# APPENDIX U

## 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 (i.e. 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$^{R}$ polyethylene bag of 4 mm thickness. The samples are placed inside two additional polyethylene ZIPLOCK$^{R}$ 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$^{o}$C or by rinsing in hexane. Samples in the foil bags are placed in two polyethylene ZIPLOCK$^{R}$ bags. After bagging, all samples are placed in non-metallic ice chests and frozen using dry ice and stored at or below -20$^{o}$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 1995-96 and 1996-97 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$^{R}$-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$^{o}$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 U-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 HNO3: 3 ml concentrated HClO4 mixture and heating the sample on a warm (75$^{o}$C) hotplate 2-3 hours. After the initial reaction, the Teflon vessel was capped and heated in a 130$^{o}$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 ll 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$^{o}$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$^{o}$C oven for 12 hours. Twenty ml of boric acid (2.5%) was added to each sediment sample before again returning to a 130$^{o}$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 ll 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 U-2).

## Synthetic Organic Compounds Analytical Techniques in Tissues - 1996

A 50 gram sample of tissue was spiked with a surrogate mixture of 4,4'-dibromo-octafluorobiphenyl, decachlorobiphenyl, and dibutylchlorendate (DBOB, DCB, DBCE) and extracted twice with acetonitrile by shaking for two hours on an orbital shaker at 300 rpm. The sample extracts were combined, filtered, and partitioned with petroleum ether. An aliquot of the petroleum ether extract was eluted through a Florisil$^{R}$ column. The Florisil$^{R}$ 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$^{R}$ 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 U-5 for 1996. A mixture of synthetic standards was eluted through the Florisil$^{R}$ column to determine the recovery and separation characteristics of the column. The distribution of synthetic organic compounds in the three fractions are listed in Table U-3. The detection limits for synthetic organics in mussels are presented in Table U-4.

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$^{R}$ with 50 percent ethyl ether/petroleum ether (Fraction 4, Table U-3). All other stations were analyzed for endosulfan I only. This fraction was also spiked with decachlorobiphenyl prior to the concentration step. All of the 50 percent extracts were diluted with iso-octane by a factor of ten prior to analysis by gas chromatography because of the high lipid content of the fraction.

## Procedure for Lipid Determination

Synthetic organic concentrations in organisms vary with lipid content so 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 sodium sulfate. 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 blowdown. 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.

## SMW Samples Analyzed with the Bay Protection Toxic Cleanup Program

The Water Pollution Control Laboratory analyzed sediment samples for the Bay Protection and Toxic Cleanup Program - Group 17b. This group of samples also included sediment samples from the 1995 SMW program. These samples were analyzed for chlorinated hydrocarbons, PCBs and PAHs.

## Polynuclear Aromatic Hydrocarbon Compounds (PAHs) and Chlorinated Hydrocarbons Analytical Techniques in Sediment

Thawed samples were stirred to a homogeneous appearance and two sample aliquots were removed, one for percent moisture determination (5 grams) and one for chemical analysis (20 grams). Sodium sulfate, activated copper and extraction surrogates were added to the bottle containing the 20 gram analytical sample which was then extracted three times with methylene chloride. After combining the three extraction aliquots, the extract was divided into two portions, one for chlorinated hydrocarbon (CH) analysis and the other for polycyclic aromatic hydrocarbon (PAH) analysis.

The CH portion was eluted through an activated Florisil column, separating the analytes into two fractions. Fraction 1 analytes were eluted with petroleum ether and fraction 2 analytes were eluted with 50% ethyl ether in petroleum ether. The two fractions were concentrated to 2 mL final volume using a Zymark Turbovap II evaporator.

The PAH aliquot was eluted through a silica gel/alumina column with methylene chloride. The extract was concentrated to 1 mL final volume using Kuderna-Danish (K-D) apparatus and a heated water bath.
Two sediment samples were spiked with solutions containing known concentrations of target analytes (chlorinated hydrocarbons, PCB congeners and PAHs) to assess accuracy and matrix effects. Percent recoveries for these analytes can be found in Tables U-10 (chlorinated hydrocarbons), Table U-11 (PCB congeners) and Table U-12 (PAHs).

Method blanks, representative of all materials and solutions contacting the sample, were prepared and analyzed. To preclude errors due to contamination, a vertical solvent was blank was prepared for each set of glassware used in the extraction and analyzed before introducing a new sample.

## Synthetic Organic Compounds Analytical Techniques in Tissue - 1997

A 25 gram sample of tissue was spiked with a synthetic organic surrgate solution (DBOB, PCB congener 207, DBCE). The sample was dried with sodium sulfate and extracted three times by shaking with methylene chloride on an orbital shaker at 300 rpm. After combining the extracts, the sample was evaportated in a Zymark$^{R}$ Turbovap to 10 mLs. A 2 mL aliquot was removed and placed in a tared planchett for lipid analysis. After the solvent evaporated, the planchett was briefly dried in a 70$^{o}$C oven to remove residual water. The percent lipid was then calculated. A 4 mL aliquot was eluted through a Florisil column as described above and four fractions (0%, 6%, 15%, and 50% petroleum ether/ethyl ether) were collected. The percent recoveries for the surrogate compounds are listed in Table U-6.

Mussel samples were spiked in duplicate with a solution containing known concentrations of target analytes to asses accuracy and matrix effects. Percent recoveries of the target analytes from the matrix spikes for 1996 and 1997 are listed in Table U-7.

Approximately 10 percent of samples were analyzed in duplicate to determine method precision. Precision results are listed in Table U-8 (1996) and Table U-9 (1997). Method blanks, representative of all materials and solutions contacting the sample, were prepared and analyzed. To preclude errors due to contamination, a vertical solvent was blank was prepared for each set of glassware used in the extraction and analyzed before introducing a new sample.

## Polynuclear Aromatic Hydrocarbon Compounds (PAHs) Analytical Techniques in Tissue

Ten grams of tissue was spiked with a PAH surrogate solution that contains eight deuterated PAH compounds. The sample was dried, placed in a stainless steel cell and extracted by forcing heated solvents (1:1 solution of methylene chloride:acetone) under pressure through the sample. The extract was solvent exchanged into methylene chloride and eluted through a gel permeation chromatograph to remove most of the lipids. The extract was further cleaned up by eluting it through a silica gel/alumina column.

Table U-13 lists the PAH dry weight detection limits for tissue. PAH surrogate recoveries are listed in Table U-14. Tables U-15 and U-16 list the matrix spike recoveries and duplicate analysis results respectively.

## Instrument and Analytical Conditions for Chlorinated Hydrocarbons

Sample extracts for chlorinated hydrocarbons were analyzed using a Varian Model 3500 gas chromatograph equipped with a Model 8035 autosampler, temperature programmable on-column injector, and dual Ni63 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 0.25 um 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 sample extracts were analyzed with a single injection using the following conditions:

| | |
|---|---|
| Injector temperature program: | Initial temperature - 70°C<br>Program rate - 300°C/min<br>Final temperature - 280°C<br>Final temperature hold time – 70 min |
| Column temperature program: | Initial temperature - 70°C<br>Program rate 1 - 15°C/min to 210°C<br>Program 1 hold time - 10 min<br>Program rate 2 - 2°C/min to 280°C |
| Final temperature hold time: | 11 min |
| | Detector temperature: 330°C |

**Instrument and Analytical Conditions for Polynuclear Aromatic Hydrocarbon Compounds (PAHs)**

Sample extracts were analyzed for PAH compounds using a Varian Saturn 4D Ion Trap GC-MS. Two microliters of sample extract were injected into a J&W Scientific DB-5MS, 60 meter x 0.25 mm I.D. fused silica capillary column with a 0.25 um 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. Injector temperature was isothermal at 300°C. The GC carrier gas was helium at a linear velocity of 37 cm/sec. Detection limits of the PAHs are reported in Table U-10. Results of duplicate analyses for PAHs in mussel and sediment are listed in Tables U-11, U-12, and U-13. Matrix spike recoveries for mussel tissue and sediment are listed in Table U-14.

