# 7.0 Laboratory Analytical Procedures

All of the chemical analytical procedures used in this program will be performed in accordance with the most current SMS, PSEP, and Ecology's Sediment Sampling and Analysis Plan Appendix (Ecology 2008) documentation, as applicable. Analytes include metals, SVOCs (PAHs, phthalates, and phenols), polychlorinated dibenzo-p-dioxin compounds (dioxins), polychlorinated dibenzofuran compounds (furans), chlorinated pesticides, total PCBs, PCB congeners, wood resin compounds, petroleum hydrocarbons, grain size, TOC, ammonia, and sulfides. In addition, three cores will be analyzed for lead-210 and cesium-137.

Each laboratory participating in this program will be National Environmental Laboratory Accreditation Program - and/or Ecology-certified to perform the analyses. Each laboratory analysis will be required to conform to accepted standard methods and internal QA/QC.

## 7.1 Chemical Analyses

Chemical analysis will be conducted by laboratories subcontracted to Herrera. The specific analyses and conventional parameters to be measured, sample preparation methods, analytical methods, TDLs, and SMS numeric criteria (SQS and CSL) are presented in Table 7-1. The TDLs listed are the laboratory reporting limits (RLs); and may be subject to modification due to elevated sample concentrations, heterogeneous samples, and potential matrix interferences that may preclude obtaining the desired quantification limit. Several organic compounds in the SMS criteria are corrected for percent OC content. In the event the laboratory is unable to meet the OC-corrected SQS and CSL numeric values, the reasons for the deviation will also be reported.

Analytical laboratory reports will be accompanied by sufficient backup data and QC results to enable independent reviewers to evaluate the quality of the data results. Analytical data will be reported in the units specified by the RLs listed in Tables 7-1, 7-2, and 7-3.

Analytical laboratory deliverables will include the following:

- Case narrative (including any problems encountered, protocol modifications, and/or corrective actions taken);
- Sample analytical and QA/QC results with units;
- All protocols used during analyses;
- Any protocol deviations from the approved sampling plan;
- Surrogate recovery results;
- MS/MSD results;

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## Table 7-1. Sediment SMS Analyte List for the Oakland Bay Sediment Study.

| Analyte | Prep Method [a] | Analytical Method [b] | Sediment RL [c,d] | SQS | CSL |
|---|---|---|---|---|---|
| Conventional Parameters | | | | | |
| Total Solids (%) | --- | PSEP [5] | 0.1 (wet weight) | --- | --- |
| Total Organic Carbon (%) | --- | PSEP [5] | 0.1 | --- | --- |
| Total Sulfides (mg/kg) | --- | PSEP [5] | 10 | --- | --- |
| Ammonia (mg/kg) | --- | PSEP [5] | 0.1 | --- | --- |
| Grain Size (%) | --- | PSEP [5] | 1 | --- | --- |
| Metals | | | mg/kg | mg/kg | |
| Arsenic | PSEP/3050B | 6010B/6020 | 19 | 57 | 93 |
| Cadmium | PSEP/3050B | 6010B/6020 | 1.7 | 5.1 | 6.7 |
| Chromium | PSEP/3050B | 6010B/6020 | 87 | 260 | 270 |
| Copper | PSEP/3050B | 6010B/6020 | 130 | 390 | 390 |
| Lead | PSEP/3050B | 6010B/6020 | 150 | 450 | 530 |
| Mercury | --- | 7471A | 0.14 | 0.41 | 0.59 |
| Silver | PSEP/3050B | 6010B/6020 | 2 | 6.1 | 6.1 |
| Zinc | PSEP/3050B | 6010B/6020 | 137 | 410 | 960 |
| Low Molecular Polycyclic Aromatic Hydrocarbons (LPAH) | | | µg/kg | mg/kg OC | |
| Naphthalene | 3540C/3550B | 8270C | 700 | 99 | 170 |
| Acenaphthylene | 3540C/3550B | 8270C | 433 | 66 | 66 |
| Acenaphthene | 3540C/3550B | 8270C | 167 | 16 | 57 |
| Fluorene | 3540C/3550B | 8270C | 180 | 23 | 79 |
| Phenanthrene | 3540C/3550B | 8270C | 500 | 100 | 480 |
| Anthracene | 3540C/3550B | 8270C | 320 | 220 | 1,200 |
| 2-Methylnaphthalene | 3540C/3550B | 8270C | 223 | 38 | 64 |
| Total LPAH | | | | 370 | 780 |
| High Molecular Polycyclic Aromatic Hydrocarbons (HPAH) | | | µg/kg | mg/kg OC | |
| Fluoranthene | 3540C/3550B | 8270C | 567 | 160 | 1,200 |
| Pyrene | 3540C/3550B | 8270C | 867 | 1,000 | 1,400 |
| Benzo(a)anthracene | 3540C/3550B | 8270C | 433 | 110 | 270 |
| Chrysene | 3540C/3550B | 8270C | 467 | 110 | 460 |
| Benzofluoranthenes, total | 3540C/3550B | 8270C | 1,067 | 230 | 450 |
| Benzo(a)pyrene | 3540C/3550B | 8270C | 533 | 99 | 210 |
| Indeno(1,2,3-c,d)pyrene | 3540C/3550B | 8270C | 200 | 34 | 88 |
| Dibenzo(a,h)anthracene | 3540C/3550B | 8270C | 77 | 12 | 33 |
| Benzo(g,h,i)perylene | 3540C/3550B | 8270C | 223 | 31 | 78 |
| Total HPAH | | | | 960 | 5,300 |
| Chlorinated Benzenes | | | µg/kg | mg/kg OC | |
| 1,2-Dichlorobenzene | 3540C/3550B | 8270C | 35 | 2.3 | 2.3 |
| 1,4-Dichlorobenzene | 3540C/3550B | 8270C | 37 | 3.1 | 9 |
| 1,2,4-Trichlorobenzene | 3540C/3550B | 8270C | 31 | 0.81 | 1.8 |
| Hexachlorobenzene | 3540C/3550B | 8270C | 22 | 0.38 | 2.3 |

