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DATA VALIDATION REPORT

Swan Island Lagoon Sediment Sampling Investigation, October 2018,
ALS Environmental Data

Prepared for:
Pacific Groundwater Group
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April 18, 2019

1.0 Introduction

Data validation was performed on the following sediment samples:

Sample ID	Sample Date/Time	Lab ID	Analyses
B5-0to26-100818	10/08/2018 15:07	K1809944-007	BEHP, TBT, PAH, OCP, TS
B3-0to21-100818	10/08/2018 12:16	K1809944-009	BEHP, TBT, PAH, OCP, TS
511-0to28-100918	10/09/2018 11:37	K1809944-010	BEHP, TBT, PAH, OCP, TS
D5-0to28-100918	10/09/2018 11:37	K1809944-011	BEHP, TBT, PAH, OCP, TS
D7-0to28-100918	10/09/2018 15:35	K1809944-018	BEHP, TBT, PAH, OCP, TS
B7-0to30-100918	10/09/2018 14:13	K1809944-019	BEHP, TBT, PAH, OCP, TS
J5-0to29-101318	10/13/2018 09:36	K1810083-001	BEHP, TBT, PAH, OCP, TS
J3-0to26-101318	10/13/2018 10:51	K1810083-002	BEHP, TBT, PAH, OCP, TS
513-0to26-101318	10/13/2018 13:13	K1810083-003	BEHP, TBT, PAH, OCP, TS
J1-0to20-101318	10/13/2018 17:11	K1810083-004	BEHP, TBT, PAH, OCP, TS
L3-0to20-101318	10/13/2018 14:33	K1810083-006	BEHP, TBT, PAH, OCP, TS
L7-0to29-101318	10/13/2018 11:36	K1810083-009	BEHP, TBT, PAH, OCP, TS
L5-0to26-101318	10/13/2018 13:13	K1810083-011	BEHP, TBT, PAH, OCP, TS
H1-0to24-101418	10/14/2018 09:09	K1810083-013	BEHP, TBT, PAH, OCP, TS
F1-0to23-101418	10/14/2018 11:08	K1810083-015	BEHP, TBT, PAH, OCP, TS
N7-0to27-101418	10/14/2018 15:10	K1810083-018	BEHP, TBT, PAH, OCP, TS
N5-0to28-101418	10/14/2018 16:46	K1810083-020	BEHP, TBT, PAH, OCP, TS
N4-0to29-101418	10/14/2018 17:23	K1810083-021	PAH, TS
B1-0to20-101418	10/14/2018 12:58	K1810083-022	BEHP, TBT, PAH, OCP, TS
D3-0to26-101018	10/10/2018 11:53	K1810088-005	BEHP, TBT, PAH, OCP, TS
F3-0to27-101118	10/11/2018 13:27	K1810088-012	BEHP, TBT, PAH, OCP, TS
F5-0to28-101118	10/11/2018 11:27	K1810088-014	BEHP, TBT, PAH, OCP, TS
F7-0to27-101118	10/11/2018 10:16	K1810088-016	BEHP, TBT, PAH, OCP, TS
H3-0to28-101118	10/11/2018 14:06	K1810088-017	BEHP, TBT, PAH, OCP, TS
H5-0to29-101118	10/11/2018 15:31	K1810088-019	BEHP, TBT, PAH, OCP, TS
512-0to28-101118	10/11/2018 11:27	K1810088-021	BEHP, TBT, PAH, OCP, TS
H7-0to24-101218	10/12/2018 15:15	K1810088-028	BEHP, TBT, PAH, OCP, TS
J7-0to26-101218	10/12/2018 15:52	K1810088-029	BEHP, TBT, PAH, OCP, TS
N3-0to26-101518	10/15/2018 08:46	K1810270-001	BEHP, TBT, PAH, OCP, TS
L1-0to30-101518	10/15/2018 10:10	K1810270-004	BEHP, TBT, PAH, OCP, TS
N1-0to30-101518	10/15/2018 11:40	K1810270-006	BEHP, TBT, PAH, OCP, TS
T1-0to30-101518	10/15/2018 15:35	K1810270-010	BEHP, TBT, PAH, OCP, TS

Sample ID	Sample Date/Time	Lab ID	Analyses
R1-0to30-101518	10/15/2018 14:37	K1810270-011	BEHP, TBT, PAH, OCP, TS
P1-0to30-101518	10/15/2018 13:50	K1810270-012	BEHP, TBT, PAH, OCP, TS
T3-0to28-101618	10/16/2018 16:30	K1810270-013	BEHP, TBT, PAH, OCP, TS
T7-0to31-101618	10/16/2018 14:00	K1810270-015	BEHP, TBT, PAH, OCP, TS
T5-0to26-101618	10/16/2018 15:13	K1810270-016	BEHP, TBT, PAH, OCP, TS
P3-0to29-101618	10/16/2018 10:50	K1810270-017	BEHP, TBT, PAH, OCP, TS
R5-0to25-101618	10/16/2018 12:09	K1810270-018	BEHP, TBT, PAH, OCP, TS
514-0to29-101618	10/16/2018 11:25	K1810270-019	BEHP, TBT, PAH, OCP, TS
P5-0to26-101618	10/16/2018 09:16	K1810270-020	BEHP, TBT, PAH, OCP, TS
R3-0to33-101718	10/17/2018 09:28	K1810270-029	BEHP, TBT, PAH, OCP, TS
K6-0to28-101918	10/19/2018 09:26	K1810302-001	TBT, TS
O4-0to28-101918	10/19/2018 13:48	K1810302-002	TS
M5-0to25-101918	10/19/2018 11:55	K1810302-003	TS
K4-0to28-101918	10/19/2018 08:11	K1810302-004	TS
O7-0to27-101918	10/19/2018 15:36	K1810302-005	TS
O5-0to27-101918	10/19/2018 14:23	K1810302-006	TS
K7-0to24-101918	10/19/2018 10:08	K1810302-007	TS
M6-0to26-101918	10/19/2018 11:18	K1810302-008	TS
M7-0to29-101918	10/19/2018 10:48	K1810302-009	TS
K5-0to27-101918	10/19/2018 08:50	K1810302-010	TS
515-0to26-101918	10/19/2018 13:11	K1810302-011	TS
O6-0to27-101918	10/19/2018 14:57	K1810302-012	TS
M4-0to26-101918	10/19/2018 13:11	K1810302-013	TS
A1-0to30-102018	10/20/2018 12:05	K1810302-016	BEHP, TBT, PAH, OCP, TS
D6-SC-64to88-102118	10/21/2018 17:40	K1810302-022	BEHP, TBT, PAH, OCP, TS
D6-SC-40to64-102118	10/21/2018 16:50	K1810302-023	BEHP, TBT, PAH, OCP, TS
H3-SC-99to114-102118	10/21/2018 14:30	K1810302-024	BEHP, TBT, PAH, OCP, TS
D6-SC-88to108-102118	10/21/2018 17:50	K1810302-025	BEHP, TBT, PAH, OCP, TS
H3-SC-46to63-102118	10/21/2018 12:50	K1810302-026	BEHP, TBT, PAH, OCP, TS
D6-SC-0to1-102118	10/21/2018 16:00	K1810302-027	BEHP, TBT, PAH, OCP, TS
H3-SC-1to2-102118	10/21/2018 11:55	K1810302-028	BEHP, TBT, PAH, OCP, TS
D6-SC-1to2-102118	10/21/2018 16:15	K1810302-029	BEHP, TBT, PAH, OCP, TS
H3-SC-29to46-102118	10/21/2018 12:40	K1810305-001	BEHP, TBT, PAH, OCP, TS
H3-SC-85to99-102118	10/21/2018 14:15	K1810305-002	BEHP, TBT, PAH, OCP, TS
R4-SC-138to142-102118	10/21/2018 20:55	K1810305-003	OCP, TS
H3-SC-0to1-102118	10/21/2018 11:45	K1810305-004	BEHP, TBT, PAH, OCP, TS
H3-SC-2to29-102118	10/21/2018 12:00	K1810305-005	BEHP, TBT, PAH, OCP, TS
H3-SC-63to85-102118	10/21/2018 13:50	K1810305-006	BEHP, TBT, PAH, OCP, TS
D6-SC-108to123-102118	10/21/2018 18:00	K1810305-007	BEHP, TBT, PAH, OCP, TS
D6-SC-2to4-102118	10/21/2018 16:30	K1810305-008	BEHP, TBT, PAH, OCP, TS
R4-SC-0to1-102118	10/21/2018 18:40	K1810305-009	BEHP, TBT, PAH, OCP, TS
R4-SC-1to2-102118	10/21/2018 18:50	K1810305-010	BEHP, TBT, PAH, OCP, TS
R4-SC-6to8-102118	10/21/2018 19:20	K1810305-011	BEHP, TBT, PAH, OCP, TS
R4-SC-2to4-102118	10/21/2018 19:00	K1810305-012	BEHP, TBT, PAH, OCP, TS
R4-SC-4to6-102118	10/21/2018 19:10	K1810305-013	BEHP, TBT, PAH, OCP, TS
R4-SC-8to10-102118	10/21/2018 19:40	K1810305-014	BEHP, TBT, PAH, OCP, TS
R4-SC-10to12-102118	10/21/2018 19:50	K1810305-015	BEHP, TBT, PAH, OCP, TS
R4-SC-120to138-102118	10/21/2018 20:45	K1810305-016	BEHP, TBT, PAH, OCP, TS
J3-SC-55to76-102218	10/22/2018 14:45	K1810417-001	BEHP, TBT, PAH, OCP, TS
J6-SC-00to10-102218	10/22/2018 18:00	K1810417-002	BEHP, TBT, PAH, OCP, TS
J3-SC-45to55-102218	10/22/2018 14:35	K1810417-003	BEHP, TBT, PAH, OCP, TS
J3-SC-36to45-102218	10/22/2018 14:25	K1810417-004	BEHP, TBT, PAH, OCP, TS
J3-SC-76to98-102218	10/22/2018 14:55	K1810417-005	BEHP, TBT, PAH, OCP, TS
J3-SC-20to36-102218	10/22/2018 13:00	K1810417-006	BEHP, TBT, PAH, OCP, TS
J6-SC-20to40-102218	10/22/2018 18:20	K1810417-007	BEHP, TBT, PAH, OCP, TS
J6-SC-10to20-102218	10/22/2018 18:10	K1810417-008	BEHP, TBT, PAH, OCP, TS
L3-SC-00to10-102218	10/22/2018 10:35	K1810417-009	BEHP, TBT, PAH, OCP, TS
J3-SC-00to10-102218	10/22/2018 12:40	K1810417-010	BEHP, TBT, PAH, OCP, TS

