APPENDIX H

Data Validation Reports
(Year 2 and Year 3)

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CALCULATION REVIEW SUMMARY

Lower Duwamish Waterway– Enhanced Natural Recovery/Activated Carbon Pilot Year Two Samples, June 2019

Prepared for:
Wood Environment and Infrastructure Solutions
3500 188th Street SW, Ste 601
Lynnwood, WA 98037-4763

September 6, 2019

1.0 Introduction

The objective of this review was to verify that the freely dissolved polychlorinated biphenyl (PCB) concentrations were accurately calculated. This review was performed by Cari Sayler.

Laboratory reported total mass per sample concentrations were converted to freely dissolved PCBs in sediment porewater by GeoSyntec Consultants of Huntington Beach, California. Data were provided in an Excel® spreadsheet named "Cfree Data_Tables only (090319).xlsx and dated 9/3/2019.

Data included the following samples and trip blanks:

Sample ID	Sample Date/Time	Lab ID
LDW-Y2-SC-S010-TB	06/24/2019 11:11	12510-001-0001-SA
LDW-Y2-SU-S010-TB	06/24/2019 11:18	12510-002-0001-SA
LDW-Y2-IN-S010-TB	06/24/2019 11:28	12510-003-0001-SA
LDW-Y2-SU-ENR+AC-CA-S010	06/24/2019 08:58	12510-004-0001-SA
LDW-Y2-SU-ENR+AC-CB-S010	06/24/2019 09:13	12510-005-0001-SA
LDW-Y2-SU-ENR+AC-CC-S010	06/24/2019 09:22	12510-006-0001-SA
LDW-Y2-SU-ENR-CA-S010	06/24/2019 10:16	12510-009-0001-SA
LDW-Y2-SU-ENR-CB-S010	06/24/2019 10:26	12510-010-0001-SA
LDW-Y2-SU-ENR-CC-S010	06/24/2019 10:35	12510-011-0001-SA
LDW-Y2-SU-S010-LCB	06/24/2019 11:00	12510-014-0001-SA
LDW-Y2-IN-ENR+AC-CA-S010	06/25/2019 09:44	12510-015-0001-SA
LDW-Y2-IN-ENR+AC-CB-S010	06/25/2019 09:58	12511-001-0001-SA
LDW-Y2-IN-ENR+AC-CC-S010	06/25/2019 10:18	12511-002-0001-SA
LDW-Y2-IN-ENR-CA-S010	06/25/2019 11:08	12511-005-0001-SA
LDW-Y2-IN-ENR-CB-S010	06/25/2019 11:25	12511-006-0001-SA
LDW-Y2-IN-ENR-CE-S010	06/25/2019 11:57	12511-009-0001-SA
LDW-Y2-IN-ENR+AC-CA-SSWI	06/24/2019 14:13	12511-010-0001-SA
LDW-Y2-IN-ENR+AC-CB-SSWI	06/24/2019 14:22	12511-011-0001-SA
LDW-Y2-IN-ENR+AC-CC-SSWI	06/24/2019 14:29	12511-012-0001-SA
LDW-Y2-IN-ENR-CA-SSWI	06/24/2019 13:31	12511-015-0001-SA
LDW-Y2-IN-ENR-CB-SSWI	06/24/2019 13:41	12512-001-0001-SA
LDW-Y2-IN-ENR-CE-SSWI	06/24/2019 14:03	12512-004-0001-SA
LDW-Y2-SC-ENR+AC-CB-S010	06/26/2019 08:44	12512-006-0001-SA
LDW-Y2-SC-ENR+AC-CC-S010	06/26/2019 08:53	12512-007-0001-SA
LDW-Y2-SC-ENR-CC-S010	06/26/2019 10:15	12512-013-0001-SA

Sample ID	Sample Date/Time	Lab ID
LDW-Y2-SC-ENR-CD-S010	06/26/2019 10:26	12512-014-0001-SA
LDW-Y2-SC-ENR-CE-S010	06/26/2019 10:40	12512-015-0001-SA
LDW-Y2-SC-ENR+AC-CA/AC-CD-S010	06/26/2019	12512-016-0001-SA
LDW-Y2-SC-ENR+AC-CB-SSWI	06/26/2019 10:56	12513-002-0001-SA
LDW-Y2-SC-ENR+AC-CC-SSWI	06/26/2019 11:02	12513-003-0001-SA
LDW-Y2-SC-ENR-CC-SSWI	06/26/2019 11:36	12513-008-0001-SA
LDW-Y2-SC-ENR-CD-SSWI	06/26/2019 11:47	12513-009-0001-SA
LDW-Y2-SC-ENR-CE-SSWI	06/26/2019 11:57	12513-010-0001-SA
LDW-Y2-SC-ENR+AC-CA/AC-CD-SSWI	06/26/2019	12513-011-0001-SA

2.0 Data Sources

Data were loaded from various sources in order to independently calculate each PCB Cfree concentration and detection limit. Spot checking and limited recalculation was done to verify the data sources as described below:

Laboratory reported concentrations: Laboratory reported mass concentrations were obtained from the laboratory electronic data deliverable (EDD). Sporadic comparisons were made to the data in spreadsheet tab “Table A3. Mass of PCBs”, and no discrepancies were noted for the PCB mass concentrations. However, the PCB mass DLs require further clarification:

Data reported in the “PCB Mass DL” columns was a mix of values. For the non-detect compounds, this column was populated with the estimated detection limit (EDL) which was found in the laboratory report under column heading of “DL” and in the EDD in the “Result” field. For detected results, this column was populated with the minimum level of quantitation (ML) which was not present on the laboratory report, but was present in the laboratory EDD in the “ML” field. It should be noted that the ML is a quantitation limit rather than a detection limit.

No discrepancies were noted between the values listed in the column and the laboratory EDLs and MLs.

SPME sampling details: SPME fiber details were obtained from the data in spreadsheet tab “Table A1. Fiber details”. Data for mass of fiber, length of fiber, % recovery, volume of PDMS, mass of PDMS were recalculated from the remaining data. No discrepancies were noted.

Reference values: Log K_{PDMS} values for each PCB were retained from the calibration study/baseline sample calculation review. These values were spot checked against reference the data in spreadsheet tab “Table A5 KPDMS”. No discrepancies were noted.

Reported PCB Cfree concentrations: Reported PCB Cfree concentrations were obtained from the data in spreadsheet tab “T1. Cfree(Final)”. This data was used for the basis of comparison to recalculated values. However, the PCB Cfree DL values contained the same mix of values that the spreadsheet tab “Table A3. Mass of PCBs”, did. For the non-detect compounds, PCB Cfree EDL were listed. For detected compounds, PCB Cfree MLs were listed.

3.0 Calculations

Formulas retained from the calibration study/baseline sample calculation review were used to recalculate PCB free concentrations.

Data calculations were performed in a Microsoft Access database. The Access Visual Basic for Applications (VBA) subroutine developed for the calibration study/baseline sample calculation review were again used to calculate the PCB free concentrations, EDLs, MLs and MDLs, and to compare the recalculated results to the reported results.

4.0 Conclusions

Concentrations: Cfree concentrations of each detected PCB was recalculated and compared to the reported values. Concentrations agreed within a reasonable variation for rounding differences. Calculated relative percent differences (RPDs) were between 0 and 5.

Reporting limits: Cfree EDLs or MLs of each PCB was recalculated and compared to the reported values. Values agreed within a reasonable variation for rounding differences. Calculated RPDs were between 0 and 5.

PRC Model: PRC Calculations were shown in tab “Table A4. ke”. Values for Ke, slope, y-intercept and R2 were recalculated with good agreement.

The PRC coefficient of determination (R2) ranged from 0.49 to 0.98 indicating poor linearity in some samples. The upper and lower confidence limits were calculated to demonstrate the potential variability in the results, and this analysis was included on the “TA6_Uncertainty Cfree” tab. It should be noted that these upper and lower confidence limits reflects variation due to PRC linearity, but not necessarily variation due to other factors such as analytical reproducibility or matrix inhomogeneity, or the number of PRC compounds included in the PRC regression.

Sample LDW-Y2-SU-S010-LCB has a p-value of 0.455, greatly exceeding the 0.05 criteria for statistical significance, with only three of the ten PRC compounds included in the regression. The variability in results for this sample likely exceeds the upper and lower confidence limits shown in tab “TA6_Uncertainty Cfree”.

Qualifiers: Qualifiers listed in the tab T1. Cfree(FINAL)’ were reviewed and were determined to be in agreement with the supplied definitions.

One clarification should be noted: A UB qualifier indicates that the background concentrations exceeded the detected concentration ‘These results should be considered not detected at the lowest available detection limit, the MDL.

Total detected PCBs: Total detected PCBs were recalculated based on the reported individual PCB Cfree concentrations, excluding spiked PRC compounds. Total detected PCBs were recalculated with good agreement.

Confidence levels: The uncertainty upper and lower confidence levels summarized in the spreadsheet tab “TA6_Uncertainty Cfree” are beyond the scope of this review and have not been recalculated.

5.0 Abbreviations and Definitions

Abbreviation	Definition
EDL	Estimated detection limit
MDL	Method detection limit
ML	Minimum level of quantitation
PDMS	Polydimethylsiloxane

Abbreviation	Definition
PRC	Performance Reference Compound
RPD	Relative percent difference
SPME	Solid phase microextraction

6.0 References

Certificate of Analysis Concentrations of Freely-dissolved Polychlorinated Biphenyls (PDBs) Measured via SP3ME Passive Samplers. Prepared for Lower Duwamish Waterway Group, Prepared by Geosyntec Consultants, March 22, 2017. This report contains data for samples collected July to September 2016.

Certificate of Analysis Concentrations of Freely-dissolved Polychlorinated Biphenyls (PDBs) Measured via SP3ME Passive Samplers. Prepared for Lower Duwamish Waterway Group, Prepared by Geosyntec Consultants, March 22, 2017. This report contains data for samples collected November 2016 to January 2017.

Polymer-water partition coefficients of hydrophobic compounds for passive sampling: Application of cosolvent models for validation. Environ. Sci. Technol. 43:7047-7054. Smedes, et al. 2009.

Quality Assurance Project Plan Enhanced Natural Recovery/Activated Carbon Pilot Study, Lower Duwamish Waterway. Prepared by AMEC Foster Wheeler Environment & Infrastructure Inc., et al. Prepared for: USEPA Region 10 and WA-DOE Northwest Regional Office, February 22, 2016.