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**Table 7-1 (continued). Sediment SMS Analyte List for the Oakland Bay Sediment Study.**

| Analyte | Prep Method [a] | Analytical Method [b] | Sediment RL [c,d] | SQS | CSL |
|---|---|---|---|---|---|
| Phthalate Esters | | | µg/kg | mg/kg OC | |
| Dimethyl phthalate | 3540C/3550B | 8270C | 24 | 53 | 53 |
| Diethyl phthalate | 3540C/3550B | 8270C | 37 | 61 | 110 |
| Di-n-butyl phthalate | 3540C/3550B | 8270C | 467 | 220 | 1,700 |
| Butyl benzyl phthalate | 3540C/3550B | 8270C | 21 | 4.9 | 64 |
| Bis(2-ethylhexyl)phthalate | 3540C/3550B | 8270C | 433 | 47 | 78 |
| Di-n-octyl phthalate | 3540C/3550B | 8270C | 2,067 | 58 | 4,500 |
| Ionizable Organic Compounds | | | µg/kg | µg/kg | |
| Phenol | 3540C/3550B | 8270C | 140 | 420 | 1,200 |
| 2-Methylphenol | 3540C/3550B | 8270C | 63 | 63 | 63 |
| 4-Methylphenol | 3540C/3550B | 8270C | 223 | 670 | 670 |
| 2,4-Dimethylphenol | 3540C/3550B | 8270C | 29 | 29 | 29 |
| Pentachlorophenol | 3540C/3550B | 8270C | 120 | 360 | 690 |
| Benzyl alcohol | 3540C/3550B | 8270C | 57 | 57 | 73 |
| Benzoic acid | 3540C/3550B | 8270C | 217 | 650 | 650 |
| Miscellaneous Compounds | | | µg/kg | mg/kg OC | |
| Dibenzofuran | 3540C/3550B | 8270C | 180 | 15 | 58 |
| Hexachlorobutadiene | 3540C/3550B | 8270C | 11 | 3.9 | 6.2 |
| N-Nitrosodiphenylamine | 3540C/3550B | 8270C | 28 | 11 | 11 |
| Total PCBs | 3540C/3550B | 8082 | 6 | 12 | 65 |

[a] Recommended sample preparation methods are: PSEP (1997a,b) and USEPA 3000 series (sample preparation methods from SW-846 [USEPA 1986])
[b] Recommended sample cleanup methods are: USEPA SW-846 Methods 3640A, 3660B, and 3665A. Alternative cleanup procedures are described in PSEP and SW-846.
[c] RL, SQS, and CSL are on a dry weight basis.
[d] The recommended RL is based on a value equal to one third of the 1988 dry weight lowest apparent effects threshold value (Barrick et al. 1988) except for the following chemicals: 1,2-dichlorobenzene, 1,2,4-trichlorobenzene, hexachlorobenzene, hexachlorobutadiene, n-nitrosodiphenylamine, 2-methylphenol, 2,4-dimethylphenol, and benzyl alcohol, for which the recommended MRL is equal to the full value of the 1988 dry weight. As reported in Table 5, Ecology 2008.

CSL = Cleanup Screening Level.
HPAH = High molecular weight polycyclic aromatic hydrocarbon compounds.
LPAH = Low molecular weight polycyclic aromatic hydrocarbon compounds.
µg/kg = Micrograms per kilogram.
mg/kg = Milligrams per kilogram.
OC = Organic carbon.
PSEP = *Recommended Protocols for Measuring Metals in Puget Sound Water, Sediment and Tissue Samples*, Puget Sound Estuary Program, April 1997.
RL = Reporting limit.
SQS = Sediment Quality Standards
SW-846 = *Test Methods for Evaluating Solid Waste, Physical Chemical Methods*, 3rd edition, USEPA, SW-846, 1986.
USEPA = United States Environmental Protection Agency.

