DATA VALIDATION REPORT

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Project/Site: Siltronic Sediment Sampling
Laboratory No: A0A0740

This report presents the validation of the data obtained during the field activities for the above referenced work assignment. The purpose of this review is to provide a Level 2A technical validation and quality control review of the following samples collected between October 7, 2019 and October 10, 2019 and submitted to APEX Laboratories, LLC. Portland, OR. In addition, raw data associated with samples SED-05-SB-2, SED-07-SB-6.35, SED-03-SB-8.45, and SED-02-SS-1.0 were evaluated as part of a Level 4 review in order to meet the 10% project requirements.

Field Sample Numbers	Laboratory ID	Analyses/Methods
SED-05-SB-2	A0A0740-01	Additional Alkylated PAH Homologs by 8270D (Modified)
SED-05-SB-5	A0A0740-02	
SED-05-SB-7	A0A0740-03	
SED-06-SB-2.0	A0A0740-04	
SED-06-SB-5.5	A0A0740-05	
SED-06-SB-8.5	A0A0740-06	
SED-04-SB-2.0	A0A0740-07	
SED-04-SB-4.75	A0A0740-08	
SED-04-SB-7.75	A0A0740-09	
SED-07-SB-2.0	A0A0740-10	
SED-07-SB-4.35	A0A0740-11	
SED-07-SB-6.35	A0A0740-12	
SED-01-SB-2.0	A0A0740-13	
SED-01-SB-5.5	A0A0740-14	

Field Sample Numbers	Laboratory ID	Analyses/Methods
SED-02-SB-2.0	A0A0740-15	Additional Alkylated PAH Homologs by 8270D (Modified)
SED-01-SB-8.65	A0A0740-16	
SED-02-SB-5.0	A0A0740-17	
SED-02-SB-8.25	A0A0740-18	
SED-03-SB-2.0	A0A0740-19	
SED-03-SB-5.0	A0A0740-20	
SED-03-SB-8.45	A0A0740-21	
SED-08-SB-2.0	A0A0740-22	
SED-08-SB-3.25	A0A0740-23	
SED-09-SB-2.0	A0A0740-24	
SED-09-SB-4.85	A0A0740-25	
SED-09-SB-6.85	A0A0740-26	
SED-10-SB-2.0	A0A0740-27	
SED-10-SB-5.2	A0A0740-28	
SED-10-SB-7.2	A0A0740-29	
SED-01-SS-1.0	A0A0740-30	
SED-02-SS-1.0	A0A0740-31	
SED-03-SS-1.0	A0A0740-32	
SED-04-SS-1.0	A0A0740-33	
SED-05-SS-1.0	A0A0740-34	
SED-06-SS-1.0	A0A0740-35	
SED-07-SS-1.0	A0A0740-36	

Field Sample Numbers	Laboratory ID	Analyses/Methods
SED-08-SS-1.0	A0A0740-37	Additional Alkylated PAH Homologs by 8270D (Modified)
SED-09-SS-1.0	A0A0740-38	
SED-10-SS-1.0	A0A0740-39	

The data submitted by the laboratory has been reviewed and verified for compliance with the Sediment Sampling Work Plan Willamette River Mile 6.55 to 6.9, West Siltronic Corporation Portland, Oregon prepared by Maul Foster & Alongi, Inc. (MFA) (May 2019) and the analytical procedures listed in the Test Methods for Evaluating Solid Wastes, SW-846, 3rd Edition and other referenced analytical methods. Data validation/data quality review was conducted in accordance with the current or most applicable versions of the National Functional Guidelines (NFG) for Superfund Organics Method Data Review (January 2017). Laboratory QC limits/acceptance limits were used to evaluate the data unless where noted.

Both a Stage 4 and Stage 2A Manual Validation as defined in the Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use, EPA-540-R-08-005, January 2009 USEPA, was performed. The data were evaluated based on the following parameters:

- Chain-of-Custody (COC)
- Case Narrative
- Field and Sample ID's
- Holding Time, including sample receipt, Preservation and Cooler Temperature
- Instrument Stability and Performance [e.g., MS tuning, interference check samples, chromatographic resolution] (Level 4)
- Calibration and Calibration Verification [e.g., initial calibration, initial calibration verification (ICV) and continuing calibration verification (CCV)] (Level 4)
- Laboratory Blanks (method blank; reagent/preparation blanks)
- Laboratory Control Samples (LCS)
- Matrix Spikes (MS)
- Laboratory Duplicates
- Field Duplicates
- Surrogate (DMC) Recovery
- Internal Standards (Level 4)
- Sample Results [e.g., Recalculation and Reduction of Results from Raw Data, Transcription Check, and Analyte Identification] (Level 4)

Data Completeness (Chain-of-Custody, Case Narrative, Field and Sample IDs)

The Level 4 data package initially submitted was incomplete as raw data quantitation reports were missing. A revised package was provided and included COC forms, a case narrative, identification of field and sample numbers, sample results, laboratory quality control results, instrument calibration; calibration verifications, sample receipt information, and all appropriate raw data.