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DATA VALIDATION REPORT

Lower Duwamish Waterway– Enhanced Natural Recovery/Activated Carbon Pilot Year Two Samples, April 2019 – July 2019

Prepared for:
Wood Environment and Infrastructure Solutions
3500 188th Street SW, Ste 601
Lynnwood, WA 98037-4763

September 6, 2019

1.0 Introduction

Data validation was performed on the following samples:

Sample ID	Sample Date/Time	Lab ID	Analyses	Matrix
LDW-Y2-SC-S010-TB	06/24/2019 11:11	12510-001-0001-SA	PCB	Solvent
LDW-Y2-SU-S010-TB	06/24/2019 11:18	12510-002-0001-SA	PCB	Solvent
LDW-Y2-IN-S010-TB	06/24/2019 11:28	12510-003-0001-SA	PCB	Solvent
LDW-Y2-SU-ENR+AC-CA-S010	06/24/2019 08:58	12510-004-0001-SA	PCB	Solvent
LDW-Y2-SU-ENR+AC-CB-S010	06/24/2019 09:13	12510-005-0001-SA	PCB	Solvent
LDW-Y2-SU-ENR+AC-CC-S010	06/24/2019 09:22	12510-006-0001-SA	PCB	Solvent
LDW-Y2-SU-ENR-CA-S010	06/24/2019 10:16	12510-009-0001-SA	PCB	Solvent
LDW-Y2-SU-ENR-CB-S010	06/24/2019 10:26	12510-010-0001-SA	PCB	Solvent
LDW-Y2-SU-ENR-CC-S010	06/24/2019 10:35	12510-011-0001-SA	PCB	Solvent
LDW-Y2-SU-S010-LCB	06/24/2019 11:00	12510-014-0001-SA	PCB	Solvent
LDW-Y2-IN-ENR+AC-CA-S010	06/25/2019 09:44	12510-015-0001-SA	PCB	Solvent
LDW-Y2-IN-ENR+AC-CB-S010	06/25/2019 09:58	12511-001-0001-SA	PCB	Solvent
LDW-Y2-IN-ENR+AC-CC-S010	06/25/2019 10:18	12511-002-0001-SA	PCB	Solvent
LDW-Y2-IN-ENR-CA-S010	06/25/2019 11:08	12511-005-0001-SA	PCB	Solvent
LDW-Y2-IN-ENR-CB-S010	06/25/2019 11:25	12511-006-0001-SA	PCB	Solvent
LDW-Y2-IN-ENR-CE-S010	06/25/2019 11:57	12511-009-0001-SA	PCB	Solvent
LDW-Y2-IN-ENR+AC-CA-SSWI	06/24/2019 14:13	12511-010-0001-SA	PCB	Solvent
LDW-Y2-IN-ENR+AC-CB-SSWI	06/24/2019 14:22	12511-011-0001-SA	PCB	Solvent
LDW-Y2-IN-ENR+AC-CC-SSWI	06/24/2019 14:29	12511-012-0001-SA	PCB	Solvent
LDW-Y2-IN-ENR-CA-SSWI	06/24/2019 13:31	12511-015-0001-SA	PCB	Solvent
LDW-Y2-IN-ENR-CB-SSWI	06/24/2019 13:41	12512-001-0001-SA	PCB	Solvent
LDW-Y2-IN-ENR-CE-SSWI	06/24/2019 14:03	12512-004-0001-SA	PCB	Solvent
LDW-Y2-SC-ENR+AC-CB-S010	06/26/2019 08:44	12512-006-0001-SA	PCB	Solvent
LDW-Y2-SC-ENR+AC-CC-S010	06/26/2019 08:53	12512-007-0001-SA	PCB	Solvent
LDW-Y2-SC-ENR-CC-S010	06/26/2019 10:15	12512-013-0001-SA	PCB	Solvent
LDW-Y2-SC-ENR-CD-S010	06/26/2019 10:26	12512-014-0001-SA	PCB	Solvent
LDW-Y2-SC-ENR-CE-S010	06/26/2019 10:40	12512-015-0001-SA	PCB	Solvent
LDW-Y2-SC-ENR+AC-CA/ AC-CD-S010	06/26/2019	12512-016-0001-SA	PCB	Solvent
LDW-Y2-SC-ENR+AC-CB-SSWI	06/26/2019 10:56	12513-002-0001-SA	PCB	Solvent
LDW-Y2-SC-ENR+AC-CC-SSWI	06/26/2019 11:02	12513-003-0001-SA	PCB	Solvent
LDW-Y2-SC-ENR-CC-SSWI	06/26/2019 11:36	12513-008-0001-SA	PCB	Solvent

Sample ID	Sample Date/Time	Lab ID	Analyses	Matrix
LDW-Y2-SC-ENR-CD-SSWI	06/26/2019 11:47	12513-009-0001-SA	PCB	Solvent
LDW-Y2-SC-ENR-CE-SSWI	06/26/2019 11:57	12513-010-0001-SA	PCB	Solvent
LDW-Y2-SC-ENR+AC-CA/ AC-CD-SSWI	06/26/2019	12513-011-0001-SA	PCB	Solvent
LDW-Y2-SU-ENR-CA-CORE	04/25/2019 12:03	12532-001-0001-SA K1903805-001	PCB, TOC, GS	Sediment
LDW-Y2-SU-ENR-CB-CORE	04/25/2019 12:15	12532-002-0001-SA K1903805-002	PCB, TOC, GS	Sediment
LDW-Y2-SU-ENR-CC-CORE	04/25/2019 12:27	12532-003-0001-SA K1903805-003	PCB, TOC, GS	Sediment
LDW-Y2-SU-ENR+AC-CA-CORE	04/25/2019 10:55	12532-004-0001-SA K1903805-004	PCB, TOC, GS	Sediment
LDW-Y2-SU-ENR+AC-CB-CORE	04/25/2019 11:07	12532-005-0001-SA K1903805-005	PCB, TOC, GS	Sediment
LDW-Y2-SU-ENR+AC-CC-CORE	04/25/2019 11:17	12532-006-0001-SA K1903805-006	PCB, TOC, GS	Sediment
LDW-Y2-IN-ENR-CA-CORE	06/26/2019 11:15	12532-007-0001-SA K1906344-001	PCB, TOC, GS	Sediment
LDW-Y2-IN-ENR-CB-CORE	06/26/2019 11:25	12532-008-0001-SA K1906344-002	PCB, TOC, GS	Sediment
LDW-Y2-IN-ENR-CE-CORE	06/26/2019 11:55	12532-009-0001-SA K1906344-003	PCB, TOC, GS	Sediment
LDW-Y2-IN-ENR+AC-CA-CORE	06/26/2019 12:00	12532-010-0001-SA K1906344-004	PCB, TOC, GS	Sediment
LDW-Y2-IN-ENR+AC-CB-CORE	07/02/2019 10:50	12532-011-0001-SA K1906344-005	PCB, TOC, GS	Sediment
LDW-Y2-IN-ENR+AC-CC-CORE	06/26/2019 12:10	12532-012-0001-SA K1906344-006	PCB, TOC, GS	Sediment
LDW-Y2-SC-ENR-CC-CORE	07/02/2019 11:30	12532-013-0001-SA K1906344-007	PCB, TOC, GS	Sediment
LDW-Y2-SC-ENR-CD-CORE	07/02/2019 11:40	12532-014-0001-SA K1906344-008	PCB, TOC, GS	Sediment
LDW-Y2-SC-ENR-CE-CORE	07/02/2019 11:45	12532-015-0001-SA K1906344-009	PCB, TOC, GS	Sediment
LDW-Y2-SC-ENR+AC-CAD-CORE	07/02/2019 11:50	12532-016-0001-SA K1906344-010	PCB, TOC, GS	Sediment
LDW-Y2-SC-ENR+AC-CB-CORE	07/02/2019 11:55	12532-017-0001-SA K1906344-011	PCB, TOC, GS	Sediment
LDW-Y2-SC-ENR+AC-CC-CORE	07/02/2019 12:00	12532-018-0001-SA K1906344-012	PCB, TOC, GS	Sediment
LDW-Y2-SC-ENR-SS	06/22/2019 17:43	12532-019-0001-SA K1906344-013	PCB, TOC, GS	Sediment
LDW-Y2-SC-ENR+AC-SS	06/26/2019 12:19	12532-020-0001-SA K1906344-014	PCB, TOC, GS	Sediment

PCB analyses were performed by Frontier Analytical Laboratory (Frontier), in El Dorado Hills, California. TOC analyses were performed by ALS Environmental (ALS) in Kelso, Washington. Grain size analyses were performed by Materials Testing & Consulting, Inc (MTC).

Validation: A full validation was performed on the PCB data. A summary validation was performed on the TOC and grain size data. Validation was performed by Cari Sayler. Data qualifiers are summarized in section 5.0 of this report.

Analytical methods: Table 3.3 of the QAPP specifies the following analytical methods:

Analysis	Method
Polychlorinated Biphenyl Congeners (PCB)	EPA 1668C
Total Organic Carbon (TOC)	EPA 9060

Analysis	Method
Grain size (GS)	ASTM D422

These methods were used with the following exception: MTC utilized the Puget Sound Estuary Protocol (PSEP) method. This is considered an acceptable substitution.

Requested analyses: Sample chain-of-custodies and sample log-in documentation were reviewed. All requested analyses were performed.

Sample number transcription: Sample IDs in the electronic data deliverable (EDD) were compared to the chain-of-custody for each sample. Sample IDs matched the chain of custody.

2.0 Polychlorinated Biphenyl Congener Analyses

Quality control analysis frequencies: The method specifies that method blank and ongoing precision and recovery (OPR) samples must be analyzed with each batch. In addition, injection standards, isotope dilution standards and cleanup standards must be measured in each field and quality control sample. These frequencies were met.

Analysis holding times: Method 1668C specifies a one year holding time between extraction and analysis, and a one year holding time from sampling to extraction for both water and Sediment samples. These holding times were met.

System performance checks: System performance criteria include: 1) The tune must demonstrate a resolving power $\geq$10,000 at m/z 330.9792 and $\geq$8,000 throughout the range. 2) The monitored m/z must be <5 ppm from theoretical for the following theoretical m/z's: 218.9856, 242.9856, 280.9825, 330.9792, 354.9797, 354.9792, and 454.9728. 3) The retention time of congener 209 must exceed 55 minutes on the SPB-Octyl column. 4) The isomer specificity check must demonstrate resolution of congeners with valleys of $\leq$40% for congeners PCB-034 from PCB-023 and PCB-187 from PCB-182 on the SPB-Octyl Column. 5) The isomer specificity check must demonstrate elution of PCB 156 and PCB 157 within 2 seconds for the SPB-Octyl Column.

The laboratory utilized a DB1 column and provided the following column-specific performance criteria: Resolution of congeners with valleys of $\leq$40% for congeners PCB-156 and 157 and PCB 209 RT$\geq$50 minutes. Additionally, congeners 106 and 118 were evaluated for coelution within 2 seconds. These criteria were met.

Instrument calibration: Initial calibration criteria include 1) maximum percent relative standard deviations (%RSD) of $\leq$20% for target compounds and $\leq$35% for labeled compounds, 2) Ion abundance ratios must be within $\pm$15% of theoretical, and 3) signal to noise ratios must be at or above 10. Continuing calibration criteria include 1) percent recoveries within 75-125% for target compounds, 65-135% for 13C-PCB-028 and 75-125% for 13C-PCB-111 and 13C-PCB-178, and 50-145% for the remaining labeled compounds. 2) Ion abundance ratios must be within $\pm$ 15% of theoretical, and 3) signal to noise ratios must be at or above 10. 4) Absolute retention times for injection internal standards must be within $\pm$ 15 seconds of the initial calibration and 5) Relative retention times (RRT) must meet method or column-specific criteria.

Signal to noise ratios and RRTs were not summarized in the raw data. SICP chromatograms were reviewed for expected retention time and noise levels. No discrepancies were noted. All remaining calibration criteria were met.

Laboratory blank results: Laboratory performance criteria in method 1668C states that the method blank must not contain any target compound at a concentration greater than either the minimum level or one-third the regulatory compliance level, whichever is greater. Additionally, the method blank must not contain any potentially interfering compound at a concentration greater than either the minimum level or one-third the regulatory compliance level, whichever is greater. This criterion was met.

