



1435 Norjohn Court, Unit 1, Burlington, ON, Canada L7L 0E6

SVOC DATA PACKAGE

Client Project Information

Project ID: 60566335
Project Description: Portland Harbour Pre- Remedial Design Investigation & Baseline Sampling
Contact: Amy Dahl

ALSE Project Information

Project ID: AECOM100
Contact: Whitney Davis
Submission ID(s): L2134269

Final Package Review by:

A handwritten signature in black ink, appearing to read "Gerry", is written over a horizontal line.

Date Reviewed: 14-Aug-18



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SECTION 1: PROJECT NARRATIVE

ALSE Project Information

Project ID: AECOM100

Contact: Whitney Davis

Submission ID(s): L2134269

Analytical Method: Chlorinated Pesticides by EPA 1699 (modified)

Client Project Information

Project ID: 60566335

Project Description: Portland Harbour Pre- Remedial Design Investigation & Baseline Sampling

Contact: Amy Dahl

ALS Sample ID	Client Sample Descriptions	Matrix	Date Sampled	Date Received	Date Extracted	Date Analyzed
L2134269-1	PDI-SC-S036-0TO1.4	Sediment	20-Jul-18	24-Jul-18	25-Jul-18	10-Aug-18
L2134269-2	PDI-SC-S036-1.4TO3.4	Sediment	20-Jul-18	24-Jul-18	25-Jul-18	10-Aug-18
L2134269-3	PDI-SC-S036-3.4TO5.2	Sediment	20-Jul-18	24-Jul-18	25-Jul-18	10-Aug-18
L2134269-4	PDI-SC-S022-0TO2	Sediment	20-Jul-18	24-Jul-18	25-Jul-18	10-Aug-18
L2134269-5	PDI-SC-S022-2TO4	Sediment	20-Jul-18	24-Jul-18	25-Jul-18	10-Aug-18
L2134269-6	PDI-SC-S022-4TO6	Sediment	20-Jul-18	24-Jul-18	25-Jul-18	10-Aug-18
WG2831102-1	Method Blank	QC	n/a	n/a	25-Jul-18	09-Aug-18
WG2831102-2	Laboratory Control Sample	QC	n/a	n/a	25-Jul-18	09-Aug-18

Comments and Notes:

a) Sample Integrity:

The samples were received in good condition at 4.2 degrees C.

b) Instrumental Analysis:

All results have been reported on a dry weight basis.

For the initial calibration, the responses from the highest calibration levels have not been included due to instrument detector saturation. The remaining six calibration levels have been employed.

For the mid-run mass resolution check, the mass peak for m/z 281 was not centered when printed. However, this peak does not belong to the time range of interest for the reported targets.

For the mid-run continuing calibration verification (CCV), the recovery of the native target 2,4'-DDD was somewhat above the method control limit. The recoveries for this target were within control for the pre and post-run CCV, as well as the laboratory control sample (LCS). However, the possibility that the results for this target may be slightly elevated cannot be precluded.

For the mid-and post-run CCVs, the recoveries of select labelled standards were below the method control limit, potentially due to a reduction in the sample introduction efficiency at the GC. Except as noted, reported native target data are not expected to be biased. The reported recoveries of the impacted labelled standards may be biased low.

I certify that this data package is in compliance with the terms and condition of the contract , both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this data package (hardcopy and/or electronic version) has been authorized by the Laboratory Manager or his designee, as verified by the following signature.

Steve Kennedy
Technical Supervisor

14-Aug-18
Date

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SECTION 2: DATA SUMMARY REPORT



1435 Norjohn Court, Unit 1, Burlington, ON, Canada L7L 0E6
Phone: 905-331-3111, FAX: 905-331-4567

Certificate of Analysis

ALS Project Contact: Whitney Davis
ALS Project ID: AECOM100
ALS WO#: L2134269
Date of Report: 14-Aug-18
Date of Sample Receipt: 24-Jul-18

Client Name: AECOM United States
Client Address: 1111 Third Avenue
Suite 1600
Seattle, WA 98101, USA
Client Contact: Amy Dahl
Client Project ID: 60566335

COMMENTS: Chlorinated Pesticides by EPA 1699 (modified)

All results have been reported on a dry weight basis.

Certified by: _____

Steve Kennedy
Steve Kennedy
Technical Supervisor

Results in this certificate relate only to the samples as submitted to the laboratory.

