

**DATA VALIDATION REPORT**

Validated by: Bill Fear, AlterEcho  
 Report Date: Revised December 26, 2019  
 Project/Site: Siltronic Sediment Sampling  
 Laboratory No: A9J0427, WO15644, B9T0140, 570-10374-1

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 10, 2019 and submitted to APEX Laboratories, LLC. Portland, OR. In addition, raw data associated with samples SED-05-SS-1.0, SED-06-SS-1.0, SED-07-SS-1.0, and SED-08-SS-1.0 were evaluated as part of a Level 4 review in order to meet project requirements for Level 4 validation.

Field Sample Numbers	Laboratory ID	Analyses/Methods
SED-05-SS-1.0	A9J0427-01 15644001 LAY795 570-10374-1	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
SED-06-SS-1.0	A9J0427-02 15644002 LAY796 570-10374-2	
SED-07-SS-1.0	A9J0427-03 15644005 LAY797 570-10374-3	
SED-07-SS-1.0-DUP	A9J0427-04 15644006 LAY798 570-10374-4	
SED-08-SS-1.0	A9J0427-05 15644007 LAY799 570-10374-5	
SED-09-SS-1.0	A9J0427-06 15644008 LAY800 570-10374-6	
SED-10-SS-1.0	A9J0427-07 15644009 LAY801	

Field Sample Numbers	Laboratory ID	Analyses/Methods
	570-10374-7	
SED-SS-RB	A9J0427-08 15644010 LAY802 570-10374-8	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 WO15644) while the Organotins samples were subcontracted to Eurofins Calscience (Work Order 570-10374-1). The samples were subcontracted to Bureau Veritas Laboratories (formerly Maxxam Analytics International (Data Package B9T0140) 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.

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)
- Blanks [e.g., method blank; initial calibration blank (ICB), and continuing calibration blank (CCB), reagent/preparation blanks, and trip blank (VOC only) if specified in method] (Only method blank evaluated for Level 2A)
- Laboratory Control Samples (LCS)
- Matrix Spike/Matrix Spike Duplicates (MS/MSD)
- Post Digestion Spikes (Metals)
- 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)
- 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 was complete 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  $4 \pm 2^{\circ}\text{C}$  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 with the exceptions noted below.

#### *Organochlorine Pesticides*

The samples were collected on October 10, 2019 but were not extracted until November 4, 2019 which exceeds the 14-day holding time by 11 days. As a result of exceeded holding times, the following results were qualified as estimated with low bias (J- or UJ):

- All Organochlorine Pesticides in all sediment samples

Various results were also Estimated Maximum Possible Concentrations (EMPCs) and have a final qualifier of “UJK”.

#### *Tributyltin*

The rinsate blank sample was collected on October 10, 2019 but was not extracted until October 18, 2019 which exceeds the 7-day holding time by one day. As a result of exceeded holding times, the following non-detected result was qualified as estimated with low bias (UJ):

- Tributyltin sample SED-SS-RB

*Alkylated PAH Homologs*

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  $4 \pm 2^\circ\text{C}$  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 33 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 Methods SW6020A Method SW8260C and Method SW8270D. No raw data issues were noted.

Resolving power and instrument performance checks were analyzed prior to the initial calibration and at the beginning of each 12-hour period of analysis. All instrument resolving power criteria were met. Peak separation between 2,3,7,8-TCDD and the other TCDD isomers met the  $\leq 25\%$  criteria. Peak separation between the 23-TrCB/34-TrCB and 182-HpCB/187-HpCB isomer pairs met the  $\leq 40\%$  criteria.

*Interference Check Samples (Metals)*

All interference check sample percent recoveries for ICSAB for Method SW6020A were within 80-120%.

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.

The method minimum RRFs were met for the SVOC compounds. All pesticide breakdown results were acceptable.

The low-level calibration verification standards for Method SW6020A were within the QC limits of 70-130%.

