

Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling Portland Harbor Superfund Site Surface Sediment – Stratified Random		
Laboratory:	TestAmerica Laboratories, Incorporated, Seattle, WA		
Laboratory Group:	580-78380-1		
Analyses/Method:	Tributyltin, Polycyclic Aromatic Hydrocarbons (PAHs), bis(2-Ethylhexyl)phthalate, and Total Solids		
Validation Level:	Stage 4		
AECOM Project Number:	60566335, Task #2.12		
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Reviewed by:	Jennifer Garner/AECOM	File Name:	580-78380-1 DVR

SUMMARY

The data quality review of 25 surface sediment samples collected between June 13 and June 25, 2018, has been completed. Samples were analyzed for tributyltin by Krone et al., PAHs by EPA Method 8270D modified by selected ion monitoring (SIM), bis(2-ethylhexyl)phthalate by EPA Method 8270D, and total solids by American Society for Testing and Materials (ASTM) Method D-2216 by TestAmerica Laboratories, Incorporated (TA) located in Tacoma, Washington. The analyses were performed in general accordance with the methods specified in EPA's *Test Methods for Evaluating Solid Waste (SW-846)*, *Annual Book of ASTM Standards*, American Society for Testing & Materials (ASTM), Philadelphia, Pennsylvania, and Krone CA et al., *A Method for Analysis of Butyltin Species and Measurement of Butyltins in Sediment and English Sole Livers from Puget Sound*, Marine Environmental Research, 1989. The laboratory provided level 2 and level 4 data packages containing sample results, and associated quality assurance (QA) and quality control (QC) data, preparation logs, and raw instrument outputs (where applicable). The following samples are associated with laboratory group 580-78380-1:

Sample ID	Laboratory ID
PDI-SG-B102-BL1	580-78380-1
PDI-SG-B304-BL1	580-78380-2
PDI-SG-B289-BL1	580-78380-3
PDI-SG-B117-BL1	580-78380-4
PDI-SG-B301-BL1	580-78380-5
PDI-SG-B297-BL1	580-78380-6
PDI-SG-B293-BL1	580-78380-7
PDI-SG-B310-BL1	580-78380-8
PDI-SG-B309-BL1	580-78380-9
PDI-SG-B314-BL1	580-78380-10
PDI-SG-B031-BL1	580-78380-12
PDI-SG-B042-BL1	580-78380-13
PDI-SG-B191-BL1	580-78380-14
PDI-SG-B114-BL1	580-78380-15
PDI-SG-B425-BL1	580-78380-16
PDI-SG-B096-BL1	580-78380-17



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Sample ID	Laboratory ID
PDI-SG-B096-BL1-D (field duplicate of PDI-SG-B096-BL1)	580-78380-18
PDI-SG-B155-BL1	580-78380-19
PDI-SG-B259-BL1	580-78380-20
PDI-SG-B274-BL1	580-78380-21
PDI-SG-B285-BL1	580-78380-22
PDI-SG-B272-BL1	580-78380-23
PDI-SG-B281-BL1	580-78380-24
PDI-SG-B278-BL1	580-78380-25
PDI-SG-B264-BL1	580-78380-26

Data validation is based on method performance criteria and QC criteria documented in the *Quality Assurance Project Plan (QAPP)*, dated March 23, 2018, as amended. If data qualification was required, data were qualified based on the definitions and use of qualifying flags outlined in the EPA documents *USEPA National Functional Guidelines for Organic Superfund Methods Data Review*, January 2017, and *USEPA National Functional Guidelines for Inorganic Superfund Methods Data Review*, January 2017. Data qualifiers assigned to results reported in this sample set are included in Table 1.

SAMPLE RECEIPT

Upon receipt by TA, the sample jar information was compared to the associated chain-of-custody (COC) and the cooler temperatures were recorded. The coolers were received at temperatures below the EPA-recommended limits of greater than 0°C and less than or equal to 6°C, at -2.1°C and -1.7°C. Data were not qualified based on low cooler temperatures. All of the samples in this laboratory group were frozen after collection until they were shipped to TA on June 27, 2018.

Sample PDI-SG-B030-BL1 was originally logged with this laboratory group as 580-78380-11, but the sample container was broken in the laboratory and insufficient volume was available for analysis. At the request of AECOM, this sample was removed from this laboratory group. Non-frozen sample volume was available and AECOM authorized TA to analyze the sample for PAHs, bis(2-ethylhexyl)phthalate, and tributyltin past the holding times indicated in the QAPP. This sample was reported with TA laboratory group 580-78153-1 and is discussed under separate cover.