Sample ID	Sample Date/Time	Lab ID	Analyses
J5-SC-60to80-102218	10/22/2018 16:50	K1810417-011	BEHP, TBT, PAH, OCP, TS
J6-SC-80to96-102218	10/22/2018 19:30	K1810417-012	BEHP, TBT, PAH, OCP, TS
J5-SC-10to20-102218	10/22/2018 16:10	K1810417-013	BEHP, TBT, PAH, OCP, TS
J6-SC-111to121-102218	10/22/2018 19:50	K1810417-014	BEHP, TBT, PAH, OCP, TS
J6-SC-60to80-102218	10/22/2018 19:00	K1810417-016	BEHP, TBT, PAH, OCP, TS
J3-SC-10to20-102218	10/22/2018 12:50	K1810417-017	BEHP, TBT, PAH, OCP, TS
L3-SC-20to40-102218	10/22/2018 11:00	K1810417-018	BEHP, TBT, PAH, OCP, TS
L3-SC-10to20-102218	10/22/2018 10:45	K1810417-019	BEHP, TBT, PAH, OCP, TS
J6-SC-96to111-102218	10/22/2018 19:40	K1810417-020	BEHP, TBT, PAH, OCP, TS
J5-SC-40to60-102218	10/22/2018 16:40	K1810417-021	BEHP, TBT, PAH, OCP, TS
J6-SC-40to60-102218	10/22/2018 18:50	K1810417-022	BEHP, TBT, PAH, OCP, TS
J5-SC-20to40-102218	10/22/2018 16:30	K1810417-023	BEHP, TBT, PAH, OCP, TS
J5-SC-00to10-102218	10/22/2018 16:00	K1810417-024	BEHP, TBT, PAH, OCP, TS
J5-SC-80to95-102218	10/22/2018 17:10	K1810417-025	BEHP, TBT, PAH, OCP, TS
J5-SC-95to110-102218	10/22/2018 17:20	K1810417-026	BEHP, TBT, PAH, OCP, TS

Analytical methods: Table 4 of the QAPP specifies the following analytical methods:

Analysis	Analytical Method
Phthalate Analysis (BEHP)	SW8270D-LL
TributylTin Analysis (TBT)	ALS Butyltin SOP
Polycyclic Aromatic Hydrocarbons Analysis (PAH)	SW8270D-SIM
Organochlorine Pesticides Analysis (OCP)	EPA 1699 M
Total Solids Analysis (TS)	EPA 160.3

These methods were used with the following exception: The laboratory referenced an in-house laboratory SOP for organochlorine pesticides which utilizes GC/MS/MS with selective reaction monitoring (SRM).

Laboratory: Analyses were performed by ALS Environmental, in Kelso Washington. Additional analyses for these samples were performed by TestAmerica Laboratories, Inc., and are discussed in a separate validation report.

Validation: A stage 4 (full) validation was performed on the sample results from laboratory report # K181088. A stage 2A (summary) validation was performed on the remaining samples. Validation was performed by Cari Sayler. Data qualifiers are summarized in section 7.0 of this report.

Sample Receipt: Sample chain-of-custodies and sample log-in documentation were reviewed. Requested analyses, where indicated, were performed with the following exception: Data for sample N4-0to29-101418 (K1810083-021) did not include BEHP, TBT, or OCP Analyses. Additionally, the total solids result for this sample was included in the report, but not the electronic data deliverable (EDD).

Sample number transcription: Sample IDs in the EDD were compared to the chain-of-custody for each sample. Sample IDs matched the chain of custody with the following exceptions: The laboratory reported equipment blank 613-102218 with a sample ID of G13-102218 in the PAH analysis. Various sample IDs included an upper case “TO” rather than the lower case “to” listed on the chain of custody. The corrected sample IDs have been used throughout this report.

2.0 Phthalate Analysis

Quality control analysis frequencies: The QAPP specifies that the following quality control samples be analyzed: a method blank, laboratory control sample (LCS), and LCS duplicate (LCSD) in each analytical batch; and a MS/MSD pair at a frequency of one per twenty samples.

EPA method 8000 requires the following quality control samples: a method blank and LCS in each analytical batch; a MS and matrix duplicate (either MSD or laboratory duplicate) at suggested frequency of one per twenty samples. Additionally, appropriate surrogates must be analyzed in each field and QC sample.

Each sample batch included a method blank and LCS. Eight of the nine batches also included, an MS/MSD pair, meeting method 8000 requirements. The additional QAPP requirement for an LCSD per batch was not met. However, no qualifiers are assigned based on the absence of this additional QC information.

QAPP field QC requirements include one blind field duplicate per twenty surface sediment samples and one equipment rinsate blank per twenty field samples or per week. Three field duplicates and weekly equipment rinsate blanks were collected and analyzed, meeting frequency requirements.

Holding times: Refrigerated sediment samples must be extracted within 14 days of collection and frozen sediment samples must be extracted within 1 year. Extracts must be analyzed within 40 days of extraction. Holding times were met with the following exception:

Sample ID	Days from extraction to Analysis	Analysis holding time (days)
D6-SC-1to2-102118 RE	42	40

Positive and non-detect results in this sample are qualified as estimated.

Additionally, the cooler receipt temperature for laboratory report #K1809944, 11.5°C, exceeded the recommended range of 0-6°C. Results for samples in the above report are qualified as estimated.

Instrument performance check (tune) ion abundances: Instrument performance check data were evaluated for the full validation data package only. Ion abundance criteria exist for 12 ions in decafluorotriphenylphosphine (DFTPP). These criteria were met in each tune provided in the data package. However, the tunes associated with the LCS KQ1815027-03 were missing from the data package. While this introduces uncertainty in the LCS recoveries, that uncertainty was considered to not have impacted the associated sample results, and no qualifiers are assigned.

Instrument calibration: Instrument calibration data were evaluated for the full validation data package only. Method criteria for initial calibrations depend on the quantitation method selected. Average response factor quantitation requires a minimum of 5 standards with maximum RSDs of ±20%. Linear regression quantitation requires a minimum of 5 standards with a minimum R^2 of 0.990, and a % recovery within 50-150% for the low standard, and within 70-130% for the remaining individual standards. Quadratic regression quantitation requires a minimum of 6 standards with a minimum R^2 of 0.990. These criteria were met.

Method criteria for continuing calibrations include a maximum percent difference of ±20% and a frequency of every 12 hours. The method allows for sporadic exceedances between 20 and 40

percent difference, with sporadic defined as less than <1/5th of compounds. Additionally the internal standard responses and retention times must be within 50-200% and ±30 seconds respectively of the initial calibration midpoint. These criteria were met for continuing calibrations provided in the data package. However, the continuing calibrations associated with the LCS KQ1815027-03 were missing from the data package. While this introduces uncertainty in the LCS recoveries, that uncertainty was considered to not have impacted the associated sample results, and no qualifiers are assigned.

Finally, analytes in both initial and continuing calibrations must meet a relative response factor minimum of 0.01. This criterion was met.

Laboratory results: Criteria for blanks are that analyte concentrations must be below the RL, or below 10% of the lowest associated sample concentration. No contamination was detected in laboratory blanks.

Surrogate recoveries: Laboratory control limits were 30-102%. Recoveries were within these limits with the following exceptions:

Sample ID	Surrogate	% Recovery	Lab Control Limit
513-0to26-101318	Terphenyl-d14	118	30 - 102

The laboratory noted matrix interference in sample 513-0to26-101318, and no qualifiers are assigned.