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Sample Analysis summary Report

Sample Name	PDI-SC-S036- 0T01.4	PDI-SC-S036- 1.4T03.4	PDI-SC-S036- 3.4T05.2	PDI-SC-S022-0T02	PDI-SC-S022-2T04	PDI-SC-S022-4T06
ALS Sample ID	L2134269-1	L2134269-2	L2134269-3	L2134269-4	L2134269-5	L2134269-6
Sample Size	5.29	6.73	6.58	6.67	6.62	6.86
Sample size units	g	g	g	g	g	g
Percent Solid	52.2%	65.4%	62.8%	66.4%	65.4%	66.5%
Sample Matrix	Sediment	Sediment	Sediment	Sediment	Sediment	Sediment
Sampling Date	20-Jul-18	20-Jul-18	20-Jul-18	20-Jul-18	20-Jul-18	20-Jul-18
Extraction Date	25-Jul-18	25-Jul-18	25-Jul-18	25-Jul-18	25-Jul-18	25-Jul-18
Target Analytes	ng/g	ng/g	ng/g	ng/g	ng/g	ng/g
2,4'-DDE	0.340	0.484	1.41	<0.0059	<0.0065	<0.018
4,4'-DDE	6.46	5.82	7.84	<0.015	<0.0081	<0.030
2,4'-DDD	0.860	3.03	25.2	<0.012	<0.010	<0.028
4,4'-DDD	2.70	9.00	35.7	<0.017	<0.011	<0.014
2,4'-DDT	2.68	0.162	1.72	<0.021	<0.014	<0.018
4,4'-DDT	4.63	0.955	12.3	<0.037	<0.045	<0.053
Extraction Standards	% Rec	% Rec	% Rec	% Rec	% Rec	% Rec
4,4'-DDE, 13C12-	71	71	59	91	87	33
4,4'-DDD, 13C12-	43	54	40	57	56	57
4,4'-DDT, 13C12-	28	36	26	41	40	30

ALS Life sciences

Quality Control Summary Report

Sample Name	Method Blank	Laboratory Control Sample
ALS Sample ID	WG2831102-1	WG2831102-2
Sample Size	5.79	1.00
Sample size units	g	n/a
Percent Solid	100.0%	50.2%
Sample Matrix	QC	QC
Sampling Date	n/a	n/a
Extraction Date	25-Jul-18	25-Jul-18
Target Analytes	ng/g	% Rec
2,4'-DDE	<0.010	112
4,4'-DDE	<0.011	96
2,4'-DDD	<0.018	102
4,4'-DDD	<0.013	98
2,4'-DDT	<0.017	79
4,4'-DDT	<0.041	96
Extraction Standards	% Rec	% Rec
4,4'-DDE, 13C12-	82	58
4,4'-DDD, 13C12-	98	78
4,4'-DDT, 13C12-	92	66

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Continuing Calibration Summary Report

Sample Name	ccv	ccv	ccv	ccv
ALS Sample ID	H6-18-RS1-053	H6-18-CCV-0745	H6-18-CCV-0747	H6-18-CCV-0749
Sample Size	1	1	1	1
Sample size units	n/a	n/a	n/a	n/a
Percent Solid	n/a	n/a	n/a	n/a
Sample Matrix	QC	QC	QC	QC
Sampling Date	n/a	n/a	n/a	n/a
Extraction Date	n/a	n/a	n/a	n/a
Target Analytes	% Rec	% Rec	% Rec	% Rec
2,4'-DDE	94	94	111	101
4,4'-DDE	92	98	95	92
2,4'-DDD	95	95	138	120
4,4'-DDD	95	100	101	94
2,4'-DDT	98	98	85	93
4,4'-DDT	89	98	98	94
Extraction Standards	% Rec	% Rec	% Rec	% Rec
4,4'-DDE, 13C12-	111	110	97	117
4,4'-DDD, 13C12-	111	110	47	71
4,4'-DDT, 13C12-	115	114	31	51

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Sample Analysis Report

Sample Name PDI-SC-S036-0T01.4
 ALS Sample ID L2134269-1
 Analysis Method EPA 1699 (mod)
 Analysis Type Sample
 Sample Matrix Sediment

Sampling Date 20-Jul-18
 Extraction Date 25-Jul-18
 Sample Size 5.29 g
 Percent Solid 52.2%
 Split Ratio 1

Approved:
R. Bakhtiari
 --e-signature--
 13-Aug-2018

Run Information **Run 1**
 Filename 6-180809A24
 Run Date 10-Aug-18 03:00
 Final Volume 1020 uL
 Dilution Factor 1
 Analysis Units ng/g
 Instrument - Column HRMS-6 HP5MSUSR163634H