Sample PDI-SG-B194-BL1 was originally included on the COC for this laboratory group, but at the request of AECOM, this sample was removed from the COC and never logged with this laboratory group. This sample was reported with TA laboratory group 580-78433-1 and is discussed under separate cover.

There was insufficient volume for the laboratory to perform total solids analysis for sample PDI-SG-B102-BL1. The sample container was broken and most of the sediment was not recoverable; however, this sample was previously submitted under a different laboratory group (580-78107-1) and total solids analysis was performed and reported. At the request of AECOM, the total solids results for PDI-SG-B102-BL1 reported in TA laboratory group 580-78107-1 were used.

ORGANIC ANALYSES

Samples were analyzed for tributyltin, PAHs, and bis(2-ethylhexyl)phthalate by the methods identified in the introduction to this report.

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1. Holding Times – Acceptable

General – Samples PDI-SG-B102-BL1, PDI-SG-B304-BL1, PDI-SG-B289-BL1, and PDI-SG-B117-BL1 were extracted between 1 and 2 days past the method-recommended holding time of 14 days for PAHs and bis(2-ethylhexyl)phthalate. The samples were frozen after sample collection and before shipment to TA and the samples were not thawed for more than 14 days; therefore, no data were qualified based on holding time exceedance.

2. Initial and Continuing Calibration Verifications – Acceptable except as noted below:

PAHs by Method 8270D-SIM – The percent difference (%D) for benzo(a)anthracene (28.9%) was outside the control limit of $\pm 20\%$ in the continuing calibration verification (CCV) associated with analytical batch 277979. Benzo(a)anthracene was detected in PDI-SG-B274-BL1, PDI-SG-B285-BL1, PDI-SG-B272-BL1, PDI-SG-B281-BL1, PDI-SG-B278-BL1, and PDI-SG-B264-BL1; therefore, TA re-analyzed these samples and reported the results in analytical group 278594. The %D for benzo(a)anthracene in the CCV associated with analytical group 278594 was acceptable; therefore, data were not qualified based on the elevated %D in the CCV associated with analytical group 277979.

3. Blanks – Acceptable except as noted below:

General – Three rinsate blanks were collected on April 16, April 21, and April 30, 2018, were reported with ALS laboratory groups K1805746 (ID K1805746-010), K1805921 (ID K1805921-004), and TA laboratory group 580-78527 (ID 580-78527-26), and are applicable to the samples collected in this laboratory group. For detailed descriptions of these rinsate blank results, see the associated data validation memoranda. Data were not qualified based on rinsate blank detections.

PAHs by Method 8270D-SIM – The following analytes were detected in the method blank extracted on June 29th, 2018, at concentrations between the method detection limits (MDLs) and the reporting limits:

Analyte	Result
2-Methylnaphthalene	0.328 ug/kg
Naphthalene	0.181 ug/kg
Phenanthrene	0.292 ug/kg

2-Methylnaphthalene, naphthalene, and phenanthrene were reported at concentrations significantly higher than the method blank concentrations in the associated samples; therefore, no data were qualified for these analytes based on these method blank results.

bis(2-Ethylhexyl)phthalate by Method 8270D – bis(2-ethylhexyl)Phthalate (6.92 ug/kg) was detected in the method blank extracted on July 3, 2018, at a concentration between the MDL and the reporting limit. The results for bis(2-ethylhexyl)phthalate were reported at concentrations significantly above the blank contamination in the associated samples; therefore, no data were qualified based on this method blank result.

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4. Surrogates – Acceptable except as noted below:

Tributyltin by Krone et al. – The percent recovery for tributyltin (0%) in the 50x dilution of PDI-SG-B259-BL1 was below the control limits of 10-113%. The dilution of PDI-SG-B259-BL1 was greater than 10x; therefore, data were not qualified based on this surrogate recovery.

PAHs by Method 8270D-SIM – The percent recovery for terphenyl-d14 (47%) in the 5x dilution of PDI-SG-B042-BL1 was below the control limits of 57-120%. The results for the PAHs reported from the 5x dilution in PDI-SG-B042-BL1 were qualified as estimated and flagged 'J' based on this surrogate recovery.

bis(2-Ethylhexyl)phthalate by Method 8270D-SIM – The percent recoveries for terphenyl-d14 in PDI-SG-B304-BL1 (176%) and PDI-SG-B310-BL1 (171%) were above the control limits of 58-120%. The result for bis(2-ethylhexyl)phthalate in PDI-SG-B310-BL1 was qualified as estimated and flagged 'J' based on this surrogate recovery. bis(2-Ethylhexyl)phthalate was not detected in PDI-SG-B304-BL1; therefore, data was not qualified based on this elevated surrogate recovery.