**Table 7-2. Sediment non-SMS Analyte List Summary for Oakland Bay Sediment Study.**

| Parameter | Analysis Method [a] | Sediment RL |
|---|---|---|
| Metals [b] | | |
| Antimony | SW 6010/6020 | 50 |
| Nickel | SW 6010/6020 | 47 |
| Chlorinated Pesticides [c] | | |
| Aldrin | SW 8081 | 1.7 |
| gamma-BHC (Lindane) | SW 8081 | 1.7 |
| Heptachlor | SW 8081 | 1.7 |
| Hexachlorobenzene | SW 8081 | 22 |
| alpha-Chlordane | SW 8081 | 1.7 |
| 4,4'-DDD | SW 8081 | 3.3 |
| 4,4'-DDE | SW 8081 | 2.3 |
| 4,4'-DDT | SW 8081 | 6.7 |
| Dieldrin | SW 8081 | 2.3 |
| Organotin Compounds [c] | | |
| Butyltin | Krone, et. al., 1989 | 4 |
| Dibutyltin | Krone, et. al., 1989 | 6 |
| TBT | Krone, et. al., 1989 | 4 |
| Wood Resin Compounds [c] | | |
| Retene | SW 8270 | 20 |
| Guaiacols | modified SW 8270 | 20 |
| 4-Chloroguaiacol | modified SW 8270 | TBD |
| 3,4-Dichloroguaiacol | modified SW 8270 | TBD |
| 4,5-Dichloroguaiacol | modified SW 8270 | 50 |
| 4,6-Dichloroguaiacol | modified SW 8270 | TBD |
| 3,4,5-Trichloroguaiacol | modified SW 8270 | 75 |
| 3,4,6-Trichloroguaiacol | modified SW 8270 | TBD |
| 4,5,6-Trichloroguaiacol | modified SW 8270 | 50 |
| Tetrachloroguaiacol | modified SW 8270 | 75 |
| Pimaric acid | modified SW 8270 | 100 |
| Sandracopimaric acid | modified SW 8270 | 100 |
| Isopimaric acid | modified SW 8270 | 100 |
| Palustric acid | modified SW 8270 | 100 |
| Dihydroabietic acid | modified SW 8270 | 100 |
| Abietic acid | modified SW 8270 | 100 |
| Neoabietic acid | modified SW 8270 | 100 |
| 9,10-Dichlorostearic acid | modified SW 8270 | TBD |
| Oleic acid | modified SW 8270 | 100 |
| Linolenic acid | modified SW 8270 | 100 |
| 12-Chlorodehydroabietic acid | modified SW 8270 | 100 |
| 14-Chlorodehydroabietic acid | modified SW 8270 | 100 |
| Dichlorodihydroabietic acid | modified SW 8270 | 100 |
| NWTPH-HCID [c] | | |
| Gasoline | NWTPH-HCID | 20 |
| Diesel #2 | NWTPH-HCID | 50 |

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**Table 7-2 (continued). Sediment non-SMS Analyte List Summary for Oakland Bay Sediment Study.**

| Parameter | Analysis Method [a] | Sediment RL |
|---|---|---|
| Motor Oil | NWTPH-HCID | 100 |
| CDD/CDF [d] | | |
| 2,3,7,8-TCDD | USEPA 1613B | 0.1 |
| Total TCDD | USEPA 1613B | 0.1 |
| 1,2,3,7,8-PeCDD | USEPA 1613B | 0.5 |
| Total PeCDD | USEPA 1613B | 0.5 |
| 1,2,3,4,7,8-HxCDD | USEPA 1613B | 0.5 |
| 1,2,3,6,7,8-HxCDD | USEPA 1613B | 0.5 |
| 1,2,3,7,8,9-HxCDD | USEPA 1613B | 0.5 |
| Total HxCDD | USEPA 1613B | 0.5 |
| 1,2,3,4,6,7,8-HpCDD | USEPA 1613B | 0.5 |
| Total HpCDD | USEPA 1613B | 0.5 |
| OCDD | USEPA 1613B | 1.0 |
| 2,3,7,8-TCDF | USEPA 1613B | 0.1 |
| Total TCDF | USEPA 1613B | 0.1 |
| 1,2,3,7,8-PeCDF | USEPA 1613B | 0.5 |
| 2,3,4,7,8-PeCDF | USEPA 1613B | 0.5 |
| Total PeCDF | USEPA 1613B | 0.5 |
| 1,2,3,4,7,8-HxCDF | USEPA 1613B | 0.5 |
| 1,2,3,6,7,8-HxCDF | USEPA 1613B | 0.5 |
| 1,2,3,7,8,9-HxCDF | USEPA 1613B | 0.5 |
| 2,3,4,6,7,8-HxCDF | USEPA 1613B | 0.5 |
| Total HxCDF | USEPA 1613B | 0.5 |
| 1,2,3,4,6,7,8-HpCDF | USEPA 1613B | 0.5 |
| 1,2,3,4,7,8,9-HpCDF | USEPA 1613B | 0.5 |
| Total HpCDF | USEPA 1613B | 0.5 |
| OCDF | USEPA 1613B | 1.0 |

[a] Krone, et. al., 1989. A method for analysis of butyltin species and measurement of butyltin in Sediment and Engish sole livers from Puget Sound. Marine Environment Research, **27**, 1-18.
[b] Metal reporting limits (RL) are on a dry weight basis in mg/g.
[c] Organic Compounds RL are on a dry weight basis in µg/g.
[d] CDD and CDF RL are on a dry weight basis in pg/g.
CDD = Chlorinated dibenzo-p-dioxin.
CDF = Chlorinated dibenzofuran.
USEPA 1613B = *Method 1613 Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS.* U.S. Environmental Protection Agency, Office of Water, Engineering and Analysis Division. October 1994.
HCID = Hydrocarbon Identification.
HxCDD = Hexachlorodibenzo-p-dioxin.
HxCDF = Hexachlorodibenzofuran.
HpCDD = Heptachlorodibenzo-p-dioxin.
HpCDF = Heptachlorodibenzofuran.
NWTPH = Northwest total petroleum hydrocarbon.
OCDD = Octachlorodibenzo-p-dioxin.
OCDF = Octachlorodibenzofuran.
PeCDD = Pentachlorodibenzo-p-dioxin.
PeCDF = Pentachlorodibenzofuran.
RL = Reporting limit.
SW = *Test Methods for Evaluating Solid Waste, Physical Chemical Methods*, 3rd edition, USEPA, SW-846, 1986.
TBD = To be determined.
TCDD = Tetrachlorodibenzo-p-dioxin.
TCDF = Tetrachlorodibenzofuran.