**State Mussel Watch Samples Analyzed with the Bay Protection Toxic Cleanup Program - Long Marine Laboratory (LML)**

The Long Marine Laboratory in Santa Cruz analyzed tissue and sediment samples for the Bay Protection Toxic Cleanup Program - Group 14 and 17a. This group of samples also included several tissue and sediment samples from the 1996 and 1997 SMW program. The following are descriptions of methods used to analyze theses samples for PAHs and chlorinated hydrocarbons.

**Analytical Techniques for Polynuclear Aromatic Hydrocarbon Compounds (PAHs) and Chlorinated Hydrocarbons (CH) Analyzed in Tissues by the Long Marine Laboratory**

A 5 gram sample of tissue is extracted two times with methylene chloride using a Tekmar Tissumizer®. Prior to extraction, sodium sulfate and

extraction surrogates are added to the sample. After combining the two extraction aliquots, the extract is divided into three portions: one for lipid weight determination, one for chlorinated hydrocarbon (CH) analysis, and one for PAHs.

The CH portion is eluted through a silica/alumina column, separating the analytes into two fractions (Table U-17). Fraction 1 (F1) analytes are eluted using 1% methylene chloride in pentane and contains > 90% of p,p'-DDE and < 10% of p,p'-DDT. Fraction 2 (F2) analytes are eluted using 1% acetone in methylene chloride. The F2 fraction undergoes additional cleanup using size-exclusion High Performance Liquid Chromatography (HPLC/SEC). The F1 and F2 fractions are concentrated to 125 uL using a combination of rotary evaporator, tube heater, and nitrogen gas evaporation.

The PAH aliquot is eluted through a silica/alumina column using 100% methylene chloride. The eluate is further cleaned using HPLC/SEC. The PAH fraction is concentrated to 125 uL, using a combination of rotary evaporator, tube heater, and nitrogen gas evaporation.

Table U-18 lists the dry weight detection limits for chlorinated hydrocarbons in sediment and tissue; Table U-19 lists the dry weight detection limits for PAHs in tissue and sediments.

Analytical Techniques for Polynuclear Aromatic Hydrocarbon Compounds (PAHs) and Chlorinated Hydrocarbons (CH) Analyzed in Sediment by the Long Marine Laboratory

Samples are removed from the freezer and allowed to thaw. Each sample is stirred to a homogeneous appearance and two 10 gram sample aliquots are removed, one for dry weight determination and one for chemical analysis.

The dry weight sample is placed into a pre-weighed aluminum planchet and dried at 100˚C for 24 hrs. The dried sample is re-weighed to determine the sample's percent moisture.

The analytical sample is extracted three times with methylene chloride in a 250-mL amber Boston round bottle on a modified rock tumbler. Prior to rolling, sodium sulfate, copper, and extraction surrogates are added to the bottle. The sodium sulfate dehydrates the sample allowing for efficient sediment extraction. The copper, which is activated with hydrochloric acid, complexes free sulfur in the sediment.

After combining the three extraction aliquots, the extract is divided into two portions, one for chlorinated hydrocarbon (CH) analysis and the other for polycyclic aromatic hydrocarbon (PAH) analysis.

The CH portion is eluted through a silica/alumina column, separating the analytes into two fractions (Table U-17). Fraction 1 (F1) is eluted with 1% methylene chloride in pentane and contains > 90% of p,p'-DDE and < 10% of p,p'-DDT. Fraction 2 (F2) analytes are eluted with 1% acetone in methylene chloride. The two fractions are exchanged into hexane and concentrated to 500 uL using a combination of rotary evaporation, controlled boiling on tube heaters, and dry nitrogen blow downs. The F1 is treated a second time with a minimal amount of activated copper to ensure complete removal of sulfur prior to GC analysis.

The extract's PAH portion is eluted through a silica/alumina column with methylene chloride. The "cleaned" eluate is exchanged into hexane and concentrated to 500 uL in the same manner as the CH fractions.

## Instrument and Analytical Conditions (Long Marine Laboratory)

The F1 and F2 fractions are analyzed by Gas Chromatography for chlorinated hydrocarbon analysis utilizing an Electron Capture Detector (GC/ECD). A single 2 uL splitless injection is directed onto two columns of different polarity (DB-17 & DB-5) to provide two dimensional confirmation of each analyte. The lowest analytical results are reported.

The PAH fraction is analyzed by Gas Chromatography Mass Spectrometry (GC/MS) for aromatic hydrocarbon analysis. A single 2 uL splitless injection is chromatographed on a DB-5 ms column and analyzed in a single ion monitoring (SIM) mode.

All analytes of interest are quantified using internal standard methodologies and are corrected for surrogate recoveries.

Quality control (QC) measures are routinely performed during sample analyses as described in the Bay Protection and Toxic Cleanup Program Quality Assurance Project Plan (BPTCP QAPP)[1]. The QC measures include the tracking of accuracy and precision as performance indices, instrument calibration verification, the dilution of samples which exceed the instrument's calibrated range and the documentation of surrogate recoveries.

Instrumental calibration is verified with continuing calibration check (CCC) solutions every 10-16 hours. Solutions purchased from the National Institute of Standards and Technology (NIST) are used to prepare mid-level CCC solutions with each analytical batch. The stability of all non-NIST analyte calibrations are monitored through the analysis of mid-level standards on 16-20 hour intervals.

All surrogates are inspected for acceptable recoveries prior to sample analysis. Samples which have recoveries exceeding the criterion of 50%-150% are subjected to re-analysis or re-extraction. Marginal recoveries which are in control yet exceed the range of 60% - 120% are closely inspected and corrective action is taken as appropriate. All corrective action is fully documented.

Prior to GC analysis, samples are spiked with a 20 fold excess of dilution internal standards. Therefore, samples exceeding the calibrated range of the instrument are subjected to a standard 1:20 dilution and reanalyzed. Deuterated fluoranthene (d12-FLA) is utilized as the PAH DIL-Istd.

Tracking of analytical precision and accuracy is accomplished through the use of method duplicates, matrix spikes, and Standard Reference Materials (SRMs) at a minimum of 5% each. Matrix spikes provide a means for assessing methodological performance for analytes not found in available SRMs. Matrix spike recoveries are presented in Tables U-20 through U-23. Tissues are

[1] Stephenson, M., M. Pucket, N. Morgan, and M. Reid. 1994. Bay Protection and Toxic Cleanup Program: Quality Assurance Project Plan. Bay Protection and Toxic Cleanup Program, State Water Resources Control Board, Sacramento, CA.

enriched such that resulting analyte levels are greater than ten times the method detection limit.

Hard copies of all chromatograms, area percent reports, and internal standard reports are generated and archived for each sample analyzed. Additionally, all phases of the analysis are archived magnetically on mini data cartridges.