5. Internal Standards – Acceptable

6. Laboratory Control Sample/Laboratory Control Sample Duplicate – Acceptable

7. Matrix Spike/Matrix Spike Duplicate (MS/MSD) – Acceptable except as noted below:

Tributyltin by Krone et al. – An MS/MSD was performed using PDI-SG-B031-BL1. Tributyltin was not recovered in the MS and the relative percent difference (RPD) for the MS/MSD pair could not be calculated. The concentration for tributyltin in the MS was just above the MDL, but the concentration for tributyltin in the MSD was just below the MDL. Due to limitations with TA's laboratory information management system, the recovery for tributyltin in the MSD could not be calculated, and the result for tributyltin in PDI-SG-B031-BL1 was qualified as estimated and flagged 'UJ' based on the MSD recovery. TA has adjusted the tributyltin spiking concentrations to prevent this from happening in future MS/MSDs.

PAHs by Method 8270D-SIM – An MS/MSD was performed using PDI-SG-B031-BL1. The percent recoveries for naphthalene in the MS (68%) and MSD (67%), and benzo[g,h,i]perylene in the MS (62%) were below the control limits of 70-120% and 63-120%, respectively. The result for naphthalene in PDI-SG-B031-BL1 was qualified as estimated and flagged 'J' based on the MS/MSD results. The percent recovery in the MSD and the RPD for the MS/MSD pair were acceptable; therefore, benzo[g,h,i]perylene was not qualified based on this MS recovery.

bis(2-Ethylhexyl)phthalate by Method 8270D-SIM – An MS/MSD was performed using PDI-SG-B031-BL1. The percent recoveries for bis(2-ethylhexyl)phthalate in the MS (171%), MSD (134%), and the RPD for the MS/MSD pair (24%) exceeded the control limits of 59-123% and 13%, respectively. bis(2-Ethylhexyl)phthalate was not detected in PDI-SG-B031-BL1; therefore, data were not qualified based on the elevated MS/MSD results.

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A field duplicate was submitted for PDI-SG-B096-BL1 and identified as PDI-SG-B096-BL1-D. The RPDs for the following analytes were greater than 50% for the field duplicate pair:

Analyte	RPD
Acenaphthylene	75%
Benzo[a]pyrene	53%
Benzo[k]fluoranthene	56%
Naphthalene	68%
Phenanthrene	67%

The sample concentrations for acenaphthylene and benzo(k)fluoranthene were less than five times the reporting limits; therefore, the results for acenaphthylene and benzo(k)fluoranthene were not qualified based on the elevated field duplicate RPDs. The results for benzo[a]pyrene, naphthalene, and phenanthrene in PDI-SG-B096-BL1 and PDI-SG-B096-BL1-D were qualified as estimated and flagged 'J' based on the elevated field duplicate RPDs.

9. Calculation Checks – Acceptable

A calculation check was performed for sample results on one sample per calibration. The review confirmed the final results were correct as reported.

10. Reporting Limits and Chromatographic Review – Acceptable except as noted below:

General – Chromatograms/spectra were reviewed to confirm target analytes were properly identified. The review confirmed target analytes were properly identified and reported by the laboratory.

One or more results were flagged 'J' by the laboratory to indicate the reported concentrations were above the MDLs but below the reporting limits. Laboratory 'J'-flagged results are considered estimated. As the result is between the MDL and the reporting limit, there is a greater level of uncertainty associated with the numerical result.

PAHs by Method 8270D-SIM – All samples required dilution prior to analysis due to the nature of the sample matrix. The elevated reporting limits for PAHs reported as not detected did not exceed the cleanup levels.

bis(2-Ethylhexyl)phthalate by Method 8270D-SIM – All samples required dilution prior to analysis due to the nature of the sample matrix. The reporting limits for bis(2-ethylhexyl)phthalate reported as not detected in PDI-SG-B102-BL1, PDI-SG-B304-BL1, PDI-SG-B289-BL1, PDI-SG-B301-BL1, PDI-SG-B297-BL1, PDI-SG-B293-BL1, PDI-SG-B314-BL1, PDI-SG-B031-BL1, PDI-SG-B042-BL1, PDI-SG-B191-BL1, PDI-SG-B114-BL1, PDI-SG-B425-BL1, PDI-SG-B096-BL1, PDI-SG-B096-D-BL1, PDI-SG-B274-BL1, and PDI-SG-B285-BL1 exceeded the cleanup level. The MDLs did not exceed the cleanup level in the samples noted above.