LCS recoveries: Laboratory control limits were 39-113%. Recoveries were within these limits.

MS recoveries: Laboratory control limits were 23-123%. Recoveries were within these limits with the following exceptions:

QC ID	Analyte	% Recovery	Lab Control Limit
L5-0to26-101318MS	Bis(2-ethylhexyl) phthalate	-46	23 - 123
L5-0to26-101318MSD	Bis(2-ethylhexyl) phthalate	-3.8	23 - 123

The native concentration in L5-0to26-101318 exceeded the spike amount added by a factor of 2.4. Therefore, variability in the native concentration is likely affecting the % recoveries, and the parent sample result is qualified as estimated rather than rejected.

MS/MSD RPDs: Laboratory control limit was <40%. RPDs were within these limits.

Sample Internal standards: Sample internal standards were evaluated for the full validation data package only. Internal standard responses and retention times in each sample must be within 50-200% and ±30 seconds, respectively, of the continuing calibration standard. These criteria were met.

Compound Quantitation: Concentrations of bis(2-ethylhexyl)phthalate and terphenyl-d14 in the samples and QC samples from the full validation data package were recalculated to verify sample quantitations. No discrepancies were noted.

Compound identifications: Chromatograms and quantitation reports in the full validation data package were reviewed for accuracy of compound identification for bis(2-ethylhexl)phthalate. No issues were noted.

Laboratory narrative/flags: No qualifiers are assigned based on the laboratory narrative or flags.

Overall assessment: With minor exceptions, documentation was found to be clear and complete. No calculation, transcription, or identification errors were noted. Calibration data demonstrated acceptable instrument performance. Quality control data showed generally acceptable precision and accuracy. Data were qualified based on an exceeded holding time, high cooler receipt temperatures, and low spike recoveries.

3.0 Tributyltin Analysis

Quality control analysis frequencies: The QAPP specifies that the following quality control samples be analyzed: a method blank, laboratory control sample (LCS), and LCS duplicate (LCSD) in each analytical batch; a MS/MSD pair at a frequency of one per twenty samples; and a laboratory duplicate at a frequency of one per ten samples.

EPA method 8000 requires the following quality control samples: a method blank and LCS in each analytical batch; a MS and matrix duplicate (either MSD or laboratory duplicate) at suggested frequency of one per twenty samples. Additionally, appropriate surrogates must be analyzed in each field and QC sample.

Each sample batch included a method blank, LCS, and MS/MSD pair, meeting method requirements. The additional QAPP requirements of LCSD and laboratory duplicate were not met. However, no qualifiers are assigned based on the absence of this additional QC information.

QAPP field QC requirements include one blind field duplicate per twenty surface sediment samples and one equipment rinsate blank per twenty field samples or per week. Three field duplicates and weekly equipment rinsate blanks were collected, meeting QAPP requirements.

Holding times: Refrigerated sediment samples must be extracted within 14 days of collection and frozen sediment samples must be extracted within 1 year. Extracts must be analyzed within 40 days of extraction. The dates that samples were frozen and pulled from the freezer to defrost prior to extraction were not documented in the laboratory packages. Communications with the laboratory indicate that two samples exceeded the 14 day refrigeration extraction holding time as follows:

Sample ID	Sample Date	Date Frozen	Date Refrigerated	Date Extracted	Total Refrigeration Time (days)
N3-0to26-101518	10/15/18	10/22/18	11/05/18	11/19/18	21
A1-0to30-102018	10/20/18	10/26/18	11/05/18	11/19/18	20

Positive and non-detect results in these two samples are qualified as estimated.

Additionally, the cooler receipt temperature for laboratory report #K1809944, 11.5°C, exceeded the recommended range of 0-6°C. Positive and non-detect results for samples in this laboratory report are also qualified as estimated.

Instrument calibration: Instrument calibration data were evaluated for the full validation data package only. Laboratory reported criteria for calibrations included a maximum initial calibration RSD of 20% and a maximum continuing calibration verification % Drift of ±25%. These criteria

were met. Retention time criteria were not provided. However, retention times were extremely stable, and no further action was taken.

Laboratory blank results: Criteria for blanks are that analyte concentrations must be below the RL, or below 10% of the lowest associated sample concentration. No contamination was detected in laboratory blanks.

Surrogate recoveries: Laboratory control limits were 10-120%. Recoveries were within these limits.

LCS recoveries: Laboratory control limits were 10-122%. Recoveries were within these limits.

MS recoveries: Laboratory control limits were 10-115%. Recoveries were within these limits with the following exceptions:

QC ID	Analyte	% Recovery	Lab Control Limit
D5-0to28-100918MS	Tri-n-butyltin Cation	153	10 - 115
D5-0to28-100918MSD	Tri-n-butyltin Cation	142	10 - 115
L5-0to26-101318MS	Tri-n-butyltin Cation	168	10 - 115
K6-0to28-101918MS	Tri-n-butyltin Cation	233	10 - 115
L5-0to26-101318MSD	Tri-n-butyltin Cation	151	10 - 115
F5-0to28-101118MSD	Tri-n-butyltin Cation	138	10 - 115

The native concentration in D5-0to28-100918 exceeded the spike amount added in the MS and MSD and no qualifiers are assigned to this sample. Tributyltin Cation is qualified as estimated in the remaining parent samples.

MS/MSD RPDs: Laboratory control limit was <40%. RPDs were within this limit.

Dual column variability: Dual column data in the in the full validation package were reviewed, with RPDs ranged from 0 to 8, showing good agreement. Qualifiers for dual column variability in the summary validation packages are assigned based on the laboratory P qualifier as follows:

Sample ID	Analyte	Result (ug/kg)	Flag	MDL (ug/kg)	RL (ug/kg)
D6-SC-88to108-102118	Tri-n-butyltin Cation	0.59	JP	0.59	1.4
N1-0TO30-101518	Tri-n-butyltin Cation	0.64	JP	0.57	1.3

These results are qualified as estimated.

Compound quantitation: Concentrations of tributyltin cation and tri-n-propyltin in the samples and QC samples from the full validation data package were recalculated to verify sample quantitations. No discrepancies were noted in the detected concentrations. It was determined that the salt to cation conversion calculation was not performed on the reporting limits. The laboratory indicated that this policy allowed each of the four butyltins to have the same reporting limit. The reporting limits were slightly higher than necessary, and no action was required.

Compound identifications: Chromatograms and quantitation reports in the full validation data package were reviewed for accuracy of tri-n-butyltin cation identification. No issues were noted.

Laboratory narrative/flags: The laboratory assigned various P qualifiers, indicating high dual column RPDs. These results are qualified as estimated.

Overall assessment: Documentation was found to be clear and complete. No calculation, transcription, or identification errors were noted. Calibration data demonstrated acceptable instrument performance. Quality control data showed generally acceptable precision and accuracy. Data were qualified based on exceeded holding times, high cooler receipt temperatures, high spike recoveries, spike duplicate, field duplicate, and dual column RPDs.

4.0 Polycyclic Aromatic Hydrocarbon Analysis

Quality control analysis frequencies: The QAPP specifies that the following quality control samples be analyzed: a method blank, laboratory control sample (LCS), and LCS duplicate (LCSD) in each analytical batch; and a MS/MSD pair at a frequency of one per twenty samples.

EPA method 8000 requires the following quality control samples: a method blank and LCS in each analytical batch; a MS and matrix duplicate (either MSD or laboratory duplicate) at suggested frequency of one per twenty samples. Additionally, appropriate surrogates must be analyzed in each field and QC sample.

Each sample batch included a method blank and LCS. Eight of the nine batches also included, an MS/MSD pair, meeting method 8000 requirements. The additional QAPP requirement for an LCSD per batch was not met. However, no qualifiers are assigned based on the absence of this additional QC information.

QAPP field QC requirements include one blind field duplicate per twenty surface sediment samples and one equipment rinsate blank per twenty field samples or per week. Three field duplicates and weekly equipment rinsate blanks were collected, meeting QAPP requirements.

Holding times: Refrigerated sediment samples must be extracted within 14 days of collection and frozen sediment samples must be extracted within 1 year. Extracts must be analyzed within 40 days of extraction. These holding times were met. However, the cooler receipt temperature for laboratory report #K1809944, 11.5°C, exceeded the recommended range of 0-6°C. Results for samples in the above report are qualified as estimated.

Instrument performance check (tune) ion abundances: Instrument performance check data were evaluated for the full validation data package only. Ion abundance criteria exist for 12 ions in decafluorotriphenylphosphine. These criteria were met in each 12 hour standard.

Instrument calibration: Instrument calibration data were evaluated for the full validation data package only. Method criteria for initial calibrations depend on the quantitation method selected. Average response factor quantitation requires a minimum of 5 standards with maximum RSDs of ±20%. Linear regression quantitation requires a minimum of 5 standards with a minimum R^2 of 0.990, and a % recovery within 50-150% for the low standard, and within 70-130% for the remaining individual standards. Quadratic regression quantitation requires a minimum of 6 standards with a minimum R^2 of 0.990.