# TABLE U-1

State Mussel Watch Program
Trace Element Detection Limits

**Tissue and Sediment**

| Element | Detection Limit (ug/g, ppm dry weight) | (ug/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 U-2

State Mussel Watch Program
Trace Element Analysis of Reference Materials (*u*g/g, dry weight)*

| | **1995-96**** | **1996-97**** |
|---|---|---|
| | 1566a - NBS Oyster | 1566a - NBS Oyster |
| Ag | 1.51±0.16<br>(1.68±0.15) | 1.71±0.15<br>(1.68±0.15) |
| Al | 204±9.54<br>(202.5±14.1) | 201±7.74<br>(202.5±14.1) |
| As | 13.5±0.751<br>(14.0±1.2) | 11.8±0.141<br>(14.0±1.2) |
| Cd | 4.32±0.10<br>(4.15±0.38) | 4.39±0.10<br>(4.15±0.38) |
| Cr | 1.11±0.08<br>(1.43±0.46) | 1.14±0.04<br>(1.43±0.46) |
| Cu | 64.7±0.80<br>(66.3±4.3) | 65.2±0.39<br>(66.3±4.3) |
| Hg | 0.064±0.004<br>(0.064±0.007) | 0.0629±0.001<br>(0.064±0.007) |
| Mn | 12.1±0.954<br>(12.3±1.5) | 12.4±0.58<br>(12.3±1.5) |
| Ni | 2.19±0.22<br>(2.25±0.44) | 2.31±0.06<br>(2.25±0.44) |
| Pb | 0.429±0.011<br>(0.371±0.014) | 0.360±0.040<br>(0.371±0.014) |
| Se | 2.12±0.45<br>2.21±0.24 | 1.85±0.0<br>(2.21±0.24) |
| Zn | 840±21<br>(830±57) | 859±30.7<br>(830±57) |

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

# TABLE U-3

State Mussel Watch Program
Distribution of Synthetic Organic Compounds Among
Four Fractions of a Standard Florisil[R] 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 | (50%) Fraction 4 |
| | oxychlordane | |
| | toxaphene | endosulfan II |
| | | endosulfan sulfate |

* Found in both 0% and 6% fractions.

# TABLE U-4

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 |
| PCT 5460 | 10 |
| tetradifon (Tedion) | 10 |
| toxaphene | 100 |
| tributyltin | 20 |

# Table U-5

State Mussel Watch Program
Percent Recovery of Surrogate Compounds for 1996

| Station Number | Station Name | DBOB | DCB | DBCE |
|---|---|---|---|---|
| 202.0 | Bodega Head | 57 | 76 | 47 |
| 400.7 | Santa Cruz Harbor/Inner | 65 | 94 | 94 |
| 401.0 | Santa Cruz Harbor/Outer | 58 | 83 | 43 |
| 404.0 | Sandholt Bridge | 56 | 84 | 117 |
| 404.0 DUP | Santholt Bridge | 59 | 85 | 120 |
| 414.0 | Pacific Grove | 58 | 83 | 110 |
| 601.0 | LA Harbor/National Steel | 57 | 82 | 75 |
| 605.0 | LA Harbor/Cabrillo Pier | 59 | 78 | 97 |
| 616.0 | LA Harbor/Consolidated Slip | 50 | 83 | 46 |
| 618.0 | LA Harbor/Angels Gate | 59 | 64 | 71 |
| 648.0 | Malibu Pier | 62 | 62 | 91 |
| 650.0 | Santa Monica Pier | 59 | 57 | 78 |
| 662.0 | Royal Palms | 58 | 77 | 92 |
| 664.0 | Cabrillo Beach | 62 | 87 | 99 |
| 713.0 | Huntington Harbor/Edinger Street | 64 | 83 | 95 |
| 715.0 | Huntington Harbor/Warner Avenue Br. | 64 | 89 | 95 |
| 715.0 DUP | Huntington Harbor/Warner Avenue Br. | 60 | 83 | 91 |
| 723.4 | Newport Bay/Turning Basin | 53 | 82 | 90 |
| 724.0 | Newport Bay/Highway 1 Bridge | 59 | 83 | 85 |
| 725.0 | Newport Bay/Crows Nest | 53 | 81 | 64 |
| 726.4 | Newport Bay/Rhine Channel/End | 54 | 84 | 68 |
| 742.0 | San Juan Creek | 67 | 86 | 81 |
| 750.0 | Oceanside | 62 | 77 | 96 |
| 882.7 | San Diego Bay/Sampson Street Pier | 51 | 82 | 86 |
| 883.1 | San Diego Bay/Chollas Creek | 53 | 84 | 90 |
| 883.6 | San Diego Bay/Seventh Street Channel | 51 | 86 | 89 |
| 883.8 | San Diego Bay/Switzer Creek | 50 | 83 | 87 |
| 886.0 | San Diego Bay/NASSCO | 55 | 85 | 96 |
| 893.0 | Laurel Street Stormdrain | 58 | 84 | 94 |
| 893.5 | B Street Pier | 62 | 86 | 98 |
| 893.5 DUP | B Street Pier | 60 | 84 | 95 |

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

DUP = Duplicate analysis.

# Table U-6

State Mussel Watch Program
Percent Recovery of Synthetic Organic Surrogate Compounds for 1997

| Station Number | Station Name | PCB Congener DBOB | 207 | DBCE |
|---|---|---|---|---|
| 1.0 | Crescent City Harbor | 70 | 88 | 63 |
| 2.0 | Crescent City/STP Outfall | 66 | 87 | 61 |
| 2.1 | Crescent City Harbor Jetty | 79 | 97 | 77 |
| 3.0 | Crescent City/Control | 67 | 87 | 72 |
| 100.0 | Mad River Slough | 89 | 96 | 57 |
| 102.6 | Humboldt Bay/J Street | 82 | 98 | 59 |
| 103.3 | Humboldt Bay/E Street | 64 | 85 | 67 |
| 103.5 | Clark Slough | 67 | 86 | 58 |
| 202.0 | Bodega Bay | 64 | 82 | 64 |
| 205.0 | Bodega Bay/Spud Pt. Marina | 69 | 73 | 67 |
| 205.1 | Bodega Bay/Porto Bodega | 61 | 78 | 97 |
| 205.3 | Bodega Bay/Mason’s Marina | 67 | 65 | 71 |
| 205.5 | Bodega Bay/Back Marsh | 68 | 88 | 74 |
| 280.0 | Russian River/Goat Rock | 64 | 85 | 69 |
| 400.6 | Santa Cruz/Natual Bridges | 68 | 86 | 64 |
| 403.0 | Elkhorn Slough/Hwy. 1 Bridge | 61 | 54 | 66 |
| 404.0 | Sandholdt Bridge | 86 | 101 | 68 |
| 404.0 DUP | Sandholdt Bridge | 64 | 85 | 66 |
| 414.0 | Pacific Grove | 63 | 84 | 74 |
| 601.0 | LA Harbor/National Steel | 54 | 50 | 62 |
| 616.0 | LA Harbor/Consolidated Slip | 65 | 57 | 61 |
| 616.0 DUP | LA Harbor/Consolidated Slip | 69 | 87 | 53 |
| 648.0 | Malibu Pier | 75 | 93 | 56 |
| 650.0 | Santa Monica Bay | 65 | 52 | 65 |
| 662.0 | Royal Palms | 77 | 102 | 67 |
| 707.0 | Anaheim Bay/Navy Harbor | 59 | 79 | 64 |
| 707.0 Dup | Anaheim Bay/Navy Harbor | 51 | 54 | 63 |
| 708.0 | Anaheim Bay/Navy Marsh | 58 | 81 | 56 |
| 708.5 | Anaheim Bay/Navy Marsh 1 | 54 | 80 | 63 |
| 713.0 | Huntington Harbor/Edinger St. | 60 | 80 | 81 |
| 715.0 | Huntington Harbor/Warner Ave. Bridge | 69 | 94 | 76 |
| 725.0 | Newport Bay/Crow’s Nest | 68 | 90 | 55 |
| 726.4 | Newport Bay/Rhine Channel/End | 70 | 96 | 62 |
| 726.6 | Newport Bay/Mariners Drive | 71 | 95 | 76 |
| 740.0 | Dana Point Harbor/Boat Yard | 68 | 89 | 61 |
| 750.0 | Oceanside | 63 | 53 | 73 |
| 883.1 | San Diego/Chollas Creek | 61 | 86 | 72 |
| 883.2 | San Diego/Chollas Creek/Mouth | 64 | 83 | 68 |
| 883.3 | San Diego/Chollas Creek/Mouth | 69 | 93 | 78 |
| 883.5 | San Diego/Tuna Docks | 65 | 59 | 63 |
| 883.6 | San Diego/7$^{th}$ Street Channel | 58 | 58 | 69 |
| 883.8 | San Diego/Switzer Creek | 56 | 84 | 60 |
| 885.1 | San Diego Bay/Paleta Creek/End | 57 | 84 | 63 |
| 894.0 | S.D.Bay/Harbor Is./E.Basin/Storm Drain | 58 | 62 | 120 |