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CONVENTIONAL ANALYSES

Sediment samples were analyzed for total solids by ASTM Method D-2216.

1. Holding Times – Acceptable except as noted below:

Total Solids by ASTM Method D-2216 – The 7-day holding time indicated for total solids in the QAPP was exceeded for the samples in this laboratory group as the samples were temporarily held in freezer storage before shipping to TA. Total solids were analyzed within the time that other analyses were performed for samples in this laboratory group. No data qualifiers were assigned based on the holding time exceedance.

2. Field Duplicate – Acceptable

A field duplicate was submitted for PDI-SG-B096-BL1 and identified as PDI-SG-B096-BL1-D. Results were comparable.

3. Laboratory Duplicate – Acceptable

Laboratory duplicates were performed using PDI-SG-B314-BL1 and PDI-SG-B155-BL1. Results were comparable.

4. Calculation Checks – Acceptable

A calculation check was performed for sample results on one sample per calibration. The review confirmed the final results were correct as reported.

5. Reporting Limits – Acceptable

OVERALL ASSESSMENT OF DATA

The data reported in this laboratory group is considered usable for meeting project objectives. The completeness for laboratory group 580-78380-1 is 100%.

Table 1
QA/QC Data Summary Review
Portland Harbor
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TestAmerica Laboratory Group: 580-78380-1

Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SG-B310-BL1	580-78380-8	SW8270D	bis(2-Ethylhexyl)phthalate	78	ug/kg	78 J	s
PDI-SG-B031-BL1	580-78380-12	SW8270DSIM	Naphthalene	27	ug/kg	27 J	m
PDI-SG-B031-BL1	580-78380-12	TA-MS-0346	Tributyltin	180 U	ug/kg	180 UJ	m
PDI-SG-B042-BL1	580-78380-13	SW8270DSIM	2-Methylnaphthalene	57	ug/kg	57 J	s
PDI-SG-B042-BL1	580-78380-13	SW8270DSIM	Acenaphthene	470	ug/kg	470 J	s
PDI-SG-B042-BL1	580-78380-13	SW8270DSIM	Acenaphthylene	250	ug/kg	250 J	s
PDI-SG-B042-BL1	580-78380-13	SW8270DSIM	Anthracene	300	ug/kg	300 J	s
PDI-SG-B042-BL1	580-78380-13	SW8270DSIM	Fluoranthene	3300	ug/kg	3300 J	s
PDI-SG-B042-BL1	580-78380-13	SW8270DSIM	Fluorene	250	ug/kg	250 J	s
PDI-SG-B042-BL1	580-78380-13	SW8270DSIM	Naphthalene	160	ug/kg	160 J	s
PDI-SG-B042-BL1	580-78380-13	SW8270DSIM	Phenanthrene	2000	ug/kg	2000 J	s
PDI-SG-B042-BL1	580-78380-13	SW8270DSIM	Pyrene	4300	ug/kg	4300 J	s
PDI-SG-B096-BL1	580-78380-17	SW8270DSIM	Benzo(a)pyrene	110	ug/kg	110 J	fd
PDI-SG-B096-BL1	580-78380-17	SW8270DSIM	Naphthalene	65	ug/kg	65 J	fd
PDI-SG-B096-BL1	580-78380-17	SW8270DSIM	Phenanthrene	200	ug/kg	200 J	fd
PDI-SG-B096-BL1-D	580-78380-18	SW8270DSIM	Benzo(a)pyrene	64	ug/kg	64 J	fd
PDI-SG-B096-BL1-D	580-78380-18	SW8270DSIM	Naphthalene	32	ug/kg	32 J	fd
PDI-SG-B096-BL1-D	580-78380-18	SW8270DSIM	Phenanthrene	100	ug/kg	100 J	fd

fd - field duplicate RPD

J - estimated value

m - matrix spike recovery

RPD - relative percent difference

s - surrogate recovery

U - Compound was analyzed for, but not detected above the value shown.

ug/kg - microgram per kilogram