Target Analytes	Ret. Time	Conc. ng/g	EDL ng/g	Flags	EMPC ng/g	LQL
2,4'-DDE	21.02	0.340	0.0048	J	0.39	
4,4'-DDE	21.95	6.46	0.013		0.39	
2,4'-DDD	22.20	0.860	0.017		0.39	
4,4'-DDD	23.17	2.70	0.025		0.39	
2,4'-DDT	23.25	2.68	0.032		0.39	
4,4'-DDT	24.22	4.63	0.11		0.39	
Extraction Standards						
4,4'-DDE, 13C12-	125	21.94	71	21-125		
4,4'-DDD, 13C12-	125	23.15	43	5-150		
4,4'-DDT, 13C12-	125	24.22	28	5-120		

EDL Indicates the Estimated Detection Limit, based on the measured background noise for this target in this sample.
 LQL Lower Quantification Limit, based on the lowest calibration level corrected for sample size, splits and dilutions.

 J indicates that a target analyte was detected below the calibrated range.

 EMPC Estimated Maximum Possible Concentration – elevated detection limit due to interference or positive id criterion failure

ALS Life sciences

Sample Analysis Report

Sample Name PDI-SC-S036-1.4TO3.4
 ALS Sample ID L2134269-2
 Analysis Method EPA 1699 (mod)
 Analysis Type Sample
 Sample Matrix Sediment

Sampling Date 20-Jul-18
 Extraction Date 25-Jul-18
 Sample Size 6.73 g
 Percent Solid 65.4%
 Split Ratio 1

Approved:
R. Bakhtiari
 --e-signature--
 13-Aug-2018

Run Information **Run 1**
 Filename 6-180809A28
 Run Date 10-Aug-18 05:13
 Final Volume 1020 uL
 Dilution Factor 1
 Analysis Units ng/g
 Instrument - Column HRMS-6 HP5MSUSR163634H

Target Analytes	Ret. Time	Conc. ng/g	EDL ng/g	Flags	EMPC ng/g	LQL
2,4'-DDE	21.00	0.484	0.0066			0.30
4,4'-DDE	21.94	5.82	0.0079			0.30
2,4'-DDD	22.17	3.03	0.0081			0.30
4,4'-DDD	23.14	9.00	0.014	M		0.30
2,4'-DDT	23.23	0.162	0.018	M,J		0.30
4,4'-DDT	24.20	0.955	0.048			0.30
Extraction Standards						
4,4'-DDE, 13C12-	125	21.92	71	21-125		
4,4'-DDD, 13C12-	125	23.14	54	5-150		
4,4'-DDT, 13C12-	125	24.20	36	5-120		

EDL Indicates the Estimated Detection Limit, based on the measured background noise for this target in this sample.
 LQL Lower Quantification Limit, based on the lowest calibration level corrected for sample size, splits and dilutions.
 M Indicates that a peak has been manually integrated.

 J indicates that a target analyte was detected below the calibrated range.

 EMPC Estimated Maximum Possible Concentration – elevated detection limit due to interference or positive id criterion failure

ALS Life sciences

Sample Analysis Report

Sample Name PDI-SC-S036-3.4T05.2
 ALS Sample ID L2134269-3
 Analysis Method EPA 1699 (mod)
 Analysis Type Sample
 Sample Matrix Sediment

Sampling Date 20-Jul-18
 Extraction Date 25-Jul-18
 Sample Size 6.58 g
 Percent Solid 62.8%
 Split Ratio 1

Approved:
R. Bakhtiari
 --e-signature--
 13-Aug-2018

Run Information **Run 1**
 Filename 6-180809A29
 Run Date 10-Aug-18 05:46
 Final Volume 1020 uL
 Dilution Factor 1
 Analysis Units ng/g
 Instrument - Column HRMS-6 HP5MSUSR163634H

Target Analytes	Ret. Time	Conc. ng/g	EDL ng/g	Flags	EMPC ng/g	LQL
2,4'-DDE	21.02	1.41	0.0064		0.31	
4,4'-DDE	21.95	7.84	0.0065		0.31	
2,4'-DDD	22.18	25.2	0.018		0.31	
4,4'-DDD	23.15	35.7	0.021		0.31	
2,4'-DDT	23.25	1.72	0.026		0.31	
4,4'-DDT	24.22	12.3	0.040		0.31	
Extraction Standards						
4,4'-DDE, 13C12-	125	21.94	59	21-125		
4,4'-DDD, 13C12-	125	23.14	40	5-150		
4,4'-DDT, 13C12-	125	24.20	26	5-120		

EDL Indicates the Estimated Detection Limit, based on the measured background noise for this target in this sample.
 LQL Lower Quantification Limit, based on the lowest calibration level corrected for sample size, splits and dilutions.