DBOB = 4,4'-dibromo-octafluorobiphenyl
DBCE = dibutylchlorendate

Dup = Duplicate analysis.

# TABLE U-7

State Mussel Watch Program
Results of Matrix Spike Analyses: 1996 and 1997
Synthetic Organic Compounds
Mussel Tissue

| | 1996 | | 1997 | |
|---|---|---|---|---|
| Station Name | Bodega Head | | Bodega Head | |
| Station Number | 202.0 | | 202.0 | |
| Species | RCM | | RCM | |
| | Percent Recovery | | Percent Recovery | |
| | MS | MSD | MS | MSD |
| Compound | | | | |
| aldrin | 69.0 | 71.2 | 84.0 | 90.4 |
| chlordane, cis | 84.7 | 89.0 | 66.4 | 80.6 |
| chlordane, trans | 81.4 | 86.1 | 54.0 | 77.1 |
| chlordene, alpha | 67.0 | 69.4 | 82.1 | 85.2 |
| chlordene, gamma | 72.0 | 72.7 | 85.8 | 83.0 |
| chlorpyrifos | 48.8 | 52.9 | 60.5 | 59.5 |
| dacthal | 62.8 | 65.1 | 35.8 | 25.9 |
| DDD, o,p′ | 91.3 | 97.0 | 78.4 | 86.6 |
| DDD, p,p′ | 92.2 | 97.9 | 78.6 | 82.7 |
| DDE, o,p′ | 89.2 | 91.1 | 83.1 | 81.8 |
| DDE, p,p′ | 87.1 | 87.8 | 66.8 | 75.1 |
| DDMU, p,p′ | 87.1 | 87.3 | 86.9 | 90.2 |
| DDT, o,p′ | 95.0 | 94.0 | 85.5 | 86.6 |
| DDT, p,p′ | 99.4 | 99.2 | 108.0 | 115.0 |
| diazinon | 59.6 | 62.7 | 26.0 | 25.9 |
| dieldrin | 59.6 | 61.2 | 45.2 | 47.9 |
| endosulfan I | 67.4 | 71.5 | 56.4 | 54.4 |
| endosulfan II | 78.6 | 82.7 | 70.8 | 71.5 |
| endosulfan sulfate | 79.1 | 83.6 | 71.1 | 87.8 |
| endrin | 83.1 | 84.9 | 55.8 | 46.6 |
| ethion | 75.5 | 77.1 | 125.0 | 133.0 |
| HCH, alpha | 48.6 | 52.0 | 48.3 | 70.5 |
| HCH, beta | 62.5 | 67.0 | 57.1 | 85.4 |
| HCH, delta | NR | NR | 39.0 | 30.6 |
| HCH, gamma | 62.4 | 64.0 | 63.1 | 75.3 |
| heptachlor | 49.3 | 53.3 | 42.8 | 20.9 |
| hexachlorobenzene | 61.6 | 62.7 | 73.4 | 80.2 |
| methoxychlor | 105.0 | 110.0 | 118.0 | 123.0 |
| nonachlor, cis | 94.7 | 101.0 | 84.1 | 92.4 |
| nonachlor, trans | 87.9 | 89.3 | 80.9 | 79.1 |
| oxadiazon | 67.2 | 67.9 | 48.6 | 46.3 |
| oxychlordane | 62.6 | 65.9 | 29.2 | 77.4 |
| parathion, ethyl | 55.4 | 57.6 | 38.9 | 47.0 |
| parathion, methyl | 49.7 | 52.1 | 33.1 | 30.3 |
| tedion | 56.2 | 57.1 | 56.7 | 50.3 |
| PCB 1254 | 72.0 | 74.8 | 71.0 | 89.8 |

RCM = Resident California Mussel.

# TABLE U-8

State Mussel Watch Program
Results of Duplicate Sample Analysis
1996 Synthetic Organic Compounds Quality Control - Mussel Tissue
(ng/g dry weight)

| Station Name | Sandholdt Bridge | | Hunting Harbor/ Warner Ave. Bridge | | San Diego Bay/ B Street Pier | |
|---|---|---|---|---|---|---|
| Station Number | 404.0 | | 715.0 | | 893.5 | |
| Species | TCM | | TCM | | TCM | |
| REPLICATE | 1 | 2 | 1 | 2 | 1 | 2 |
| Compound | | | | | | |
| Aldrin | | | | | | |
| chlordane, cis | 33.9 | 32.4 | 30.7 | 30.8 | 8.80 | 8.07 |
| chlordane, trans | 29.1 | 28.5 | 30.6 | 29.6 | 7.04 | 6.64 |
| chlordene, alpha | | | 1.96 | 1.84 | | |
| chlordene, gamma | | | 1.49 | 1.55 | | |
| chlorpyrifos | 14.2 | 11.9 | 4.91 | 5.70 | | |
| dacthal | 47.4 | 47.5 | | | | |
| DDD, o,p' | 89.0 | 85.3 | 18.8 | 18.1 | | |
| DDD, p,p' | 264 | 254 | 39.5 | 38.5 | 8.45 | 6.84 |
| DDE, o,p' | 45.1 | 44.5 | 6.06 | 5.50 | | |
| DDE, p,p' | 1870 | 1830 | 248 | 228 | 34.3 | 34.0 |
| DDMU, p,p' | 55.6 | 58.6 | 10.1 | 8.18 | | |
| DDT, o,p' | 98.9 | 116 | | | | |
| DDT, p,p' | 756 | 731 | 5.15 | 4.40 | 5.16 | 4.91 |
| diazinon | | | | | | |
| dieldrin | 231 | 229 | 8.55 | 9.10 | 4.47 | 2.27 |
| endosulfan I | 3.71 | 1.44 | | | | |
| endosulfan II | 10.2 | 9.5 | | | | |
| endosulfan sulfate | | | | | | |
| endrin | 28.8 | 27.1 | | | | |
| ethion | | | | | | |
| HCH, alpha | | | | | | |
| HCH, beta | | | | | <RL | 5.07 |
| HCH, delta | | | | | | |
| HCH, gamma | | | | | | |
| heptachlor | | | | | | |
| heptachlor epoxide | 3.39 | 3.52 | 1.09 | 0.905 | | |
| hexachlorobenzene | | | | | | |
| methoxychlor | | | | | | |
| nonachlor,cis | 16.8 | 14.6 | 18.4 | 17.8 | 6.08 | 5.41 |
| nonachlor, trans | 29.3 | 31.8 | 30.0 | 29.3 | 6.73 | 6.09 |
| oxadiazon | 7.25 | 4.64 | 2.13 | 2.39 | | |
| oxychlordane | | | 1.43 | 1.33 | | |
| parathion, ethyl | | | | | | |
| parathion, methyl | | | | | | |
| tedion | | | | | | |
| toxaphene | 1670 | 1460 | | | | |
| PCB 1254 | 247 | 277 | 184 | 161 | 428 | 427 |
| PCB 1260 | | 15.2 | 15.4 | | | |
| Percent moisture | 86.1 | 86.3 | 87.9 | 88.3 | 85.8 | 86.3 |
| Percent lipid | 0.922 | 0.987 | 0.456 | 0.449 | 0.522 | 0.474 |

