Appendix D

Analytical Reports and Quality Assurance Review

Appendix D – Laboratory Analytical Reports and Quality Assurance/Quality Control Results Summary

1.0 Introduction

This appendix documents the results of a quality assurance (QA) review of the analytical data for sediment samples that were collected during the Phase II Environmental Site Assessment for Mirror Pond, in Bend, Oregon.

The sediment samples collected were analyzed by ESC Lab Sciences of Mount Juliet, TN.

The QA review outlines the applicable quality control criteria utilized during the data review process, as well as any deviations from those criteria. Examination and validation of the laboratory summary reports include:

- Analytical methods;
- Reporting limits;
- Detection limits and estimated concentrations;
- Sample holding times;
- Custody records and sample receipt;
- Spikes, blanks, and surrogates; and
- Duplicates.

The QA review did not include a review of calibration or raw data. Section 2.0 lists the analytical methods used in sample analysis. Section 3.0 defines the QA terms used in this report. Section 4.0 provides the QA results.

2.0 Analytical Methods

2.1 Sediment Analyses

- Diesel/oil-range hydrocarbons by Northwest Method NWTPH-Dx,
- VOCs by EPA Method 8260B,
- PAHs by EPA Method 8270C-SIM,
- Metals (antimony, arsenic, barium, cadmium, chromium, lead, copper, mercury, selenium, silver and zinc) by EPA 6010/6020 method,
- Mercury by EPA 7471b
- Polychlorinated biphenyls (PCBs) Aroclors by EPA Method 8082.

- Pesticides by EPA 8081
- Herbicides by EPA 8151
- Total Organic Carbon (TOC)– EPA 5310B mod of EPA 9060

3.0 Quality Assurance Objectives and Review Procedures

The general QA objectives for this project were to develop and implement procedures for obtaining, evaluating, and confirming the usability of environmental data of a specified quality. To collect such information, analytical data must have an appropriate degree of accuracy and reproducibility, samples collected must be representative of actual field conditions, and samples must be collected and analyzed using unbroken chain-of-custody (COC) procedures.

Reporting limits and analytical results were compared to action levels for each parameter. Precision, accuracy, representativeness, completeness, and comparability parameters used to indicate data quality are defined below.

Reporting Limits. Method reporting limits (MRLs) are set by the laboratory and are based on instrumentation abilities, sample matrix, and suggested MRLs by the EPA or DEQ. In some cases, the MRLs are raised due to high concentrations of analytes in the samples or matrix interferences. MRLs are generally consistent with industry standards and below promulgated regulatory standards when possible.

Detection Limits and Estimated Concentrations. The method detection limit (MDL) is the lowest quantity of a substance that can be distinguished from the absence of that substance within a stated confidence limit. The MDL is estimated from the mean of the blank, the standard deviation of the blank and some confidence factor.

Holding Times. Holding times are the length of time a sample can be stored after collection and prior to analysis without significantly affecting the analytical results. Holding times vary by analyte, sample matrix, and analytical methodology.

Custody Records and Sample Receipt. COC refers to the document or paper trail showing the collection, custody, control, transfer, analysis, and disposition of physical materials. The sample receipt identifies the condition of samples upon arrival at the analytical laboratory.

Method Blanks. A method, or laboratory, blank is a sample prepared in the laboratory along with the field samples and analyzed for the same parameters at the same time. It is used to assess for laboratory introduced contamination.

Laboratory Control Sample. A laboratory control sample (LCS) is analyzed by the laboratory to assess the accuracy of the analytical equipment. The sample is prepared from an analyte-free matrix that is then spiked with known levels of the constituents of interest (i.e., a standard). The concentrations are measured and the results compared to the known spiked levels. This comparison is expressed as percent recovery.

Laboratory Control Sample Duplicate. A laboratory control sample duplicate [LCSD]) is prepared and analyzed along with the LCS. The LCS and LCSD data are compared to assess the precision of the analytical method (i.e., the relative percent difference [RPD]).

Matrix Spike Analyses. Matrix spike (MS) analyses are performed on samples submitted to the laboratory that are of the same matrix as the field sample. The MS sample is spiked with known levels of the constituents of interest and analyzed to assess the potential for matrix interference with recovery or detection of the constituents of interest and the accuracy of the determination. The spiked sample results are compared to the expected result (i.e., sample concentration plus spike amount) and reported as percent recovery.

Lab Duplicate. A laboratory duplicate is a second analysis of a QA/QC sample, which serves as an internal check on laboratory quality as well as potential variability of the sample matrix. The laboratory duplicate is analyzed and compared to the primary sample results to assess the precision of the analytical method. This comparison can be expressed by the RPD between the primary and duplicate samples.