Method criteria for continuing calibrations include a maximum percent difference of ±20% and a frequency of every 12 hours. The method allows for sporadic exceedances between 20 and 40 percent difference, with sporadic defined as less than <1/5th of compounds. Additionally the internal standard responses and retention times must be within 50-200% and ±30 seconds respectively of the initial calibration midpoint.

Finally, common target analytes in both initial and continuing calibrations must meet method relative response factor minimums which range from 0.01 to 0.80. Remaining analytes must meet a minimum response factor of 0.01.

All of the above criteria were met in the full validation data package. However, the narrative from laboratory report K1810417 indicates that the pyrene result was outside the ±20% difference limit in the CCV as follows:

Standard ID	Analysis Date	Analyte	% Difference	Control Limit
\MS14\1112F002.D	11/12/2018	Pyrene	-25%	±20%

Positive and non-detect results for this analyte in the two samples associated with this CCV are qualified as estimated.

Laboratory blank results: Performance criteria for blanks are that analyte concentrations must be below the RL, or below 10% of the lowest associated sample concentration. These criteria were met. However, concentrations below the reporting limit may have an impact on low level sample results. Contamination was detected in laboratory blanks at levels below the reporting limit as follows:

Blank ID	Analyte	Concentration (ug/kg)	RL (ug/kg)
KWG1805263-4	Phenanthrene	0.038J	0.25
KWG1805529-4	Pyrene	0.038J	0.25
KWG1805529-4	Benzo(a)anthracene	0.065J	0.25
KWG1805529-4	Chrysene	0.046J	0.25
KWG1805529-4	Benzo(b)fluoranthene	0.073J	0.25
KWG1805529-4	Benzo(k)fluoranthene	0.053J	0.25
KWG1805529-4	Benzo(a)pyrene	0.036J	0.25
KWG1805529-4	Indeno(1,2,3-cd)pyrene	0.085J	0.25
KWG1805529-4	Benzo(g,h,i)perylene	0.098J	0.25
KWG1805479-4	Phenanthrene	0.045J	0.25
KWG1805479-4	Fluoranthene	0.036J	0.25
KWG1805479-4	Pyrene	0.033J	0.25
KWG1805479-4	Benzo(a)anthracene	0.036J	0.25
KWG1805597-4	Dibenzofuran	0.034J	0.25
KWG1805597-4	Fluorene	0.039J	0.25
KWG1805597-4	Phenanthrene	0.066J	0.25
KWG1805796-6	Benzo(a)anthracene	0.035J	0.25
KWG1805796-6	Benzo(a)anthracene	0.035J	0.25

Sample concentrations within 5 times these levels should be considered not detected, and are qualified U. Sample concentrations between 5 and 10 times these levels are qualified as estimated.

Surrogate recoveries: Laboratory control limits ranged from 23-110 to 27-115%. Recoveries were within these limits.

LCS recoveries: Laboratory control limits ranged from 14-125 to 65-97%. Recoveries were within these limits.

LCS/LCSD RPDs: Laboratory control limit was <30%. RPDs were within this limit.

MS recoveries: The laboratory-reported control limits were the method 8000 specified 70-130% default for all organic methods. However, semivolatile organics do not typically recover with this efficiency, and qualification was performed based on a more typical 30-120% recovery limit. Recoveries were within 30-120% with the following exceptions:

QC ID	Analyte	% Recovery	Adjusted Control Limit
D5-0TO28-100918MS	Chrysene	122	30 - 120
L5-0TO26-101318MS	Phenanthrene	-56	30 - 120
L5-0TO26-101318MS	Fluoranthene	-112	30 - 120
L5-0TO26-101318MS	Pyrene	-43	30 - 120
L5-0TO26-101318MS	Chrysene	22	30 - 120
L5-0TO26-101318MS	Benzo(b)fluoranthene	27	30 - 120
J5-SC-00TO10-102218MS	Phenanthrene	286	30 - 120
J5-SC-00TO10-102218MS	Fluoranthene	392	30 - 120
J5-SC-00TO10-102218MS	Pyrene	369	30 - 120
J5-SC-00TO10-102218MS	Benzo(a)anthracene	179	30 - 120
J5-SC-00TO10-102218MS	Chrysene	244	30 - 120
J5-SC-00TO10-102218MS	Benzo(b)fluoranthene	257	30 - 120
J5-SC-00TO10-102218MS	Benzo(k)fluoranthene	164	30 - 120
J5-SC-00TO10-102218MS	Benzo(a)pyrene	214	30 - 120
J5-SC-00TO10-102218MS	Indeno(1,2,3-cd)pyrene	192	30 - 120
J5-SC-00TO10-102218MS	Benzo(g,h,i)perylene	171	30 - 120
J3-SC-55TO76-102218MS	Fluoranthene	121	30 - 120
J3-SC-55TO76-102218MS	Pyrene	142	30 - 120
J3-SC-55TO76-102218MS	Benzo(a)anthracene	125	30 - 120
L5-0TO26-101318MSD	Phenanthrene	-68.2	30 - 120
L5-0TO26-101318MSD	Fluoranthene	-132.4	30 - 120
L5-0TO26-101318MSD	Pyrene	-66.2	30 - 120
L5-0TO26-101318MSD	Chrysene	11.5	30 - 120
L5-0TO26-101318MSD	Benzo(b)fluoranthene	19.6	30 - 120
J5-SC-00TO10-102218MSD	Phenanthrene	182.8	30 - 120
J5-SC-00TO10-102218MSD	Fluoranthene	286.7	30 - 120
J5-SC-00TO10-102218MSD	Pyrene	232.8	30 - 120
J5-SC-00TO10-102218MSD	Benzo(a)anthracene	138.3	30 - 120
J5-SC-00TO10-102218MSD	Chrysene	180.5	30 - 120
J5-SC-00TO10-102218MSD	Benzo(b)fluoranthene	164.8	30 - 120
J5-SC-00TO10-102218MSD	Benzo(k)fluoranthene	126.6	30 - 120
J5-SC-00TO10-102218MSD	Benzo(a)pyrene	140.6	30 - 120
J5-SC-00TO10-102218MSD	Indeno(1,2,3-cd)pyrene	140.6	30 - 120
J5-SC-00TO10-102218MSD	Benzo(g,h,i)perylene	127.3	30 - 120
J3-SC-55TO76-102218MSD	Fluoranthene	209.7	30 - 120
J3-SC-55TO76-102218MSD	Pyrene	67.5	30 - 120
J3-SC-55TO76-102218MSD	Benzo(a)anthracene	69.7	30 - 120
J3-SC-55TO76-102218MSD	Chrysene	68.6	30 - 120
J3-SC-55TO76-102218MSD	Benzo(b)fluoranthene	65.8	30 - 120
J3-SC-55TO76-102218MSD	Benzo(a)pyrene	69.5	30 - 120

These analytes are qualified as estimated in the native samples.

MS/MSD RPDs: The laboratory control limit was <40%. RPDs were within these limits with the following exceptions:

QC ID	Analyte	RPD	Lab Control Limit
J3-SC-55TO76-102218MSD	Fluoranthene	52	40
J3-SC-55TO76-102218MSD	Pyrene	65	40
J3-SC-55TO76-102218MSD	Benzo(a)anthracene	56	40
J3-SC-55TO76-102218MSD	Chrysene	48	40
J3-SC-55TO76-102218MSD	Benzo(b)fluoranthene	47	40
J3-SC-55TO76-102218MSD	Benzo(a)pyrene	49	40

Positive results for these analytes are qualified as estimated in the native sample.

Sample Internal standards: Sample internal standards were evaluated for the full validation data package only. Internal standard responses and retention times in each sample must be within 50-200% and ±30 seconds, respectively, of the continuing calibration standard. These criteria were met.

Compound quantitation: Concentrations of Dibenzofuran, Benzo(b)fluoranthene, and Fluorene-d10 in the samples and QC samples from the full validation data package were recalculated to verify sample quantitations. No discrepancies were noted.

Compound identifications: Chromatograms and quantitation reports in the full validation data package were reviewed for accuracy of compound identification for Dibenzofuran and Benzo(b) fluoranthene. No issues were noted.

Laboratory narrative/flags: Various acenaphthylene, benzo(a)anthracene, and dibenzofuran results were flagged with an X, and noted in the laboratory narrative as containing matrix interference with high bias. These results are qualified as estimated.

Conflicting definitions of the X flag applied to the Benzo(b)fluoranthene and Benzo(k) fluoranthene results in two samples were received from the laboratory. The original narrative indicated that Benzo(b)fluoranthene and Benzo(k)fluoranthene co-eluted in the X-flagged samples. Email correspondence indicated that Benzo(b)fluoranthene co-eluted with only Benzo(j)fluoranthene, and the X flag was meant to indicate matrix interference as described above. These results are also qualified as estimated.

