Appendix D. Data Validation Report

APPENDIX D

DATA VALIDATION REPORT

BLAKELY HARBOR PARK SEDIMENT INVESTIGATION

INTRODUCTION

A comprehensive quality assurance/quality control (QA/QC) program was followed by Leidos and NewFields during the Blakely Harbor Park Sediment Investigation to ensure that analytical results and the decisions based on these results are representative of the environmental conditions.

LABORATORY QUALITY CONTROL ASSESSMENT

The analytical data generated during the investigation underwent a quality assurance review and data validation. Validation conducted by Leidos included a minimum of Stage 2b validation for all SMS chemical data and Stage 4 validation for the dioxin/furan congener data. Validation was conducted using most recent EPA (USEPA 2005, 2008, 2009, and 2010), and PSEP (PTI 1989) guidelines. In addition, data were verified based on guidelines and specifications in the Blakely Harbor Park Sediment Investigation SAP/QAPP (Leidos and NewFields 2019). All samples were submitted to Eurofins TestAmerica, Seattle, and results were submitted in three sample delivery groups (SDGs): 280-125895-1, 580-87377-1, and 580-87377-2.

DATA VALIDATON SUMMARY

The following summary provides the information necessary to determine data usability for decision-making. The data were verified and validated and the qualifiers described below were applied to data as appropriate. Results that were rejected (R) were not used for decision-making. Results that were qualified as non-detect (U) or estimated (J or UJ) for various reasons during the validation process encountered minor analytical problems, but are considered fully usable for decision-making. The validation summary presented below is limited to QA/QC outliers that resulted in qualification of the data during the validation process. If outliers did not result in qualification of the data (e.g. high recoveries with non-detect values require no data validation qualifiers), then they are not discussed as these instances did not impact data usability.

The following data validation qualifiers were applied to the results as dictated by QC outliers:

- *U* — The analyte was analyzed for but was not detected above the reported sample quantitation limit. These results are qualitatively acceptable.
- *J* — The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample. These results are qualitatively acceptable but considered as estimates.
- *UJ* — The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample. These results are qualitatively acceptable but considered as estimates.
- *R* — The sample results were rejected due to serious deficiencies in the ability to analyze the sample and meet QC criteria. The presence or absence of the analyte cannot be verified.

The following methods were used for sample analysis of the samples collected during the Blakely Harbor Park Sediment Investigation:

Analytical Name	Analytical Method
Total Solids	ASTM D2216
Total Volatile Solids	EPA 160.4
Ammonia Nitrogen	EPA 350.1
Grain Size	PSEP Plumb
Metals (As, Ad, Cr, Cu, Pb, Ni, Se, Ag, Zn)	SW 6020A/B
Metals (Hg)	SW 7470A/7471B
Total Organic Carbon	SW 9060
Sulfide	SW 9034
PCB Aroclors	SW 8082A
SVOCs	SW 8270D
Dioxins/Furans	EPA 1613B

Table 1 (provided at the end of this section) provides a summary of data that were qualified as a result of the validation and indicates the validation qualifiers, reason codes, and potential bias applied to the data. Reason code definitions are provided at the end of Table 1. The following items (as applicable) have been addressed during the validation review:

- Sample custody and sample receipt, integrity, and preservation
- Sample handling and preparation
- Sample holding times
- Sample dilutions
- Blanks
- Instrument performance checks
- Initial and continuing calibrations
- Initial and continuing calibration blanks
- MS/MSDs
- LCS and/or LCSDs
- Laboratory duplicates
- Serial dilutions
- Post-digestion spikes
- Surrogate and labeled compound recovery
- Internal standards

GENERAL DATA PACKAGE COMMENTS

The laboratory submitted all required deliverables. The laboratory followed adequate corrective action processes and all anomalies were discussed in the case narrative with a few minor exceptions. All samples were received in good condition, properly preserved, and at the appropriate temperature.

GENERAL DATA VALIDATION COMMENTS

All analytical data received from the laboratory underwent some sort of data verification and/or data validation. Total solids, total volatile solids, ammonia nitrogen, and grain size underwent EPA Stage 1 (EPA1) verification and validation. EPA1 requires all samples be checked for completeness of results and adherence to all sample receipt condition checks.

Metals, mercury, total organic carbon (TOC), sulfide, PCB Aroclors, and SVOCs underwent EPA Stage 2B (EPA2B) verification and validation. EPA2B requires all of EPA1 plus review of method QC summary forms plus instrument QC forms and preparation logs. Dioxins/furans underwent EPA Stage 4 (EPA4) verification and validation. EPA4 requires all of EPA1, EPA2B, and review of the raw data

necessary to recalculate sample results (including recalculations of a subset of sample results), standard traceability logs, and the review of data necessary to qualitatively evaluate the results (e.g., instrument chromatograms and spectra).