The COC forms were properly filled out including signatures, date and time of sampling, sampling identification, analyses requested, and custody transfers between different parties were signed and dated. The samples collected were appropriately identified and analyzed as per the COC.

Case narratives or a list of laboratory flags (Notes and Definitions) were provided and QC anomalies and QC outliers were noted.

Holding Times, Preservation and Cooler Temperature

The samples were received by the laboratory in good condition and within the recommended temperature range of 4 ± 2°C or just below, but not frozen.

Analytical holding times were assessed to determine whether the method holding time requirements were met by the laboratory. Upon arrival at the laboratory, the alkylated PAH homologs volume was frozen to a temperature of -18°C rather than being stored at a temperature of 4 ± 2°C because it was unknown whether the alkylated PAH homologs analysis would be needed. The eventual extraction of the alkylated PAH homologs analysis for these samples was performed 28 to 31 days after sample collection which was within the one year holding time for frozen samples as indicated by Table 4-2 of the Sediment Sampling Work Plan. No qualification of the results was required because the extraction was performed within the extended, one year holding time.

Instrument Stability and Performance (e.g., MS tuning, interference check samples, chromatographic resolution) (Level 4)

Tunes / Instrument Performance
The instruments were tuned prior to calibration and calibration verification at the correct frequency. All instrument tune criteria were met for Method SW8270D. No raw data issues were noted.

Initial and Continuing Calibrations (Level 4)

The instruments associated with the Level 4 samples were calibrated at the required frequency and with the appropriate number of standards. The lowest calibration standards were at or near the laboratory reporting or quantitation limits. The relative standard deviation (%RSDs) were less than method calibration requirements or the correlation coefficients were greater than 0.99.

Initial Calibration Verification (ICV)
All second source ICV method criteria were met.

Continuing Calibration Verification
The CCV standards were analyzed at the correct frequency. All CCV or continuing calibration method criteria were met.

Laboratory Blanks (method blank; reagent/preparation blanks)

The method blanks were prepared and analyzed as appropriate and at the required frequency. No contaminants were found in the laboratory method blanks.

Field Blanks

The field blanks were not analyzed for these additional PAH analytes.

Laboratory Control Samples

At least one laboratory control sample (LCS) analysis was analyzed with each QC prep batch. Accuracy and precision were evaluated using these analyses.

All LCS recoveries were within the laboratory QC limits.

Matrix Spikes (MS)

MS analyses were performed on samples SED-04-SS-1.0 and SED-10-SS-1.0. All recoveries were within the laboratory QC limits.

Laboratory Duplicates

The laboratory analyzed a laboratory duplicate on samples SED-05-SB-2 and SED-01-SB-2.0. All laboratory duplicate criteria were met.

The laboratory flagged the result for C1-Decalin in sample SED-01-SB-2.0 for not meeting duplicate criteria. However, no qualification was required because the results or the difference between the results were less than the reporting limits and the result are acceptable.

Field Duplicates

The field duplicates were not analyzed for the additional PAH analysis.

Surrogate (DMC) Recovery (Organics)

Surrogate compounds were appropriately added to all samples and QC samples for this analysis. The surrogate percent recoveries were within laboratory QC limits for all analyses.

Internal Standards

Internal standards (IS) were added to every field sample, standard, and QC sample for the additional organic GCMS analysis. All internal standard acceptance criteria were met for these analyses.

Sample Results

All appropriate raw data associated with samples SED-05-SB-2, SED-07-SB-6.35, SED-03-SB-8.45, and SED-02-SS-1.0 were included. The raw data were evaluated to verify reduction of the sample results, calibrations, blank, and QC results to the results summary forms. Calculations were performed to verify quantitation accuracy. The appropriate sample sizes, final volumes, dilution or run factors were used and no transcription or calculation errors were observed. The results and reporting limits or detection limits were correctly reported.

These samples were diluted due to high target or non-target concentrations. The non-detected results for these samples are at elevated detection limits due to the dilutions performed on these samples.

Overall Assessment

The analytical data are acceptable and usable as reported without qualifications.

DATA QUALIFIER DEFINITIONS

For the purpose of Data Validation, the following validation qualifiers and associated definitions are provided for use by the data validator to summarize the data quality.

Data Qualifier	Description
Standard Data Qualifiers	
U	The analyte was analyzed for, but was not detected at or above the associated value.
UJ	The analyte was not detected. The reported sample quantitation limit is considered estimated for QC reasons.
J	The analyte was detected. The reported numerical value is considered estimated for QC reasons.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
R	The sample result is rejected as unusable due to serious deficiencies in one or more QC criteria. The analyte may or may not be present in the sample.
K	Estimated Maximum Possible Concentration (EMPC)