*Initial Calibration Verification (ICV)*

All second source ICV method criteria were met.

*Continuing Calibration Verification*

The CCV standards were analyzed at the correct frequency for each method as applicable. All CCV or continuing calibration method criteria were met with the exceptions noted below.

*Semivolatile Organic Compounds*

Several continuing calibration percent differences or percent drifts exceeded 20% in the continuing calibration associated with the Level 4 review samples. The following non-detected results are qualified as estimated (UJ) because of associated continuing calibration percent differences or percent drift outliers:

Analytical sequence 9J21035

- 2,4-Dichlorophenol, di-n-octylphthalate, and hexachlorocyclopentadiene in sample SED-06-SS-1.0

Analytical sequence 9J21036

- 2,4-Dinitrophenol, 4,6-dinitro-2-methylphenol, aniline, benzoic acid, 2-nitrophenol, and 3,3'-dichlorobenzidine in samples SED-05-SS-1.0 and SED-08-SS-1.0

Analytical sequence 9J22033

- 4-Chloroaniline, bis(2-chloroethyl) ether, aniline, benzoic acid, and 3,3'-dichlorobenzidine in sample SED-07-SS-1.0

*Metals*

A CCV for selenium exceeded the QC limits of 90-110%. No data were qualified as the associated selenium results were reported from reanalysis which met QC limits.

Laboratory Blanks (method blank; instrument blanks (e.g., initial calibration blank, CCB, if specified in method); reagent/preparation blanks)

The method blanks, calibration 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 calibration blanks associated with these sample analyses with the exceptions noted below.

*Semivolatile Organic Compounds*

Numerous compounds were detected in the method blank for QC Batch 9101073. However, the associated rinsate blank sample results were non-detected and therefore data are not qualified.

*Dioxin/Furan*

Numerous Dioxin/Furans were detected in the method blanks for QC Prep Batch 42119 and 42129. Several of these blank results were reported as 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 in samples SED-07-SS-01-DUP, SED-08-SS-01, and SED-10-SS-01
- 1,2,3,4,7,8-HxCDD in samples SED-07-SS-01-DUP, SED-08-SS-01, SED-09-SS-01, and SED-10-SS-01
- 1,2,3,6,7,8-HxCDD in samples SED-05-SS-01 and SED-08-SS-01
- 1,2,3,7,8,9-HxCDD in samples SED-06-SS-01, SED-07-SS-01-DUP, SED-08-SS-01, and SED-10-SS-01
- 1,2,3,7,8-PeCDF in sample SED-07-SS-01-DUP
- 2,3,4,7,8-PeCDF in sample SED-05-SS-01
- 1,2,3,7,8,9-HxCDF in samples SED-05-SS-01, SED-07-SS-01-DUP, and SED-08-SS-01
- 2,3,4,6,7,8-HxCDF and 1,2,3,4,7,8,9-HpCDF in samples SED-05-SS-01, SED-08-SS-01, and SED-10-SS-01
- Total-PeCDD and Total- HxCDD in sample SED-05-SS-01

*PCB Congeners*

Various PCB congeners were detected in the method blanks for QC Prep Batches 42225 and 42253. The following sample results are less than five times the method blank concentration (adjusted for sample size) and are qualified as Non-Detects (U) at the sample concentration due to method blank contamination.

- 52-TeCB, 61/66/70/76-TeCB, 110/115-PeCB, 129/138/163-HxCB, 147/149-HxCB, 153/168-HxCB, 170-HpCB, 174-HpCB, 180/193-HpCB, 194-OcCB, 198-OcCB, 206-NoCB, and 209-DeCB in sample SED-SS-RB (associated with MB - 42225)

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

*Organochlorine pesticides*

Hexachlorobenzene (0.011 ng/g) and Methoxychlor (0.0126 ng/g) were detected in the method blank for QC Prep Batch 6423301. No qualification is needed since all sample results were greater than 5 times the method blank concentration (adjusted for sample size) or were not detected.