The following discussion only includes results that received data validation qualifiers and reason codes during the data validation and verification process. All items listed above were reviewed during the validation process; however, if one of the items listed above was found to be acceptable (e.g., initial calibration results), then it is not discussed below.

Total Solids, Total Volatile Solids, Ammonia Nitrogen and Grain Size

No data were qualified due to the data validation process. Overall completeness for total solids, total volatile solids, ammonia nitrogen, and grain size data was excellent at 100% and all data are considered fully usable for decision-making.

Semivolatile Organic Compounds (SVOCs) Data Usability Assessment

Three SVOC results were qualified as estimated (J/UJ) because the continuing calibration verification (CCV) was below SW 8270D method acceptance limits as shown in Table 1 with reason code C05 L. Two SVOC results were qualified as non-detect (U) due to laboratory method blank contamination as shown in Table 1 with reason code F01. Nine SVOC results were qualified as estimated (J) due to MS/MSD recoveries above the upper control limit (UCL) as shown in Table 1 with reason code H01 H. Seventeen SVOC results were qualified as estimated (J/UJ) due to MS/MSD recoveries below the lower control limit (LCL) as shown in Table 1 with reason code H02 L. Two SVOC results were qualified as rejected (R) due to MS/MSD recoveries below 10% for benzyl alcohol in samples BH2-02-S and BH2-34-S as shown in Table 1 with reason code H03 L. Seven SVOC results were qualified as estimated (J) due to MS/MSD relative percent differences (RPDs) that exceeded the allowable limit as shown in Table 1 with reason code H04. Six SVOC results were qualified as estimated (J/UJ) due to LCS recoveries above the UCL as shown in Table 1 with reason code P01 H. Twenty-one SVOC results were qualified as estimated (UJ) due to LCS recoveries below the LCL as shown in Table 1 with reason code P02 L.

All SVOC sample extracts required dilutions ranging from 10X, 20X, and 50X prior to analysis due to sample matrix interferences or the high level of target and non-target compounds in the sample. The chromatograms were examined to determine if the samples warranted the dilutions performed by the laboratory. It was observed that a type of background interference was introducing a large number of peaks, resembling a “humpogram” which is often associated with some form of waste oil. Thus, it was determined that the dilutions were necessary. Even some samples with relatively low TOC results were found to have matrix interference that could possibly be due to oil or wood waste.

Results that were qualified as estimated (J/UJ) for various reasons encountered minor analytical problems, but are considered fully usable for decision-making. Results qualified as rejected (R) encountered serious analytical deficiencies and are not considered usable for decision-making. The overall completeness for SVOC data was excellent at 99.9%, and exceeded the completeness goal of 95%.

Polychlorinated Biphenyl (PCB) Aroclor Data Usability Assessment

Ten PCB results were qualified as estimated (J/UJ) due to CCV recoveries below the LCL as shown in Table 1 with reason code G02 L. Seven PCB results were qualifies as estimated (UJ) due to surrogate recoveries below the LCL as shown in Table 1 with reason code G02 L. Four PCB results were qualified as estimated (J) due to internal standard area counts below the LCL as shown in Table 1 with reason code K01 L. Four PCB results were qualified as estimated (J) because the RPD values between the two PCB columns exceeded 40%. All PCB data are considered fully usable for decision-making. The overall completeness for the PCB data was excellent at 100%.

Dioxin/Furan Data Usability Assessment

Twenty low-level dioxin/furan results were qualified as non-detect (U) due to laboratory method blank contamination as shown in Table 1 with reason code F01. All dioxin/furan data were considered usable for decision-making and the overall completeness was excellent at 100%.

Metals and Mercury Data Usability Assessment

Seven chromium results were qualified as estimated (J) due to CCV recoveries below the LCL as shown in Table 1 with reason code D05 L. One mercury result was qualified as estimated (J) due to MS/MSD recoveries above the UCL as shown in Table 1 with reason code H01 H. All metals and mercury data are considered fully usable for decision-making. The overall completeness for metals and mercury data was excellent at 100%.

Total Organic Carbon (TOC) Data Usability Assessment

No TOC data were qualified during the data validation process. All TOC data are considered fully usable for decision-making and the completeness was excellent at 100%.

Sulfide Data Usability Assessment

Two sulfide results were qualified as estimated (J/UJ) due to MS/MSD recoveries below the LCL as shown in Table 1 with reason code H02 L. These estimated data points are considered fully usable for decision-making. Overall completeness for sulfide data was excellent at 100%.