Extracted internal standard (surrogate) recoveries: Method criteria are 5-145% for labeled congeners between C13-PCB-001 and C13-PCB-054 and 10-145% for labeled congeners between C13-PCB-077 and C13-PCB-209. Recoveries were within these limits with the following exceptions:

Sample ID	Compound	% Recovery	Lab Control Limit
LDW-Y2-SU-S010-LCB	13C-PCB-015	159	5 - 145
LDW-Y2-IN-ENR+AC-CA-S010	13C-PCB-001	0.3	5 - 145
LDW-Y2-IN-ENR+AC-CA-S010	13C-PCB-003	4.2	5 - 145
LDW-Y2-IN-ENR+AC-CA-S010	13C-PCB-015	474	5 - 145

Additionally, as noted in the case narrative, these two samples were briefly evaporated to dryness and reconstituted in hexane. Both Injection internal standard responses and extracted internal standard responses within these two samples were erratic throughout chlorination levels one through four, with the lowest responses in the first and second chlorination level standards.

In these two samples, non-detect results are rejected as unusable for chlorination level one and two congeners (PCB-1 through PCB-15) and detected results are qualified as estimated. Both positive and non-detect results are qualified as estimated for chlorination level three and four congeners (PCB-16 through PCB-81) and for the total PCB concentration.

Cleanup standard recoveries: Method criteria are 5-145% for C13-PCB-028 and 10-145% for C13-PCB-111 and C13-PCB-178. Cleanup standard recoveries were within laboratory control limits.

OPR recoveries: Method criteria for OPR recoveries are 60-135% for 27 representative target compounds. OPR recoveries were within these limits.

Compound Identification: Method criteria for compound identification include: 1) The signals of the characteristic ions must maximize within the same 2 scans. 2) The signal to noise ratio must be greater than 2.5. 3) Ion abundance ratios must be within ± 15% of theoretical, or within ±15% of the calibration verification standard. 4) Relative retention times must meet method or column-specific criteria.

Criteria were reviewed for each Toxic WHO Congener. Neither the signal to noise ratio nor the individual signal in height and noise levels were included in the raw data for detected compounds. SICP chromatograms in these reports were reviewed and no evidence of noise interference was found.

No discrepancies were noted with the remaining identification criteria.

Compound Quantitation: Sample concentrations were recalculated to verify sample quantitations. No quantitation discrepancies were noted.

Second column confirmation: Second column confirmation was not required to separate congeners 156 and 157 due to the use of the DB1 Column.

Second column confirmation was not performed to separate congeners 106 and 118. Since congener 106 is not a component of any of the commercial Aroclor mixtures, no further action was deemed necessary.

Estimated detection limits: Peak heights for one of the two isotope dilution standards were not present in the original data package. Resubmissions were requested, and received. Estimated detection limits (EDLs) were recalculated for PCB-169 in each sample and method blanks. No discrepancies were noted.

All sediment EDLs met QAPP target reporting limits of 4 pg/g. With 4 exceptions, solvent EDLs ranged from 0.59 to 17.6 pg/sample. The EDLs for PCBs 1 through 4 in sample LDW-Y2-IN-ENR+AC-CA-S010 were very high due to the low recovery of the labeled compound used for quantitation. The EDLs for PCBs 1 through 3 in this sample were rejected as unusable, and the result for PCB 4 was estimated at 623 pg/sample.

Toxicity equivalent quantity (TEQ): TEQ calculations were not required for this project.

Laboratory narrative: No additional qualifiers are assigned based on the laboratory narrative.

Overall assessment: With minor exceptions, resubmitted documentation was found to be clear and complete. No discrepancies were noted in analyte identification or result quantitation. Calibration data and system performance checks demonstrate acceptable instrument performance. With minor exceptions, quality control results indicate acceptable accuracy.

Polychlorinated biphenyl data are acceptable for use as qualified.

3.0 Total Organic Carbon (TOC) Analyses

Quality control analysis frequencies: Each sample was analyzed in duplicate. A method blank, laboratory control sample (LCS), matrix spike (MS), and matrix spike duplicate (MSD) was analyzed in each batch, meeting frequency requirements.

Holding times: TOC must be analyzed within 28 days. Samples should be shipped and maintained at temperatures between 0 and 6° Celsius. These criteria were met with one exception:

Sample ID	Sample Date	Analysis Date	Elapsed days
LDW-Y2-SC-ENR-SS	6/22/2019	7/24/2019	32

The total organic carbon result in this sample should be considered estimated.

Laboratory blank results: Criteria for method blanks are that analyte concentrations must be below the PQL, or below 10% of the lowest associated sample concentration. This criterion was met for all method blanks.

LCS results: The LCS recovery control limit was 72-122%. This criterion was met.

MS recoveries: The MS and MSD recovery control limit was 70-122%. This criterion was met.

Sample replicate variability: The RPD between the first and second replicate analysis of each sample was below 20%.

Matrix spike duplicate variability: The MS/MSD control limit for RPDs was <20%. This criterion was met.

Total organic carbon results are acceptable for use as qualified.

4.0 Grain Size Analyses

Quality control analysis frequencies: Each batch included a laboratory triplicate, meeting frequency requirements.

Holding times: Sediment samples must be analyzed within 6 months of collection. Samples were analyzed within the holding time.

Laboratory triplicate RSDs: Triplicate RSDs were below 25%.

Grain size data are acceptable for use as reported.

5.0 Qualifier Summary Table

Client ID	Analyte(s)	Qualifier	Reason
LDW-Y2-IN-ENR+AC-CA-S010	PCB-1 through PCB-81 detections, Total PCBs	J	Sample evaporated to dryness
LDW-Y2-IN-ENR+AC-CA-S010	PCB-1 through PCB-15 non-detects	R	Sample evaporated to dryness
LDW-Y2-IN-ENR+AC-CA-S010	PCB-16 through PCB-81 non-detects	UJ	Sample evaporated to dryness
LDW-Y2-SU-S010-LCB	PCB-1 through PCB-81 detections, Total PCBs	J	Sample evaporated to dryness
LDW-Y2-SU-S010-LCB	PCB-1 through PCB-15 non-detects	R	Sample evaporated to dryness
LDW-Y2-SU-S010-LCB	PCB-16 through PCB-81 non-detects	UJ	Sample evaporated to dryness
LDW-Y2-SC-ENR-SS	Total Organic Carbon	J	Hold time exceeded

6.0 Abbreviations and Definitions

DV Qualifier	Definition
U	The material was analyzed for, but was not detected above the level of the associated value.
UY	The reporting limit was elevated due to chromatographic overlap with related compounds. The material was analyzed for, but was not detected above the level of the associated value.
J	The analyte was positively identified. The associated numerical value is the approximate concentration of the analyte in the sample.
N	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification.
UJ	The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.

DV Qualifier	Definition
R	The sample result is rejected. The presence or absence of the analyte cannot be verified and data are not usable.
R1	This sample result has been rejected in favor of a more accurate, precise or conservative result. The other result should be used.
R2	This sample result has been rejected in favor of a more accurate, precise or conservative result from another analytical method. The other result should be used.

Abbreviation	Definition
DV	Data validation
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
EDL	Estimated detection limit
EMPC	Estimated maximum possible concentration
IDS	Isotope dilution standard
MS	Matrix spike
MSD	Matrix spike duplicate
NA	Not Applicable
OPR	Ongoing Precision and Recovery
RL	Reporting limit
RPD	Relative percent difference
RRM	Regional reference material
RSD	Relative standard deviations
SRM	Standard reference material

7.0 References

National Functional Guidelines For Inorganic Superfund Data Review, Office of Superfund Remediation and Technology Innovation, U.S. Environmental Protection Agency, August 2014, USEPA-540-R-13-001.

National Functional Guidelines for High Resolution Superfund Methods Data Review, Office of Superfund Remediation and Technology Innovation, U.S. Environmental Protection Agency, April 2016, EPA-542-B-16-001.

Method 1668C: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS, US Environmental Protection Agency, Office of Water Engineering and Analysis Division, April 2010.

Quality Assurance Project Plan Enhanced Natural Recovery/Activated Carbon Pilot Study, Lower Duwamish Waterway. Prepared by AMEC Foster Wheeler Environment & Infrastructure Inc., et al. Prepared for USEPA Region 10 and WA-DOE Northwest Regional Office, February 22, 2016.

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CALCULATION REVIEW SUMMARY

Lower Duwamish Waterway– Enhanced Natural Recovery/Activated Carbon Pilot Year Three Samples, September 2020 Data

Prepared for:
Wood Environment and Infrastructure Solutions
3500 188th Street SW, Ste 601
Lynnwood, WA 98037-4763

December 16, 2020

1.0 Introduction

The objective of this review was to verify that the freely dissolved polychlorinated biphenyl (PCB) concentrations were accurately calculated. This review was performed by Cari Sayler.

Laboratory reported total mass per sample concentrations were converted to freely dissolved PCBs in sediment porewater by GeoSyntec Consultants of Huntington Beach, California. Data were provided in an Excel® spreadsheet named "Y3 LDW SPME Cfree Summary Tables (draft 112320).xlsx" and dated 11/23/2020. A resubmission was requested and provided in an Excel® spreadsheet named "Y3 LDW SPME Cfree Summary Tables (draft 121420) v2.xlsx" and dated 12/14/2020. This resubmission can be considered a complete replacement for the original data, but included changes to only two samples: LDW-Y3-SC-ENR+AC-CB-S010-LONG and LDW-Y3-SC-ENR+AC-CC-S010-LONG.

Data included the following samples and trip blanks:

Sample ID	Sample Date/Time	Lab ID
LDW-Y3-EXTRA-S010-TB	09/29/2020 15:22	13371-001-0001-SA
LDW-Y3-IN-ENR+AC-CA-S010	09/29/2020 09:53	13371-002-0001-SA
LDW-Y3-IN-ENR+AC-CB-S010	09/29/2020 10:06	13371-003-0001-SA
LDW-Y3-IN-ENR+AC-CC-S010	09/29/2020 10:15	13371-004-0001-SA
LDW-Y3-IN-ENR-CB-S010	09/29/2020 10:50	13371-008-0001-SA
LDW-Y3-IN-ENR-CC-S010	09/29/2020 11:01	13371-009-0001-SA
LDW-Y3-IN-ENR-CD-S010	09/29/2020 11:13	13371-010-0001-SA
LDW-Y3-IN-S010-TB	09/28/2020 15:15	13371-012-0001-SA
LDW-Y3-SC-ENR+AC-CA-S010	09/29/2020 13:42	13371-013-0001-SA
LDW-Y3-SC-ENR+AC-CA-S010-LONG	09/29/2020 15:44	13371-014-0001-SA
LDW-Y3-SC-ENR+AC-CB-S010	09/29/2020 13:51	13371-015-0001-SA
LDW-Y3-SC-ENR+AC-CB-S010-LONG	09/29/2020 15:54	13371-016-0001-SA
LDW-Y3-SC-ENR+AC-CC-S010	09/29/2020 13:59	13371-017-0001-SA
LDW-Y3-SC-ENR+AC-CC-S010-LONG	09/29/2020 16:02	13371-018-0001-SA
LDW-Y3-SC-ENR+AC-S010-DEP	09/28/2020 14:49	13372-003-0001-SA
LDW-Y3-SC-ENR-CA-S010	09/29/2020 14:36	13372-004-0001-SA
LDW-Y3-SC-ENR-CA-S010-LONG	09/29/2020 16:27	13372-005-0001-SA
LDW-Y3-SC-ENR-CC-S010	09/29/2020 14:46	13372-008-0001-SA
LDW-Y3-SC-ENR-CC-S010-LONG	09/29/2020 16:44	13372-009-0001-SA
LDW-Y3-SC-ENR-CD-S010	09/29/2020 15:01	13372-010-0001-SA

