

# Measurement Procedures

Ecology arranged for the MEL to analyze samples for the majority of toxic chemicals listed in Appendix E. Ecology and MEL subcontracted directly with specialty laboratories for testing not performed by the MEL.

Analytical methods and reporting limits for all target parameters are also provided in Appendix E. Detailed information on specific analytical procedures that were used for this project is provided below.

## **BNAs and Herbicides**

*Analyzed using USEPA SW-846 Method 8270*

Samples were analyzed by gas chromatography/mass spectrometry (GC/MS) following extraction and, if necessary, appropriate sample cleanup and derivatization procedures. Sample extracts were injected into a gas chromatograph (GC), equipped with a capillary column, which utilizes a temperature program to separate analytes which are then detected with a mass spectrometer. Analytes were identified by comparing electron impact spectra to the spectra of known standards. Analytes were quantified by comparing the response of a major ion relative to an internal standard using a calibration curve developed for each GC/MS.

## **PAHs**

*Analyzed using USEPA SW-846 Method 8270 SIM*

Method 8270 SIM is a modification of method 8270. Selected ion monitoring (SIM) enhances sensitivity by setting the mass spectrometer to detect specific ions rather than a range of ions. Sensitivity is generally increased by a factor of ten over standard mass spectrometer measurements. The primary disadvantage of SIM is a loss of qualitative information (unable to compare spectra).

## **Pesticides**

*Analyzed using USEPA SW-846 Method 8081*

Samples were analyzed by gas chromatography/electron capture detector (GC/ECD) following extraction and, if necessary, appropriate sample cleanup procedures. Sample extracts were injected into a GC, equipped with a capillary column, which utilizes a temperature program to separate analytes which were then detected with either an electron capture detector (ECD) or electrolytic conductivity detector (ELCD). Analytes were identified by comparing the retention time of target compounds with retention times of known standards on two dissimilar columns. Analytes were quantified by comparing the sample peak response using a calibration curve developed for each target compound.

## **PBDEs**

*Analyzed using EPA method GC/HRMS 1614*

Samples were analyzed for PBDE congeners using U.S. EPA SW-846 Method 1614 (Brominated Diphenyl Ethers in Water, Soil, Sediment, and Tissue by high resolution gas chromatography/high resolution mass spectrometry [HRGC/HRMS]) rather than U.S. EPA

Method 1668 as specified in the QAPP. This variation was acceptable because it provided equivalent or better data than required to meet project data quality objectives.

### **PCBs**

*Analyzed using EPA method GC/HRMS 1668*

Samples were analyzed using GC/HRMS following extraction and, if necessary, appropriate sample cleanup procedures. Sample extracts were injected into a GC, equipped with a capillary column, which utilizes a temperature program to separate analytes which were then detected with a HRMS. Congeners were identified by comparing the retention time and ion-abundance ratio of target compounds and associated labeled analog compounds with retention times and ion-abundance ratio of known standards. Congeners were quantified using the isotopic dilution quantitation technique, comparing the area of the quantification ion to that of the <sup>13</sup>C-labelled standard and correcting for response factors.

### **Metals**

*Analyzed using USEPA Method 200.8*

Samples were analyzed for dissolved metals following filtration of the whole water samples using a 0.45 micron fiber filter. Total metals analyses were conducted using unfiltered water samples.

Metals analyzed using Method 200.8 include: aluminum, arsenic, barium, beryllium, cadmium, cobalt, copper, lead, manganese, nickel, selenium, thallium, tin, and zinc. Samples were analyzed by inductively coupled plasma-mass spectrometry (ICP-MS) following acid extraction. Sample extracts injected into the ICP-MS were quantified by comparing instrument response to a calibration curve developed for each analyte. Results were reported for total (unfiltered) and filtered metals.

### **Calcium and Magnesium**

*Analyzed using USEPA Method 200.7*

Samples were analyzed for dissolved calcium and magnesium following filtration of the whole water samples using a 0.45 micron fiber filter. Total calcium and magnesium analyses were conducted using unfiltered water samples.

Samples were analyzed by inductively coupled plasma-atomic emission spectroscopy (ICP-AES) following acid extraction. Sample extracts injected into the ICP-AES were quantified by comparing instrument response to a calibration curve developed for each analyte. Results were reported for total (unfiltered) and filtered calcium and magnesium.

### **Mercury**

*Analyzed using either EPA Method 245.7 or EPA Method 7470*

Samples were analyzed for dissolved mercury following filtration of the whole water samples using a 0.45 micron fiber filter. Total mercury analyses were conducted using unfiltered water samples.

Samples may have been analyzed using Method 245.7 “Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry”. Samples would be oxidized and the ionic mercury would then be reduced to form volatile mercury. Volatile mercury is purged from the sample solution into a cold-vapor atomic adsorption (CVAA). Concentration is determined by measuring sample fluorescence against the fluorescence of known standards.

Samples may also have been analyzed using Method 7470 “Mercury in Liquid Waste (Manual Cold Vapor Technique)”. This technique is similar to Method 245.7 but uses different reagents to oxidize and then reduce the mercury. Instead of measuring fluorescence, this method measures absorbance using an atomic absorption spectrophotometer. Concentration is determined by measuring sample absorbance against the absorbance of known standards.

### **Total Petroleum Hydrocarbons (as gasoline)**

*Analyzed using Washington Department of Ecology Method NWTPH-Gx*

Samples were purged using a purge/trap concentrator and analyzed by gas chromatograph using a flame ionization detector (GC/FID). Volatile petroleum hydrocarbon concentrations as gasoline were measured by integrating sample responses (e.g., peak areas) to responses from a calibration curve developed using a regular unleaded gasoline standard. The range of peaks associated with gasoline generally includes compounds in the molecular weight range of benzene to naphthalene.