Surrogate Recovery. Surrogates are organic compounds that are similar in chemical composition to the analytes of interest and spiked into environmental and batch QC samples prior to sample preparation and analysis. Surrogate recoveries for environmental samples are used to evaluate matrix interference on a sample-specific basis.

Field Duplicate. A field duplicate is a second field sample collected from a sampling location (e.g.., a well or sediment core). Field duplicate samples serve as a check on laboratory quality as well as potential variability of the sample matrix. The field duplicate is analyzed and compared to the primary sample to assess the precision of the analytical method. This comparison can be expressed by the RPD between the original and duplicate samples.

Trip Blanks. A trip blank is a sample prepared in the laboratory that is shipped along with the sample bottles to the field, kept with the sediment and groundwater samples during collection, and shipped back to the laboratory with the field samples. The trip blank is analyzed for constituents of interest, along with the

primary samples, to assess if detected contaminants may have been the result of contamination of the samples during transport or storage.

4.0 QA/QC Review Results

The data reviewed includes sediment samples collected from Mirror Pond from June 7 and 8, 2016. Samples were analyzed using the methods listed in Section 2.1.

Reporting Limits. With the exception of the TPH-Dx analyses, MRLs were not elevated for samples. For TPH-diesel range and residual range values, the analysis required dilutions ranging from 20 time to 50 times. As a result, reporting limits were elevated. No data are flagged.

Detection Limits and Estimated Concentrations. Concentrations were reported below method reporting limits at concentrations that are estimated. The results are flagged with a "J".

Holding Times. The samples were analyzed within applicable holding times.

Custody Records and Sample Receipt. The samples were received below the required temperature of 4°C and consistent with the accompanying COC.

Method Blanks. No compounds were detected in the method blanks. No data are flagged.

Laboratory Control Sample. Percent recoveries of the LCS were within control limits.

Laboratory Control Sample Duplicate. Percent recoveries of the LCSD were within control limits. LCS/LCSD RPDs were below acceptable limits for QA samples associated with several TPH-Dx samples. The RPD between the associated MS/MSD was within acceptable limits, establishing the precision of the analysis; therefore, no data were flagged.

Matrix Spike Analyses. With the following exceptions, the RPD for the MS and MS duplicate (MSD) were within control limits:

- The MS and/or MSD recoveries for acetone, acrylonitrile, benzene, bromobenzene, bromomethane, sec-butylbenzene, tert-butylbenzene, carbon tetrachloride, chlorobenzene, chloroethane, 2-choroethyl vinyl ether, chloroform, chloromethane, 2-chlorotoluene, 4-chlorotoluene, 1,2-dibromo-3-chloropropane, dichrlordifluromethane, 1,1-dichloroethane, 1-2, dichloroethane, 1,1-dichloroethene, cis-1,2-dichloroethene, trans-1,2-dichloroethene, 1,2-dichloropropane, 1,3-dichloropropane, 2,2-dichloropropane, di-isopropyl ether, ethylbenzene, isopropylbenzene, p-isopropyltoluene, 2-butanone (MEK), methylene chloride, 4-methyl-2-

pentanone (MIBK), methyl tert-butyl ether, n-propylbenzene, tetrachloroethene ,toluene, 1,1,2-trichlorotrifluoroethane, 1,1,1-trichloroethane, trichlorofluoromethane, 1,2,4-trimethylbenzene, 1,3,5-trimethylbenzene, vinyl chloride, and total xylenes are above control limits. The associated LCS and LCSD duplicate sample results are within control limits, so no data are flagged.

- The MS and/or MSD recoveries for dinoseb, 4,4-DDD, 4,4-DDT, and heptachlor, are below control limits. The associated LCS and LCSD duplicate sample results are within control limits, so no data are flagged.
- The MS/MSD RPD for several samples QA/QC samples were outside of the acceptable range for precision. The associated LCS and LCSD duplicate sample results are within control limits, indicating the precision of the analysis is acceptable.

Lab Duplicate. No lab duplicate was analyzed.

Surrogate Recovery. With the following exceptions, surrogate recoveries were within control limits for samples:

- For composite sample SS-5 - SS-6 (4 -5 ft), one of four surrogate recoveries was below control limits. The applicable sample analytes have been flagged with a "J2" qualifier.

Field Duplicate. A field duplicate was not collected.

Trip Blank. Xylenes were detected in the trip blank at an estimated concentration of 1.74 mg/kg. Xylenes were not detected in project samples; therefore, no data are flagged.

Conclusion. In conclusion, the overall QA objectives have been met and the data are of adequate quality for use in this project.