Sample ID	Sample Date/Time	Lab ID
LDW-Y3-SC-ENR-CD-S010-LONG	09/29/2020 16:53	13372-011-0001-SA
LDW-Y3-SC-ENR-S010-DEP	09/28/2020 14:40	13372-014-0001-SA
LDW-Y3-SC-S010-TB	09/28/2020 15:00	13372-015-0001-SA
LDW-Y3-SU-ENR+AC-CA-S010	09/28/2020 09:58	13372-016-0001-SA
LDW-Y3-SU-ENR+AC-CA-S010-BIO	09/28/2020 13:21	13372-017-0001-SA
LDW-Y3-SU-ENR+AC-CB-S010-BIO	09/28/2020 13:30	13372-019-0001-SA
LDW-Y3-SU-ENR+AC-CC-S010	09/28/2020 10:22	13372-020-0001-SA
LDW-Y3-SU-ENR+AC-CC-S010-BIO	09/28/2020 13:38	13373-001-0001-SA
LDW-Y3-SU-ENR+AC-CD-S010	09/28/2020 10:33	13373-002-0001-SA
LDW-Y3-SU-ENR-CA-S010	09/28/2020 10:50	13373-004-0001-SA
LDW-Y3-SU-ENR-CA-S010-BIO	09/28/2020 13:55	13373-005-0001-SA
LDW-Y3-SU-ENR-CB-S010	09/28/2020 10:57	13373-006-0001-SA
LDW-Y3-SU-ENR-CB-S010-BIO	09/28/2020 14:02	13373-007-0001-SA
LDW-Y3-SU-ENR-CC-S010	09/28/2020 11:03	13373-008-0001-SA
LDW-Y3-SU-ENR-CC-S010-BIO	09/28/2020 14:11	13373-009-0001-SA
LDW-Y3-SU-S010-LCB	09/28/2020 11:47	13373-012-0001-SA
LDW-Y3-SU-S010-TB	09/28/2020 15:06	13373-013-0001-SA
LDW-Y3-LBS-WAT-S010-SPME	09/28/2020 13:47	13373-014-0001-SA

2.0 Data Sources

Data were loaded from various sources in order to independently calculate each PCB Cfree concentration and detection limit. Spot checking and limited recalculation was done to verify the data sources as described below:

Laboratory reported concentrations: Laboratory reported mass concentrations were obtained from the laboratory electronic data deliverable (EDD). Sporadic comparisons were made to the data in spreadsheet tab “Table A3. Mass of PCBs”, and no discrepancies were noted for the PCB mass concentrations. However, the PCB mass DLs require further clarification:

Data reported in the “PCB Mass DL” columns was a mix of values. For the non-detect compounds, this column was populated with the estimated detection limit (EDL) which was found in the laboratory report under column heading of “DL” and in the EDD in the “Result” field. For detected results, this column was populated with the minimum level of quantitation (ML) which was not present on the laboratory report, but was present in the laboratory EDD in the “ML” field. It should be noted that the ML is a quantitation limit rather than a detection limit.

No discrepancies were noted between the values listed in the column and the laboratory EDLs and MLs.

SPME sampling details: SPME fiber details were obtained from the data in spreadsheet tab “Table A1. Fiber details”. Data for mass of fiber, length of fiber, % recovery, volume of PDMS, and mass of PDMS were recalculated from the remaining data. The following discrepancy was noted:

The recalculated PDMS volumes for samples LDW-Y3-SC-ENR+AC-CB-S010-LONG and LDW-Y3-SC-ENR+AC-CC-S010-LONG were slightly different than the values were in the original submission. GeoSyntec was contacted and corrections were submitted on December 14, 2020.

Reference values: Log K_{PDMS} values for each PCB were retained from the calibration study/baseline sample calculation review. These values were spot checked against reference

the data in spreadsheet tab “Table A5 KPDMS” of the original submission. No discrepancies were noted.

Reported PCB Cfree concentrations: Reported PCB Cfree concentrations were obtained from the data in spreadsheet tab “T1. Cfree(Final)”. This data was used for the basis of comparison to recalculated values. However, the PCB Cfree DL values contained the same mix of values that the spreadsheet tab “Table A3. Mass of PCBs”, did. For the non-detect compounds, PCB Cfree EDL were listed. For detected compounds, PCB Cfree MLs were listed.

3.0 Calculations

Formulas retained from the calibration study/baseline sample calculation review were used to recalculate PCB free concentrations.

Data calculations were performed in a Microsoft Access database. The Access Visual Basic for Applications (VBA) subroutine developed for the calibration study/baseline sample calculation review were again used to calculate the PCB free concentrations, EDLs, MLs and MDLs, and to compare the recalculated results to the reported results.

4.0 Conclusions

Concentrations: Cfree concentrations of each detected PCB was recalculated and compared to the resubmitted values. Concentrations agreed within a reasonable variation for rounding differences. Calculated relative percent differences (RPDs) were between 0 and 5.

Reporting limits: Cfree EDLs or MLs of each PCB was recalculated and compared to the reported and resubmitted values. These agreed within a reasonable variation for rounding differences. Calculated relative percent differences (RPDs) were between 0 and 5.

PRC Model: PRC Calculations were shown in tab “Table A4. ke”. Values for Ke, slope, y-intercept and R2 were recalculated with good agreement.

The PRC coefficient of determination (R2) ranged from 0.31 to 0.99 indicating poor linearity in some samples. The upper and lower confidence limits were calculated to demonstrate the potential variability in the results, and this analysis was included on the “TA6_Uncertainty Cfree” tab. It should be noted that these upper and lower confidence limits reflects variation due to PRC linearity, but not necessarily variation due to other factors such as analytical reproducibility or matrix inhomogeneity, or the number of PRC compounds included in the PRC regression.

Eight samples had a p-value exceeding the 0.05 criteria for statistical significance and 5 samples utilized fewer than half of the PRC compounds:

Sample ID	Number of PRCs Used	p-Value	r^2
LDW-Y3-SC-ENR+AC-CB-S010-LONG	2	NA	NA
LDW-Y3-IN-ENR-CD-S010	3	0.1275	0.960433
LDW-Y3-SU-ENR-CB-S010-BIO	3	0.3118	0.778683
LDW-Y3-SC-ENR+AC-CA-S010-LONG	4	0.069	0.866833
LDW-Y3-SC-ENR+AC-CC-S010-LONG	4	0.1126	0.759576
LDW-Y3-SC-ENR+AC-S010-DEP	4	0.0134	0.973346
LDW-Y3-SU-ENR+AC-CA-S010-BIO	4	0.0854	0.836436
LDW-Y3-SU-ENR+AC-CB-S010-BIO	4	0.0053	0.989512
LDW-Y3-SU-ENR+AC-CC-S010-BIO	4	0.0176	0.965048

Sample ID	Number of PRCs Used	p-Value	r^2
LDW-Y3-SC-ENR-CD-S010-LONG	8	0.057817	0.477225
LDW-Y3-SU-ENR-CC-S010-BIO	8	0.1441	0.319769
LDW-Y3-SC-ENR-S010-DEP	9	0.099333	0.340039

The variability in results for these sample may exceed the upper or lower confidence limits shown in tab “TA6_Uncertainty Cfree”.

Qualifiers: Qualifiers listed in the tab T1. Cfree(FINAL)’ were reviewed and were determined to be in agreement with the supplied definitions.

One clarification should be noted: A UB qualifier indicates that the background concentration exceeded the detected concentration. These results should be considered not detected at the lowest available detection limit, the method detection limit (MDL). However, the PCB CFree DL column in in Table 1 Cfree (final) shows the CFree Minimum Level, not the CFree MDL. Laboratory mass method detection limits were converted to CFree values for the samples shown below:

Sample ID	Analyte	PCB CFree MDL (pg/L)	PCB CFree ML (pg/L)	Qualifier
LDW-Y3-IN-ENR+AC-CA-S010	PCB-011	0.565	5.1	UB J
LDW-Y3-IN-ENR+AC-CA-S010	PCB-131	0.0929	1.3	UB C,J L
LDW-Y3-IN-ENR+AC-CA-S010	PCB-207	0.0137	0.45	UB L
LDW-Y3-IN-ENR+AC-CB-S010	PCB-011	0.462	4.2	UB
LDW-Y3-IN-ENR+AC-CB-S010	PCB-131	0.0418	0.57	UB C
LDW-Y3-IN-ENR+AC-CB-S010	PCB-197	0.00955	0.17	UB L
LDW-Y3-IN-ENR+AC-CB-S010	PCB-207	0.0031	0.1	UB L
LDW-Y3-IN-ENR+AC-CB-S010	PCB-209	0.00202	0.06	UB L
LDW-Y3-IN-ENR+AC-CC-S010	PCB-011	0.512	4.6	UB J
LDW-Y3-IN-ENR+AC-CC-S010	PCB-131	0.0813	1.1	UB C,J L
LDW-Y3-IN-ENR-CB-S010	PCB-197	0.203	3.6	UB L
LDW-Y3-IN-ENR-CB-S010	PCB-207	0.116	3.8	UB L
LDW-Y3-IN-ENR-CC-S010	PCB-011	0.661	6	UB
LDW-Y3-IN-ENR-CD-S010	PCB-131	0.135	1.8	UB C L
LDW-Y3-LBS-WAT-S010-SPME	PCB-011	0.67	6.1	UB
LDW-Y3-LBS-WAT-S010-SPME	PCB-207	0.00076	0.025	UB L
LDW-Y3-LBS-WAT-S010-SPME	PCB-209	0.000354	0.011	UB L
LDW-Y3-SC-ENR+AC-CA-S010	PCB-131	0.148	2	UB C L
LDW-Y3-SC-ENR+AC-CA-S010-LONG	PCB-131	0.948	13	UB C L
LDW-Y3-SC-ENR+AC-CB-S010	PCB-131	0.215	2.9	UB C L
LDW-Y3-SC-ENR+AC-CB-S010-LONG	PCB-131	0.366	4.94	UB C L
LDW-Y3-SC-ENR+AC-CC-S010	PCB-131	0.052	0.7	UB C L
LDW-Y3-SC-ENR+AC-CC-S010	PCB-209	0.00217	0.065	UB L
LDW-Y3-SC-ENR+AC-CC-S010-LONG	PCB-131	0.996	13.5	UB C L
LDW-Y3-SC-ENR+AC-S010-DEP	PCB-131	0.428	5.8	UB C L
LDW-Y3-SC-ENR-CA-S010	PCB-011	0.625	5.7	UB
LDW-Y3-SC-ENR-CA-S010	PCB-207	0.0232	0.76	UB L
LDW-Y3-SC-ENR-CA-S010-LONG	PCB-011	0.549	5	UB
LDW-Y3-SC-ENR-CA-S010-LONG	PCB-131	0.0505	0.68	UB C L
LDW-Y3-SC-ENR-CA-S010-LONG	PCB-197	0.011	0.2	UB L
LDW-Y3-SC-ENR-CA-S010-LONG	PCB-207	0.00348	0.11	UB L
LDW-Y3-SC-ENR-CA-S010-LONG	PCB-209	0.0022	0.066	UB L
LDW-Y3-SC-ENR-CC-S010	PCB-131	0.139	1.9	UB C L
LDW-Y3-SC-ENR-CC-S010-LONG	PCB-131	0.0342	0.46	UB C
LDW-Y3-SC-ENR-CC-S010-LONG	PCB-207	0.00181	0.059	UB L
LDW-Y3-SC-ENR-CC-S010-LONG	PCB-209	0.00106	0.032	UB L
LDW-Y3-SC-ENR-CD-S010	PCB-131	0.114	1.5	UB C L