Table 1 – Blakely Harbor Qualified Data

Sample ID	Analytical Parameter	Result	Result Units	Analytical Method	Data Validation Qualifier	Data Validation Reason Code
BH2-06-S	Chromium	28	mg/Kg	SW6020A	J	D05 L
BH2-09-S	PCB-Aroclor 1254	0.0078	mg/Kg	SW8082A	J	K01 L
BH2-20-S	PCB-Aroclor 1254	0.0085	mg/Kg	SW8082A	J	M08
BH2-21-S	PCB-Aroclor 1254	0.0027	mg/Kg	SW8082A	J	M08
BH2-22-S	PCB-Aroclor 1254	0.0024	mg/Kg	SW8082A	J	M08
BH2-17-S	PCB-Aroclor 1254	0.0062	mg/Kg	SW8082A	J	C05 L
BH2-14-S	Carbazole	46	ug/Kg	SW8270D	J	P01 H
BH2-14-S	PCB-Aroclor 1254	0.0034	mg/Kg	SW8082A	J	C05 L
BH2-07-S	PCB-Aroclor 1254	0.0019	mg/Kg	SW8082A	UJ	C05 L
BH2-03-S	PCB-Aroclor 1254	0.0021	mg/Kg	SW8082A	UJ	C05 L
BH2-03-S	Chromium	27	mg/Kg	SW6020A	J	D05 L
BH2-02-S	1,2,4-Trichlorobenzene	13	ug/Kg	SW8270D	UJ	H02 L
BH2-02-S	1,2-Dichlorobenzene	26	ug/Kg	SW8270D	UJ	H02 L
BH2-02-S	1,4-Dichlorobenzene	18	ug/Kg	SW8270D	UJ	H02 L
BH2-02-S	2-Methylnaphthalene	20	ug/Kg	SW8270D	J	H02 L
BH2-02-S	m,p-Cresol (2:1 ratio)	400	ug/Kg	SW8270D	J	H02 L
BH2-02-S	Anthracene	51	ug/Kg	SW8270D	J	H01 H
BH2-02-S	Benz[a]anthracene	130	ug/Kg	SW8270D	J	H01 H, H04 H
BH2-02-S	Benzo(a)pyrene	100	ug/Kg	SW8270D	J	H01 H, H04 H
BH2-02-S	Benzo(ghi)perylene	73	ug/Kg	SW8270D	J	H01 H, H04 H
BH2-02-S	Benzofluoranthene	150	ug/Kg	SW8270D	J	H01 H, H04 H
BH2-02-S	Benzyl Alcohol	170	ug/Kg	SW8270D	R	H03 L
BH2-02-S	Chrysene	120	ug/Kg	SW8270D	J	H02 L, H04 H
BH2-02-S	Dibenzo(a,h)anthracene	26	ug/Kg	SW8270D	J	H04 H
BH2-02-S	Fluoranthene	310	ug/Kg	SW8270D	J	H01 H, H04 H
BH2-02-S	Hexachlorobutadiene	33	ug/Kg	SW8270D	UJ	H02 L
BH2-02-S	Indeno(1,2,3-cd)pyrene	84	ug/Kg	SW8270D	J	H01 H
BH2-02-S	Naphthalene	180	ug/Kg	SW8270D	J	H02 L
BH2-02-S	Phenanthrene	190	ug/Kg	SW8270D	J	H01 H
BH2-02-S	Pyrene	330	ug/Kg	SW8270D	J	H01 H
BH2-02-S	PCB-Aroclor 1254	0.0041	mg/Kg	SW8082A	J	K01 L, C05 L
BH2-01-S	PCB-Aroclor 1254	0.0012	mg/Kg	SW8082A	UJ	C05 L
BH2-01-S	Chromium	19	mg/Kg	SW6020A	J	D05 L