EMPC Estimated Maximum Possible Concentration – elevated detection limit due to interference or positive id criterion failure

ALS Life sciences

Sample Analysis Report

Sample Name PDI-SC-S022-0T02
 ALS Sample ID L2134269-4
 Analysis Method EPA 1699 (mod)
 Analysis Type Sample
 Sample Matrix Sediment

Sampling Date 20-Jul-18
 Extraction Date 25-Jul-18
 Sample Size 6.67 g
 Percent Solid 66.4%
 Split Ratio 1

Approved:
R. Bakhtiari
 --e-signature--
 13-Aug-2018

Run Information **Run 1**
 Filename 6-180809A25
 Run Date 10-Aug-18 03:33
 Final Volume 1020 uL
 Dilution Factor 1
 Analysis Units ng/g
 Instrument - Column HRMS-6 HP5MSUSR163634H

Target Analytes	Ret. Time	Conc. ng/g	EDL ng/g	Flags	EMPC ng/g	LQL
2,4'-DDE	NotFnd	<0.0059	0.0059	U		0.31
4,4'-DDE	21.94	<0.015	0.0092	M,J,R	0.015	0.31
2,4'-DDD	NotFnd	<0.012	0.012	U		0.31
4,4'-DDD	NotFnd	<0.017	0.017	U		0.31
2,4'-DDT	NotFnd	<0.021	0.021	U		0.31
4,4'-DDT	NotFnd	<0.037	0.037	U		0.31
Extraction Standards ng						
4,4'-DDE, 13C12-	125	21.94	91	21-125		
4,4'-DDD, 13C12-	125	23.14	57	5-150		
4,4'-DDT, 13C12-	125	24.20	41	5-120		

EDL Indicates the Estimated Detection Limit, based on the measured background noise for this target in this sample.
 LQL Lower Quantification Limit, based on the lowest calibration level corrected for sample size, splits and dilutions.
 M Indicates that a peak has been manually integrated.
 U Indicates that this compound was not detected above the EDL.

 J indicates that a target analyte was detected below the calibrated range.
 R Indicates that the ion abundance ratio for this compound did not meet the acceptance criterion.

 EMPC Estimated Maximum Possible Concentration – elevated detection limit due to interference or positive id criterion failure

ALS Life sciences

Sample Analysis Report

Sample Name PDI-SC-S022-2TO4
 ALS Sample ID L2134269-5
 Analysis Method EPA 1699 (mod)
 Analysis Type Sample
 Sample Matrix Sediment

Sampling Date 20-Jul-18
 Extraction Date 25-Jul-18
 Sample Size 6.62 g
 Percent Solid 65.4%
 Split Ratio 1

Approved:
R. Bakhtiari
 --e-signature--
 13-Aug-2018

Run Information **Run 1**
 Filename 6-180809A26
 Run Date 10-Aug-18 04:06
 Final Volume 1020 uL
 Dilution Factor 1
 Analysis Units ng/g
 Instrument - Column HRMS-6 HP5MSUSR163634H

Target Analytes	Ret. Time	Conc. ng/g	EDL ng/g	Flags	EMPC ng/g	LQL
2,4'-DDE	NotFnd	<0.0065	0.0065	U		0.31
4,4'-DDE	NotFnd	<0.0081	0.0081	U		0.31
2,4'-DDD	NotFnd	<0.010	0.010	U		0.31
4,4'-DDD	NotFnd	<0.011	0.011	U		0.31
2,4'-DDT	NotFnd	<0.014	0.014	U		0.31
4,4'-DDT	NotFnd	<0.045	0.045	U		0.31
Extraction Standards						
4,4'-DDE, 13C12-	125	21.94	87	21-125		
4,4'-DDD, 13C12-	125	23.14	56	5-150		
4,4'-DDT, 13C12-	125	24.20	40	5-120		

EDL Indicates the Estimated Detection Limit, based on the measured background noise for this target in this sample.
 LQL Lower Quantification Limit, based on the lowest calibration level corrected for sample size, splits and dilutions.
 U Indicates that this compound was not detected above the EDL.
 EMPC Estimated Maximum Possible Concentration – elevated detection limit due to interference or positive id criterion failure