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**Table 7-3. Tissue Analyte List Summary for Oakland Bay Sediment Study.**

| Parameter [a] | Analysis Method | Tissue RL |
|---|---|---|
| Metals [a] | | |
| Antimony | SW 6010/6020 | 3 |
| Arsenic | SW 6010/6020 | 0.25 |
| Cadmium | SW 6010/6020 | 0.5 |
| Chromium | SW 6010/6020 | 1.3 |
| Copper | SW 6010/6020 | 1 |
| Lead | SW 6010/6020 | 1.5 |
| Mercury | SW 7471 | 0.02 |
| Nickel | SW 6010/6020 | 1 |
| Silver | SW 6010/6020 | 1 |
| Zinc | SW 6010/6020 | 2.5 |
| Polycyclic Aromatic Hydrocarbons (PAH) [b] | | |
| Naphthalene | SW 8270 | 20 |
| Acenaphthylene | SW 8270 | 20 |
| Acenaphthene | SW 8270 | 20 |
| Fluorene | SW 8270 | 20 |
| Phenanthrene | SW 8270 | 20 |
| Anthracene | SW 8270 | 20 |
| 2-Methylnaphthalene | SW 8270 | 20 |
| Fluoranthene | SW 8270 | 20 |
| Pyrene | SW 8270 | 20 |
| Benzo(a)anthracene | SW 8270 | 20 |
| Chrysene | SW 8270 | 20 |
| Benzofluoranthenes | SW 8270 | 20 |
| Benzo(a)pyrene | SW 8270 | 20 |
| Indeno(1,2,3-c,d)pyrene | SW 8270 | 20 |
| Dibenzo(a,h)anthracene | SW 8270 | 20 |
| Benzo(g,h,i)perylene | SW 8270 | 20 |
| Chlorinated Benzenes b | | |
| 1,2-Dichlorobenzene | SW 8270 | 20 |
| 1,4-Dichlorobenzene | SW 8270 | 20 |
| 1,2,4-Trichlorobenzene | SW 8270 | 20 |
| Hexachlorobenzene | SW 8270 | 20 |
| Phthalate Esters b | | |
| Dimethyl phthalate | SW 8270 | 20 |
| Diethyl phthalate | SW 8270 | 20 |
| Di-n-butyl phthalate | SW 8270 | 20 |
| Butyl benzyl phthalate | SW 8270 | 20 |
| Bis(2-ethylhexyl)phthalate | SW 8270 | 20 |
| Di-n-octyl phthalate | SW 8270 | 20 |
| Ionizable Organic Compounds [b] | | |
| Phenol | SW 8270 | 20 |
| 2-Methylphenol | SW 8270 | 20 |

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**Table 7-3 (continued). Tissue Analyte List Summary for Oakland Bay Sediment Study.**

| Parameter [a] | Analysis Method | Tissue RL |
|---|---|---|
| 4-Methylphenol | SW 8270 | 20 |
| 2,4-Dimethylphenol | SW 8270 | 20 |
| Pentachlorophenol | SW 8270 | 100 |
| Benzyl alcohol | SW 8270 | 20 |
| Benzoic acid | SW 8270 | 200 |
| Miscellaneous Compounds [b] | | |
| Dibenzofuran | SW 8270 | 20 |
| Hexachlorobutadiene | SW 8270 | 20 |
| N-Nitrosodiphenylamine | SW 8270 | 20 |
| Aroclors b | | |
| Aroclor 1016 | SW 8082 | 10 |
| Aroclor 1242 | SW 8082 | 10 |
| Aroclor 1248 | SW 8082 | 10 |
| Aroclor 1254 | SW 8082 | 10 |
| Aroclor 1260 | SW 8082 | 10 |
| Total PCBs | SW 8082 | 10 |
| Chlorinated Pesticides [b] | | |
| Aldrin | SW 8081 | 1 |
| gamma-BHC (Lindane) | SW 8081 | 1 |
| Heptachlor | SW 8081 | 1 |
| Hexachlorobenzene | SW 8081 | 1 |
| alpha-Chlordane | SW 8081 | 1 |
| 4,4'-DDD | SW 8081 | 2 |
| 4,4'-DDE | SW 8081 | 2 |
| 4,4'-DDT | SW 8081 | 2 |
| Dieldrin | SW 8081 | 2 |
| CB [c] | | |
| 3,3',4,4'-TeCB (PCB-77) | USEPA 1668 | 20 |
| 3,4,4',5-TeCB (PCB-81) | USEPA 1668 | 20 |
| 2,3,3',4,4'-PeCB (PCB-105) | USEPA 1668 | 20 |
| 2,3,4,4',5-PeCB (PCB-114) | USEPA 1668 | 20 |
| 2, 3',4,4',5-PeCB (PCB-118) | USEPA 1668 | 20 |
| 2',3,4,4',5-PeCB (PCB-123) | USEPA 1668 | 20 |
| 3,3',4,4',5-PeCB (PCB-126) | USEPA 1668 | 20 |
| 2,3,3',4,4',5-HxCB (PCB-156) | USEPA 1668 | 20 |
| 2,3,3',4,4',5'-HxCB (PCB-157) | USEPA 1668 | 20 |
| 2,3',4,4',5,5'-HxCB (PCB-167) | USEPA 1668 | 20 |
| 3,3',4,4',5,5'-HxCB (PCB-169) | USEPA 1668 | 20 |
| 2,3,3',4,4',5,5'-HPCB (PCB-189) | USEPA 1668 | 20 |
| PCDD/PCDF [c] | | |
| 2,3,7,8-TCDD | USEPA 1613B | 0.2 |
| Total TCDD | USEPA 1613B | 0.2 |