### **Total Petroleum Hydrocarbons (as diesel and as lube oil)**

*Analyzed using Washington Department of Ecology Method NWTPH-Dx*

Samples were solvent extracted and analyzed by GC/FID. Semi-volatile petroleum hydrocarbon concentrations as diesel were measured by integrating sample responses (e.g., peak areas) to responses from a calibration curve developed using a #2 diesel standard. The range of peaks associated with diesel generally includes compounds in the molecular weight range of jet fuels through #2 diesel. Semi-volatile petroleum hydrocarbon concentrations as lube oil were measured by integrating sample responses (e.g., peak areas) to responses from a calibration curve developed using a non-synthetic SAE 30 weight motor oil standard. The range of peaks associated with lube oil generally includes compounds such as lubricating oils, heavy fuel oils and mineral oils.

### **Oil and Grease, HEM**

*Analyzed using USEPA Method 1664, Rev. A*

This method measures oil and grease, HEM by extracting a sample with n-hexane then desiccating the extract and weighing the residue. The materials extracted include: non-volatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases and related materials.

Silica gel treated HEM (SGT-HEM; non-polar material) may have been analyzed by re-dissolving the residue and using silica gel to remove polar compounds, then desiccating the sample and weighing the residue. Lower reporting limits may be achieved by increasing the volume of the water sample. In addition to the standard analysis described here, a “Diesel [& Lube Oil] Extract of Oil and Grease, HEM” was conducted. The final weighed residue of the gravimetric Oil and Grease, HEM analyses was re-suspended in methylene chloride and the material analyzed using the GC portion of the NWTPH-Dx method described above.

## **Hardness**

*Measured using Standard Methods method 2340 B*

Total hardness was calculated after determining calcium and magnesium concentrations in the sample. Calcium and magnesium were analyzed following USEPA Method 200.8 (see above).

## **Ammonia Nitrogen**

*Measured using Standard Methods method 4500-NH3 H*

A water sample was injected into a flow injection analyzer (FIA) and following reaction with various reagents, producing a blue-colored dye. The intensity of the dye's absorbance is measured with an absorbance detector. Concentration was measured by comparing sample absorbance to a standard curve of absorbance determined from known ammonia standard concentrations.

## **Nitrate and Nitrite Nitrogen**

*Measured using Standard Methods method 4500-NO3 I*

A water sample was passed through a copperized cadmium column to reduce nitrate ( $\text{NO}_3$ ) to nitrite ( $\text{NO}_2$ ). The sample was then injected into a FIA and following reaction with various reagents, produces a magenta-colored dye. Total  $\text{NO}_3 + \text{NO}_2$  concentration was measured by comparing sample absorbance to a standard curve of absorbance determined from known nitrate standard concentrations.

## **Total Nitrogen**

*Measured using Standard Methods method 4500-N B*

A water sample was injected into a FIA. In-line ultraviolet/persulfate digestion and oxidation followed by reaction with various reagents, produces a pink-colored dye. The intensity of the dye's absorbance is measured with an absorbance detector. Concentration is measured by comparing sample absorbance to a standard curve of absorbance determined from known standard concentrations of nitrate and nitrite compounds. This method measures nearly all forms of organic and inorganic nitrogen.

## **Dissolved Organic Carbon**

*Measured using Standard Methods method 5310 B*

There are several options for measuring dissolved organic carbon in this method; all of which are similar to the description below.

A field-filtered water sample is first treated by acidification and purging to remove inorganic carbon. The sample is then injected into a high temperature reaction chamber containing an oxidative catalyst. Organic carbon is oxidized to carbon dioxide ( $\text{CO}_2$ ) which is carried in a gas stream to a non-dispersive infrared analyzer which measures the concentration of the  $\text{CO}_2$ . Concentration is determined by comparing measured  $\text{CO}_2$  sample concentration to a standard concentration curve determined from known standard concentrations of potassium biphthalate.

### **Total Organic Carbon**

*Measured using Standard Methods method 5310 B*

The method is the same as for dissolved organic carbon above; only the filtering step is eliminated.

### **Orthophosphate Phosphorus**

*Measured using Standard Methods method 4500-P G*

Water sample is injected into a FIA and following reaction with various reagents, produces a blue complex. The intensity of the complex's absorbance is measured with an absorbance detector. Orthophosphate (PO<sub>4</sub>) concentration is measured by comparing sample absorbance to a standard curve of absorbance determined from known orthophosphate standard concentrations.

### **Total Phosphorus**

*Measured using Standard Methods method 4500-P F*

A water sample is digested using acid and persulfate to convert the phosphate in organic and inorganic compounds to orthophosphate. Following neutralization the sample is injected into a FIA and following reaction with various reagents produces a complex that is reduced with ascorbic acid to form a new blue complex. The intensity of the complex's absorbance is measured with an absorbance detector. Total phosphorus concentration is measured by comparing sample absorbance to a standard curve of absorbance determined from known orthophosphate standards that have been carried through the entire procedure.

### **Total Suspended Solids**

*Measured using Standard Methods method 2540 D*

Water sample is filtered through a weighed glass-fiber filter. The filter and residue are dried at 103 to 105°C. The increase in weight is the mass of the total suspended solids.

### **Field Parameters (dissolved oxygen, pH, specific conductance, and temperature)**

*Measured using a YSI 556 meter*

The YSI 556 multi-parameter meter is a hand held instrument. Dissolved oxygen is measured using an internal polarographic sensor. pH is measured using a glass combination electrode. Specific conductance is measured using a 4-electrode conductivity cell. Temperature is determined with a precision thermistor. The meter is calibrated according to manufacturer specifications prior to use.

*This page is purposely left blank*