Field Blanks

Sample SED-SS-RB was an equipment rinsate blank collected with these samples. Low level positive equipment rinsate blank results did not impact any detected sediment sample results.

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 LCS or LCSD recoveries of several compounds in analytical batch 9101073 were below the control limits. The following non-detected results were qualified as estimated (UJ) due to the low LCS recoveries.

- Acenaphthene, 1-Methylnaphthalene, 2-Methylnaphthalene, Naphthalene, Hexachloroethane, Hexachlorobutadiene, 1,2-Dichlorobenzene, 1,3-Dichlorobenzene, 1,4-Dichlorobenzene, 2-Chloronaphthalene, 1,2,4-Trichlorobenzene, 4-Chlorophenyl phenyl ether in sample SED-SS-RB

The LCS recovery for 4,6-Dinitro-2-methylphenol and the LCS/LCSD RPD for benzoic acid in analytical batch 9101073 and the LCS recoveries for 4-Nitroaniline and 3,3'-Dichlorobenzidine in analytical batch 9101492 were above the control limits. Qualification was not appropriate because these compounds were not detected in the associated 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 or above the control limits. Qualification was not appropriate because this compound was not detected in any of the samples.

#### *Chlorinated Herbicides*

The LCS recoveries for four compounds were greater than the QC limit for QC batch W9J0978. However, qualification was not needed as the affected compounds were not detected in the associated sample.

#### 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 SED-06-SS-1.0 or SED-10-SS-1.0 for a various 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.

#### *Semivolatile Organic Compounds*

The compounds 2,4-dinitrophenol, hexachlorocyclopentadiene, 3-nitroaniline, benzoic acid, 3,3'-dichlorobenzidine were not recovered in the MS performed on sample SED-10-SS-1.0, resulting in 0% recoveries. The following non-detected results were rejected (R) due to the 0% recoveries.

- 2,4-Dinitrophenol, hexachlorocyclopentadiene, 3-nitroaniline, benzoic acid, 3,3'-dichlorobenzidine in sample SED-10-SS-1.0

The MS/MSD results for sample SED-06-SS-1.0 were not applicable as the QC samples were analyzed at 100 times dilution and the results are below the linear calibration or the unspiked sample results were significantly above the spiking levels.

#### *Chlorinated Herbicides*

The MS/MSD RPD for Dinoseb was greater than the QC limit of 35% for sample SED-06-SS-1.0. However, qualification was not needed as the affected compound was not detected in the associated sample.

#### *Total Cyanide*

The MS/MSD recoveries for *Total Cyanide* (50%/43%) for sample SED-06-SS-1.0 were below the QC limits (64-136%). The following detected results were qualified as estimated with low bias (J-) due to the low MS/MSD recoveries.

- Total Cyanide in all sediment samples

*Dioxin/Furan*

The MS/MSD recoveries for 1,2,3,4,6,7,8-HpCDD for sample SED-06-SS-1.0 at –62.4% and –64.8% were below the QC limits (70-130%) and the MS recovery for 2,3,7,8-TCDF from the confirmation analysis at 135% exceeded the QC limits. The following detected results were qualified as estimated (J) due to the MS/MSD accuracy anomalies.

- 2,3,7,8-TCDF (confirmation result) and 1,2,3,4,6,7,8-HpCDD in sample SED-06-SS-1.0

MS/MSD recoveries for OCDD were not applicable due to the high native concentrations of this analyte in the unspiked parent sample. No data additional validation qualifiers are added to the data.

*PCB Congeners*

The MS recovery for 118-PeCB at 171% exceeded the QC limit (50-150%) for sample SED-06-SS-1.0. The following detected result was qualified as estimated (J) due to the MS accuracy anomaly.

