DATA VALIDATION REPORT

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Report Date: Revised December 23, 2019
Project/Site: Siltronic Sediment Sampling
Laboratory No: A9J0277, WO15627, B9S5599, 570-9778-1

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 and rinsate blank collected on October 7, 2019 and submitted to APEX Laboratories, LLC. Portland, OR.

Field Sample Numbers	Laboratory ID	Analyses/Methods
SED05-SB-2	A9J0277-01 15627001 KZY764 570-9778-1	Diesel and Oil Hydrocarbons by NWTPH-Dx Semivolatiles and PAHs by GC/MS SW8270D Alkylated PAH Homologs by 8270D (Modified) Total metals and mercury (ICP-MS) by SW6020A Cyanide - Total (solid) by ASTM D7511 Total Organic Carbon (solid) by EPA 9060A Mod Total Solid Determination by PSEP-TS Grain Size by ASTM D 422M/PSET Parameters Percent Dry Weight by SW8000C Chlorinated Herbicides by 8151A Percent Solids by EPA 160.3M Dioxins and Furans by Method 1613B PCB Congeners by Method 1668C Organochlorine Pesticides by BRL SOP 00014/1, GC/MS/MS (EPA Method 1699 Modified) Organotins (Tributyltin) by GC/MS SIM
SED05-SB-5	A9J0277-03 15627002 KZY765 570-9778-2	
SED05-SB-7	A9J0277-04 15627003 KZY766 570-9778-3	
SED05-SB-RB	A9J0277-05 15627004 KZY767 570-9778-4	Diesel and Oil Hydrocarbons by NWTPH-Dx Semivolatiles and PAHs by GC/MS SW8270D Total metals and mercury (ICP-MS) by SW6020A Cyanide – Total (aqueous) by EPA 335.4 Total Organic Carbon by SM5310C Chlorinated Herbicides by 8151A Dioxins and Furans by Method 1613B PCB Congeners by Method 1668C Organochlorine Pesticides by BRL SOP 00014/1, GC/MS/MS (EPA Method 1699 Modified) Organotins (Tributyltin) by GC/MS SIM

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), the NFG for Superfund Inorganics Method Data Review (January 2017), and the NFG for High Resolution Superfund Methods Data Review (April 2016), along with the Region 10 Data Validation and Review Guidelines for Polychlorinated Dibenzo-p-Dioxin and Polychlorinated Dibenzofuran Data (PCDD/PCDF) Using Method 1613B, and SW846 Method 8290A, May 2014, modified for the method criteria. Laboratory QC limits/acceptance limits were used to evaluate the data unless where noted. Based on discussions with the data users, AlterEcho did not verify the toxic equivalencies (TEQs) listed for Dioxins and Furans in the laboratory reports since these factors will not be used for data reporting. Also, AlterEcho did not verify the Total PCB Congener concentrations listed in the laboratory reports since the data user plans to recalculate the Total PCB Congener concentrations using the validated data.

The herbicide samples were subcontracted to Weck Laboratories, Inc. and reported in the Apex Laboratories report. The Dioxins and Furans and PCB Congener samples were subcontracted to Cape Fear Analytical, LLC (Work Order WO15627) while the Organotins samples were subcontracted to Eurofins Calscience (Work Order 570-9778-1). The samples were subcontracted to Bureau Veritas Laboratories (formerly Maxxam Analytics International (Data Package B9S5599) for Organochlorine Pesticides by BRL SOP 00014/1, GC/MS/MS (EPA Method 1699 Modified). Samples were shipped and received under proper custody and preservation.

A 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 on the samples. The data were evaluated based on the following parameters:

- Chain-of-Custody
- Case Narrative
- Field and Sample ID's
- Holding Time, including sample receipt, Preservation and Cooler Temperature
- Laboratory Blanks (method blank; reagent/preparation blanks)
- Field Blanks
- Laboratory Control Samples
- Matrix Spike/Matrix Spike Duplicates
- Post Digestion Spikes
- Laboratory Duplicates
- Field Duplicates
- Serial Dilution Samples (Metals)
- Surrogate (DMC) Recovery (Organics)
- Labeled Compounds and Clean-Up Standards (Dioxins/Furans, PCB Congeners, and Organochlorine Pesticides and Toxaphene)
- Sample Results

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

The Level 2A data package was reviewed and included chain-of-custody (COC) forms, a case narrative, identification of field and sample numbers, sample results, laboratory quality control results, and sample receipt information. Raw data and instrument performance and calibration data are not evaluated for Level 2A data validation.

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. The holding times were met as all samples were prepared and/or analyzed within the method suggested holding times except as noted below.