TCM = Transplanted California Mussel.

# TABLE U-9

State Mussel Watch Program
Results of Duplicate Sample Analysis
1997 Synthetic Organic Compounds Quality Control - Mussel Tissue
(ng/g dry weight)

| Station Name | Sandholdt Bridge | | LA Harbor/ Consolidated Slip | | Anaheim Bay/ Navy Harbor | |
|---|---|---|---|---|---|---|
| Station Number | 404.0 | | 616.0 | | 707.0 | |
| Species | TCM | | TCM | | TCM | |
| REPLICATE | 1 | 2 | 1 | 2 | 1 | 2 |
| Compound | | | | | | |
| Aldrin | | | | | | |
| chlordane, cis | 38.2 | 34.6 | 23.3 | 24.8 | 17.4 | 17.4 |
| chlordane, trans | 32.9 | 25.8 | 19.2 | 20.7 | 17.3 | 16.4 |
| chlordene, alpha | 2.24 | 1.69 | 1.31 | 1.96 | 1.49 | ND |
| chlordene, gamma | | | | | | |
| chlorpyrifos | 9.51 | 8.93 | ND | 5.32 | 9.07 | 8.28 |
| dacthal | 23.8 | 26.2 | 2.57 | 2.78 | | |
| DDD, o,p' | 89.8 | 83.5 | 20.0 | 20.9 | 12.3 | 12.1 |
| DDD, p,p' | 384 | 368 | 63.0 | 68.2 | 25.7 | 22.9 |
| DDE, o,p' | 38.2 | 31.1 | 8.19 | 16.6 | 14.9 | 9.41 |
| DDE, p,p' | 2000 | 1500 | 272 | 228 | 271 | 240 |
| DDMU, p,p' | 43.8 | 38.0 | 15.8 | 16.5 | 20.9 | 17.2 |
| DDT, o,p' | 214 | 152 | 4.16 | 6.06 | 5.08 | ND |
| DDT, p,p' | 785 | 728 | 39.6 | 41.7 | 24.9 | 24.5 |
| diazinon | | | | | | |
| dieldrin | 238 | 230 | 7.68 | 8.16 | 10.1 | 10.5 |
| endosulfan I | 2.77 | 3.81 | | | | |
| endosulfan II | 10.9 | 11.7 | | | | |
| endosulfan sulfate | | | | | | |
| endrin | 18.7 | 18.1 | | | | |
| ethion | | | | | | |
| HCH, alpha | | | | | | |
| HCH, beta | | | | | | |
| HCH, delta | | | | | | |
| HCH, gamma | | | | | | |
| heptachlor | | | | | | |
| heptachlor epoxide | 1.14 | 1.38 | | | | |
| hexachlorobenzene | | | | | | |
| methoxychlor | | | | | | |
| nonachlor,cis | 19.0 | 17.4 | 10.4 | 11.3 | 9.12 | 7.4 |
| nonachlor, trans | 38.7 | 30.2 | 15.5 | 18.4 | 16.1 | 11.4 |
| oxadiazon | | | 25.3 | 27.0 | 6.6 | 3.99 |
| oxychlordane | | | | | | |
| parathion, ethyl | | | | | | |
| parathion, methyl | | | | | | |
| tedion | | | | | | |
| toxaphene | 1970 | 1940 | 342 | 348 | | |
| PCB 1248 | | | 54.9 | 89.8 | | |
| PCB 1254 | 243 | 152 | 372 | 460 | | |
| PCB 1260 | 34.4 | 25.7 | 26.8 | 28.3 | | |
| Percent moisture | 88.4 | 88.4 | 87.5 | 87.4 | 83.6 | 83.6 |
| Percent lipid | 0.844 | 0.853 | 0.819 | 0.877 | 1.39 | 1.33 |

TCM = Transplanted California Mussel.

# TABLE U-10

Bay Protection Toxic Cleanup - Group 17b
Results of Matrix Spike Analyses in Sediment: Chlorinated Hydrocarbons

| Compound | Matrix Spike Percent Recovery | Matrix Spike Duplicate Percent Recovery |
|---|---|---|
| Aldrin | 66 | 64 |
| Chlordane, cis | 52 | 51 |
| Chlordane, trans | 83 | 82 |
| Chlordene, alpha | 73 | 72 |
| Chlordene, gamma | 70 | 68 |
| Chlorpyrifos | 117 | 115 |
| Dacthal | 85 | 83 |
| DDD, o,p’ | 93 | 91 |
| DDD, p,p’ | 79 | 78 |
| DDE, o,p’ | 81 | 80 |
| DDE, p,p’ | 85 | 84 |
| DDT, o,p’ | 85 | 84 |
| DDT, p,p’ | 90 | 88 |
| DDMU, p,p’ | 87 | 85 |
| Oxadiazon | 91 | 89 |
| Dieldrin | 94 | 93 |
| Endosulfan I | 83 | 82 |
| Endosulfan II | 92 | 90 |
| Endosulfan sulfate | 95 | 93 |
| Endrin | 77 | 75 |
| HCH, alpha | 70 | 61 |
| HCH, beta | 117 | 114 |
| HCH, gamma | 63 | 62 |
| HCH, delta | 82 | 80 |
| Heptachlor | 43 | 42 |
| Heptachlor epoxide | 83 | 82 |
| Hexachlorobenzene | 65 | 64 |
| Methoxychlor | 89 | 87 |
| Nonachlor, cis | 88 | 86 |
| Nonachlor, trans | 84 | 82 |
| Oxychlordane | 82 | 80 |

# TABLE U-11

Bay Protection Toxic Cleanup - Group 17b
Results of Matrix Spike Analyses in Sediment: PCB Congeners

| PCB Congener No. | Matrix Spike Percent Recovery | Matrix Spike Duplicate Percent Recovery |
|---|---|---|
| 8 | 64 | 67 |
| 18 | 70 | 73 |
| 28 | 75 | 75 |
| 44 | 84 | 83 |
| 52 | 77 | 74 |
| 66 | 88 | 87 |
| 101 | 81 | 80 |
| 105 | 88 | 91 |
| 118 | 88 | 87 |
| 128 | 89 | 90 |
| 138 | 83 | 82 |
| 153 | 82 | 88 |
| 170 | 99 | 98 |
| 180 | 92 | 90 |
| 187 | 90 | 98 |
| 195 | 96 | 95 |
| 206 | 98 | 97 |