- 118-PeCB in sample SED-06-SS-1.0

*Organochlorine pesticides*

The MS/MSD RPDs for Aldrin and Endrin aldehyde exceeded the QC limit for sample SED-06-SS-1.0. However, no qualification is required as these compounds were not detected in the sample.

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 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 SED-05-SS-1.0 or SED-10-SS-1.0 for several analyses. All laboratory duplicate criteria were met with the exception noted below.

#### *Diesel Range Organics*

A laboratory duplicate was performed on sample SED-05-SS-1.0. The RPD between parent and laboratory duplicate for oil exceeded 30%. However, since the concentrations were less than 5x the reporting limit and the difference between the sample and duplicate results were less than the reporting limit, data qualification was not appropriate.

#### *Semivolatile Organic Compounds*

The results for the following compounds in sample SED-10-SS-1.0 are qualified as estimated (J) because the associated laboratory duplicate RPDs exceeded 30% or the difference between the sample and duplicate result was greater than the reporting limit.

- Benzo(g,h,i)perylene, indeno(1,2,3-cd)pyrene, benzo(b)fluoranthene, benzo(k)fluoranthene, chrysene, benzo(a)pyrene, and benz(a)anthracene in sample SED-10-SS-1.0

The laboratory also flagged results for anthracene, dibenzofuran, acenaphthylene, dibenz(a,h)anthracene, and hexachloroethane in sample SED-10-SS-1.0 for not meeting duplicate criteria. However, no qualification was required for these compounds because the results or the difference between the results were less than the reporting limits.

#### *Total Organic Carbon (solid) by EPA 9060A Mod*

The RPD between parent and duplicate results for the laboratory duplicate performed on sample SED-06-SS-1.0 in analytical batch 9101455 at 30% was above the control limits of 20%. The following results have been qualified as estimated (J) for laboratory duplicate precision anomalies.

- Total organic carbon for all sediment samples

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

Sample SED-07-SS-1.0-DUP is a field duplicate of sample SED-07-SS-1.0. All field duplicate precision criteria were met as the RPDs for results greater than the reporting limit were less than 50% or the difference between the sample and field duplicate results was less than the reporting limits with the exceptions noted below:

#### *Dioxin/Furan*

These samples did not demonstrate acceptable precision as the majority of the results exceeded field duplicate criteria. It should be noted that the original sample was analyzed at a 10X dilution but the duplicate was analyzed undiluted. The following results were qualified as estimated (J) or (UJ) due to exceeded field duplicate precision:

- All Dioxin/Furan results for samples SED-07-SS-1.0 and SED-07-SS-1.0-DUP

Note that results were also qualified for EMPCs and blank contamination.

#### *Organochlorine pesticides*

The duplicate RPD for o,p-DDD exceeded the QC limit of 50%. As a result of exceeded precision, the following detected results were qualified as estimated (J):

- o,p-DDD in samples SED-07-SS-1.0 and SED-07-SS-1.0-DUP

These results are also qualified for holding times and have a qualifier of (J-).

### Serial Dilution Samples (Metals)

A serial dilution was not provided for the total and dissolved metals for this 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*

Surrogate recoveries for samples SED-05-SS-01, SED-06-SS-01, and SED-09-SS-01 were considered diluted below the linear calibration range due to the 100X and 40X dilutions. Therefore any recoveries outside QC limits were not used to qualify any data.

### 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 with the exceptions noted below.

#### *Dioxin/Furan*

The recovery for the labeled compound 13C-1,2,3,7,8,9-HxCDF was less than the QC limits (29-147%) for samples SED-06-SS-1.0 (18.9%). As a result of the low recoveries, the following detected results were qualified as estimated (J-).

- 1,2,3,7,8,9-HxCDF and Total HxCDF in sample SED-06-SS-1.0

The result for Total HxCDF has a final qualifier of “JK” as the concentration is also effected by EMPCs.