Sample ID	Analyte	PCB CFree MDL (pg/L)	PCB CFree ML (pg/L)	Qualifier
LDW-Y3-SC-ENR-CD-S010	PCB-209	0.0283	0.84	UB L
LDW-Y3-SC-ENR-CD-S010-LONG	PCB-011	0.632	5.7	UB
LDW-Y3-SC-ENR-CD-S010-LONG	PCB-131	0.0719	0.97	UB C L
LDW-Y3-SC-ENR-CD-S010-LONG	PCB-197	0.018	0.32	UB L
LDW-Y3-SC-ENR-CD-S010-LONG	PCB-207	0.00603	0.2	UB L
LDW-Y3-SC-ENR-CD-S010-LONG	PCB-209	0.00405	0.12	UB L
LDW-Y3-SC-ENR-S010-DEP	PCB-131	0.0747	1	UB C L
LDW-Y3-SC-ENR-S010-DEP	PCB-172	0.0348	0.51	UB L
LDW-Y3-SC-ENR-S010-DEP	PCB-207	0.00449	0.15	UB L
LDW-Y3-SC-ENR-S010-DEP	PCB-209	0.00271	0.081	UB L
LDW-Y3-SU-ENR+AC-CA-S010	PCB-131	0.0334	0.45	UB C
LDW-Y3-SU-ENR+AC-CA-S010-BIO	PCB-011	0.878	7.9	UB
LDW-Y3-SU-ENR+AC-CA-S010-BIO	PCB-131	0.316	4.3	UB C L
LDW-Y3-SU-ENR+AC-CB-S010-BIO	PCB-011	0.589	5.3	UB
LDW-Y3-SU-ENR+AC-CB-S010-BIO	PCB-131	0.276	3.7	UB C L
LDW-Y3-SU-ENR+AC-CC-S010	PCB-131	0.0199	0.27	UB C
LDW-Y3-SU-ENR+AC-CC-S010	PCB-207	0.000644	0.021	UB
LDW-Y3-SU-ENR+AC-CC-S010-BIO	PCB-011	0.53	4.8	UB
LDW-Y3-SU-ENR+AC-CC-S010-BIO	PCB-131	0.102	1.4	UB C L
LDW-Y3-SU-ENR+AC-CD-S010	PCB-131	0.0279	0.38	UB C
LDW-Y3-SU-ENR+AC-CD-S010	PCB-207	0.00138	0.045	UB L
LDW-Y3-SU-ENR-CA-S010	PCB-131	0.0219	0.3	UB C
LDW-Y3-SU-ENR-CA-S010	PCB-197	0.00288	0.051	UB
LDW-Y3-SU-ENR-CA-S010-BIO	PCB-011	0.85	7.7	UB
LDW-Y3-SU-ENR-CA-S010-BIO	PCB-131	0.0563	0.76	UB C L
LDW-Y3-SU-ENR-CA-S010-BIO	PCB-209	0.0013	0.039	UB L
LDW-Y3-SU-ENR-CB-S010	PCB-131	0.0233	0.31	UB C
LDW-Y3-SU-ENR-CB-S010	PCB-197	0.00307	0.055	UB
LDW-Y3-SU-ENR-CB-S010	PCB-207	0.000775	0.025	UB L
LDW-Y3-SU-ENR-CB-S010	PCB-209	0.000392	0.012	UB L
LDW-Y3-SU-ENR-CB-S010-BIO	PCB-131	0.334	4.5	UB C L
LDW-Y3-SU-ENR-CC-S010	PCB-131	0.0245	0.33	UB C
LDW-Y3-SU-ENR-CC-S010	PCB-197	0.0036	0.064	UB L
LDW-Y3-SU-ENR-CC-S010	PCB-207	0.000958	0.031	UB L
LDW-Y3-SU-ENR-CC-S010	PCB-209	0.000511	0.015	UB L
LDW-Y3-SU-ENR-CC-S010-BIO	PCB-011	1.25	11	UB
LDW-Y3-SU-ENR-CC-S010-BIO	PCB-131	0.151	2	UB C L
LDW-Y3-SU-ENR-CC-S010-BIO	PCB-197	0.0365	0.65	UB L
LDW-Y3-SU-ENR-CC-S010-BIO	PCB-207	0.012	0.39	UB L
LDW-Y3-SU-ENR-CC-S010-BIO	PCB-209	0.00785	0.23	UB L
LDW-Y3-SU-S010-LCB	PCB-207	0.00137	0.045	UB L
LDW-Y3-SU-S010-LCB	PCB-209	0.00076	0.023	UB L

Total detected PCBs: Total detected PCBs were recalculated based on the reported individual PCB Cfree concentrations, excluding spiked PRC compounds. Total detected PCBs were recalculated with good agreement.

Confidence levels: The uncertainty upper and lower confidence levels summarized in the spreadsheet tab "TA6_Uncertainty Cfree" are beyond the scope of this review and have not been recalculated.

5.0 Abbreviations and Definitions

Abbreviation	Definition
EDL	Estimated detection limit
MDL	Method detection limit

Abbreviation	Definition
ML	Minimum level of quantitation
PDMS	Polydimethylsiloxane
PRC	Performance Reference Compound
RPD	Relative percent difference
SPME	Solid phase microextraction

6.0 References

Certificate of Analysis Concentrations of Freely-dissolved Polychlorinated Biphenyls (PDBs) Measured via SP3ME Passive Samplers. Prepared for Lower Duwamish Waterway Group, Prepared by Geosyntec Consultants, March 22, 2017. This report contains data for samples collected July to September 2016.

Certificate of Analysis Concentrations of Freely-dissolved Polychlorinated Biphenyls (PDBs) Measured via SP3ME Passive Samplers. Prepared for Lower Duwamish Waterway Group, Prepared by Geosyntec Consultants, March 22, 2017. This report contains data for samples collected November 2016 to January 2017.

Polymer-water partition coefficients of hydrophobic compounds for passive sampling: Application of cosolvent models for validation. Environ. Sci. Technol. 43:7047-7054. Smedes, et al. 2009.

Quality Assurance Project Plan Enhanced Natural Recovery/Activated Carbon Pilot Study, Lower Duwamish Waterway. Prepared by AMEC Foster Wheeler Environment & Infrastructure Inc., et al. Prepared for: USEPA Region 10 and WA-DOE Northwest Regional Office, February 22, 2016.

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DATA VALIDATION REPORT

Lower Duwamish Waterway– Enhanced Natural Recovery/Activated Carbon Pilot Study, Year Three Samples, July 2020 – October 2020

Prepared for:
Wood Environment and Infrastructure Solutions
3500 188th Street SW, Ste 601
Lynnwood, WA 98037-4763

December 18, 2020

1.0 Introduction

Data validation was performed on the following samples:

Sample ID	Sample Date/Time	Lab ID	Analyses
Sediment Samples			
LDW-Y3-IN-ENR+AC-CA-CORE	10/01/2020 10:50	13376-006-0001-SA, K2008786-006	PCB,TOC, BC,GS
LDW-Y3-IN-ENR+AC-CB-CORE	10/01/2020 10:55	13376-007-0001-SA, K2008786-007	PCB,TOC, BC,GS
LDW-Y3-IN-ENR+AC-CC-CORE	10/01/2020 11:00	13376-008-0001-SA, K2008786-008	PCB,TOC, BC,GS
LDW-Y3-IN-ENR-CB-CORE	10/01/2020 10:20	13376-002-0001-SA, K2008786-002	PCB,TOC, BC,GS
LDW-Y3-IN-ENR-CC-CORE	10/01/2020 10:25	13376-003-0001-SA, K2008786-003	PCB,TOC, BC,GS
LDW-Y3-IN-ENR-CD-CORE	10/01/2020 10:30	13376-004-0001-SA, K2008786-004	PCB,TOC, BC,GS
LDW-Y3-SC-ENR+AC-CA-CORE	10/16/2020 12:25	13416-015-0001-SA, K2009513-016	PCB,TOC, BC,GS
LDW-Y3-SC-ENR+AC-CA-ULM	10/16/2020 12:30	13416-006-0001-SA, K2009513-006	PCB,TOC, BC,GS
LDW-Y3-SC-ENR+AC-CB-CORE	10/16/2020 11:55	13416-016-0001-SA, K2009513-017	PCB,TOC, BC,GS
LDW-Y3-SC-ENR+AC-CB-ULM	10/16/2020 12:00	13416-007-0001-SA, K2009513-007	PCB,TOC, BC,GS
LDW-Y3-SC-ENR+AC-CC-CORE	10/16/2020 11:30	13416-017-0001-SA, K2009513-018	PCB,TOC, BC,GS
LDW-Y3-SC-ENR+AC-CC-ULM	10/16/2020 11:35	13416-008-0001-SA, K2009513-008	PCB,TOC, BC,GS
LDW-Y3-SC-ENR+AC-SS	08/11/2020 14:55	13417-002-0001-SA, K2006959-002	PCB,TOC, BC,GS
LDW-Y3-SC-ENR-CA-CORE	10/16/2020 12:55	13416-010-0001-SA, K2009513-011	PCB,TOC, BC,GS
LDW-Y3-SC-ENR-CA-ULM	10/16/2020 13:00	13416-001-0001-SA, K2009513-001	PCB,TOC, BC,GS
LDW-Y3-SC-ENR-CC-CORE	10/16/2020 12:10	13416-012-0001-SA, K2009513-013	PCB,TOC, BC,GS
LDW-Y3-SC-ENR-CC-ULM	10/16/2020 12:15	13416-003-0001-SA, K2009513-003	PCB,TOC, BC,GS
LDW-Y3-SC-ENR-CD-CORE	10/16/2020 11:00	13416-013-0001-SA, K2009513-014	PCB,TOC, BC,GS
LDW-Y3-SC-ENR-CD-ULM	10/16/2020 11:05	13416-004-0001-SA, K2009513-004	PCB,TOC, BC,GS
LDW-Y3-SC-ENR-SS	08/11/2020 15:15	13417-001-0001-SA, K2006959-001	PCB,TOC, BC,GS
LDW-Y3-SU-ENR+AC-CA-CORE	07/24/2020 10:25	13237-006-0001-SA, K2006339-004	PCB,TOC,BC,GS
LDW-Y3-SU-ENR+AC-CB-CORE	07/24/2020 10:30	K2006339-005	TOC,GS
LDW-Y3-SU-ENR+AC-CC-CORE	07/24/2020 10:40	13237-008-0001-SA, K2006339-006	PCB,TOC, BC,GS
LDW-Y3-SU-ENR+AC-CD-CORE	07/24/2020 10:45	13237-009-0001-SA, K2009513-010	PCB,TOC, BC,GS
LDW-Y3-SU-ENR-CA-CORE	07/24/2020 09:25	13237-001-0001-SA, K2006339-001	PCB,TOC, BC,GS
LDW-Y3-SU-ENR-CB-CORE	07/24/2020 09:30	13237-002-0001-SA, K2006339-002	PCB,TOC, BC,GS
LDW-Y3-SU-ENR-CC-CORE	07/24/2020 09:40	13237-003-0001-SA, K2006339-003	PCB,TOC, BC,GS
Solvent Samples			
LDW-Y3-EXTRA-S010-TB	09/29/2020 15:22	13371-001-0001-SA	PCB
LDW-Y3-IN-ENR+AC-CA-S010	09/29/2020 09:53	13371-002-0001-SA	PCB
LDW-Y3-IN-ENR+AC-CB-S010	09/29/2020 10:06	13371-003-0001-SA	PCB