Alkylated PAH Homologs by 8270D (Modified)
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 samples SED05-SB-2, SED05-SB-5, SED05-SB-7 and SED05-SB-RB was performed 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.

Organotins (Tributyltin) by GC/MS SIM
The extraction of the equipment rinse blank sample was performed eight days after sample collection. Since the seven day extraction holding time was exceeded, the following not detected result has been qualified as at an estimated reporting limit (UJ).

- Tributyltin for sample SED05-SB-RB

Laboratory Blanks (method blank; reagent/preparation blank)

The method blanks and preparation blanks were prepared and analyzed as appropriate and at the required frequency. No contaminants were found in the laboratory method blanks and preparation blanks associated with these sample analyses with the exceptions noted below.

Semivolatile Organic Compounds
Naphthalene was detected in the method blank for QC Batch 9101003. However, the associated sample result was non-detected and therefore data are not qualified.

Chlorinated Herbicides
There were detections of 2,4,5-TP (Silvex); dicamba; dichloroprop; pentachlorophenol, and picloram in the method blank associated with batch W9J0915. Qualification was not appropriate because these analytes were not detected in the associated samples.

Dioxin/Furan
Numerous Dioxin/Furans were detected in the method blanks for QC Prep Batch 42063 and 42119. The majority of these blank results were reported as Estimated Maximum Possible Concentrations (EMPCs). The following results less than five times the method blank concentration are qualified as Non-Detected (U) at the sample concentration due to method blank contamination.

- 1,2,3,7,8-PeCDD and 1,2,3,4,7,8-HxCDD in sample SED05-SB-2
- 1,2,3,7,8-PeCDD, 1,2,3,4,7,8-HxCDD, and 1,2,3,7,8,9-HxCDF in sample SED05-SB-5
- 1,2,3,7,8-PeCDD and 1,2,3,4,7,8-HxCDD in sample SED05-SB-7

PCB Congeners
Several PCB congeners were detected in the method blank for QC Prep Batch 42073. The following sample results are less than five times the method blank concentration and are qualified as Non-Detects (U) at the sample concentration due to method blank contamination.

- 11-DiCB, 18/30-TrCB, 20/28-TrCB, 21/33-TrCB, 22-TrCB, 31-TrCB, 44/47/65-TeCB, 49/69-TeCB, 52-TeCB, 61/66/70/76-TeCB, 66-TeCB, 86/87/97/109/119/125-PeCB, 90/101/113-PeCB, 95-PeCB, 105-PeCB, 110/115-PeCB, 118-PeCB, 129/138/163-HxCB, 132-HxCB, 135/151-HxCB, 147/149-HxCB, 153/168-HxCB, 156/157-HxCB, 174-HpCB, 180/193-HpCB, and 187-HpCB in sample SED05-SB-RB

Numerous PCB congeners were detected in the method blank for QC Prep Batch 42154. However, no data were qualified as all associated sediment sample results were greater than five times the blank values or were not detected.

Note: Several of the sample results in the bullet items above were qualified as Estimated Maximum Possible Concentrations (EMPCs) by the laboratory. Since these results were qualified as not detected (U) due to method blank contamination, no additional action was required.

Field Blanks

Sample SED05-SB-RB was an equipment rinsate blank collected with these samples. No sample results were qualified for rinsate blank contamination because the associated sample results were greater than the reporting limit and 5 times the blank value or were non-detected.

Laboratory Control Samples

At least one laboratory control sample (LCS) analysis was analyzed per QC batch and for each analysis. A laboratory control sample duplicate (LCSD) was also analyzed with several methods if laboratory duplicates or matrix spikes were not performed. Accuracy and precision were evaluated using these analyses.

All LCS and LCSD recoveries were within the laboratory QC limits and all precision criteria were met as the RPDs were within laboratory QC limits with the exceptions noted below.

Semivolatile Organic Compounds
The RPD between LCS and LCSD recoveries for 1,2,4-trichlorobenzene; 1,2-dichlorobenzene; 1,3-dichlorobenzene; 1,4-dichlorobenzene; hexachlorobutadiene; hexachlorocyclopentadiene, and hexachloroethane in analytical batch 9101003 were above the control limit of less than 30%. Qualification was not appropriate because these compounds were not detected in the affected sample.

The LCS recoveries of carbazole, 4-nitroaniline, and 3,3'-dichlorobenzidine in analytical batch 9101307 were above the control limits. Qualification was not appropriate because these compounds were not detected in the affected samples.

The laboratory indicated that due to erratic or low blank spike recoveries, results for 3,3'-dichlorobenzidine are considered Estimated Values. However, the LCS/LCSD recoveries of 3,3'-dichlorobenzidine were within the control limits for analytical batch

9101003 or above the control limits in analytical batch 9101307. Qualification was not taken because this compound was not detected in any of the samples.