#### *PCB Congeners*

The recoveries for the labeled compounds 13C-205-OcCB for sample SED-10-SS-1.0 and 13C-206-NoCB for samples SED-7-SS-1.0 and SED-7-SS-1.0-DUP exceeded the QC limits (10-145%). As a result of the elevated recoveries, the following detected results were qualified as estimated with high bias (J+) and non-detected results were qualified as estimated (UJ).

- All OcCB congeners except for 202-OcCB in sample SED-10-SS-1.0
- 206-NoCB and 207-NoCB in samples SED-7-SS-1.0 and SED-7-SS-1.0-DUP

Note that 205-OcCB in sample SED-10-SS-1.0 was qualified as an EMPC and has a final qualifier of “UJK”.

### Internal Standards

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

### Sample Results

All appropriate raw data 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.

According to the case narrative or lab notes, various analyses for these samples were diluted or re-extracted with reduced sample size due to high target concentrations, high non target matrix interference, sample matrix, or due to the appearance of the final extract. 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 noted that the oil and diesel results samples SED-7-SS-1.0, SED-7-SS-1.0-DUP, and SED-9-SS-1.0 are estimated due to overlap from the other result. These results are considered estimated concentrations with a final qualifier of (J).

The laboratory also indicated that the remaining oil results 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.

#### *Semivolatile Organic Compounds*

The laboratory indicated that the benzo(k)fluoranthene results associated with samples SED-05-SS-1.0, SED-06-SS-1.0, and SED-08-SS-1.0 are estimated because the peak separation for structural isomers is insufficient for accurate quantification. The affected results have been qualified as estimated (J).

#### *Dioxin/Furan*

The results for Total-TeCDF in sample SED-09-SS-1.0 was flagged for exceeding the instrument linear calibration range. The result is considered an estimated quantity and is qualified as estimated (J). This result is also impacted by EMPCs, see below.

A few dioxin and furan results were reported as EMPCs and were qualified with the laboratory "K" flag denoting an EMPC value. The results were below the PQL and were reported by the laboratory as estimated (JK) values. The following EMPCs not previously qualified as non-detected due to method blank contamination were 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.

- 2,3,7,8-TCDD in sample SED-10-SS-1.0
- 2,3,7,8-TCDF in samples SED-07-SS-1.0 and SED-07-SS-1.0-DUP

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 TeCDF and Total HxCDF in sample SED-05-SS-1.0
- Total TeCDD, Total PeCDF, and Total HxCDF in sample SED-06-SS-1.0
- Total TeCDD, Total PeCDD, Total HxCDD, Total TeCDF, and Total HxCDF in sample SED-07-SS-1.0
- Total TeCDD, Total PeCDD, Total HxCDD, Total TeCDF, Total PeCDF, and Total HxCDF in sample SED-07-SS-1.0-DUP
- Total TeCDD, Total PeCDD, Total TeCDF, Total PeCDF, and Total HxCDF in sample SED-08-SS-1.0
- Total TeCDD, Total HxCDD, Total TeCDF, and Total HpCDF in sample SED-09-SS-1.0
- Total TeCDD, Total PeCDD, Total TeCDF, Total PeCDF and Total HxCDF in sample SED-10-SS-1.0

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.

Various dioxin and furan and their associated labeled compounds were flagged as “Q” by the laboratory indicating that quantitative interference resulted in an estimated value. The following results that were not already qualified due to low labeled compound recoveries or as an EMPC are qualified as estimated (J or UJ).