ALS Life sciences

Sample Analysis Report

Sample Name PDI-SC-S022-4TO6
 ALS Sample ID L2134269-6
 Analysis Method EPA 1699 (mod)
 Analysis Type Sample
 Sample Matrix Sediment

Sampling Date 20-Jul-18
 Extraction Date 25-Jul-18
 Sample Size 6.86 g
 Percent Solid 66.5%
 Split Ratio 1

Approved:
R. Bakhtiari
 --e-signature--
 13-Aug-2018

Run Information **Run 1**
 Filename 6-180809A27
 Run Date 10-Aug-18 04:40
 Final Volume 1020 uL
 Dilution Factor 1
 Analysis Units ng/g
 Instrument - Column HRMS-6 HP5MSUSR163634H

Target Analytes	Ret. Time	Conc. ng/g	EDL ng/g	Flags	EMPC ng/g	LQL
2,4'-DDE	21.00	<0.018	0.018	M,U	0.017	0.30
4,4'-DDE	21.94	<0.030	0.024	M,J,R	0.030	0.30
2,4'-DDD	NotFnd	<0.028	0.028	U		0.30
4,4'-DDD	NotFnd	<0.014	0.014	U		0.30
2,4'-DDT	NotFnd	<0.018	0.018	U		0.30
4,4'-DDT	NotFnd	<0.053	0.053	U		0.30
Extraction Standards ng						
4,4'-DDE, 13C12-	125	21.94	33	21-125		
4,4'-DDD, 13C12-	125	23.14	57	5-150		
4,4'-DDT, 13C12-	125	24.20	30	5-120		

EDL Indicates the Estimated Detection Limit, based on the measured background noise for this target in this sample.
 LQL Lower Quantification Limit, based on the lowest calibration level corrected for sample size, splits and dilutions.
 M Indicates that a peak has been manually integrated.
 U Indicates that this compound was not detected above the EDL.

 J indicates that a target analyte was detected below the calibrated range.
 R Indicates that the ion abundance ratio for this compound did not meet the acceptance criterion.

 EMPC Estimated Maximum Possible Concentration – elevated detection limit due to interference or positive id criterion failure

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SECTION 3: METHOD SUMMARY

OC Pesticide METHOD SUMMARY
Method EPA 1699

Introduction:

This summary is to provide ALSE Burlington OC pesticide method details in order to provide persons reviewing or validating this data package sufficient information to re-construct the sample calculation, data verification and review. It incorporates the analysis of organochlorine pesticides via EPA method 1699. Deviations from this reference method are documented in ALS Standard Operating Procedures (available upon request) and in this Method Summary.

Any deviations to what is listed herein or in the ALS Standard Operating Procedures would be listed in the project narrative.

To avoid the confusion and conflicting nomenclature within the performance based methods, we have defined the labeled standards in terms relating to the time of addition to the sample or extract. Therefore;

- Laboratory Surrogate (when provided/requested by the client) are added prior to sample extraction
- The Field or Sampling Standards (where used) are added prior to field sampling
- The Extraction Standards are added prior to extraction
- The GPC Recovery Standard is added (when used) prior to Gel Permeation Chromatographic cleanup
- The Clean-up Standards (where used) are added prior to extract clean-up
- The Injection Standards are added prior to extract injection.

Additional method information, such as Instrumental Descriptors, is documented in ALS Standard Operating Procedures and available upon request.

Calibration Standard Levels:

Seven levels of standard are available for calibration as listed in Table 1. These targets give a wide range of responses on the analytical instruments, thus it is expected that for any given target, either the lowest standard level(s) or the highest standard level(s) may be excluded due to poor response, poor linearity, or detector saturation. With seven levels of standard, it is expected that at least 5 points can be used for calibration for each target.