Sample ID	Sample Date/Time	Lab ID	Analyses
LDW-Y3-IN-ENR+AC-CC-S010	09/29/2020 10:15	13371-004-0001-SA	PCB
LDW-Y3-IN-ENR-CB-S010	09/29/2020 10:50	13371-008-0001-SA	PCB
LDW-Y3-IN-ENR-CC-S010	09/29/2020 11:01	13371-009-0001-SA	PCB
LDW-Y3-IN-ENR-CD-S010	09/29/2020 11:13	13371-010-0001-SA	PCB
LDW-Y3-IN-S010-TB	09/28/2020 15:15	13371-012-0001-SA	PCB
LDW-Y3-SC-ENR+AC-CA-S010	09/29/2020 13:42	13371-013-0001-SA	PCB
LDW-Y3-SC-ENR+AC-CA-S010-LONG	09/29/2020 15:44	13371-014-0001-SA	PCB
LDW-Y3-SC-ENR+AC-CB-S010	09/29/2020 13:51	13371-015-0001-SA	PCB
LDW-Y3-SC-ENR+AC-CB-S010-LONG	09/29/2020 15:54	13371-016-0001-SA	PCB
LDW-Y3-SC-ENR+AC-CC-S010	09/29/2020 13:59	13371-017-0001-SA	PCB
LDW-Y3-SC-ENR+AC-CC-S010-LONG	09/29/2020 16:02	13371-018-0001-SA	PCB
LDW-Y3-SC-ENR+AC-S010-DEP	09/28/2020 14:49	13372-003-0001-SA	PCB
LDW-Y3-SC-ENR-CA-S010	09/29/2020 14:36	13372-004-0001-SA	PCB
LDW-Y3-SC-ENR-CA-S010-LONG	09/29/2020 16:27	13372-005-0001-SA	PCB
LDW-Y3-SC-ENR-CC-S010	09/29/2020 14:46	13372-008-0001-SA	PCB
LDW-Y3-SC-ENR-CC-S010-LONG	09/29/2020 16:44	13372-009-0001-SA	PCB
LDW-Y3-SC-ENR-CD-S010	09/29/2020 15:01	13372-010-0001-SA	PCB
LDW-Y3-SC-ENR-CD-S010-LONG	09/29/2020 16:53	13372-011-0001-SA	PCB
LDW-Y3-SC-ENR-S010-DEP	09/28/2020 14:40	13372-014-0001-SA	PCB
LDW-Y3-SC-S010-TB	09/28/2020 15:00	13372-015-0001-SA	PCB
LDW-Y3-SU-ENR+AC-CA-S010	09/28/2020 09:58	13372-016-0001-SA	PCB
LDW-Y3-SU-ENR+AC-CA-S010-BIO	09/28/2020 13:21	13372-017-0001-SA	PCB
LDW-Y3-SU-ENR+AC-CB-S010-BIO	09/28/2020 13:30	13372-019-0001-SA	PCB
LDW-Y3-SU-ENR+AC-CC-S010	09/28/2020 10:22	13372-020-0001-SA	PCB
LDW-Y3-SU-ENR+AC-CC-S010-BIO	09/28/2020 13:38	13373-001-0001-SA	PCB
LDW-Y3-SU-ENR+AC-CD-S010	09/28/2020 10:33	13373-002-0001-SA	PCB
LDW-Y3-SU-ENR-CA-S010	09/28/2020 10:50	13373-004-0001-SA	PCB
LDW-Y3-SU-ENR-CA-S010-BIO	09/28/2020 13:55	13373-005-0001-SA	PCB
LDW-Y3-SU-ENR-CB-S010	09/28/2020 10:57	13373-006-0001-SA	PCB
LDW-Y3-SU-ENR-CB-S010-BIO	09/28/2020 14:02	13373-007-0001-SA	PCB
LDW-Y3-SU-ENR-CC-S010	09/28/2020 11:03	13373-008-0001-SA	PCB
LDW-Y3-SU-ENR-CC-S010-BIO	09/28/2020 14:11	13373-009-0001-SA	PCB
LDW-Y3-SU-S010-LCB	09/28/2020 11:47	13373-012-0001-SA	PCB
LDW-Y3-SU-S010-TB	09/28/2020 15:06	13373-013-0001-SA	PCB
LDW-Y3-LBS-WAT-S010-SPME	09/28/2020 13:47	13373-014-0001-SA	PCB
Tissue Samples			
LDW-Y3-LBS-SU-ENR-A-CLAM	09/24/2020	13378-007-0001-SA	PCB
LDW-Y3-LBS-SU-ENR-B-CLAM	09/24/2020	13378-014-0001-SA	PCB
LDW-Y3-LBS-SU-ENR-C-CLAM	09/24/2020	13378-021-0001-SA	PCB
LDW-Y3-LBS-SU-ENR+AC-A-CLAM	09/24/2020	13379-007-0001-SA	PCB
LDW-Y3-LBS-SU-ENR+AC-B-CLAM	09/24/2020	13379-014-0001-SA	PCB
LDW-Y3-LBS-SU-ENR+AC-C-CLAM	09/24/2020	13379-021-0001-SA	PCB
LDW-Y3-LBS-SU-ENR-A-WORM	09/24/2020	13380-007-0001-SA	PCB
LDW-Y3-LBS-SU-ENR-B-WORM	09/24/2020	13380-014-0001-SA	PCB
LDW-Y3-LBS-SU-ENR-C-WORM	09/24/2020	13380-021-0001-SA	PCB
LDW-Y3-LBS-SU-ENR+AC-A-WORM	09/24/2020	13380-028-0001-SA	PCB
LDW-Y3-LBS-SU-ENR+AC-B-WORM	09/24/2020	13383-007-0001-SA	PCB
LDW-Y3-LBS-SU-ENR+AC-C-WORM	09/24/2020	13383-014-0001-SA	PCB
LDW-Y3-LBS-CLAM-BAS	09/24/2020	13383-018-0001-SA	PCB
LDW-Y3-LBS-WORM-BAS	09/24/2020	13383-022-0001-SA	PCB

PCB analyses were performed by Frontier Analytical Laboratory (Frontier), in El Dorado Hills, California. TOC analyses were performed by ALS Environmental (ALS) in Kelso, Washington. Grain size analyses were performed by Materials Testing & Consulting, Inc (MTC). Black carbon analyses were performed by the University of Maryland, Baltimore County.

Validation: A full validation was performed on the PCB data. A summary validation was performed on the TOC and grain size data. A limited validation was performed on the black carbon data. Validation was performed by Cari Sayler. Data qualifiers are summarized in section 6.0 of this report.

Analytical methods: Table 3.3 of the QAPP specifies the following analytical methods:

Analysis	Method
Polychlorinated Biphenyl Congeners (PCB)	EPA 1668C
Total Organic Carbon (TOC)	EPA 9060
Black Carbon (BC)	Gustafsson et al. (1997)
Grain size (GS)	ASTM D422

These methods were used with the following exception: MTC utilized the Puget Sound Estuary Protocol (PSEP) method. This is considered an acceptable substitution. Additionally, The laboratory report did not specify a version for the PCB congener analysis, referencing only "EPA Method 1668. Data were validated using criteria from version EPA 1668C. No method was specified in the black carbon data, and could not be reviewed.

Requested analyses: Sample chain-of-custodies and sample log-in documentation were reviewed. All requested analyses were performed.

Sample number transcription: Sample IDs in the electronic data deliverable (EDD) were compared to the chain-of-custody for each sample. Sample IDs matched the chain of custody with one exception: LDW-Y3-SC-ENR-CA-ULM was listed in the MTC (grain size) laboratory EDD and report as LDW-YS-SC-ENR-CA-ULM.

2.0 Polychlorinated Biphenyl Analyses

Quality control analysis frequencies: The method specifies that method blank and ongoing precision and recovery (OPR) samples must be analyzed with each batch. In addition, injection standards, isotope dilution standards and cleanup standards must be measured in each field and quality control sample. These frequencies were met.

Analysis holding times: Method 1668C specifies a one year holding time between extraction and analysis, and a one year holding time from sampling to extraction for both water and Sediment samples. These holding times were met.

System performance checks: System performance criteria include: 1) The tune must demonstrate a resolving power $\geq$10,000 at m/z 330.9792 and $\geq$8,000 throughout the range. 2) The monitored m/z must be <5 ppm from theoretical for the following theoretical m/z's: 218.9856, 242.9856, 280.9825, 330.9792, 354.9797, 354.9792, and 454.9728. 3) The retention time of congener 209 must exceed 55 minutes on the SPB-Octyl column. 4) The isomer specificity check must demonstrate resolution of congeners with valleys of $\leq$40% for congeners PCB-034 from PCB-023 and PCB-187 from PCB-182 on the SPB-Octyl Column. 5) The isomer specificity check must demonstrate elution of PCB 156 and PCB 157 within 2 seconds for the SPB-Octyl Column.

The laboratory utilized a DB1 column and provided the following column-specific performance criteria: Resolution of congeners with valleys of $\leq$40% for congeners PCB-156 and 157 and PCB 209 RT$\geq$50 minutes. Additionally, congeners 106 and 118 were evaluated for coelution within 2 seconds. These criteria were met.

Instrument calibration: Initial calibration criteria include 1) maximum percent relative standard deviations (%RSD) of ≤20% for target compounds and ≤35% for labeled compounds, 2) Ion abundance ratios must be within ±15% of theoretical, and 3) signal to noise ratios must be at or above 10. Continuing calibration criteria include 1) percent recoveries within 75-125% for target compounds, 65-135% for 13C-PCB-028 and 75-125% for 13C-PCB-111 and 13C-PCB-178, and 50-145% for the remaining labeled compounds. 2) Ion abundance ratios must be within ± 15% of theoretical, and 3) signal to noise ratios must be at or above 10. 4) Absolute retention times for injection internal standards must be within ± 15 seconds of the initial calibration and 5) Relative retention times (RRT) must meet method or column-specific criteria. These criteria were met.

Laboratory blank results: Laboratory performance criteria in method 1668C states that the method blank must not contain any target compound at a concentration greater than either the minimum level or one-third the regulatory compliance level, whichever is greater. Additionally, the method blank must not contain any potentially interfering compound at a concentration greater than either the minimum level or one-third the regulatory compliance level, whichever is greater. These criteria were met.