- Total PeCDF in samples SED-05-SS-1.0, SED-07-SS-1.0
- Total PeCDD in sample SED-06-SS-1.0
- 1,2,3,7,8,9-HxCDF, Total PeCDD, Total PeCDF, and Total HxCDF in sample SED-09-SS-1.0
- 1,2,3,7,8,9-HxCDF in sample SED-10-SS-1.0

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. All but one of the results were also below the PQL and 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 (UK or 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, 8-DiCB, 16-TrCB, 55-TeCB, 120-PeCB, 150-HxCB, and 152-HxCB in sample SED-05-SS-1.0
- 1-MoCB, 3-MoCB, 6-DiCB, 11-DiCB, 15-DiCB, 54-TeCB, 63-TeCB, 67-TeCB, 137-HxCB, 154-HxCB, and 208-NoCB in sample SED-06-SS-1.0
- 10-DiCB, 122-PeCB, 123-PeCB, and 181-HpCB in sample SED-07-SS-1.0
- 11-DiCB, 36-TrCB, and 41-TeCB in sample SED-07-SS-1.0-DUP
- 3-MoCB, 36-TrCB, 114-PeCB, 172-HpCB, and 186-HpCB in sample SED-08-SS-1.0
- 41-TeCB, 85/116/117-PeCB, 94-PeCB, and 150-HxCB in sample SED-09-SS-1.0
- 7-DiCB, 34-TrCB, 79-TeCB, 103-PeCB, 122-PeCB, and 205-OcCB in sample SED-10-SS-1.0
- 11-DiCB, 20/28-TrCB, 31-TrCB, 105-PeCB, 118-PeCB, 135/151-HxCB, and 187-HpCB in sample SED-SS-RB

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

Various PCBs and associated labeled compounds were flagged as “Q” by the laboratory indicating that quantitative interference resulted in an estimated value. The following results that were not already qualified due to labeled compound recoveries or as an EMPC are qualified as estimated (J).

- 89-PeCB, 135/151-HxCB, 144-HxCB, 156/157-HxCB, and 172-HpCB in sample SED-05-SS-1.0

*Organochlorine pesticides*

The results for p,p-DDT in sample SED-09-SS-1.0 from the 20X dilution exceeds the instrument linear calibration range. The result is considered an estimated quantity and is qualified as estimated (J).

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, g-Chlordane, Aldrin, and o,p-DDE in sample SED-05-SS-1.0
- a-Chlordane, g-Chlordane, Aldrin, o,p-DDE, Dieldrin, and trans-Nonachlor in sample SED-06-SS-1.0
- Aldrin, o,p-DDE, Dieldrin, and Endosulfan sulfate in sample SED-07-SS-1.0
- Aldrin, o,p-DDE, Endosulfan sulfate, cis-Nonachlor, and trans-Nonachlor in sample SED-07-SS-1.0-DUP
- Aldrin, Endosulfan sulfate, and Endrin in sample SED-08-SS-1.0
- Aldrin, beta-BHC, delta-BHC, Dieldrin, Endosulfan sulfate, Endosulfan II, and trans-Nonachlor in sample SED-09-SS-1.0
- Endosulfan II in sample SED-SS-RB

#### *Grain Size by ASTM D 422M/PSET Parameters*

The laboratory note/narrative for most samples indicated that the No. 4 sieve (gravel) and No. 10 sieve (coarse sand) grain size fractions contained organic materials. See the grain size case narratives included at the end of the Apex Laboratories report.

#### Overall Assessment

The analytical data are acceptable and usable as reported with the minor qualifications noted above, with the exception of the rejected results noted below. All sediment Organochlorine Pesticides were qualified due to exceeded holding times. Additional results were qualified due to CCV, LCS, labeled compound recoveries, field duplicate precision, MS/MSD, laboratory duplicate accuracy and precision issues and results above the calibration range. Results for Dioxin/Furans, Organochlorine Pesticides and PCB Congeners were also qualified as not detected due to blank contamination or as EMPCs. The semivolatile results for 2,4-Dinitrophenol, hexachlorocyclopentadiene, 3-nitroaniline, benzoic acid and 3,3'-dichlorobenzidine in sample SED-10-SS-1.0 were rejected due to 0% MS/MSD recovery.

**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.

<b>Data Qualifier</b>	<b>Description</b>
<b>Standard Data Qualifiers</b>	
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)