Table 1: Calibration Standards (conc in ng/mL)

	CS1	CS2	CS3	CS4	CS5	CS6	CS7
Natives							
Hexachlorobutadiene	2	7.5	20	50	150	400	1200
1,2,4,5-Tetrachlorobenzene	2	7.5	20	50	150	400	1200
1,2,3,4-Tetrachlorobenzene	2	7.5	20	50	150	400	1200
Pentachlorobenzene	2	7.5	20	50	150	400	1200
Hexachlorobenzene	2	7.5	20	50	150	400	1200
3,4,5,6-Tetrachloroveratrole	2	7.5	20	50	150	400	1200
Pentachloroanisole	2	7.5	20	50	150	400	1200
alpha-BHC	2	7.5	20	50	150	400	1200
beta-BHC	2	7.5	20	50	150	400	1200
gamma-BHC	2	7.5	20	50	150	400	1200
delta-BHC	2	7.5	20	50	150	400	1200
Pentachloronitrobenzene	2	7.5	20	50	150	400	1200
Heptachlor	2	7.5	20	50	150	400	1200
Aldrin	2	7.5	20	50	150	400	1200
4,4'-DDNU	2	7.5	20	50	150	400	1200
Dacthal	2	7.5	20	50	150	400	1200
Chlorpyrifos	10	37.5	100	250	750	2000	6000
Octachlorostyrene	2	7.5	20	50	150	400	1200
Heptachlor Epoxide B	2	7.5	20	50	150	400	1200
Heptachlor Epoxide A	2	7.5	20	50	150	400	1200
Oxychlordane	2	7.5	20	50	150	400	1200
4,4'-DDMU	2	7.5	20	50	150	400	1200
trans-Chlordane	2	7.5	20	50	150	400	1200
cis-Chlordane	2	7.5	20	50	150	400	1200
trans-Nonachlor	2	7.5	20	50	150	400	1200
Dieldrin	2	7.5	20	50	150	400	1200
Endrin	2	7.5	20	50	150	400	1200
cis-Nonachlor	2	7.5	20	50	150	400	1200
Endosulfan I	2	7.5	20	50	150	400	1200
Endosulfan II	2	7.5	20	50	150	400	1200
Endosulfan Sulfate	2	7.5	20	50	150	400	1200
24'-DDE	2	7.5	20	50	150	400	1200
44'-DDE	2	7.5	20	50	150	400	1200
24'-DDD	2	7.5	20	50	150	400	1200
44'-DDD	2	7.5	20	50	150	400	1200
24'-DDT	2	7.5	20	50	150	400	1200
44'-DDT	2	7.5	20	50	150	400	1200
Endrin Aldehyde	2	7.5	20	50	150	400	1200
Endrin Ketone	2	7.5	20	50	150	400	1200
Methoxychlor	2	7.5	20	50	150	400	1200
Dicofol	20	75	200	500	1500	4000	12000
Mirex	2	7.5	20	50	150	400	1200
Parlar-26	2	7.5	20	50	150	400	1200
Parlar-50	2	7.5	20	50	150	400	1200
Parlar-62	2	7.5	20	50	150	400	1200

Laboratory Surrogate	1,3-Dibromobenzene	20	20	20	20	20	20	20
	Endrin Ketone	2	7.5	20	50	150	400	1200
Field Surrogate	1,3,5-Tribromobenzene	20	20	20	20	20	20	20
	1,2,4,5-Tetrabromobenzene	20	20	20	20	20	20	20
	delta-BHC	2	7.5	20	50	150	400	1200
GPC Recovery Standard	13C12-PCB-133	100	100	100	100	100	100	100
Extraction Standard	13C6-Pentachlorobenzene	250	250	250	250	250	250	250
	13C6-Hexachlorobenzene	250	250	250	250	250	250	250
	13C6-alpha-BHC	250	250	250	250	250	250	250
	d6-gamma-BHC	250	250	250	250	250	250	250
	13C10-Heptachlor	250	250	250	250	250	250	250
	13C10-Oxychlorane	250	250	250	250	250	250	250
	13C10-trans-Nonachlor	250	250	250	250	250	250	250
	13C12-Dieldrin	250	250	250	250	250	250	250
	13C12-Endrin	250	250	250	250	250	250	250
	13C9-Endosulfan-II	250	250	250	250	250	250	250
	13C12-44'-DDE	250	250	250	250	250	250	250
	13C12-44'-DDD	250	250	250	250	250	250	250
	13C12-44'-DDT	250	250	250	250	250	250	250
	d6-Methoxychlor	250	250	250	250	250	250	250
	13C10-Mirex	250	250	250	250	250	250	250
Injection Standard	13C12-PCB-9	100	100	100	100	100	100	100
	13C12-PCB-52	100	100	100	100	100	100	100
	13C12-PCB-101	100	100	100	100	100	100	100

Calibration and Quality Control Limits

The calibration and QC Sample control limits are presented in Table 2 below. For the lowest standard used for initial calibration, and for each calibration verification CS3, the signal to noise ratio for each ion for both labelled and non-labelled analytes must be greater than or equal to 10:1