Extracted internal standard (surrogate) recoveries: Method criteria are 5-145% for labeled congeners between C13-PCB-001 and C13-PCB-054 and 10-145% for labeled congeners between C13-PCB-077 and C13-PCB-209. Recoveries were within these limits.

Cleanup standard recoveries: Method criteria are 5-145% for C13-PCB-028 and 10-145% for C13-PCB-111 and C13-PCB-178. Cleanup standard recoveries were within laboratory control limits.

OPR recoveries: Method criteria for OPR recoveries are 60-135% for 27 representative target compounds. OPR recoveries were within these limits.

Compound Identification: Method criteria for compound identification include: 1) The signals of the characteristic ions must maximize within the same 2 scans. 2) The signal to noise ratio must be greater than 2.5. 3) Ion abundance ratios must be within ± 15% of theoretical, or within ±15% of the calibration verification standard. 4) Relative retention times must meet method or column-specific criteria.

Criteria were reviewed for each Toxic WHO Congener. Neither the signal to noise ratio nor the individual signal in height and noise levels were included in the raw data for detected compounds. SICP chromatograms in these reports were reviewed and no evidence of noise interference was found.

PCB-197 in sample LDW-Y3-LBS-SU-ENR-C-WORM did not meet the ion abundance ratio criteria and was appropriately flagged M by the laboratory to indicate a maximum possible concentration. In order to clarify that this value is an estimate and that it should be included in the total PCB calculation. A validation qualifier of “M,J” is assigned.

No other identification discrepancies were noted.

Compound Quantitation: Sample concentrations were recalculated to verify sample quantitations. No quantitation discrepancies were noted.

Second column confirmation: Second column confirmation was not required to separate congeners 156 and 157 due to the use of the DB1 Column.

Second column confirmation was not performed to separate congeners 106 and 118. Since congener 106 is not a component of any of the commercial Aroclor mixtures, no further action was deemed necessary.

Estimated detection limits: Peak heights for one of the two isotope dilution standards were not present in the original data package. Resubmissions were requested, and received. Estimated detection limits (EDLs) were recalculated for PCB-169 in each sample and method blank. No discrepancies were noted.

All sediment EDLs met QAPP target reporting limits of 4 pg/g. Solvent EDLs ranged from 2.55 to 19.2 pg/sample. Tissue EDLs ranged from 0.425 pg/g wet weight to 2.91 pg/g wet weight.

Toxicity equivalent quantity (TEQ): TEQ calculations were not required for this project.

Laboratory narrative: No additional qualifiers are assigned based on the laboratory narrative.

Overall assessment: Documentation was found to be clear and complete. No discrepancies were noted in analyte identification or result quantitation. Calibration data and system performance checks demonstrate acceptable instrument performance. Quality control results indicate acceptable accuracy.

Polychlorinated biphenyl data are acceptable for use as qualified.

3.0 Total Organic Carbon (TOC) Analyses

Quality control analysis frequencies: Each sample was analyzed in duplicate. A method blank, laboratory control sample (LCS), matrix spike (MS), and matrix spike duplicate (MSD) was analyzed in each batch, meeting frequency requirements.

Holding times: TOC must be analyzed within 28 days. Samples should be shipped and maintained at temperatures between 0 and 6° Celsius. These criteria were met with one exception:

Sample ID	Sample Date	Analysis Date	Elapsed days
LDW-Y3-SU-ENR+AC-CD-CORE	7/24/2020	11/06/2020	105

The total organic carbon result in this sample should be considered estimated.

Laboratory blank results: Criteria for method blanks are that analyte concentrations must be below the PQL, or below 10% of the lowest associated sample concentration. This criterion was met for all method blanks.

LCS results: The LCS recovery control limit was 72-122%. This criterion was met.

MS recoveries: The MS and MSD recovery control limit was 70-122%. This criterion was met.

Sample replicate variability: The RPD between the first and second replicate analysis of each sample was below 20%.

Matrix spike duplicate variability: The MS/MSD control limit for RPDs was <20%. This criterion was met.

Total organic carbon results are acceptable for use as qualified.

4.0 Black Carbon (TOC) Analyses

Limited information was provided for the black carbon analysis. Data were presented in an excel spreadsheet titled "LDW_Y3_BC-Analysis.xlsx". No analysis date or analytical method was specified. Holding times and method-specific QC criteria (if any) could not be evaluated.

Standard recoveries: Two standards were analyzed. Recoveries were not provided, but were calculated based on the provided true value. The calculated recoveries were 95.7% and 93.8%, and are well within the QAPP accuracy goal of 75-125%.

Duplicate RPDs: Two sample duplicates were analyzed. RPDs were not provided, but were calculated as follows:

Sample ID	Sample Black Carbon Content	Duplicate Black Carbon Content	RPD
LDW-Y3-SU-ENR+AC-CC-CORE	0.46%	0.51%	8.5%
LDW-Y3-IN-ENR+AC-CC-CORE	2.02%	2.20%	8.2%
LDW-Y3-SC-ENR+AC-CC-CORE	3.02%	4.34%	36.0%
LDW-Y3-SC-ENR+AC-CC-ULM	3.40%	3.68%	7.8%

The RPD for LDW-Y3-SC-ENR+AC-CC-CORE exceeds the QAPP precision goal of ±25%, and this result is qualified as estimated.

Black carbon data are acceptable for use as qualified.

5.0 Grain Size Analyses

Quality control analysis frequencies: Each batch included a laboratory triplicate, meeting frequency requirements.

Holding times: Sediment samples must be analyzed within 6 months of collection. Samples were analyzed within the holding time.

Laboratory triplicate RSDs: Triplicate RSDs were below 25% with one exception:

Lab SDG	QC ID	Analyte	RSD
S20-0769	LDW-Y3-IN-ENR-CD-CORE LT	>10 Clay	45.3

Grain size data are acceptable for use as qualified.

6.0 Qualifier Summary Table

Client ID	Analyte(s)	Qualifier	Reason
Polychlorinated Biphenyl Analyses			
LDW-Y3-LBS-SU-ENR-C-WORM	PCB-197	M,J	Estimated maximum possible concentration
Total Organic Carbon Analysis			
LDW-Y3-SU-ENR+AC-CD-CORE	TOC	J	Holding time exceeded

Client ID	Analyte(s)	Qualifier	Reason
Black Carbon Analyses			
LDW-Y3-SC-ENR+AC-CC-CORE	Black Carbon	J	High duplicate RPD
Grain Size Analyses			
LDW-Y3-IN-ENR-CB-CORE	>10 Clay	J	High triplicate RSD
LDW-Y3-IN-ENR-CC-CORE	>10 Clay	J	High triplicate RSD
LDW-Y3-IN-ENR-CD-CORE	>10 Clay	J	High triplicate RSD
LDW-Y3-IN-ENR+AC-CC-CORE	>10 Clay	J	High triplicate RSD
LDW-Y3-IN-ENR+AC-CB-CORE	>10 Clay	J	High triplicate RSD
LDW-Y3-IN-ENR+AC-CA-CORE	>10 Clay	J	High triplicate RSD

7.0 Abbreviations and Definitions

DV Qualifier	Definition
U	The material was analyzed for, but was not detected above the level of the associated value.
UY	The reporting limit was elevated due to chromatographic overlap with related compounds. The material was analyzed for, but was not detected above the level of the associated value.
J	The analyte was positively identified. The associated numerical value is the approximate concentration of the analyte in the sample.
M,J	The analyte did not meet all identification criteria. The associated value should be considered an estimated maximum possible concentration.
N	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification.
UJ	The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.
R	The sample result is rejected. The presence or absence of the analyte cannot be verified and data are not usable.
R1	This sample result has been rejected in favor of a more accurate, precise or conservative result. The other result should be used.
R2	This sample result has been rejected in favor of a more accurate, precise or conservative result from another analytical method. The other result should be used.

Abbreviation	Definition
DV	Data validation
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
EDL	Estimated detection limit
EMPC	Estimated maximum possible concentration
IDS	Isotope dilution standard
MS	Matrix spike
MSD	Matrix spike duplicate
NA	Not Applicable
OPR	Ongoing Precision and Recovery
RL	Reporting limit
RPD	Relative percent difference
RRM	Regional reference material
RSD	Relative standard deviations

Abbreviation	Definition
SRM	Standard reference material

8.0 References

National Functional Guidelines For Inorganic Superfund Data Review, Office of Superfund Remediation and Technology Innovation, U.S. Environmental Protection Agency, August 2014, USEPA-540-R-13-001.

National Functional Guidelines for High Resolution Superfund Methods Data Review, Office of Superfund Remediation and Technology Innovation, U.S. Environmental Protection Agency, April 2016, EPA-542-B-16-001.

Method 1668C: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS, US Environmental Protection Agency, Office of Water Engineering and Analysis Division, April 2010.

Quality Assurance Project Plan Enhanced Natural Recovery/Activated Carbon Pilot Study, Lower Duwamish Waterway. Prepared by AMEC Foster Wheeler Environment & Infrastructure Inc., et al. Prepared for USEPA Region 10 and WA-DOE Northwest Regional Office, February 22, 2016.

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DATA VALIDATION REPORT

Lower Duwamish Waterway– Enhanced Natural Recovery/Activated Carbon Pilot Study, Year Three Additional Sediment Samples, January 2021

Prepared for:
Wood Environment and Infrastructure Solutions
3500 188th Street SW, Ste 601
Lynnwood, WA 98037-4763

February 21, 2021

1.0 Introduction

Data validation was performed on the following sediment samples:

Sample ID	Sample Date/Time	Lab ID	Analyses
LDW-Y3-LBS-SU-ENR-A-CORE	01/22/2021 10:25	13559-001-0001-SA, K2100698-001	PCB,TOC, BC,GS
LDW-Y3-LBS-SU-ENR-B-CORE	01/22/2021 11:50	13559-002-0001-SA K2100698-002	PCB,TOC, BC,GS
LDW-Y3-LBS-SU-ENR-C-CORE	01/22/2021 10:40	13559-003-0001-SA K2100698-003	PCB,TOC, BC,GS
LDW-Y3-LBS-SU-ENR+AC-A-CORE	01/22/2021 10:55	13559-004-0001-SA K2100698-004	PCB,TOC, BC,GS
LDW-Y3-LBS-SU-ENR+AC-B-CORE	01/22/2021 11:15	13559-005-0001-SA K2100698-005	PCB,TOC, BC,GS
LDW-Y3-LBS-SU-ENR+AC-C-CORE	01/22/2021 11:30	13559-006-0001-SA K2100698-006	PCB,TOC, BC,GS

PCB analyses were performed by Frontier Analytical Laboratory (Frontier), in El Dorado Hills, California. TOC analyses were performed by ALS Environmental (ALS) in Kelso, Washington. Grain size analyses were performed by Materials Testing & Consulting, Inc (MTC). Black carbon analyses were performed by the University of Maryland, Baltimore County.

Validation: A full validation was performed on the PCB data. A summary validation was performed on the TOC and grain size data. A limited validation was performed on the black carbon data. Validation was performed by Cari Sayler. No qualifiers were assigned as a result of this validation.