Overall assessment: With minor exceptions, documentation was found to be clear and complete. No calculation, transcription, or identification errors were noted. Calibration data demonstrated generally acceptable instrument performance. Blank contamination resulted in U qualification of some concentrations below the reporting limit and one estimated concentration. Data were estimated based on high cooler receipt temperatures, continuing calibration % differences, analytical interferences, low, very low and high spike recoveries, and spike duplicate and field duplicate variability.

5.0 Organochlorine Pesticide Analyses

Quality control analysis frequencies: The laboratory SOP specifies that method blank, laboratory control sample (LCS), matrix spike (MS), and MS duplicate (MSD) must be analyzed with each batch. In addition, labeled standards must be measured in each field and quality control sample. The QAPP also specifies that a laboratory duplicate must be analyzed 1 per 20 samples.

Each sample batch included a method blank, LCS, MS and MSD. The additional QAPP requirement for a laboratory duplicate was not met. However, no qualifiers are assigned based on the absence of this additional QC information.

QAPP field QC requirements include one blind field duplicate per twenty surface sediment samples and one equipment rinsate blank per twenty field samples or per week. Three field duplicates and weekly equipment rinsate blanks were collected, meeting QAPP requirements.

Holding times: Refrigerated sediment samples must be extracted within 14 days of collection and frozen sediment samples must be extracted within 1 year. Extracts must be analyzed within 40 days of extraction. These holding times were met. However, the cooler receipt temperature for laboratory report #K1809944, 11.5°C, exceeded the recommended range of 0-6°C. Results for samples in the above report are qualified as estimated.

System performance checks: System performance checks were evaluated for the full validation data package only. The laboratory SOP and/or draft SOP update specifies that the tune must meet manufacturer's recommendations for PFTBA (perfluorotributylamine), and that endrin and 4,4'-DDT breakdown must not exceed 20%. These criteria were met.

Instrument calibration: Instrument calibration data were evaluated for the full validation data package only. The following criteria were evaluated, and may be from the laboratory SOP or method 1668C: 1) An initial calibration (ICAL) with a minimum of 5 points and maximum percent relative standard deviations (%RSD) of ≤20%, or use of an alternate calibration model with an R^2 >0.990. 2) An initial calibration verification (ICV) standard with a percent recovery within 75-125% for target analytes and 50-200% recovery for labeled compounds. 3) A continuing calibration verification (CCV) analyzed at the beginning of each 12 hour shift, with percent recoveries within 75-125% for target analytes and 50-200% recovery for labeled compounds. 4) Both initial and continuing calibrations must have relative response factors >0.01 5) Additionally, CCV injection internal standard responses must be within 50-200% of the ICAL midpoint and retention times must be within +/- 30 seconds of the ICAL midpoint. These criteria were met in the full validation package.

However, the narrative from laboratory report K1809944 indicates that 3 compounds were outside the 75-125% recovery limit in the CCV as follows:

Standard ID	Analysis Date	Analyte	%Recovery	Control Limit
MS42\102418F004.D	10/24/2018	2,4'-DDD	69.6	75-125%
MS42\102418F004.D	10/24/2018	4,4'-DDE	62.1	75-125%
MS42\102418F004.D	10/24/2018	Dieldrin	70.3	75-125%

Positive and non-detect results for these analytes in samples associated with this CCV are qualified as estimated.

Laboratory blank results: Criteria for blanks are that analyte concentrations must be below the RL, or below 10% of the lowest associated sample concentration. These criteria were met. However, concentrations below the reporting limit may have an impact on low level sample results. Contamination was detected in laboratory blanks at levels below the reporting limit as follows:

Blank ID	Analyte	Concentration (ng/L)	RL (ng/L)
KQ1815643-03	2,4'-DDD	0.13J	0.5

Blank ID	Analyte	Concentration (ng/L)	RL (ng/L)
KQ1815643-03	2,4'-DDT	0.24J	0.5
KQ1815643-03	4,4'-DDD	0.17J	0.5
KQ1815643-03	4,4'-DDT	0.19J	0.5

Sample concentrations within 5 times these levels should be considered not detected, and are qualified U.

Labeled compound recoveries: Laboratory control limits ranged from 5-120% to 13-200%. 4,4'DDT-d4 was not recovered in the following samples:

Sample ID	Compound	% Recovery	Lab Control Limit
H3-SC-0TO1-102118	S_4,4'-DDT-d4	0	13 - 200
H3-SC-2TO29-102118	S_4,4'-DDT-d4	0	13 - 200

The laboratory noted interference. Associated results have been rejected due to identification issues (see "Compound identifications:" below), and no further qualification is necessary.

The remaining labeled compound recoveries ranged from 24 to 160% and were within laboratory control limits.

LCS recoveries: Laboratory control limits ranged from 59-138 to 85-113%. Sediment LCS recoveries were within these limits.

LCS/LCSD RPDs: Laboratory control limit was <30%. RPDs were within this limit.

MS recoveries: Recoveries which exceeded laboratory control limits are discussed below.

QC ID	Analyte	% Recovery	Lab Control Limit
D6-SC-40to64-102118MS	4,4'-DDE	10	35 - 162
D6-SC-40to64-102118MSD	4,4'-DDE	0	35 - 162
J3-SC-20to36-102218MSD	4,4'-DDE	192.3	35 - 162
D6-SC-40to64-102118MS	Aldrin	3327	52 - 151
D6-SC-40to64-102118MSD	Aldrin	3402	52 - 151
J3-SC-20to36-102218MS	Aldrin	0	52 - 151
J3-SC-20to36-102218MSD	Aldrin	0	52 - 151
J5-SC-20to40-102218MS	Aldrin	835	52 - 151
J5-SC-20to40-102218MSD	Aldrin	1278.6	52 - 151
D5-0to28-100918MS	Aldrin	1200	52 - 151
D5-0to28-100918MSD	Aldrin	454.9	52 - 151
F5-0to28-101118MS	Aldrin	285	52 - 151
F5-0to28-101118MSD	Aldrin	0	52 - 151
L5-0to26-101318MS	Aldrin	313	52 - 151
L5-0to26-101318MSD	Aldrin	466	52 - 151
J3-SC-20to36-102218MS	2,4'-DDT	0	55 - 161
J3-SC-20to36-102218MS	Dieldrin	0	28 – 150
J3-SC-20to36-102218MSD	2,4'-DDT	0	55 – 161
J3-SC-20to36-102218MSD	Dieldrin	0	28 - 150
J3-SC-20to36-102218MSD	gamma-BHC (Lindane)	62.1	64 - 135
D5-0to28-100918MS	2,4'-DDT	-11	55 - 161
D5-0to28-100918MSD	2,4'-DDT	16.2	55 - 161

Concentrations of 4,4'-DDE in samples D6-SC-40to64-102118 and J3-SC-20TO36-102218 exceeded 4 times the spike amount, and qualifiers are not required.

Where recoveries are above the control limit, concentrations in the native sample are qualified as estimated and non-detects are considered unaffected. Where recoveries are below the control limit but above 20%, both detected concentrations and non-detects in the native sample are qualified as estimated.

Where recoveries are below 20%, detected concentrations are qualified as estimated and non-detect results are rejected as unusable, with the following exception: Variability in the 2,4'-DDT concentration in sample D5-0to28-100918 likely contributed to the low recoveries in the matrix spikes, and the native result is qualified as estimated rather than rejected.

Because 12 of the 18 MS/MSD Aldrin recoveries were within QC limits, qualifiers for this compound are applied to all samples in the extraction batch and not just the native sample.

MS/MSD RPDs: Laboratory control limit was <40%. RPDs were within this limit with the following exceptions:

QC ID	Analyte	RPD	Lab Control Limit
D5-0TO28-100918 MSD RE	ALDRIN	90	40
J5-SC-20TO40-102218 MSD	ALDRIN	42	40

Aldrin is qualified as estimated in the native samples.

Field duplicate variability: For field duplicate pairs with concentrations above five times the reporting limit, RPDs were below 50% with the following exceptions:

Field duplicate (FD) ID	Analyte	FD Result (ug/kg)	Sample Result (ug/kg)	RPD
511-0TO28-100918 / D5-0TO28-100918	2,4'-DDT	0.13 U	2.1	177
512-0TO28-101118 RE / F5-0to28-101118	2,4'-DDT	0.14 U	1.9	173

These analytes are qualified as estimated in the sample and field duplicate.

For field duplicate pairs with concentrations below five times the reporting limit, absolute differences were within ± two times the reporting limit.

Compound Quantitation: Compound quantitations were evaluated for the full validation data package only. Concentrations of 4,4'-DDT, gamma chlordane, and 4,4'-DDT-d4 were recalculated to verify sample quantitations. No discrepancies were noted.

Compound identifications: Extracted transition current profiles and quantitation reports in the full validation data package were reviewed for accuracy of compound identification for 4,4'-DDT, and gamma chlordane. No issues were noted.

Identification issues were observed during evaluation of labeled compound recoveries of samples H3-SC-0TO1-102118 and H3-SC-2TO29-102118. Evaluation of the extracted transition current profiles indicate associated 2,4'-DDT and 4,4'-DDT results may not have been identified or reported accurately, and these results are rejected as unusable.