	Calibration		Samples and QC Samples	
	Initial Cal. %RSD	Cal. Ver. %Exp	LCS % Rec	Samples % Rec
Natives				
Hexachlorobutadiene	35	70-130	5-200	
1,2,4,5-Tetrachlorobenzene	35	70-130	5-200	
1,2,3,4-Tetrachlorobenzene	35	70-130	5-200	
Pentachlorobenzene	20	70-130	5-200	
Hexachlorobenzene	20	75-125	10-150	
3,4,5,6-Tetrachloroveratrole	35	70-130	20-200	
Pentachloroanisole	35	70-130	20-200	
alpha-BHC	20	75-125	50-120	
beta-BHC	35	75-125	50-120	
gamma-BHC	20	75-125	50-120	
delta-BHC	35	75-125	50-120	
Pentachloronitrobenzene	35	70-130	20-200	
Heptachlor	20	75-125	50-120	
Aldrin	35	75-125	50-120	
4,4'-DDNU	35	75-125	20-160	
Dacthal	35	50-150	20-200	
Chlorpyrifos	35	75-125	19-163	
Octachlorostyrene	35	70-130	50-175	
Heptachlor Epoxide B	35	70-130	20-200	
Heptachlor Epoxide A	35	75-125	50-120	
Oxychlordane	20	75-125	50-120	
4,4'-DDMU	35	75-125	20-160	
trans-Chlordane	35	75-125	50-120	
cis-Chlordane	35	75-125	50-120	
trans-Nonachlor	20	75-125	50-120	
Dieldrin	20	75-125	50-120	
Endrin	20	75-125	50-120	
cis-Nonachlor	35	75-125	50-120	
Endosulfan I	35	75-125	50-120	
Endosulfan II	20	75-125	5-200	
Endosulfan Sulfate	35	75-125	50-200	
24'-DDE	35	75-125	24-123	
44'-DDE	20	75-125	50-120	
24'-DDD	35	75-125	50-120	
44'-DDD	20	75-125	42-120	
24'-DDT	35	75-125	50-120	
44'-DDT	20	75-125	50-120	
Endrin Aldehyde	35	70-130	20-200	
Endrin Ketone	35	75-125	50-134	
Methoxychlor	20	75-125	50-120	
Dicofol	35	50-150	20-200	
Mirex	20	75-125	50-120	
Parlar-26	35	70-130	20-200	
Parlar-50	35	70-130	20-200	
Parlar-62	35	70-130	20-200	

Laboratory Surrogate	1,3-Dibromobenzene	35	50-150	50-150	40-120
	Endrin Ketone	35	50-150	50-150	40-150
Field Surrogate	1,3,5-Tribromobenzene	35	50-150	50-150	60-120
	1,2,4,5-Tetrabromobenzene	35	50-150	50-150	60-120
	delta-BHC	35	50-150	50-150	60-120
GPC Recovery Standard	13C12-PCB-133	35	50-150	50-150	50-120
Extraction Standard	13C6-Pentachlorobenzene	35	70-130	5-120	5-120
	13C6-Hexachlorobenzene	35	70-130	5-120	5-120
	13C6-alpha-BHC	35	70-130	13-138	16-129
	d6-gamma-BHC	35	70-130	5-124	11-120
	13C10-Heptachlor	35	70-130	5-128	5-120
	13C10-Oxychlorane	35	70-130	5-144	23-135
	13C10-trans-Nonachlor	35	70-130	17-154	36-139
	13C12-Dieldrin	35	70-130	19-161	40-151
	13C12-Endrin	35	70-130	20-157	35-155
	13C9-Endosulfan-II	35	70-130	5-144	15-148
	13C12-44'-DDE	35	70-130	26-169	47-160
	13C12-44'-DDD	35	70-130	13-200	5-150
	13C12-44'-DDT	35	70-130	13-200	5-120
	d6-Methoxychlor	35	70-130	8-200	5-120
	13C10-Mirex	35	70-130	5-138	5-120

Additional Continuing Calibration Details:

After initial calibration is established, a CS4 standard is injected as a Continuing Calibration Verification (CCV) at the beginning of every 12 hour shift in which samples are analyzed. If the following performance criteria are met, analysis of samples may proceed:

- Ion abundance ratios are within their respective theoretical limits (see Table 3)
- All targets have a s/n ratio of at least 10:1
- The RT of each analyte is within 15 seconds of that in the initial calibration
- Endin and DDT breakdown is less than 20% (see Section 5.2.4.2)
- The %Diff is within the CCV limits (see Table 2)

If these performance criteria are not met, GC maintenance is performed or the system is adjusted and a new CCV is injected, or a new initial calibration is run.