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**Table 7-3 (continued). Tissue Analyte List Summary for Oakland Bay Sediment Study.**

| Parameter [a] | Analysis Method | Tissue RL |
|---|---|---|
| 1,2,3,7,8-PeCDD | USEPA 1613B | 1.0 |
| Total PeCDD | USEPA 1613B | 1.0 |
| 1,2,3,4,7,8-HxCDD | USEPA 1613B | 1.0 |
| 1,2,3,6,7,8-HxCDD | USEPA 1613B | 1.0 |
| 1,2,3,7,8,9-HxCDD | USEPA 1613B | 1.0 |
| Total HxCDD | USEPA 1613B | 1.0 |
| 1,2,3,4,6,7,8-HpCDD | USEPA 1613B | 1.0 |
| Total HpCDD | USEPA 1613B | 1.0 |
| OCDD | USEPA 1613B | 2.0 |
| 2,3,7,8-TCDF | USEPA 1613B | 0.2 |
| Total TCDF | USEPA 1613B | 0.2 |
| 1,2,3,7,8-PeCDF | USEPA 1613B | 1.0 |
| 2,3,4,7,8-PeCDF | USEPA 1613B | 1.0 |
| Total PeCDF | USEPA 1613B | 1.0 |
| 1,2,3,4,7,8-HxCDF | USEPA 1613B | 1.0 |
| 1,2,3,6,7,8-HxCDF | USEPA 1613B | 1.0 |
| 1,2,3,7,8,9-HxCDF | USEPA 1613B | 1.0 |
| 2,3,4,6,7,8-HxCDF | USEPA 1613B | 1.0 |
| Total HxCDF | USEPA 1613B | 1.0 |
| 1,2,3,4,6,7,8-HpCDF | USEPA 1613B | 1.0 |
| 1,2,3,4,7,8,9-HpCDF | USEPA 1613B | 1.0 |
| Total HpCDF | USEPA 1613B | 1.0 |
| OCDF | USEPA 1613B | 2.0 |

[a] Metal reporting limits (RL) are on a dry weight basis in mg/g.
[b] Organic Compounds reporting limits (RL) are on a dry weight basis in µg/g.
[d] CDD and CDF reporting limits (RL) are on a dry weight basis in pg/g.
CDD = Chlorinated dibenzo-p-dioxin.
CDF = Chlorinated dibenzofuran.
USEPA 1613B = Method 1613 Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS. USEPA, Office of Water, Engineering and Analysis Division. October 1994.
USEPA 1668 = Method 1668 Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS. U.S. Environmental Protection Agency, Office of Water, Engineering and Analysis Division. December 1999.
HCID = Hydrocarbon Identification.
HxCB = Hexachlorobipenyl.
HxCD = Hexachlorodibenzo-p-dioxin.
HxCDF =Hexachlorodibenzofuran.
HpCB = Heptachlorobipenyl.
HpCDD = Heptachlorodibenzo-p-dioxin.
HpCDF = Heptachlorodibenzofuran.
NWTPH = Northwest total petroleum hydrocarbon.
OCDD = Octachlorodibenzo-p-dioxin.
OCDF = Octachlorodibenzofuran.
PeCB = Pentachlorobipenyl.
PeCDD = Pentachlorodibenzo-p-dioxin.
PeCDF = Pentachlorodibenzofuran.
RL = Reporting limit.
SW = Test Methods for Evaluating Solid Waste, Physical Chemical Methods, 3rd edition, USEPA, SW-846, 1986.
TBD = To be determined.
TeCB = Tetrachlorobipenyl.
TCDD = Tetrachlorodibenzo-p-dioxin.
TCDF = Tetrachlorodibenzofuran.

- Laboratory duplicate/triplicate results;
- Blank results;
- Sample custody records (including original COC forms); and
- Analytical results in electronic Environmental Information Management (EIM) system.

## 7.2 Bioassay Analyses

This section describes specific procedures for the suite of bioassays used for SMS biological analysis. Sediment collected at predetermined locations will be submitted for bioassay testing. Bioassay testing requires that test samples be matched and tests run with appropriate reference sediments to factor out background conditions and sediment grain-size effects on bioassay organisms. The contractor will collect the identified reference sediments at the same time that other samples are collected. The location of the reference sediment sampling location will be recorded to the nearest 0.1 second (NAD 83).

All sediment samples selected for bioassay analysis will be stored at 4 °C with no headspace until bioassay testing commences. All bioassays will commence within 14 days from collection of the first grab sample of the sediment composite to be tested. The laboratory will maintain COC procedures throughout biological testing.

Bioassay testing will be initiated as soon as possible after the last sample (to define a batch) is received by the laboratory. This includes obtaining test organisms and control/reference sediments in a timely manner.

Three bioassays (Table 7-4) including amphipod mortality, larval development, and juvenile polychaete growth, will be conducted on each sample identified for biological testing. All biological testing will be conducted in strict compliance with Recommended Guidelines for Conducting Laboratory Bioassays on Puget Sound Sediments (PSEP 1995), with appropriate modifications as specified in the annual review process. General biological testing procedures and specific procedures for each sediment bioassay are summarized in the following sections.

The specific QA/QC measures employed as part of the biological analyses are discussed in detail in Section 5.0.