The laboratory resubmitted data packages for 6 of the 7 reports indicating that the 2,4'-DDT and 4,4'-DDD peaks were mis-identified (switched) in the initial calibration, and therefore also in the associated continuing calibrations, samples and QC samples. Resubmission results were reviewed as follows:

1) Data package K1809944 was reviewed to confirm resubmission was not required. A different initial calibration was used and the elution order matched the elution order of the resubmitted data for other batches. Therefore no changes were required.

2) Sample results in the resubmitted EDDs were compared to the original EDDs for compounds other than 2,4'-DDT and 4,4'-DDD, and an additional 18 result differences, most very slight, were identified and sent to the laboratory for evaluation. The significant differences are shown below.

Sample ID	Analyte	Original Result	Resubmitted Result
L5-0TO26-101318	ALDRIN	ND	3.8
H3-SC-2TO29-102118	4,4'-DDE	ND	25
J6-SC-00TO10-102218	4,4'-DDT	0.27	1.9

In conjunction with the laboratory, it was determined that the resubmitted result for 4,4'-DDE in sample H3-SC-2TO29-102118, 25 ug/kg, was the correct value, and the original result for remaining two discrepancies were correct.

3) Sample results for 2,4'-DDT in the resubmitted EDD were compared to 4,4'-DDD results in the original EDD and 4,4'-DDE in the resubmitted EDD were compared to 2,4'-DDT results in the original EDD to identify problem areas for further review. Results with resubmitted concentrations which differed by more than 20% from the originally reported concentration for the mis-identified analyte were recalculated to verify quantitations. Recalculated values matched the resubmitted concentrations. It was noted that the relative response factor (RRF) in the resubmitted results for the 11/16 initial calibration differed significantly from the original results. The 11/16/18 RRF in the resubmitted results showed better agreement with the 11/06/18 RRF, and no further review was performed.

4) Results with resubmitted detect/non-detect status which differed from the originally reported result for the mis-identified analyte were evaluated to verify sample identification These results included the following:

Sample ID	Resub Analyte	Resub Result	Original Analyte	Original Result	RPD
B1-0to20-101418	2,4'-DDT	0.094 U	4,4'-DDD	0.46	132.1
N3-0to26-101518	4,4'-DDD	0.067 U	2,4'-DDT	0.59	159.2
N3-0to26-101518	2,4'-DDT	0.59	4,4'-DDD	0.067 U	159.2
P1-0to30-101518	2,4'-DDT	0.23	4,4'-DDD	0.064 U	112.9
D6-SC-88to108-102118	4,4'-DDD	0.043 J	2,4'-DDT	0.094 U	74.5
H3-SC-46to63-102118	4,4'-DDD	0.54	2,4'-DDT	0.094 U	140.7
H3-SC-1to2-102118	2,4'-DDT	0.096 U	4,4'-DDD	21	198.2
R4-SC-1to2-102118	2,4'-DDT	0.1 U	4,4'-DDD	8.1	195.1

- In three samples B1-0to20-101418, H3-SC-1to2-102118, and R4-SC-1to2-102118, one peak was identified and quantitated in the original submission as both 4,4'-DDD and 2,4'-DDT. This was corrected in the resubmission.

- In sample N3-0to26-101518, the mis-identification of 2,4'-DDT and 4,4'-DDT was not corrected. The results for these two analytes in this sample are corrected as follows:

Sample ID	Analyte	Resub Result	Corrected Result
N3-0to26-101518	4,4'-DDD	0.067 U	0.73
N3-0to26-101518	2,4'-DDT	0.59	0.094 U

- In sample P1-0to30-101518, the data system did not identify the peak at retention time 11.82 as a detection in the original submission. However, the transition ratio is close to the lower limit in the resubmission, and the transition ratio may not have met identification criteria in the original submission. The identification in the resubmission is acceptable.

- In sample D6-SC-88to108-102118, the concentration was below the MDL for 2,4'-DDT, but above the MDL for 4,4'-DDD, and is correct in the resubmission.

- In sample H3-SC-46to63-102118, the resubmitted 4,4'-DDD result has a transition ratio outside of limits. This result should be considered not detected with an elevated reporting limit at the reported concentration and is qualified "U".

5) Additionally, two issues were noted in the QC results:

- Identifications of 2,4'-DDT and 4,4'-DDD were not corrected for the matrix spike and matrix spike duplicate of J5-SC-20-40-102218. Recalculated % recoveries were still within limits, and no further action was required.

- 2,4'-DDT was reported as not detected for the matrix spike and matrix spike duplicate of J3-SC-20to36-102218, even though both analytes were reported in the original submission. Quantitation reports and chromatograms were reviewed. In the initial submission, the main peak was reported as 2,4'-DDT and a shoulder at the edge of the peak was integrated separately and reported as 4,4'-DDD. In the resubmission, the entire peak was reported as 4,4'-DDD. Interference was present, as noted in the laboratory narrative, and interpretation of the proper integration is subjective. Data qualifiers have been updated to reflect the resubmission recoveries.

The number of identification issues, including the retention time switch and the identification of the same peak as two separate compounds, introduces a higher level of uncertainty in the reported results. If a higher confidence in data identifications is required, it is recommended that additional validation focused on identification review be performed on critical sample results.

Additionally, a stage 4 validation is limited in the information available for review, and must rely on criteria present in the method, and/or the elution order reported by the laboratory in the calibration standards. A method validation may be useful in evaluating the laboratory in-house method. Two recommendations which may improve future data quality are:

- That the laboratory SOP be updated with retention time and/or relative retention time criteria, so that Identification errors are more likely to be caught during review.

- That the laboratory evaluate increasing the run time in order to reduce the overlap between retention time windows and eliminate the identification of a single peak as two different compounds. For reference, the minimum methoxyclor retention time in method 1699 is 39 minutes. The methoxyclor retention time in the reported data was approximately 14 minutes.

Laboratory narrative/flags: No qualifiers are assigned based on the laboratory narrative or flags.

Overall assessment: Calibration data demonstrated generally acceptable instrument performance. Quality control data showed widespread interferences and poor accuracy in the available matrix spike data, resulting in the rejection of non-detect results. Blank contamination resulted in U qualification of three concentrations below the reporting limit. Data were estimated based on high cooler receipt temperatures, continuing calibration % differences, and field duplicate variability.

Additionally several identification issues were noted and corrected or rejected with this data set. Four results associated with labeled standards which were not recovered were potentially mis-reported and were rejected as unusable. An identification error in the initial calibration used for most of the sample results was noted by the laboratory and corrected data resubmitted. Review of the resubmitted data showed additional identification errors, decreasing the overall confidence in the sample results. Further review of critical sample results may be warranted.

6.0 Total Solids Analyses

Quality control analysis frequencies: A laboratory duplicate was analyzed in each batch as appropriate for the method.

QAPP field QC requirements include one blind field duplicate per twenty surface sediment samples. Four field duplicates were collected, meeting QAPP requirements. Three weekly equipment rinsate blanks were also collected and analyzed for total suspended solids, but these results were not evaluated.

Holding times: The QAPP specifies a holding time of 7 days for refrigerated samples and 1 year for frozen samples. Samples were analyzed between 9 and 21 days after sampling, meeting the QAPP specified holding time for frozen samples.

These holding times were exceeded for the following samples:

Sample ID	LabID	Days refrigerated from sampling to analysis	Analysis holding time (days)
D3-0to26-101018	K1810088-005	8	7
N3-0to26-101518	K1810270-001	8	7
L1-0to30-101518	K1810270-004	11	7
N1-0to30-101518	K1810270-006	11	7
T1-0to30-101518	K1810270-010	11	7
R1-0to30-101518	K1810270-011	11	7
P1-0to30-101518	K1810270-012	11	7
T3-0to28-101618	K1810270-013	10	7
T7-0to31-101618	K1810270-015	10	7

Sample ID	LabID	Days refrigerated from sampling to analysis	Analysis holding time (days)
T5-0to26-101618	K1810270-016	10	7
P3-0to29-101618	K1810270-017	10	7
R5-0to25-101618	K1810270-018	10	7
514-0to29-101618	K1810270-019	10	7
P5-0to26-101618	K1810270-020	10	7
R3-0to33-101718	K1810270-029	9	7

The total solids result in these samples are qualified as estimated.

Additionally, the cooler receipt temperature for laboratory report #K1809944, 11.5°C, exceeded the recommended range of 0-6°C. Results for samples in the above report are qualified as estimated.

During review of holding times, it was observed that 1) Analysis dates in some data packages were incorrect and 2) raw data was missing from the data package for the samples analyzed on 10/26/2018 in laboratory report K1810270 and the samples analyzed on 10/24/2018 in laboratory report K1810302. Updated data packages were requested from the laboratory.

Instrument calibration: Instrument calibration data were evaluated for the full validation data package only. The measured calibration values were well within ± 0.1 grams, demonstrating acceptable balance precision.

Laboratory duplicate results: Laboratory duplicate RPDs were within the laboratory control limit of 20%.

Field duplicate results: All field duplicate RPDs were below 20%.

