	33	15	13
anthracene				
1-methylphenanthrene				
fluoranthene	69	67		
pyrene	ND	78	ND	26
benz[a]anthracene				
chrysene	55	44		
benzo[b]fluoranthene				
benzo[k]fluoranthene				
benzo[e]pyrene				
benzo[a]pyrene				
perylene				
indeno[1,2,3-cd]pyrene				
dibenz[a,h]anthracene				
benzo[ghi]perylene				
percent moisture	88.2	88.5	76.1	NA
percent lipid	0.443	0.484	1.59	NA

TCM = Transplanted California Mussel. ND = Not Detected. NA = Not Analyzed.

**TABLE R-12**

State Mussel Watch Program  
 Results of Duplicate Sample Analysis: 1994 Polynuclear Aromatic Hydrocarbons Quality Control  
 Sediment  
 (ng/g dry weight)

Station Name	LA Harbor/ National Steel 601.0 SED	
Station No.	1	2
<u>REPLICATE</u>		
<u>COMPOUNDS</u>		
naphthalene	34	36
1-methylnaphthalene	16	14
2-methylnaphthalene	51	54
biphenyl	20	14
2,6-dimethylnaphthalene	27	18
acenaphthylene	ND	ND
acenaphthene	18	17
2,3,5-trimethylnaphthalene	11	11
fluorene	24	24
phenanthrene	180	200
anthracene	100	110
1-methylphenanthrene	21	26
fluoranthene	390	420
pyrene	380	490
benz[a]anthracene	230	260
chrysene	430	490
benzo[b]fluoranthene	740	830
benzo[k]fluoranthene	150	190
benzo[e]pyrene	350	400
benzo[a]pyrene	440	510
perylene	120	130
indeno[1,2,3-cd]pyrene	140	120
dibenz[a,h]anthracene	75	46
benzo[ghi]perylene	110	120
percent moisture	46.2	45.8

SED = Sediment.

ND = Not Detected.

**TABLE R-13**

State Mussel Watch Program  
 Results of Duplicate Sample Analysis: 1995 Polynuclear Aromatic Hydrocarbons Quality Control  
 Mussel Tissue\*  
 (ng/g dry weight)

Station Name	Mission Bay/ Landfill 2	
Station No.	868.6 TCM	
REPLICATE	1	2
<u>COMPOUNDS</u>		
naphthalene	41	41
1-methylnaphthalene	24	25
2-methylnaphthalene	100	100
biphenyl		
2,6-dimethylnaphthalene	13	12
acenaphthylene	5.2	6.0
acenaphthene		
2,3,5-trimethylnaphthalene		
fluorene		
phenanthrene	18	23
anthracene		
1-methylphenanthrene		
fluoranthene	28	33
pyrene	23	34
benz[a]anthracene		
chrysene	15	ND
benzo[b]fluoranthene		
benzo[k]fluoranthene		
benzo[e]pyrene		
benzo[a]pyrene		
perylene		
indeno[1,2,3-cd]pyrene		
dibenz[a,h]anthracene		
benzo[ghi]perylene		
percent moisture	NA	NA

TCM = Transplanted California Mussel.

ND = Not Detected.

NA = Not Analyzed.

\* Duplicate sample analysis was not performed on sediment in 1995.

**TABLE R-14**

State Mussel Watch Program  
 Results of Matrix Spike Analyses: 1993-95 Polynuclear Aromatic Hydrocarbons (PAHs)

Station Name	1994	1995
Station Number	LA Harbor/Cabrillo Pier	Trinidad Head
Species	605.0 SED	10.0 RCM
	Percent Recovery*	Percent Recovery
<b>COMPOUNDS</b>		
naphthalene	100	100
1-methylnaphthalene	80	not spiked
biphenyl	63	not spiked
2,6-dimethylnaphthalene	30	not spiked
acenaphthylene	86	110
acenaphthene	121	120
2,3,5-trimethylnaphthalene	63	not spiked
fluorene	100	130
phenanthrene	100	130
anthracene	96	110
1-methylphenanthrene	140	not spiked
fluoranthene	120	120
pyrene	200	100
benz[a]anthracene	180	130
chrysene	230	130
benzo[b]fluoranthene	360	100
benzo[k]fluoranthene	160	100
benzo[e]pyrene	210	not spiked
benzo[a]pyrene	250	100
perylene	410	not spiked
indeno[1,2,3-cd]pyrene	160	110
dibenz[a,h]anthracene	110	120
benzo[ghi]perylene	200	120

SED = Sediment.

RCM = Resident California Mussel.

\* The percent recovery of several spiked PAHs exceeded 150% in the 1994 sediment sample. The unspiked sample contained concentrations of these PAH compounds at 2.5 to 10 times the amount spiked. For example, the concentration of perylene in the unspiked sample was 3.47 ppm while the amount of perylene spiked was only 0.332 ppm. The recovery of 2,6-dimethylnaphthalene was low, reported concentrations for this compound are qualified as estimates only.