# TABLE U-12

Bay Protection Toxic Cleanup - Group 17b
Results of Matrix Spike Analyses in Sediment: PAHs

| Compound | Matrix Spike Percent Recovery | Matrix Spike Duplicate Percent Recovery |
|---|---|---|
| naphthalene | 102 | 102 |
| 1-methylnaphthalene | 100 | 99 |
| 2-methylnaphthalene | 101 | 102 |
| biphenyl | 96 | 95 |
| 2,6-dimethylnaphthalene | 91 | 91 |
| acenaphthylene | 98 | 99 |
| acenaphthene | 96 | 97 |
| 2,3,5-trimethylnaphthalene | 100 | 102 |
| fluorene | 102 | 100 |
| phenanthrene | 105 | 101 |
| anthracene | 117 | 104 |
| 1-methylphenanthrene | 129 | 123 |
| fluoranthene | 96 | 75 |
| pyrene | 95 | 79 |
| benzo[a]anthracene | 112 | 93 |
| chrysene | 114 | 93 |
| benzo[e]fluroanthene | 195 | 140 |
| benzo[k]fluroanthene | 24 | 40 |
| benzo[e]pyrene | 117 | 98 |
| benzo[a]pyrene | 126 | 102 |
| perylene | 98 | 83 |
| indeno[123-cd)pyrene | 122 | 78 |
| dibenzo[a]anthracene | 106 | 82 |
| benzo[g,h,i]perylene | 117 | 88 |

# TABLE U-13

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 U-14

State Mussel Watch Program
Percent Recovery of Deuterated PAH Surrogate Compounds for 1997

| Station Number | Station Name | d8-NPH | d10-BPH | d10-ACE | d10-PYR | d12-BAA | d12-BEP | d12-PER | d12-BGP |
|---|---|---|---|---|---|---|---|---|---|
| 1.0 | Crescent City Harbor | 60 | 107 | 93 | 135 | 230 | 134 | 106 | 123 |
| 2.1 | Crescent City/Harbor Jetty | 72 | 113 | 119 | 119 | 136 | 123 | 103 | 109 |
| 3.0 | Crrescent City Control | 29 | 45 | 60 | 52 | 69 | 58 | 56 | 40 |
| 100.0 | Mad River Slough | 25 | 45 | 60 | 47 | 76 | 65 | 64 | 53 |
| 102.6 | Humboldt Bay/J Street | 52 | 65 | 63 | 59 | 66 | 50 | 44 | 33 |
| 103.3 | Humboldt Bay/E Street | 26 | 89 | 96 | 93 | 119 | 104 | 99 | 88 |
| 103.5 | Clark Slough | 44 | 54 | 62 | 78 | 72 | 66 | 63 | 64 |
| 202.0 | Bodega Bay | 20 | 47 | 61 | 57 | 68 | 62 | 57 | 56 |
| 205.0 | Bodega Bay/Spud Point Marina | 56 | 83 | 102 | 99 | 82 | 77 | 67 | 71 |
| 205.1 | Bodega Bay/Port Bodega | 34 | 70 | 75 | 65 | 94 | 62 | 64 | 47 |
| 205.3 | Bodega Bay/Mason’s Marina | 10 | 52 | 54 | 51 | 73 | 64 | 58 | 55 |
| 205.5 | Bodega Bay/Back Marsh | 19 | 59 | 81 | 73 | 101 | 86 | 80 | 61 |
| 280.0 | Russian River/Goat Rock | 49 | 90 | 136 | 120 | 148 | 142 | 132 | 120 |
| 400.6 | Santa Cruz/Natural Bridges | 40 | 82 | 99 | 91 | 118 | 89 | 85 | 65 |
| 403.0 | Elkhorn Slough/Hwy 1 Bridge | 55 | 96 | 115 | 101 | 119 | 84 | 84 | 62 |
| 414.0 | Pacific Grove | 66 | 81 | 85 | 104 | 96 | 95 | 69 | 100 |
| 404.0 | Sandholdt Bridge | 67 | 139 | 143 | 141 | 171 | 132 | 124 | 105 |
| 404.0 Dup | Sandholdt Bridge | 55 | 128 | 132 | 135 | 156 | 134 | 125 | 111 |
| 650.0 | Santa Monica Pier | 16 | 45 | 61 | 60 | 76 | 65 | 61 | 56 |
| 707.0 | Anaheim Bay/Navy Harbor | 6 | 85 | 115 | 103 | 188 | 130 | 119 | 132 |
| 707.0 Dup | Anaheim Bay/Navy Harbor | 28 | 79 | 93 | 83 | 116 | 92 | 87 | 71 |
| 708.5 | Anaheim Bay/Navy Marsh 1 | 19 | 50 | 77 | 74 | 100 | 76 | 77 | 63 |
| 740.0 | Dana Point Harbor/Boat Yard | 62 | 78 | 95 | 126 | 108 | 107 | 101 | 102 |
| 750.0 | Oceanside | 62 | 75 | 85 | 100 | 92 | 92 | 62 | 89 |
| 883.1 | San Diego Bay/Chollas Ck | 73 | 74 | 88 | 103 | 91 | 90 | 71 | 67 |
| 883.2 | San Diego Bay/Chollas Ck/Mouth | 82 | 82 | 88 | 108 | 98 | 81 | 75 | 63 |
| 883.3 | San Diego Bay/Chollas Ck/End | 71 | 69 | 84 | 110 | 74 | 77 | 53 | 70 |
| 883.5 | San Diego Bay/Tuna Docks | 73 | 77 | 89 | 107 | 94 | 88 | 77 | 82 |
| 883.6 | San Diego Bay/7$^{th}$ St. Channel | 75 | 78 | 87 | 97 | 86 | 80 | 72 | 73 |
| 883.8 | San Diego Bay/Switzer Ck | 63 | 76 | 82 | 93 | 77 | 72 | 54 | 62 |
| 885.1 | San Diego Bay/Paleta Ck/End | 70 | 80 | 90 | 99 | 54 | 61 | 48 | 51 |
| 885.3 | San Diego Bay/7$^{th}$ St Ch/Mid | 66 | 72 | 76 | 105 | 96 | 90 | 79 | 78 |
| 888.0 | San Diego Bay/Coronado Bridge | 38 | 43 | 46 | 64 | 49 | 60 | 34 | 50 |
| 894.0 | SD Bay/Harbor Is/E.Basin/Storm | 61 | 66 | 74 | 96 | 78 | 76 | 58 | 67 |

d8-NPH = naphthalene-d8
d10-BPH = biphenyl-d10
d10-ACE = acenaphthene-d10
d10-PYR = pyrene-d10
d12-BAA = benzo[a]anthracene-d12
d12-BEP = benzo[e]pyrene-d12
d12-PER = perylene-d12
d12-BGP = benzo[g,h,i]perylene-d12

DUP = Duplicate analysis.