Compound Quantitation: Sample concentrations of total solids in the full validation samples were recalculated reviewed to verify sample quantitations. No discrepancies were noted.

Overall assessment: With exceptions, documentation was found to be clear and complete. Quality control results demonstrate generally acceptable precision. Review of the full validation data indicates accurate quantitation. Data were estimated due to exceeded holding times.

Total solids results are acceptable for use as qualified.

7.0 Qualifier Summary Table

Client ID	Analyte(s)	Qualifier	Reason
511-0to28-100918	Bis(2-ethylhexyl)phthalate	J	High cooler temperature
B3-0to21-100818	Bis(2-ethylhexyl)phthalate	J	High cooler temperature
B5-0to26-100818	Bis(2-ethylhexyl)phthalate	J	High cooler temperature
B7-0to30-100918	Bis(2-ethylhexyl)phthalate	J	High cooler temperature
D5-0to28-100918	Bis(2-ethylhexyl)phthalate	J	High cooler temperature
D6-SC-1to2-102118 RE	Bis(2-ethylhexyl)phthalate	J	Analysis holding time exceeded
D7-0to28-100918	Bis(2-ethylhexyl)phthalate	J	High cooler temperature
L5-0to26-101318	Bis(2-ethylhexyl)phthalate	J	Very low MS and MSD recoveries
511-0to28-100918 RE	Tri-n-butyltin cation	J	High cooler temperature
512-0to28-101118	Tri-n-butyltin cation	J	High FD RPD
A1-0to30-102018	Tri-n-butyltin cation	UJ	Extraction holding time exceeded
B3-0to21-100818	Tri-n-butyltin cation	J	High cooler temperature
B5-0to26-100818	Tri-n-butyltin cation	J	High cooler temperature
B7-0to30-100918	Tri-n-butyltin cation	J	High cooler temperature

Client ID	Analyte(s)	Qualifier	Reason
D5-0to28-100918 RE	Tri-n-butyltin cation	J	High cooler temperature
D6-SC-88to108-102118	Tri-n-butyltin cation	J	High dual column RPD
D7-0to28-100918	Tri-n-butyltin cation	J	High cooler temperature
F5-0to28-101118	Tri-n-butyltin cation	J	High MSD recovery, High FD RPD
K6-0to28-101918	Tri-n-butyltin cation	J	High MS recovery
L5-0to26-101318	Tri-n-butyltin cation	J	High MS and MSD recoveries
N1-0to30-101518	Tri-n-butyltin cation	J	High dual column RPD
N3-0to26-101518	Tri-n-butyltin cation	J	Extraction holding time exceeded
511-0to28-100918	2-Methylnaphthalene, Acenaphthylene, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(g,h,i)perylene, Benzo(k)fluoranthene, Chrysene, Dibenzo(a,h)anthracene, Dibenzofuran, Fluoranthene, Indeno(1,2,3-cd)pyrene, Naphthalene, Phenanthrene, Pyrene	J	High cooler temperature
511-0to28-100918	Acenaphthene, Anthracene, Fluorene	J	High cooler temperature, High FD difference
513-0to26-101318	Fluoranthene, Phenanthrene	J	High FD RPD
611-101118	2-Methylnaphthalene, Acenaphthene, Benzo(a)anthracene, Dibenzofuran, Fluoranthene, Fluorene, Naphthalene, Phenanthrene, Pyrene	UJ	Lab blank contamination
613-102218	Benzo(a)anthracene, Naphthalene	UJ	Lab blank contamination
A1-0to30-102018	Acenaphthylene	J	Interference
A1-0to30-102018	Benzo(a)anthracene	UJ	Lab blank contamination
B3-0to21-100818	2-Methylnaphthalene, Acenaphthene, Acenaphthylene, Anthracene, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(g,h,i)perylene, Benzo(k)fluoranthene, Chrysene, Dibenzo(a,h)anthracene, Dibenzofuran, Fluoranthene, Fluorene, Indeno(1,2,3-cd)pyrene, Naphthalene, Phenanthrene, Pyrene	J	High cooler temperature
B5-0to26-100818	2-Methylnaphthalene, Acenaphthene, Acenaphthylene, Anthracene, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(g,h,i)perylene, Benzo(k)fluoranthene, Chrysene, Dibenzo(a,h)anthracene, Dibenzofuran, Fluoranthene, Fluorene, Indeno(1,2,3-cd)pyrene, Naphthalene, Phenanthrene, Pyrene	J	High cooler temperature
B7-0to30-100918	2-Methylnaphthalene, Acenaphthene, Acenaphthylene, Anthracene, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(g,h,i)perylene, Benzo(k)fluoranthene, Chrysene, Dibenzo(a,h)anthracene, Dibenzofuran, Fluoranthene, Fluorene, Indeno(1,2,3-cd)pyrene, Naphthalene, Phenanthrene, Pyrene	J	High cooler temperature
D5-0to28-100918	2-Methylnaphthalene, Acenaphthylene, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(g,h,i)perylene, Benzo(k)fluoranthene, Dibenzo(a,h)anthracene, Dibenzofuran, Fluoranthene, Indeno(1,2,3-cd)pyrene, Naphthalene, Phenanthrene, Pyrene	J	High cooler temperature
D5-0to28-100918	Acenaphthene, Anthracene, Fluorene	J	High cooler temperature, High FD difference
D5-0to28-100918	Chrysene	J	High cooler temperature, High MS recovery
D6-SC-108to123-102118	Acenaphthylene	J	Interference

Client ID	Analyte(s)	Qualifier	Reason
D7-0to28-100918	2-Methylnaphthalene, Acenaphthene, Acenaphthylene, Anthracene, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(g,h,i)perylene, Benzo(k)fluoranthene, Chrysene, Dibenzo(a,h)anthracene, Dibenzofuran, Fluoranthene, Fluorene, Indeno(1,2,3-cd)pyrene, Naphthalene, Phenanthrene, Pyrene	J	High cooler temperature
H3-SC-85to99-102118	Benzo(a)anthracene	J	Interference
H3-SC-99to114-102118	Benzo(a)anthracene	J	Interference
J3-SC-00to10-102218	Dibenzofuran	J	Interference
J3-SC-10to20-102218	Acenaphthylene	J	Interference
J3-SC-20to36-102218	Dibenzofuran	J	Interference
J3-SC-36to45-102218	Benzo(b)fluoranthene, Dibenzofuran	J	Interference
J3-SC-36to45-102218	Benzo(k)fluoranthene	UJ	Interference
J3-SC-45to55-102218	Dibenzofuran	J	Interference
J3-SC-55to76-102218	Benzo(a)anthracene, Pyrene	J	High MS recovery, High MS/MSD RPD
J3-SC-55to76-102218	Benzo(a)pyrene, Benzo(b)fluoranthene, Chrysene	J	High MS/MSD RPD
J3-SC-55to76-102218	Fluoranthene	J	High MS and MSD recoveries, High MS/MSD RPD
J5-SC-00to10-102218	Acenaphthene	J	High MS recovery
J5-SC-00to10-102218	Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(g,h,i)perylene, Benzo(k)fluoranthene, Chrysene, Fluoranthene, Indeno(1,2,3-cd)pyrene, Phenanthrene, Pyrene	J	High MS and MSD recoveries
J5-SC-10to20-102218	Dibenzofuran	J	Interference
J5-SC-20to40-102218	Dibenzofuran	J	Interference
J5-SC-40to60-102218	Pyrene	J	Low continuing calibration %D
J5-SC-60to80-102218	Acenaphthylene, Dibenzofuran	J	Interference
J5-SC-95to110-102218	Acenaphthylene	J	Interference
J6-SC-10to20-102218	Dibenzofuran	J	Interference
J6-SC-20to40-102218	Dibenzofuran	J	Interference
J6-SC-40to60-102218	Dibenzofuran	J	Interference
J6-SC-60to80-102218	Pyrene	J	Low continuing calibration %D
J6-SC-80to96-102218	Acenaphthylene	J	Interference
J6-SC-96to111-102218	Acenaphthylene	J	Interference
L1-0to30-101518	Dibenzofuran	UJ	Lab blank contamination
L1-0to30-101518	Fluorene	J	Lab blank contamination
L3-SC-00to10-102218	Acenaphthylene, Dibenzofuran	J	Interference
L3-SC-10to20-102218	Acenaphthylene	J	Interference
L3-SC-20to40-102218	Acenaphthylene	J	Interference
L5-0to26-101318	Benzo(b)fluoranthene, Chrysene	J	Low MS recovery, Very low MSD recovery
L5-0to26-101318	Fluoranthene, Phenanthrene	J	Very low MS and MSD recoveries, High FD RPD
L5-0to26-101318	Pyrene	J	Very low MS and MSD recoveries
N1-0to30-101518	Acenaphthylene	J	Interference
N1-0to30-101518	Dibenzofuran, Fluorene	UJ	Lab blank contamination
R4-SC-8to10-102118	Benzo(b)fluoranthene	J	Interference
R4-SC-8to10-102118	Benzo(k)fluoranthene	UJ	Interference
T1-0to30-101518	Acenaphthylene	J	Interference
T1-0to30-101518	Dibenzofuran, Fluorene, Phenanthrene	UJ	Lab blank contamination
511-0to28-100918	2,4'-DDD, 4,4'-DDT, Aldrin, cis-Nonachlor, gamma-BHC (Lindane), Heptachlor, Oxychlordane	UJ	High cooler temperature
511-0to28-100918	2,4'-DDE, 4,4'-DDD, alpha-Chlordane, Chlordane, gamma, trans-Nonachlor	J	High cooler temperature
511-0to28-100918	2,4'-DDT	UJ	High cooler temperature, High FD difference
511-0to28-100918	4,4'-DDE	J	High cooler temperature, Low continuing calibration %D
511-0to28-100918	Dieldrin	UJ	High cooler temperature, Low continuing calibration %D
512-0to28-101118	4,4'-DDD	UJ	High FD Difference
512-0to28-101118	4,4'-DDT	J	High FD Difference