Chlorinated Herbicides
The LCS/LCSD RPDs for 2,4-DB and 4-nitrophenol in analytical batch W9J0721 were greater than the QC limit of 25%. However, qualification was not needed as the analytes were not detected in the samples.

Organochlorine pesticides
The LCS recoveries Endosulfan II (13%) and Endosulfan sulfate (15%), were below the QC limits in the LCS associated with batch 6385055. The following analytes have been qualified as estimated (UJ) due to the low LCS recoveries.

- Endosulfan II and Endosulfan sulfate in samples SED05-SB-2, SED05-SB-5, and SED05-SB-7

Note that these results were also qualified as EMPCs in sample SED05-SB-2.

The LCS recovery for Mirex (174%) was flagged as greater than the QC limits in the LCS associated with batch 6385055. However, qualification for high bias was not required as the analyte was not detected in the samples. Note that it appears that the incorrect QC limits of 50-200% were reported on the laboratory Quality Assurance Report.

Matrix Spike/Matrix Spike Duplicates (MS/MSD)

MS/MSD analyses were not requested on a sample from this SDG. However, the laboratory did perform a MS or MS/MSD on sample SED05-SB-2 for a few analyses. All MS/MSD recoveries were within the laboratory QC limits and all precision criteria were met as the RPDs were within laboratory QC limits with the exceptions noted below.

Chlorinated Herbicides
A MS/MSD was performed on sample SED05-SB-2 in analytical batch W9J0815. The RPD between MS and MSD recoveries of several analytes were above the control limit of 25%. Qualification was not appropriate because the affected analytes were not detected in the unspiked parent sample.

Organochlorine pesticides
The laboratory indicated that the recoveries for o,p-DDD, p,p-DDD, p,p-DDE, o,p-DDT, and p,p-DDT were not calculated (applicable) due to the high native concentrations of these analytes in the unspiked parent sample. No data validation qualifiers are added to the data.

The laboratory also indicated that the recoveries for Aldrin and o,p-DDE were not calculated due to matrix interferences. These two analytes were reported and qualified as EMPCs at elevated detection limits. No additional qualifiers were added to the data.

The laboratory also provided MS and/or MSD analyses that were performed on unknown samples from other SDGs or work orders for several analyses. Typically, sample data are not qualified using MS/MSD results from unknown samples or samples from other SDGs. Additionally, for organic analyses only the unspiked parent sample is usually qualified for the MS/MSD results unless a systematic issue is noted. Therefore, these MS/MSD analyses were not evaluated and no data in this SDG were qualified using only the matrix spike results from unknown non-site samples or site samples from other SDGs. Refer to the LCS/LCSD for precision and accuracy data.

Post Digestion Spikes (Metals)

A post digestion spike (PDS) was not provided or required.

Laboratory Duplicates

Duplicate analyses were not requested on the samples from this sample delivery group. The laboratory analyzed a laboratory duplicate on sample SED05-SB-2 for several analyses. All laboratory duplicate criteria were met with the exceptions noted below.

Semivolatile Organic Compounds
A laboratory duplicate was performed on sample SED05-SB-2. The laboratory noted that the RPD for fluorene could not be calculated. Qualification was not appropriate because this compound was not detected in the parent sample and the concentration detected in the laboratory duplicate was below the reporting limit.

Organochlorine pesticides
Duplicate RPDs exceeded 25% for o,p-DDT and p,p-DDT for the laboratory duplicate on sample SED05-SB-2. As a result of the exceeded precision criteria, the following detected results were qualified as estimated (J):

- o,p-DDT and p,p-DDT in sample SED05-SB-2

The laboratory also provided duplicate analyses that were performed on unknown samples from other SDGs or work orders. Other duplicate results were not evaluated as they were performed on unknown or non-site samples.

Field Duplicates

A field duplicate was not collected with these samples.

Serial Dilution Samples (Metals)

A serial dilution was not provided for the total and dissolved metals for the level 2A review.

Surrogate (DMC) Recovery (Organics)

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

Semivolatile Organic Compounds
The surrogates 2-Fluorobiphenyl and 2-Fluorophenol were recovered below the control limits in sample SED05-SB-RB. The following results have been qualified as estimated (UJ) due to low surrogate recovery anomalies.

- All SVOC results in sample SED05-SB-RB

Labeled Compounds and Clean-Up Standards (Dioxins/Furans, PCB Congeners, and Organochlorine Pesticides)

The recoveries of the labeled compounds and clean-up standards met the method or laboratory criteria.

Sample Results

Raw data and sample quantitation were not evaluated for this 2A review. The results and reporting limits or detection limits were correctly reported with the correct units and appeared to be adjusted for sample size and dilution.