Sample ID	Analytical Parameter	Result	Result Units	Analytical Method	Data Validation Qualifier	Data Validation Reason Code
BH2-05-S	PCB-Aroclor 1254	0.0032	mg/Kg	SW8082A	J	K01 L, C05 L
BH2-05-S	Chromium	20	mg/Kg	SW6020A	J	D05 L
BH2-04-S	PCB-Aroclor 1254	0.0016	mg/Kg	SW8082A	J	K01 L, C05 L
BH2-04-S	Chromium	20	mg/Kg	SW6020A	J	D05 L
BH2-04-D	PCB-Aroclor 1254	0.0017	mg/Kg	SW8082A	UJ	C05 L
BH2-04-D	Chromium	22	mg/Kg	SW6020A	J	D05 L
BH2-10-S	PCB-Aroclor 1254	0.0010	mg/Kg	SW8082A	UJ	C05 L
BH2-10-S	Chromium	8.9	mg/Kg	SW6020A	J	D05 L
BH2-11-S	2,4-Dimethylphenol	86	ug/Kg	SW8270D	UJ	P02 L
BH2-13-S	2,4-Dimethylphenol	87	ug/Kg	SW8270D	UJ	P02 L
BH2-15-S	2,4-Dimethylphenol	43	ug/Kg	SW8270D	UJ	P02 L
BH2-15-S	PCB-Aroclor 1254	0.0010	mg/Kg	SW8082A	J	M08
BH2-12-S	2,4-Dimethylphenol	79	ug/Kg	SW8270D	UJ	P02 L
BH2-40-S	2,4-Dimethylphenol	20	ug/Kg	SW8270D	UJ	P02 L
BH2-39-S	2,4-Dimethylphenol	40	ug/Kg	SW8270D	UJ	P02 L
BH2-38-S	2,4-Dimethylphenol	22	ug/Kg	SW8270D	UJ	P02 L
BH2-37-S	2,4-Dimethylphenol	91	ug/Kg	SW8270D	UJ	P02 L
BH2-36-S	2,4-Dimethylphenol	20	ug/Kg	SW8270D	UJ	P02 L
BH2-35-S	2,4-Dimethylphenol	20	ug/Kg	SW8270D	UJ	P02 L
BH2-34-S	1,2,4-Trichlorobenzene	7.8	ug/Kg	SW8270D	UJ	H02 L
BH2-34-S	1,2-Dichlorobenzene	16	ug/Kg	SW8270D	UJ	H02 L
BH2-34-S	1,4-Dichlorobenzene	11	ug/Kg	SW8270D	UJ	H02 L
BH2-34-S	2,4-Dimethylphenol	20	ug/Kg	SW8270D	UJ	P02 L
BH2-34-S	Benzofluoranthene	110	ug/Kg	SW8270D	J	H02 L
BH2-34-S	Benzyl Alcohol	100	ug/Kg	SW8270D	R	H03 L
BH2-34-S	Chrysene	110	ug/Kg	SW8270D	J	H02 L
BH2-34-S	Fluoranthene	170	ug/Kg	SW8270D	J	H02 L
BH2-34-S	Hexachlorobutadiene	20	ug/Kg	SW8270D	UJ	H02 L
BH2-34-S	Naphthalene	15	ug/Kg	SW8270D	J	H02 L
BH2-34-S	Pyrene	190	ug/Kg	SW8270D	J	H02 L
BH2-34-S	Mercury	0.075	mg/Kg	SW7471A	J	H01 H
BH2-33-S	2,4-Dimethylphenol	21	ug/Kg	SW8270D	UJ	P02 L
BH2-32-S	2,4-Dimethylphenol	110	ug/Kg	SW8270D	UJ	P02 L
BH2-32-S	Carbazole	1000	ug/Kg	SW8270D	J	P01 H
BH2-31-S	2,4-Dimethylphenol	39	ug/Kg	SW8270D	UJ	P02 L