# TABLE U-15

State Mussel Watch Program
Results of Matrix Spike Analyses: 1997 Polynuclear Aromatic Hydrocarbons (PAHs)

| Station Name | Bodega Head | | Crescent City/ STP Outfall | |
|---|---|---|---|---|
| Station Number | 202.0 | | 2.0 | |
| Species | TCM | | TCM | |
| | Percent Recovery | | Percent Recovery | |
| | MS | MSD | MS | MSD |
| Compound | | | | |
| naphthalene | 100 | 99 | 115 | 107 |
| 1-methylnaphthalene | 96 | 99 | 97 | 97 |
| 2-methylnaphthalene | 104 | 109 | 105 | 100 |
| biphenyl | 100 | 103 | 104 | 101 |
| 2,6-dimethylnaphthalene | 105 | 105 | 99 | 96 |
| acenaphthylene | 102 | 99 | 102 | 93 |
| acenaphthene | 97 | 99 | 100 | 102 |
| 2,3,5-trimethylnaphthalene | 100 | 102 | 105 | 102 |
| fluorene | 118 | 119 | 115 | 107 |
| phenanthrene | 111 | 113 | 115 | 111 |
| anthracene | 94 | 92 | 62 | 58 |
| 1-methylphenanthrene | 123 | 123 | 96 | 102 |
| fluoranthene | 124 | 128 | 92 | 110 |
| pyrene | 116 | 121 | 94 | 105 |
| benz[a]anthracene | 109 | 109 | 98 | 94 |
| chrysene | 119 | 121 | 124 | 127 |
| benzo[b]fluoranthene | 101 | 103 | 96 | 99 |
| benzo[k]fluoranthene | 104 | 101 | 97 | 99 |
| benzo[e]pyrene | 115 | 89 | 94 | 97 |
| benzo[a]pyrene | 86 | 86 | 72 | 66 |
| perylene | 104 | 97 | 87 | 77 |
| indeno[1,2,3-cd]pyrene | 101 | 87 | 90 | 87 |
| dibenz[a,h]anthracene | 82 | 74 | 92 | 88 |
| benzo[ghi]perylene | 94 | 85 | 92 | 89 |

TCM = Transplanted California Mussel

# TABLE U-16

State Mussel Watch Program
Results of Duplicate Sample Analysis: 1997 Polynuclear Aromatic Hydrocarbons Quality Control
(ng/g dry weight)

| Station Name | Sandholdt Bridge | | Anaheim Bay/ Navy Harbor | | LA Harbor/ Consolidated | |
|---|---|---|---|---|---|---|
| Slip | | | | | | |
| Station Number | 404.0 | | 707.0 | | 616.0 | |
| Species | TCM | | TCM | | TCM | |
| REPLICATE | 1 | 2 | 1 | 2 | 1 | 2 |
| Compound | | | | | | |
| naphthalene | 55.3 | 94.7 | <50 | <50 | <50 | <50 |
| 1-methylnaphthalene | 10.7 | 16.1 | <10 | <10 | <10 | <10 |
| 2-methylnaphthalene | 23.9 | 33.7 | 12.7 | 16.5 | 56.2 | 71.7 |
| biphenyl | <10 | <10 | <10 | <10 | <10 | <10 |
| 2,6-dimethylnaphthalene | 10.7 | 12.3 | 10.4 | 10.3 | 35.6 | 44.7 |
| acenaphthylene | <10 | <10 | <10 | <10 | 27.5 | 27.8 |
| acenaphthene | <10 | <10 | 24.3 | 23.4 | 27.5 | 40.1 |
| 2,3,5-trimethylnaphthalene | <10 | <10 | 14.0 | 12.7 | 58.2 | 47.8 |
| fluorene | 24.0 | 23.9 | 36.8 | 38.7 | 21.0 | 20.1 |
| phenanthrene | 106 | 107 | 180 | 167 | 77.6 | 95.6 |
| anthracene | 10.4 | 11.6 | 51.1 | 51.6 | 35.6 | 43.2 |
| 1-methylphenanthrene | 24.4 | 25.3 | 34.9 | 29.8 | 116 | 78.7 |
| fluoranthene | 37.4 | 41.0 | 117 | 144 | 464 | 703 |
| pyrene | 38.0 | 41.7 | 126 | 143 | 608 | 848 |
| benz[a]anthracene | 10.1 | 10.1 | 29.2 | 30.5 | 182 | 215 |
| chrysene | 17.0 | 16.4 | 43.6 | 49.3 | 440 | 388 |
| benzo[b]fluoranthene | <10 | <10 | 22.3 | 17.1 | 199 | 236 |
| benzo[k]fluoranthene | <10 | <10 | <10 | <10 | 79.7 | 83.8 |
| benzo[e]pyrene | <10 | <10 | 16.3 | 13.9 | 205 | 242 |
| benzo[a]pyrene | <10 | <10 | <10 | <10 | <10 | <10 |
| perylene | <10 | <10 | <10 | <10 | <10 | <10 |
| indeno[1,2,3-cd]pyrene | <10 | <10 | <10 | <10 | 48.6 | 30.3 |
| dibenz[a,h]anthracene | <10 | <10 | <10 | <10 | <10 | <10 |
| benzo[ghi]perylene | <10 | <10 | <10 | <10 | 39.4 | 56.3 |
| percent moisture | 88.4 | 88.4 | 83.6 | 83.6 | 90.0 | 90.0 |

TCM = Transplanted California Mussel.

# TABLE U-17

Bay Protection Toxic Cleanup Program
Distribution of Synthetic Organic Compounds Among
Two Fractions of a Silica/Alumina Column

| Fraction 1 | Fraction 2 | Fraction 1 & 2 |
|---|---|---|
| Aldrin | Chlordane, cis | DDE, p,p′ |
| Chlordene, alpha | Chlordane, trans | DDT, p,p′ |
| Chlordene, gamma | Chlorpyrifos | DDMU, p,p′ |
| DDE, o,p′ | Dacthal | Nonachlor, trans |
| DDT, o,p′ | DDD, o,p′ | |
| Heptachlor | DDD, p,p′ | |
| Hexachlorobenzene | DCBP, p,p′ | |
| Mirex | Methoxychlor | |
| Dieldrin | | |
| Endosulfan I | | |
| Endosulfan II | | |
| Endosulfan sulfate | | |
| Endrin | | |
| HCH, alpha | | |
| HCH, beta | | |
| HCH, gamma | | |
| HCH, delta | | |
| Heptachlor epoxide | | |
| Nonachlor, cis | | |
| Oxychlordane | | |

# TABLE U-18

Bay Protection Toxic Cleanup Program
Synthetic Organic Compounds Analyzed and Their Detection Limits

| Compound | Detection Limit (ng/g, ppb dry weight) Sediment | Detection Limit (ng/g, ppb dry weight) Tissue |
|---|---|---|
| Aldrin | 0.5 | 1.0 |
| Chlordene, alpha | 0.5 | 1.0 |
| Chlordene, gamma | 0.5 | 1.0 |
| DDE, o,p′ | 1.0 | 3.0 |
| DDT, o,p′ | 1.0 | 4.0 |
| Heptachlor | 0.5 | 1.0 |
| Hexachlorobenzene | 0.2 | 1.0 |
| Mirex | 0.5 | 1.0 |
| DDE, p,p′ | 1.0 | 1.0 |
| DDT, p,p′ | 1.0 | 4.0 |
| DDMU, p,p′ | 2.0 | 5.0 |
| Nonachlor, trans | 0.5 | 1.0 |
| Chlordane, cis | 0.5 | 1.0 |
| Chlordane, trans | 0.5 | 1.0 |
| Chlorpyrifos | 1.0 | 4.0 |
| Dacthal | 0.2 | 2.0 |
| DDD, o,p′ | 1.0 | 5.0 |
| DDD, p,p′ | 0.4 | 3.0 |
| DCBP, p,p′ | 3.0 | 25 |
| Methoxychlor | 1.5 | 15 |
| Dieldrin | 0.5 | 1.0 |
| Endosulfan I | 0.5 | 1.0 |
| Endosulfan II | 1.0 | 3.0 |
| Endosulfan sulfate | 2.0 | 5.0 |
| Endrin | 2.0 | 6.0 |
| HCH, alpha | 0.2 | 1.0 |
| HCH, beta | 1.0 | 3.0 |
| HCH, gamma | 0.2 | 0.8 |
| HCH, delta | 0.5 | 2.0 |
| Heptachlor epoxide | 0.5 | 1.0 |
| Nonachlor, cis | 0.5 | 1.0 |
| Oxychlordane | 0.5 | 1.0 |

