

**APPENDIX A  
CHEMICAL DATA QUALITY REVIEW AND  
CERTIFICATES OF ANALYSIS**

## APPENDIX A CHEMICAL DATA QUALITY REVIEW AND CERTIFICATES OF ANALYSIS

### A.1 Sediment Chemical Analysis

Twelve sediment samples and twelve subsamples for volatile organics and sulfide, were collected between February 17 and 23, 1999. The samples were submitted to MultiChem Analytical Services (MultiChem) of Seattle, WA for analysis of the following:

- ▶ Total Metals (EPA Method 6000/7000);
- ▶ Semivolatile Organics (EPA Method 8270, and 8270 SIM);
- ▶ Volatile Organics (EPA Method 8260);
- ▶ Pesticides/PCBs (EPA Method 8081/8082);
- ▶ Total Organic Carbon (EPA Method 9060 mod);
- ▶ Total Sulfide (PSEP);
- ▶ Ammonia (PSEP);
- ▶ Total Volatile Solids (PSEP); and
- ▶ Total Solids (PSEP).

The samples were also submitted to Columbia Analytical Services (CAS) of Kelso, WA for analysis of the following:

- ▶ Pore water Tributyltin (TBT, Krone et al., 1989);

The following criteria were evaluated in the standard data quality review process for the results:

- ▶ Holding times;
- ▶ Method blanks;
- ▶ Reporting limits;
- ▶ Surrogate recoveries;
- ▶ Blank spike/blank spike duplicate (BS/BSD) recoveries;
- ▶ Matrix spike/matrix spike duplicate (MS/MSD) recoveries; and
- ▶ Laboratory duplicates relative percent differences (RPDs).

Two batches of samples (SDG 909028 and 909026) were received by MultiChem at temperatures below the recommended  $4 \pm 2$  °C. Results were not qualified based on sample temperature receipt.

**Total Metals.** All required holding times were met. Reporting limits were below DMMP SLs. Zinc was detected in the digestion blank; however, associated

sample results were greater than five times the blank contamination so no results were qualified. BS and MS recoveries were within control limits. Serial dilution RPDs for zinc in several samples were greater than 10 percent. Associated sample results were qualified as estimated (J). Laboratory duplicate RPDs were acceptable with the exception of copper associated with C6, C11, and C12. Associated sample results were qualified as estimated (J).

**Semivolatile Organics.** All required holding times were met. Reporting limits were below DMMP SLs. Result for the individual isomers of benzofluoranthene could not be reported due to poor chromatographic resolution; results qualified with T indicate the result reported is the sum of the benzo(b) and benzo(k) isomers. Method blank contamination was present for phenol, benzoic acid, bis(2-ethylhexyl)phthalate, and di-n-butylphthalate. Associated sample results less than five times the phenol and benzoic acid contamination and ten times the phthalate contamination were qualified as not detected (U). Surrogate recoveries were within control limits with the following exceptions. 2,4,6-tribromophenol was below control limits in two method blanks. 2-fluorobiphenyl was below control limits in several samples. 2-fluorophenol and nitrobenzene was below control limits in C11. Qualifiers were not assigned since the other surrogate recoveries were acceptable.

BS/BSD recoveries were within control limits with the following exceptions. Hexachloroethane, 2,4-dimethylphenol, pentachlorophenol, and 1,2,4-trichlorobenzene recoveries were below control limits in the BS/BSD. BS/BSD and MS/MSD RPDs for these compounds were also out of control limits. MS/MSD recoveries of fluoranthene, benzo (a) pyrene, phenanthrene, 2,4-dimethylphenol, pentachlorophenol, and hexachloroethane were below control limits. 2,4-dimethylphenol and pentachlorophenol results from associated samples were already qualified as estimated by the laboratory. Hexachloroethane and results associated with low BS and MS recoveries were qualified as estimated (U/J).

BS/BSD RPDs of several compounds were out of control limits. No results were qualified since the associated BS recoveries and MS/MSD RPDs were acceptable. MS/MSD RPDs of several compounds were out of control limits. MS recoveries of several PAHs in sample C8 were not calculable due to sample inhomogeneity. Results were not qualified based on MS/MSD recoveries alone.

**Volatile Organics.** All required holding times were met. Reporting limits were below DMMP SLs. No method blank contamination was detected. Surrogate MS/MSD and BS recoveries were within laboratory control limits. MS/MSD RPDs were within control limits.

**Pesticides/PCBs.** All required holding times were met. Reporting limits were below DMMP SLs. No method blank contamination was detected. Surrogate and BS/BSD recoveries were within laboratory control limits with the following exceptions. Aldrin and heptachlor BS recoveries were below control limits. Results were not qualified since MS/MSD recoveries were acceptable. Recovery of TCMX in C4 was below control limits. No results were qualified since the remaining surrogate recovery was acceptable. Recoveries of several pesticides in the MS/MSD were below control limits because the MSD extract went dry during the concentrating step. The associated MS/MSD RPDs were also out of control limits. No results were qualified based on MS/MSD recoveries alone. Laboratory duplicate RPDs were within control limits.

**Total Organic Carbon.** All required holding times were met. No method blank contamination was detected. BS and MS recoveries were acceptable. Laboratory triplicates were not performed. The laboratory duplicate RPD associated with samples C11, C12, and C6 was out of control limits. Associated results were qualified as estimated (J).

**Total Sulfide.** All required holding times were met. No method blank contamination was present. MS recoveries were acceptable. Laboratory duplicate RPDs were within control limits.

**Ammonia.** All required holding times were met. No method blank contamination was present. BS and MS recoveries were acceptable. Laboratory duplicate RPDs were within control limits.

**Total Volatile Solids/Total Solids.** All required holding times were met. No method blank contamination was present. Laboratory duplicate RPDs were within control limits.

**Pore Water Tributyltin.** All required holding times were met. Pore water was extracted within seven days. Reporting limits were below DMMP SLs. No method blank contamination was present. Surrogate recoveries were within control limits. LCS and MS/MSD recoveries were acceptable.

## ***A.2 Tissue Chemical Analysis***

Thirty-six tissue samples from bioaccumulation test on Comp-1 and Comp-2 were collected on April 1, 2000. The samples were submitted to Columbia Analytical Services of Kelso, WA for analysis of Butyltins (TBT) and Lipids (Gravimetric).

The following criteria were evaluated in the standard data quality review process for the results:

- ▶ Holding times;
- ▶ Method blanks;
- ▶ Surrogate recoveries;
- ▶ Laboratory control sample recoveries; and
- ▶ Matrix spike/matrix spike duplicate (ms/msd) recoveries and relative percent differences (RPD).

All required holding times were met. No method blank contamination was present. Surrogate recoveries were within laboratory control limits. LCS and MS/MSD recoveries and RPDs were within control limits.

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**CERTIFICATES OF ANALYSIS  
COLUMBIA ANALYTICAL SERVICES**