Sample ID	Analytical Parameter	Result	Result Units	Analytical Method	Data Validation Qualifier	Data Validation Reason Code
BH2-30-S	2,4-Dimethylphenol	21	ug/Kg	SW8270D	UJ	P02 L
BH2-29-S	2,4-Dimethylphenol	22	ug/Kg	SW8270D	UJ	P02 L
BH2-26-S	2,4-Dimethylphenol	61	ug/Kg	SW8270D	UJ	P02 L
BH2-25-S	2,4-Dimethylphenol	60	ug/Kg	SW8270D	UJ	P02 L
BH2-25-D	2,4-Dimethylphenol	64	ug/Kg	SW8270D	UJ	P02 L
BH2-24-S	2,4-Dimethylphenol	25	ug/Kg	SW8270D	UJ	P02 L
BH2-24-S	PCB-Aroclor 1016	0.0011	mg/Kg	SW8082A	UJ	G02 L
BH2-24-S	PCB-Aroclor 1221	0.0014	mg/Kg	SW8082A	UJ	G02 L
BH2-24-S	PCB-Aroclor 1232	0.0014	mg/Kg	SW8082A	UJ	G02 L
BH2-24-S	PCB-Aroclor 1242	0.00075	mg/Kg	SW8082A	UJ	G02 L
BH2-24-S	PCB-Aroclor 1248	0.00055	mg/Kg	SW8082A	UJ	G02 L
BH2-24-S	PCB-Aroclor 1254	0.0012	mg/Kg	SW8082A	UJ	G02 L
BH2-24-S	PCB-Aroclor 1260	0.0012	mg/Kg	SW8082A	UJ	G02 L
BH2-16-S	2,4-Dimethylphenol	96	ug/Kg	SW8270D	UJ	P02 L
BH2-15-ER	Benzyl Alcohol	2.6	ug/L	SW8270D	UJ	P01 H, F01
BH2-15-ER	Benzoic Acid	1.5	ug/L	SW8270D	J	C05 L, P08
BH2-15-ER	Di(2-ethylhexyl) phthalate	12	ug/L	SW8270D	J	P01 H
BH2-16-ER	Benzoic Acid	0.81	ug/L	SW8270D	UJ	C05 L
BH2-16-ER	Di(2-ethylhexyl) phthalate	10	ug/L	SW8270D	J	P01 H
BH2-16-RB	Benzyl Alcohol	0.87	ug/L	SW8270D	UJ	P01 H, F01
BH2-16-RB	Benzoic Acid	0.81	ug/L	SW8270D	UJ	C05 L
BH2-06-S	Sulfide	39	mg/Kg	SW9034	J	H02 L
BH2-2-S	Sulfide	9.5	mg/Kg	SW9034	UJ	H02 L
BH2-06-S	1,2,3,7,8,9-HxCDF	0.58	pg/g	EPA1613B	U	F01
BH2-09-S	1,2,3,7,8,9-HxCDF	0.41	pg/g	EPA1613B	U	F01
BH2-09-S	OCDF	12	pg/g	EPA1613B	U	F01
BH2-08-S	1,2,3,7,8,9-HxCDF	0.26	pg/g	EPA1613B	U	F01
BH2-08-S	1,2,3,4,7,8,9-HpCDF	0.37	pg/g	EPA1613B	U	F01
BH2-21-S	1,2,3,7,8,9-HxCDF	0.40	pg/g	EPA1613B	U	F01
BH2-18-S	1,2,3,7,8,9-HxCDF	0.44	pg/g	EPA1613B	U	F01
BH2-18-S	OCDF	12	pg/g	EPA1613B	U	F01
BH2-18-D	1,2,3,7,8,9-HxCDF	0.38	pg/g	EPA1613B	U	F01
BH2-18-D	OCDF	11	pg/g	EPA1613B	U	F01
BH2-03-S	1,2,3,7,8,9-HxCDF	0.63	pg/g	EPA1613B	U	F01
BH2-39-S	1,2,3,4,7,8-HxCDD	0.55	pg/g	EPA1613B	U	F01

Sample ID	Analytical Parameter	Result	Result Units	Analytical Method	Data Validation Qualifier	Data Validation Reason Code
BH2-39-S	1,2,3,7,8,9-HxCDF	0.33	pg/g	EPA1613B	U	F01
BH2-39-S	1,2,3,4,7,8,9-HpCDF	0.19	pg/g	EPA1613B	U	F01
BH2-39-S	OCDF	6.0	pg/g	EPA1613B	U	F01
BH2-37-S	1,2,3,7,8,9-HxCDF	0.57	pg/g	EPA1613B	U	F01
BH2-31-S	1,2,3,4,7,8-HxCDD	0.55	pg/g	EPA1613B	U	F01
BH2-31-S	1,2,3,7,8,9-HxCDF	0.22	pg/g	EPA1613B	U	F01
BH2-31-S	1,2,3,4,7,8,9-HpCDF	0.24	pg/g	EPA1613B	U	F01
BH2-31-S	OCDF	5.9	pg/g	EPA1613B	U	F01

Bias Code Definitions

Bias Codes	Definition
H	Bias in the sample result is believed to be high.
L	Bias in the sample result is believed to be low.

Reason Code Definitions

Reason Codes	Definition
C05	Continuing calibration % difference was not acceptable (Organics).
D05	CCV recovery was below the lower control limit (Inorganics).
F01	Sample data were qualified as a result of the method blank.
G02	Surrogate chemical recovery was below the lower control limit.
H01	MS/MSD recovery was above the upper control limit.
H02	MS/MSD recovery was below the lower control limit.
H03	MS/MSD recovery was <10%.
H04	MS/MSD pairs exceed the RPD limit.
K01	Internal Standard Area counts were outside the control limits.
M08	The RPD between the two pesticide/PCB column checks was >40%.
P01	LCS recovery was above the upper control limit.
P02	LCS recovery was below the lower control limit.
P08	Professional judgment was used to qualify the data due to high LCS/LCSD RPD value.