**Table 7-4. Bioassay Suite for the Oakland Harbor Sediment Study.**

| Bioassay Test | Test Organisms |
|---|---|
| 10-day Amphipod Mortality Test | *Eohaustorius estuaries* or *Rhepoxynius abronius* |
| 48-hour Larval Development Test [a] (bivalve) | *Mytilus galloprovincialus* or *Dendraster excentricus* |
| 20-day Juvenile Polychaete Growth Test | *Neanthes arenaceodentata* |

[a] Actual test length may vary based on larval development stage.

### 7.2.1 Amphipod Mortality Bioassay

This test involves exposing *Rhepoxynius abronius* or *Eohaustorius estuarius* to test sediment for 10 days and counting the surviving animals at the end of the exposure period. Daily emergence data and the number of amphipods failing to rebury at the end of the test will be recorded as well. The control sediment has a performance standard of 10 percent mortality. The reference sediment has a performance standard of 25 percent mean mortality.

*E. estuarius* is the preferred test organism for sediments with percent fines greater than 60 percent. *R. abronius* is the preferred amphipod species for coarser-grained sediments (less than 60 percent fines). Based on historical grain size data from previous investigations, both *E. estuarius* and *R. abronius* will be used in the investigation.

No treatment for confounding factors will be performed on the sediment sample during the bioassay procedure. Ammonia reference toxicant tests may be conducted if elevated ammonia concentration is suspected in test sediments.

### 7.2.2 Larval Development Bioassay

This test monitors larval development of a suitable echinoderm or molluscan species (e.g., *Dendraster excentricus* or *Mytilus galloprovincialus*) in the presence of test sediment. *M. galloprovincialis* is the preferred species, followed by *D. excentricus*. The sediment larval bioassay has a variable endpoint (not necessarily 48 hours) that is determined by the developmental stage of organisms in a sacrificial seawater control (PSEP 1995). At the end of the test, larvae from each test sediment exposure are examined to quantify abnormality and mortality. The seawater control has a performance standard of 70 percent mean normal survivorship. Initial counts will be made for a minimum of five 10 ml (0.34 oz) aliquots. Final counts for seawater control, reference sediment, and test sediment will be made on 10 ml (0.34 oz) aliquots.

No treatment for confounding factors will be performed on the sediment sample during the bioassay procedure. Ammonia reference toxicant tests may be conducted if elevated ammonia concentration is suspected in test sediments.

### 7.2.3 Juvenile Polychaete Growth Bioassay

This sublethal, static-renewal toxicity test can be used to determine the relative toxicity of marine sediments using the juvenile polychaete, *Neanthes arenaceodentata*. The test is conducted in accordance with the methods described by PSEP (1995) and modifications to the test approved by the Dredged Material Management Program (DMMP) agencies.

The toxicity test involves a 20-day exposure to sediments and the response of the organisms to test sediments as compared to their response in control (clean) and reference sediment. The test endpoint is mean individual growth (expressed as mg/individual/day).

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The control sediment has a performance standard of 10 percent mortality. The reference sediment has a performance standard of 80 percent of the control growth. The DMMP agencies have established a target control growth performance guideline of greater than or equal to 0.72 mg/individual/day. The *N. arenaceodentata* negative control performance guideline is a target growth rate of greater than or equal to 0.72 mg/individual/day; the negative control performance standard is greater than 0.38 mg/individual/day (below which the test is considered a QC failure). Use of worms smaller than 0.25 mg (dry weight) at the beginning of the test will also be considered a QC failure.

### 7.2.4 Full-Spectrum Lighting

Under certain conditions, when PAHs are exposed to ultraviolet (UV) radiation of sufficient quality and quantity, photo-activation may occur (Kosian et al.1998). Photo-activation has been demonstrated to result in increased acute and chronic toxicity (Arfsten et al.1996). Benthic and aquatic organisms exposed to selected PAHs and simultaneously to specific wavelengths and intensities of UV radiation may be at significantly greater risk to toxic effects than organisms exposed to the same PAHs absent the UV radiation (Ahrens 2002). When the following site conditions are encountered, all bioassays will be performed in the presence of full-spectrum lighting that includes UV wavelengths of sufficient intensity to mimic conditions at the site (Ecology 2008):

1. If either sediment depth is 12 ft or less (4 m) than MLLW for more than 25 percent of the surface sediment or surface sediment covers more than 1/2 acre; or

2. The presence or presumed presence of any of the photo-activated PAHs listed in Table 7-5 is expected (Nagpal 1993).

**Table 7-5. Photo-activated Polycyclic Aromatic Hydrocarbons**

| PAHs | PAHs |
|---|---|
| Anthracene | Benz[c]acridine |
| Acridine | Benzathrone |
| Phenazine | Benzo[a]pyrene |
| Fluoranthene | Benzo[e]pyrene |
| 1H-benzo[a]fluorene | Perylene |
| 1H-benzo[b]fluorene | Dibenz[a,h]acridine |
| Pyrene | Dibenz[a,h]anthracene |
| Benz[a]anthracene | Dibenz[a,j]anthracene |
| Benz[b]anthracene | Benzo[b]chrysene |
| Chrysene | Dibenz[a,c]phenazine |
| Benzo[k]fluoranthene | Benzo[b]triphenyline |
| Benz[a]acridine | Benzo[g,h,i]perylene |

Based on historical data from previous sampling events in Oakland Bay, all bioassays should be conducted using full-spectrum lighting.