# TABLE U-19

Bay Protection Toxic Cleanup Program
Polynuclear Aromatic Hydrocarbons (PAHs)
Analyzed and Their Detection Limits

| Compound | Tissue dry weight (ng/g, ppb) | Sediment dry weight (ng/g, ppb) |
|---|---|---|
| naphthalene | 10 | 5 |
| 1-methylnaphthalene | 10 | 5 |
| 2-methylnaphthalene | 10 | 5 |
| biphenyl | 10 | 5 |
| 2,6-dimethylnaphthalene | 10 | 5 |
| acenaphthylene | 10 | 5 |
| acenaphthene | 10 | 5 |
| 2,3,5-trimethylnaphthalene | 10 | 5 |
| fluorene | 10 | 5 |
| phenanthrene | 10 | 5 |
| anthracene | 10 | 5 |
| 1-methylphenanthrene | 10 | 5 |
| fluoranthene | 10 | 5 |
| pyrene | 10 | 5 |
| benz[a]anthracene | 10 | 5 |
| chrysene | 10 | 5 |
| benzo[b]fluoranthene | 10 | 5 |
| benzo[k]fluoranthene | 10 | 5 |
| benzo[e]pyrene | 10 | 5 |
| benzo[a]pyrene | 10 | 5 |
| perylene | 10 | 5 |
| indeno[1,2,3-cd]pyrene | 15 | 5 |
| dibenz[a,h]anthracene | 15 | 5 |
| benzo[ghi]perylene | 15 | 5 |

# TABLE U-20

Bay Protection Toxic Cleanup - Group 14
Results of Matrix Spike Analyses: Polynuclear Aromatic Hydrocarbons (PAHs)

| Compound | Percent Recovery TCM | Percent Recovery TCM |
|---|---|---|
| naphthalene | 94 | 99 |
| 1-methylnaphthalene | 101 | 105 |
| 2-methylnaphthalene | 104 | 108 |
| biphenyl | 107 | 110 |
| 2,6-dimethylnaphthalene | 107 | 110 |
| acenaphthylene | 97 | 103 |
| acenaphthene | 98 | 103 |
| 2,3,5-trimethylnaphthalene | 102 | 107 |
| fluorene | 104 | 109 |
| phenanthrene | 106 | 110 |
| anthracene | 109 | 115 |
| 1-methylphenanthrene | 114 | 118 |
| fluoranthene | 102 | 106 |
| pyrene | 91 | 95 |
| benz[a]anthracene | 95 | 98 |
| chrysene | 84 | 90 |
| benzo[b]fluoranthene | 101 | 106 |
| benzo[k]fluoranthene | 94 | 99 |
| benzo[e]pyrene | 97 | 99 |
| benzo[a]pyrene | 97 | 104 |
| perylene | 99.5 | 100 |
| indeno[1,2,3-cd]pyrene | 101 | 107 |
| dibenz[a,h]anthracene | 99 | 107 |
| benzo[ghi]perylene | 94 | 99 |

TCM = Transplanted California Mussel

# TABLE U-21

Bay Protection Toxic Cleanup Group 17A
Results of Matrix Spike Analyses: Chlorinated Hydrocarbons

| Compound | Percent Recovery TCM |
|---|---|
| Aldrin | 108 |
| Chlordane, cis | 76 |
| Chlordane, trans | 85 |
| Chlordene, alpha | 97 |
| Chlordene, gamma | 94 |
| Chlorpyrifos | 89 |
| Dacthal | 80 |
| DDE, o,p’ | 97 |
| DDE, p,p’ | 106 |
| DDD, o,p’ | 76 |
| DDD, p,p’ | 114 |
| DDT, o,p’ | 94 |
| DDT, p,p’ | 85 |
| DDMU, p,p’ | 109 |
| Diclorobenzophenone | 103 |
| Dieldrin | 74 |
| Endrin | 82 |
| Endosulfan I | 79 |
| Endosulfan II | 76 |
| Endosulfan sulfate | 84 |
| Hexachlorobenzene | 100 |
| HCH, alpha | 97 |
| HCH, beta | 90 |
| HCH, gamma | 84 |
| HCH, delta | 94 |
| Heptachlor | 106 |
| Heptachlor epoxide | 83 |
| Nonachlor, cis | 85 |
| Nonachlor, trans | 89 |
| Methoxychlor | 77 |
| Mirex | 101 |
| Oxadiazon | 76 |
| Oxychlordane | 73 |

TCM = Transplanted California Mussel

# TABLE U-22

Bay Protection Toxic Cleanup - Group 17A
Results of Matrix Spike Analyses: Polychlorinated Biphenyls (PCBs)

| PCB Congener No. | Percent Recovery TCM |
|---|---|
| 5 | 106 |
| 8 | 112 |
| 15 | NR |
| 18 | 108 |
| 27 | 93 |
| 28 | 116 |
| 29 | 109 |
| 31 | 111 |
| 44 | 104 |
| 49 | 105 |
| 52 | 112 |
| 66 | 114 |
| 70 | 108 |
| 74 | 81 |
| 87 | 92 |
| 95 | 104 |
| 97 | 83 |
| 99 | 102 |
| 101 | 108 |
| 105 | 108 |
| 110 | 105 |
| 118 | 114 |
| 128 | 105 |
| 132 | 101 |
| 137 | 86 |
| 138 | 108 |
| 149 | 105 |
| 151 | 108 |
| 153 | 108 |
| 156 | 95 |
| 157 | 102 |
| 158 | 93 |
| 170 | 113 |
| 174 | 103 |
| 177 | 112 |
| 180 | 109 |
| 183 | 105 |
| 187 | 106 |
| 189 | 109 |
| 194 | 107 |
| 195 | 109 |
| 201 | 118 |
| 203 | 108 |
| 206 | 104 |
| 209 | 98 |
| PCT 5460 | 96 |

TCM = Transplanted California Mussel

# TABLE U-23

Bay Protection Toxic Cleanup - Group 17A
Results of Matrix Spike Analyses: Polynuclear Aromatic Hydrocarbons (PAHs)

| Compound | Percent Recovery TCM |
|---|---|
| naphthalene | 99 |
| 1-methylnaphthalene | 95 |
| 2-methylnaphthalene | 94 |
| biphenyl | 95 |
| 2,6-dimethylnaphthalene | 100 |
| acenaphthylene | 98 |
| acenaphthene | 100 |
| 2,3,5-trimethylnaphthalene | 97 |
| fluorene | 98 |
| phenanthrene | 98 |
| anthracene | 89 |
| 1-methylphenanthrene | 100 |
| fluoranthene | 90 |
| pyrene | 90 |
| benz[a]anthracene | 73 |
| chrysene | 80 |
| benzo[b]fluoranthene | 77 |
| benzo[k]fluoranthene | 89 |
| benzo[e]pyrene | 91 |
| benzo[a]pyrene | 76 |
| perylene | 83 |
| indeno[1,2,3-cd]pyrene | 92 |
| dibenz[a,h]anthracene | 84 |
| benzo[ghi]perylene | 90 |

TCM = Transplanted California Mussel