According to the case narrative or lab notes, various analyses for these samples were diluted due to high target concentrations, high non target matrix interference, or sample matrix. The non-detected results for these analyses are at elevated detection limits due to the dilutions performed on these samples. Additionally, the reporting limits for several individual analytes were raised to account for interference from co-eluting analytes

present in the sample or dilution. These analytes are reported as not-detected at the raised detection limit/reporting limit.

Diesel Range Organics
The laboratory indicated that the oil results for samples SED05-SB-2, SED05-SB-5, and SED05-SB-7 are elevated due to the presence of individual analyte peaks in the quantitation range that are not representative of the fuel pattern reported. No qualification is required.

Dioxin/Furan
The results for OCDD in samples SED05-SB-5 and SED05-SB-7 were flagged for exceeding the instrument linear calibration range. These two results are considered estimated quantities and are qualified as estimated (J).

The following total dioxin and furan results were flagged (K) by the laboratory indicating the result was impacted by an EMPC. Results also below the PQL were flagged as (JK). These total results are considered estimated quantities and are qualified as an estimated value (JK).

- Total TeCDD, Total PeCDD, Total TeCDF, and Total PeCDF in sample SED05-SB-2
- Total TeCDD, Total PeCDD, Total TeCDF, Total PeCDF, and Total HxCDF in sample SED05-SB-5
- Total TeCDD, Total HxCDD Total TeCDF, and Total PeCDF in sample SED05-SB-7

The above total results were greater than the results for the individual dioxin/furan congeners or were impacted by both EMPCs and confirmed homologues and the results were qualified as estimated (JK) rather than as not detected.

A few total dioxin and furan results indicated above were also flagged as "Q" by the laboratory indicating that quantitative interference resulted in an estimated value. No additional qualification was required.

Confirmatory runs for 2,3,7,8-TCDF were analyzed for these samples and the detected results for 2,3,7,8-TCDF greater than the PQLs were confirmed by the second analysis. The results for 2,3,7,8-TCDF from both analyses were reported on the EDD. The confirmation results which should be reported were reported from the November 5, 2019 analysis.

PCB Congeners
Several PCB Congener results were reported as EMPCs and were qualified with the laboratory "K" flag denoting an EMPC value. Results which were also below the PQL

were reported by the laboratory as estimated (JK) values. The following EMPCs that were not previously qualified as non-detected due to method blank contamination are qualified as estimated non-detects (UJK) at the reported concentration in accordance with EPA Region 10 PCDD/PCDF DV guidelines and NFG use of regional guidance and/or professional judgment in evaluating these results.

- 1-MoCB, 10-DiCB, 35-TrCB, 54-TeCB, 96-PeCB, 126-PeCB, and 145-HxCB in sample SED05-SB-2
- 35-TrCB, 60-TeCB, 79-TeCB, 121-PeCB, 123-PeCB, 181-HpCB, and 182-HpCB in sample SED05-SB-5
- 57-TeCB and 121-PeCB in sample SED05-SB-7
- 99-PeCB in sample SED05-SB-RB

Note: The final Total PCB Congeners values should be adjusted based on blank contamination and EMPC actions noted in the previous sections.

Organochlorine pesticides
The laboratory indicated that several results were an EMPC / NDR as the peak detected does not meet ratio criteria and has resulted in an elevated detection limit. Results were reported as non-detected. The following results were qualified as estimated detection limit (UJK) to be consistent with the qualification of EMPCs:

- a-Chlordane, Aldrin, Endosulfan II, Endosulfan sulfate, and o,p-DDE in sample SED05-SB-2
- a-Chlordane, Aldrin, Dieldrin, Endrin, and o,p-DDE in sample SED05-SB-5
- a-Chlordane, Aldrin, Endrin, and o,p-DDE in sample SED05-SB-7
- Endosulfan II in sample SED05-SB-RB

Grain Size by ASTM D 422M/PSET Parameters
The laboratory note/narrative for samples SED05-SB-2 and SED05-SB-5 indicated that the No. 4 sieve (gravel) and No. 10 sieve (coarse sand) grain size fractions contained an abundance of organic material while the note/narrative for sample SED05-SB-7 indicated that the No. 4 sieve (gravel) grain size fraction consists entirely of organic material and the No. 10 sieve (coarse sand) grain size fraction contains abundant organic material.

Overall Assessment

The analytical data are acceptable and usable as reported with the minor qualifications noted above. Some results were qualified due to low surrogate and LCS recoveries or duplicate precision. Results for Dioxin/Furans, Organochlorine Pesticides and PCB Congeners were qualified as not detected due to blank contamination or as EMPCs.

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)