Client ID	Analyte(s)	Qualifier	Reason
512-0to28-101118	Aldrin	R	High batch MS and very low batch MSD recoveries
613-102218	2,4'-DDT, 4,4'-DDD, 4,4'-DDT	UJ	Lab blank contamination
B3-0to21-100818	2,4'-DDD, 4,4'-DDD, alpha-Chlordane, Chlordane, gamma, trans-Nonachlor	J	High cooler temperature
B3-0to21-100818	2,4'-DDE, 2,4'-DDT, 4,4'-DDT, Aldrin, cis-Nonachlor, gamma-BHC (Lindane), Heptachlor, Oxychlordane	UJ	High cooler temperature
B3-0to21-100818	4,4'-DDE	J	High cooler temperature, Low continuing calibration %D
B3-0to21-100818	Dieldrin	UJ	High cooler temperature, Low continuing calibration %D
B5-0to26-100818	2,4'-DDD, 4,4'-DDD, alpha-Chlordane, Chlordane, gamma, trans-Nonachlor	J	High cooler temperature
B5-0to26-100818	2,4'-DDE, 2,4'-DDT, 4,4'-DDT, Aldrin, cis-Nonachlor, gamma-BHC (Lindane), Heptachlor, Oxychlordane	UJ	High cooler temperature
B5-0to26-100818	4,4'-DDE	J	High cooler temperature, Low continuing calibration %D
B5-0to26-100818	Dieldrin	UJ	High cooler temperature, Low continuing calibration %D
B7-0to30-100918	2,4'-DDD, 2,4'-DDT, 4,4'-DDD, 4,4'-DDT, alpha-Chlordane, Chlordane, gamma, trans-Nonachlor	J	High cooler temperature
B7-0to30-100918	2,4'-DDE, Aldrin, cis-Nonachlor, gamma-BHC (Lindane), Heptachlor, Oxychlordane	UJ	High cooler temperature
B7-0to30-100918	4,4'-DDE	J	High cooler temperature, Low continuing calibration %D
B7-0to30-100918	Dieldrin	UJ	High cooler temperature, Low continuing calibration %D
D3-0to26-101018	Aldrin	R	High batch MS and very low batch MSD recoveries
D5-0to28-100918	2,4'-DDD, 4,4'-DDT, Aldrin, cis-Nonachlor, gamma-BHC (Lindane), Heptachlor, Oxychlordane	UJ	High cooler temperature
D5-0to28-100918	2,4'-DDE, 4,4'-DDD, alpha-Chlordane, Chlordane, gamma, trans-Nonachlor	J	High cooler temperature
D5-0to28-100918	2,4'-DDT	J	High cooler temperature, High FD difference
D5-0to28-100918	4,4'-DDE	J	High cooler temperature, Low continuing calibration %D
D5-0to28-100918	Dieldrin	UJ	High cooler temperature, Low continuing calibration %D
D7-0to28-100918	2,4'-DDD, 2,4'-DDT, 4,4'-DDD, alpha-Chlordane, Chlordane, gamma, trans-Nonachlor	J	High cooler temperature
D7-0to28-100918	2,4'-DDE, 4,4'-DDT, Aldrin, cis-Nonachlor, gamma-BHC (Lindane), Heptachlor, Oxychlordane	UJ	High cooler temperature
D7-0to28-100918	4,4'-DDE	J	High cooler temperature, Low continuing calibration %D
D7-0to28-100918	Dieldrin	UJ	High cooler temperature, Low continuing calibration %D
F3-0to27-101118	Aldrin	R	High batch MS and very low batch MSD recoveries
F5-0to28-101118	4,4'-DDD, 4,4'-DDT	J	High FD Difference
F7-0to27-101118	Aldrin	R	High batch MS and very low batch MSD recoveries
H3-0to28-101118	Aldrin	R	High batch MS and very low batch MSD recoveries
H3-SC-0to1-102118	4,4'-DDT	R	Analyte mis-reported
H3-SC-2to29-102118	2,4'-DDT, 4,4'-DDT	R	Analyte mis-reported
H3-SC-46to63-102118	4,4'-DDD	U	Interference
H5-0to29-101118	Aldrin	R	High batch MS and very low batch MSD recoveries
H7-0to24-101218	Aldrin	R	High batch MS and very low batch MSD recoveries
J3-SC-00to10-102218	Aldrin	R	Very low batch MS and MSD recoveries

Client ID	Analyte(s)	Qualifier	Reason
J3-SC-10to20-102218	Aldrin	R	Very low batch MS and MSD recoveries
J3-SC-20to36-102218	2,4'-DDT	R	Very low MS and MSD recovery
J3-SC-36to45-102218	Aldrin	R	Very low batch MS and MSD recoveries
J3-SC-45to55-102218	Aldrin	R	Very low batch MS and MSD recoveries
J3-SC-55to76-102218	Aldrin	R	Very low batch MS and MSD recoveries
J3-SC-76to98-102218	Aldrin	R	Very low batch MS and MSD recoveries
J5-SC-10to20-102218	Aldrin	R	Very low batch MS and MSD recoveries
J5-SC-40to60-102218	Aldrin	R	Very low batch MS and MSD recoveries
J5-SC-60to80-102218	Aldrin	R	Very low batch MS and MSD recoveries
J6-SC-00to10-102218	Aldrin	R	Very low batch MS and MSD recoveries
J6-SC-10to20-102218	Aldrin	R	Very low batch MS and MSD recoveries
J6-SC-111to121-102218	Aldrin	R	Very low batch MS and MSD recoveries
J6-SC-20to40-102218	Aldrin	R	Very low batch MS and MSD recoveries
J6-SC-60to80-102218	Aldrin	R	Very low batch MS and MSD recoveries
J6-SC-80to96-102218	Aldrin	R	Very low batch MS and MSD recoveries
J6-SC-96to111-102218	Aldrin	R	Very low batch MS and MSD recoveries
J7-0to26-101218	Aldrin	R	High batch MS and very low batch MSD recoveries
L3-SC-00to10-102218	Aldrin	R	Very low batch MS and MSD recoveries
L3-SC-10to20-102218	Aldrin	R	Very low batch MS and MSD recoveries
L3-SC-20to40-102218	Aldrin	R	Very low batch MS and MSD recoveries
512-0to28-101118	Total Solids	J	Holding time exceeded
514-0to29-101618	Total Solids	J	Holding time exceeded
D3-0to26-101018	Total Solids	J	Holding time exceeded
F3-0to27-101118	Total Solids	J	Holding time exceeded
F5-0to28-101118	Total Solids	J	Holding time exceeded
F7-0to27-101118	Total Solids	J	Holding time exceeded
H3-0to28-101118	Total Solids	J	Holding time exceeded
H5-0to29-101118	Total Solids	J	Holding time exceeded
L1-0to30-101518	Total Solids	J	Holding time exceeded
N1-0to30-101518	Total Solids	J	Holding time exceeded
N3-0to26-101518	Total Solids	J	Holding time exceeded
P1-0to30-101518	Total Solids	J	Holding time exceeded
P3-0to29-101618	Total Solids	J	Holding time exceeded
P5-0to26-101618	Total Solids	J	Holding time exceeded
R1-0to30-101518	Total Solids	J	Holding time exceeded
R3-0to33-101718	Total Solids	J	Holding time exceeded
R5-0to25-101618	Total Solids	J	Holding time exceeded
T1-0to30-101518	Total Solids	J	Holding time exceeded
T3-0to28-101618	Total Solids	J	Holding time exceeded
T5-0to26-101618	Total Solids	J	Holding time exceeded
T7-0to31-101618	Total Solids	J	Holding time exceeded

8.0 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.
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
ARI	Analytical Resources, Inc.
CRDL	Contract required detection limit
DV	Data validation
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
EDL	Estimated detection limit
EMPC	Estimated maximum possible concentration
MS	Matrix spike
MSD	Matrix spike duplicate
NA	Not Applicable
RL	Reporting limit
RPD	Relative percent difference
RRM	Regional reference material
RSD	Relative standard deviations
SRM	Standard reference material
SRM	Selective reaction monitoring

9.0 References

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