Standard fluorescent laboratory lighting fixtures are not full spectrum and do not produce "natural" wavelengths and intensity of light. Therefore the laboratory must use two light sources with different radiation characteristics. The full spectrum fluorescent lamp needed must include the following (Ecology 2008):

1. UV-B output (280 nanometers (nm) < λ < 315 nm) photo-activating wavelengths.

2. UV-A output (315 nm < λ < 400 nm), this may have an effect upon burial and feeding behavior.

3. Correct Color temperature: 'warm' red to 'cold' blue expressed in degrees Kelvin. Daylight at noon is typically estimated at 5,500 °K.

4. High Color Rendering Index (CRI): Color rendering is the degree to which a light source shows the true color of objects it illuminates. This is measured on a CRI rated from 0–100. A normal fluorescent lamp rates 54 on the CRI scale. High quality fluorescent lamps will rate 90–98 on the same scale.

In addition to the quality of the lamp, its proximity to the animal, its output intensity, and duration of use are also critical. It is absolutely critical that nothing is placed between the envelope of the lamp tube and the recipient test organism or vessel. UV-B is greatly attenuated by glass, plastic, and ultra-fine mesh. The amount of UV-B received is also diminished with distance. It is recommended that any UV-B tubes be no further than 12 in (30 cm) away from the organism or vessel (Ecology 2008).

The recommended lab conditions for full spectrum testing include:

- Light intensity: 50–100 foot candles;
- Light duration: 16:8 (light/dark);
- Overlying water depth: not greater than 15 cm (6 in);
- Lamp to water surface distance: not greater than 30 cm (12 in); and
- UV wavelength range: 3–8 percent UV-B (280 nm < λ < 315 nm), (3-5 percent preferred)
- 20–35 percent UV-A (315 nm < λ < 400 nm).

### 7.2.5 Bioassay Interpretation

Test interpretations consist of endpoint comparisons to controls and reference on an absolute percentage basis as well as statistical comparison to reference. The SMS biological effects criteria are presented in Table 7-6.

**Table 7-6. SMS Biological Effect Criteria.**

| Biological Test | Sediment Quality Standards | Cleanup Screening Levels |
|---|---|---|
| Amphipod Mortality | The test sediment has a significantly higher (t-test, P≤0.05) mean mortality than the reference sediment, and the test sediment mean mortality is more than 25% greater, on an absolute basis, than the reference sediment mean mortality. | The test sediment has a significantly higher (t-test, P≤0.05) mean mortality than the reference sediment, and the test sediment mean mortality is more than 30% greater, on an absolute basis, than the reference sediment mean mortality. |
| Larval Development | The test sediment has a mean survivorship of normal larvae that is significantly less (t-test, P≤0.1) than the mean normal survivorship in the reference sediment, and the mean normal survivorship in the test sediment is less than 85% of the mean normal survivorship in the reference sediment. | The test sediment has a mean survivorship of normal larvae that is significantly less (t-test, P≤0.1) than the mean normal survivorship in the reference sediment, and the mean normal survivorship in the test sediment is less than 70% of the mean normal survivorship in the reference sediment. |
| Juvenile Polychaete Growth | The mean individual growth rate of polychaetes in the test sediment is less than 70% of the mean individual growth rate of the polychaetes in the reference sediment, and the test sediment mean individual growth rate is statistically different (t-test, P P≤0.05) from the reference sediment mean individual growth rate. | The mean individual growth rate of polychaetes in the test sediment is less than 50% of the mean individual growth rate of the polychaetes in the reference sediment, and the test sediment mean individual growth rate is statistically different (t-test, P P≤0.05) from the reference sediment mean individual growth rate. |

## 7.3 Radioisotope Analyses

Laboratory analysis will consist of lead-210 and cesium-137 radioisotope activity measurements. Percent dry weight and lead-210 in disintegrations per minute per gram (dpm/g) and cesium-137 in dpm/g, will be determined for each sample. Lead-210 analysis will be performed on every third 2 cm (0.8 in) section; cesium-137 will be performed on two core sections.

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# 8.0 Data Analysis and Reporting

This section describes data analysis and reporting requirements for the data collection activities described above.

## 8.1 Data Analysis

### 8.1.1 Analysis of Sediment Chemistry Data

The analysis of chemistry data will include a comparison of the results to SMS numeric criteria. Sediment chemistry data will be summarized and presented in tables indicating sampling locations and detected contaminants and any detection limits that exceed SQS and/or CSL numeric criteria, along with any data qualifiers assigned by the laboratory or during data validation efforts. Sample locations with chemistry exceeding numeric criteria will be mapped to delineate any areas that may require cleanup or other remedial action.

### 8.1.2 Fingerprinting Analysis of Sediment Data

A screening-level "fingerprinting" evaluation of TPH, PAH, and dioxin/furan sediment data will be conducted to provide a preliminary indication of the usefulness of the analytical data to differentiate between sources of contaminants. Three lines of forensic evidence will be qualitatively investigated: TPH, PAHs, and dioxin/furans (PCDD/PCDFs).

Petroleum hydrocarbons will be analyzed using the TPH method NWTPH-HCID for Hydrocarbon Identification. NWTPH-HCID is a qualitative and semi-quantitative screening tool that will be used to confirm the presence and type of petroleum product in a sediment sample. Results are qualitatively reported as gasoline, diesel, or heavy oils. The method is most useful for elimination of the need for more detailed petroleum analyses where NWTPH-HCID results indicate TPH concentrations are below regulatory limits. The reporting limits for sediment are 20 mg/kg for gasoline, 50 mg/kg for #2 diesel, and 100 mg/kg for motor oil. Pattern matching with known reference product chromatograms is used to identify (i.e. "fingerprint") the type of hydrocarbon. A laboratory analyst will categorize the TPH based on chromatogram identification. Herrera personnel will also visually evaluate the sample chromatograms to identify unique patterns, if any, associated with potential sources.