Analytical methods: Table 3.3 of the QAPP specifies the following analytical methods:

Analysis	Method
Polychlorinated Biphenyl Congeners (PCB)	EPA 1668C
Total Organic Carbon (TOC)	EPA 9060
Black Carbon (BC)	Gustafsson et al. (1997)
Grain size (GS)	ASTM D422

These methods were used with the following exception: MTC utilized the Puget Sound Estuary Protocol (PSEP) method. This is considered an acceptable substitution. Additionally, The laboratory report did not specify a version for the PCB congener analysis, referencing only "EPA Method 1668. Data were validated using criteria from version EPA 1668C. No method was specified in the black carbon data, and could not be reviewed.

Requested analyses: Sample chain-of-custodies and sample log-in documentation were reviewed. All requested analyses were performed.

Sample number transcription: Sample IDs in the electronic data deliverable (EDD) were compared to the chain-of-custody for each sample. Sample IDs matched the chain of custody.

2.0 Polychlorinated Biphenyl Analyses

Quality control analysis frequencies: The method specifies that method blank and ongoing precision and recovery (OPR) samples must be analyzed with each batch. In addition, injection standards, isotope dilution standards and cleanup standards must be measured in each field and quality control sample. These frequencies were met.

Analysis holding times: Method 1668C specifies a one year holding time between extraction and analysis, and a one year holding time from sampling to extraction for both water and Sediment samples. These holding times were met.

System performance checks: System performance criteria include: 1) The tune must demonstrate a resolving power $\geq$10,000 at m/z 330.9792 and $\geq$8,000 throughout the range. 2) The monitored m/z must be <5 ppm from theoretical for the following theoretical m/z's: 218.9856, 242.9856, 280.9825, 330.9792, 354.9797, 354.9792, and 454.9728. 3) The retention time of congener 209 must exceed 55 minutes on the SPB-Octyl column. 4) The isomer specificity check must demonstrate resolution of congeners with valleys of $\leq$40% for congeners PCB-034 from PCB-023 and PCB-187 from PCB-182 on the SPB-Octyl Column. 5) The isomer specificity check must demonstrate elution of PCB 156 and PCB 157 within 2 seconds for the SPB-Octyl Column.

The laboratory utilized a DB1 column and previously provided the following column-specific performance criteria: Resolution of congeners with valleys of $\leq$40% for congeners PCB-156 and 157 and PCB 209 RT$\geq$50 minutes. Additionally, congeners 106 and 118 were evaluated for coelution within 2 seconds. These criteria were met.

Instrument calibration: Initial calibration criteria include 1) maximum percent relative standard deviations (%RSD) of $\leq$20% for target compounds and $\leq$35% for labeled compounds, 2) Ion abundance ratios must be within $\pm$15% of theoretical, and 3) signal to noise ratios must be at or above 10. Continuing calibration criteria include 1) percent recoveries within 75-125% for target compounds, 65-135% for 13C-PCB-028 and 75-125% for 13C-PCB-111 and 13C-PCB-178, and 50-145% for the remaining labeled compounds. 2) Ion abundance ratios must be within $\pm$ 15% of theoretical, and 3) signal to noise ratios must be at or above 10. 4) Absolute retention times for injection internal standards must be within $\pm$ 15 seconds of the initial calibration and 5) Relative retention times (RRT) must meet method or column-specific criteria. These criteria were met.

Laboratory blank results: Laboratory performance criteria in method 1668C states that the method blank must not contain any target compound at a concentration greater than either the

minimum level or one-third the regulatory compliance level, whichever is greater. Additionally, the method blank must not contain any potentially interfering compound at a concentration greater than either the minimum level or one-third the regulatory compliance level, whichever is greater. These criteria were met.

Extracted internal standard (surrogate) recoveries: Method criteria are 5-145% for labeled congeners between C13-PCB-001 and C13-PCB-054 and 10-145% for labeled congeners between C13-PCB-077 and C13-PCB-209. Recoveries were within these limits for reported samples.

Cleanup standard recoveries: Method criteria are 5-145% for C13-PCB-028 and 10-145% for C13-PCB-111 and C13-PCB-178. Cleanup standard recoveries were within these limits for reported samples.

OPR recoveries: Method criteria for OPR recoveries are 60-135% for 27 representative target compounds. OPR recoveries were within these limits.

Compound Identification: Method criteria for compound identification include: 1) The signals of the characteristic ions must maximize within the same 2 scans. 2) The signal to noise ratio must be greater than 2.5. 3) Ion abundance ratios must be within ± 15% of theoretical, or within ±15% of the calibration verification standard. 4) Relative retention times must meet method or column-specific criteria.

Criteria were reviewed for each Toxic WHO Congener. Neither the signal to noise ratio nor the individual signal in height and noise levels were included in the raw data for detected compounds. SICP chromatograms in these reports were reviewed and no evidence of noise interference was found.

No other identification discrepancies were noted.

Compound Quantitation: Sample concentrations were recalculated to verify sample quantitations. No quantitation discrepancies were noted.

Second column confirmation: Second column confirmation was not required to separate congeners 156 and 157 due to the use of the DB1 Column.

Second column confirmation was not performed to separate congeners 106 and 118. Since congener 106 is not a component of any of the commercial Aroclor mixtures, no further action was deemed necessary.

Estimated detection limits: Peak heights for one of the two isotope dilution standards (IDS) were not present in the original data package. Estimated detection limits (EDLs) were recalculated for PCB-169 in each sample and method blank using an altered formula which included the single IDS height and a ratio of the two IDS areas. The recalculated EDLs agreed within 2 significant figures, and no further action was taken.

All sediment EDLs met QAPP target reporting limits of 4 pg/g.

Toxicity equivalent quantity (TEQ): TEQ calculations were not required for this project.

Laboratory narrative: No additional qualifiers are assigned based on the laboratory narrative.

Overall assessment: With a minor exception, documentation was found to be clear and complete. No discrepancies were noted in analyte identification or result quantitation. Calibration data and system performance checks demonstrate acceptable instrument performance. Quality control results indicate acceptable accuracy.

Polychlorinated biphenyl data are acceptable for use as reported.

3.0 Total Organic Carbon (TOC) Analyses

Quality control analysis frequencies: Each sample was analyzed in duplicate. A method blank, laboratory control sample (LCS), matrix spike (MS), and matrix spike duplicate (MSD) was analyzed in each batch, meeting frequency requirements.

Holding times: TOC must be analyzed within 28 days. Samples should be shipped and maintained at temperatures between 0 and 6° Celsius. These criteria were met.

Laboratory blank results: Criteria for method blanks are that analyte concentrations must be below the PQL, or below 10% of the lowest associated sample concentration. This criterion was met.

LCS results: The LCS recovery control limit was 72-122%. This criterion was met.

MS recoveries: The MS and MSD recovery control limit was 70-122%. This criterion was met.

Sample replicate variability: The RPD between the first and second replicate analysis of each sample was below 20%.

Matrix spike duplicate variability: The MS/MSD control limit for RPDs was <20%. This criterion was met.

Total organic carbon results are acceptable for use as reported.

4.0 Black Carbon (TOC) Analyses

Limited information was provided for the black carbon analysis. Data were presented in an excel spreadsheet titled “LDW-Y3_BC-Analysis_8Feb2021.xlsx”. No analysis date or analytical method was specified. Holding times and method-specific QC criteria (if any) could not be evaluated.

Standard recoveries: Two standards were analyzed. Recoveries were not provided, but were calculated based on the provided true value. The calculated recoveries were 95.9% and 92.0%, and are well within the QAPP accuracy goal of 75-125%.

Duplicate RPDs: One sample duplicate was analyzed. The RPD was not provided, but calculated for the provided black carbon content. The calculated RPD was 24.1%, within the QAPP precision goal of ±25%.

Black carbon data are acceptable for use as reported.

5.0 Grain Size Analyses

Quality control analysis frequencies: This batch included a laboratory triplicate, meeting frequency requirements.

Holding times: Sediment samples must be analyzed within 6 months of collection. Samples were analyzed within the holding time.

Laboratory triplicate RSDs: Triplicate RSDs were below 25%.

Grain size data are acceptable for use as reported.

6.0 Abbreviations and Definitions

DV Qualifier	Definition
U	The material was analyzed for, but was not detected above the level of the associated value.
UY	The reporting limit was elevated due to chromatographic overlap with related compounds. The material was analyzed for, but was not detected above the level of the associated value.
J	The analyte was positively identified. The associated numerical value is the approximate concentration of the analyte in the sample.
M,J	The analyte did not meet all identification criteria. The associated value should be considered an estimated maximum possible concentration.
N	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification.
UJ	The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.
R	The sample result is rejected. The presence or absence of the analyte cannot be verified and data are not usable.
R1	This sample result has been rejected in favor of a more accurate, precise or conservative result. The other result should be used.
R2	This sample result has been rejected in favor of a more accurate, precise or conservative result from another analytical method. The other result should be used.

Abbreviation	Definition
DV	Data validation
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
EDL	Estimated detection limit
EMPC	Estimated maximum possible concentration
IDS	Isotope dilution standard
MS	Matrix spike
MSD	Matrix spike duplicate
NA	Not Applicable
OPR	Ongoing Precision and Recovery
RL	Reporting limit
RPD	Relative percent difference
RRM	Regional reference material
RSD	Relative standard deviations

Abbreviation	Definition
SRM	Standard reference material

7.0 References

National Functional Guidelines For Inorganic Superfund Data Review, Office of Superfund Remediation and Technology Innovation, U.S. Environmental Protection Agency, August 2014, USEPA-540-R-13-001.

National Functional Guidelines for High Resolution Superfund Methods Data Review, Office of Superfund Remediation and Technology Innovation, U.S. Environmental Protection Agency, April 2016, EPA-542-B-16-001.

Method 1668C: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS, US Environmental Protection Agency, Office of Water Engineering and Analysis Division, April 2010.

Quality Assurance Project Plan Enhanced Natural Recovery/Activated Carbon Pilot Study, Lower Duwamish Waterway. Prepared by AMEC Foster Wheeler Environment & Infrastructure Inc., et al. Prepared for USEPA Region 10 and WA-DOE Northwest Regional Office, February 22, 2016.

Table H1
Data Qualifier Definitions

Qualifier	Definition	Description
C	Co-eluting congener	Concentration represents total concentration of all congeners that coelute with qualified congener.
CXXX	Co-elutes with the indicated congener	Analyte coelutes with another congener, see numbered congener for concentration.
J	Estimated	Analyte was detected at a level below the instrument quantitation limit. Concentration is considered estimated.
U	Non-detect	Analyte was not detected, concentration is the estimated detection limit.
L	Percent to steady state less than 20%	Extent of PCB equilibration between porewater and SPME sampler is less than 20%. The reported C_{free} value has higher uncertainty due to the larger value used to estimate a steady state concentration in the passive sampler.
R	Rejected	The sample result is rejected. The presence or absence of the analyte cannot be verified and data are not usable.
UB	Background concentration exceeds detected concentration	The background concentration of PCBs (not PRCs) that were detected in trip blanks exceeded the detected concentration and no PCB free concentration was reported. These results should be considered not detected at the lowest available detection limit, the MDL.

Notes:

Qualifiers listed include both laboratory and data validation qualifiers.

Abbreviations:

C_{free} = freely dissolved concentrations
MDL = Method detection limit
PCB = Polychlorinated biphenyl
PRC = Performance recovery compound
SPME = Solid-phase micro extraction