Both PAHs and PCDD/PCDFs have characteristic patterns and distributions in materials. For example, petroleum and wood combustion sources have different PAH analyte patterns, while wood industry and smelter activities generate different patterns of PCDD/PCDFs. The sediment data will be evaluated for relative analyte concentrations of PAHs and PCDD/PCDFs and compared to published data on the relative ratios in potential source materials. PAH and PCDD/PCDF analyte distribution in samples from potential source areas will also be visually evaluated to identify unique patterns, if any, associated with those sources.

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While the analytical data should meet the data quality objectives necessary to provide qualitative screening for guidance regarding the utility of the data for source differentiation, given the limited number of samples at each potential source quantitative evaluation of the data on a statistically significant basis will likely not be possible.

### 8.1.3 Analysis of Biological Data

The analysis of biological data will include comparison to SMS biological effects criteria. Toxicity test data results will be summarized and presented in tables indicating sampling locations and test results that exceed SQS and/or CSL biological effects interpretive criteria, along with the results of statistical comparisons to reference sediment test results. The sampling locations with sediment toxicity exceeding the SMS criteria will be mapped to delineate any areas that may require cleanup or other remedial action.

### 8.1.4 Tissue Residue Chemistry

The analysis of chemistry data will include a comparison of the results to the surface and subsurface sediment results. Tissue samples will be analyzed for SVOCs, chlorinated pesticides, metals, dioxin/furan compounds, and coplanar dioxin-like PCB congeners. The concentration of dioxin/furan compounds and PCB congeners will be normalized to 2,3,7,8-TCDD toxicity using toxic equivalent factors (TEFs) updated by the World Health Organization (WHO) in 2005 (Van den Berg et al. 2006). The toxic equivalent quotient is equivalent to the sum of the concentrations of individual congeners multiplied by their TEF (potency relative to 2,3,7,8-TCDD). Non-detected values will be assessed as half of the MDL for data evaluation purposes. The tissue sample data results will be reported as an addendum to the sediment quality investigation.

### 8.1.5 Radioisotope Dating

Sedimentation rate information, including sediment age in years, year of deposition, sediment accumulation rate (cm/yr), and sedimentation rate ($g/cm^2/yr$) will be determined and reported. The sedimentation rate is normally derived from lead-210 results; however, in some cases the cesium-137 data may be used to determine the sedimentation rate and sediment ages. Cesium-137 results are normally used to verify dates determined with sedimentation rates.

## 8.2 Sediment Investigation Report

A written SIR documenting all activities associated with collection, management, chemical analyses, and biological testing of collected samples will be prepared. The report will include recommendations for further action or investigation based on data comparisons to established evaluation criteria. Chemical, biological, and QA/QC reports will be included as appendices. As a minimum, the following will be included in the report:



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- Description of sampling and analysis activities;
- Protocols used during sampling and testing and an explanation of any deviations from SAP protocols;
- Physical descriptions of samples;
- Methods used for station positioning, sample collection locations reported in latitude and longitude to the nearest tenth of a second (NAD 83);
- Map showing actual locations of sampling stations and results of data comparisons to SMS and other criteria;
- Chain-of-custody records;
- Chemistry and biological testing results and laboratory reports;
- Comparison of data results to interpretive criteria;
- Fingerprinting analysis of sediment data results and interpretation;
- Radioisotope results and interpretation;
- QA/QC summary; and
- Data validation reports.

A geomorphic assessment of the shoreline and marine waters of Shelton Harbor and the southern shoreline of Oakland Bay will be based on site observations, historical assessment, core logs, and the geophysical surveys. This analysis will include the geomorphic ramifications of human activities cataloged in the historical analysis and the probable fate of past sediment-bound pollutant discharges discovered previously. It will include a listing of the physical processes potentially responsible for transport of existing polluted areas, as compiled from previous hydrographic studies in the area (e.g., Oakland Bay Dye Study), those processes interpreted from the geophysical survey and sedimentological features found in the core logs. In so doing, the assessment will provide the framework to interpret the results of the geophysical surveys and document the limits of particle-born pollutant transport in the study area. A technical memorandum will be produced describing geomorphic conditions in the study area with attached georeferenced graphics illustrating all salient features.

## 8.3 Human Health and Ecological Risk Assessment

Both an ERA and an HHRA will be prepared for the Oakland Bay study area. The purpose of the ERA and HHRA is to determine if contaminants in sediment pose risks to ecological receptors or people using the site. Historic and current contaminant sources to the study area include industrial facilities, commercial activities, and municipal sources. The results of the ERA and

HHRA will be used to determine if remedial measures are necessary to protect and/or restore the natural environment, and, if so, aid in the selection of appropriate remediation goals.

The ERA will include sections on ecological characterization; problem formulation; risk evaluation for marine plants, invertebrates, fish, and wildlife, and uncertainty evaluation. The HHRA will include sections on site characterization; problem formulation; risk evaluation for four categories of users (subsistence fisher, recreational fisher, recreational user, and residential user); and uncertainty analysis.

Chemical concentration data obtained through planned sampling activities (sediment and shellfish) will be used to model estimated chemical exposure to human and ecological users. Estimated exposure values will be compared to literature-based effect levels for human and ecological receptors to determine whether there is potential risk.

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