Mid-run Calibration Verification:

While the EPA 1699 does not require a post-run calibration verification standard to be run, it is recognized that responses and/or relative responses of some targets may change significantly during HRMS analysis due to sample related contamination of GC or MS components. This problem is compounded by chemical dissimilarities between some targets and their quantification reference standards in the case of internal standard quantification. Enhanced quantification and a measure of confidence in sample results obtained during an analytical shift can be attained by injecting a CS4 calibration verification (VER) standard in the middle of, and at the end of a 12-hour run, and quantifying samples against the average of bracketing calibration standards where improved results would be achieved.

a) Mid-Run VER:

If this analysis meets the performance criteria for a pre-run CCV, then all of the samples preceding the mid-run VER can be quantified vs. the initial calibration, and analysis can proceed. If the mid-run VER does not meet pre-run CCV criteria, the preceding samples can be quantified vs. bracketing calibration runs (using the pre-run CCV and mid-run VER as a two-point calibration) and analysis can proceed, provided that the following criteria are met:

- Ion abundance ratios are within their respective theoretical limits (see Table 1) or within 15% of the ratios in the pre-run CCV
- All targets have a s/n ratio of at least 10:1
- The RT of each analyte is within 15 seconds of that in the initial calibration
- Endin and DDT breakdown is less than 20%
- The %RPD of the mid-run VER vs. the pre-run CCV meets the CCV %Diff limits (See Table 2)

If the mid-run VER does not meet the above criteria either, analysis cannot continue without corrective action (samples analyzed after the mid-run VER in an automated sequence must be re-analyzed). The samples preceding the failing mid-run VER may be flagged and reported, but must be assessed for impact on data quality:

- If a failing native target is present in any of the preceding samples above the Method Detection Limit (or above the client's lower required Detection Limit, if known), that sample must be re-analyzed for that target.
- If a failing native target's Estimated Detection Limit is above the Method Detection Limit (or above the client's lower required Detection Limit, if known) due to deterioration of system performance, that sample must be re-analyzed for that target.

a) Post-Run VER:

If this analysis meets the performance criteria for a pre-run CCV, then all of the samples preceding the post-run VER can be quantified vs. the initial calibration. If the post-run VER does not meet pre-run CCV criteria, the preceding samples can be quantified vs. bracketing calibration runs (using the post-run VER and mid-run VER as a two-point calibration) provided that the following criteria are met:

- Ion abundance ratios are within their respective theoretical limits (see Table 1) or within 15% of the ratios in the mid-run CCV
- All targets have a s/n ratio of at least 10:1
- The RT of each analyte is within 15 seconds of that in the initial calibration
- Endin and DDT breakdown is less than 20%
- The %RPD of the post-run VER vs. the mid-run VER meets the CCV %Diff limits (See Table 2)

If the post-run VER does not meet the above criteria either, the samples preceding the failing post-run VER may be flagged and reported, but must be assessed for impact on data quality:

- If a failing native target is present in any of the preceding samples above the Method Detection Limit (or above the client's lower required Detection Limit, if known), that sample must be re-analyzed for that target.
- If a failing native target's Estimated Detection Limit is above the Method Detection Limit (or above the client's lower required Detection Limit, if known) due to deterioration of system performance, that sample must be re-analyzed for that target.

Reporting Limits:

Unless indicated in the otherwise, native target data is reported down to 2.5:1 signal to noise for each isomer grouping for each extract injection. This is consistent to SW846 8290 defined protocols (i.e. EDL or Estimated Detection Limit) and is commonly applied throughout the industry to any and all performance based HRMS methods.

Method Blank:

The method blank levels must be below the response to the lowest calibration standard used for initial calibration.

MS/MSD (where required):

The % relative difference between the MS and MSD spike recoveries should be less than or equal to 20%.

Instrument/Run Performance Criteria:

a) Chromatographic Performance

For the DB-5 column, 44'-DDT and 24'-DDT (or the labelled analogues) must be uniquely resolved to a valley height of less than 60% of the shorter of the two peaks.

b) DDT and Endrin Breakdown

A custom standard (HROCP-GC_BD#1) is injected to measure the breakdown of endrin and DDT during the run. This standard must be injected at the beginning and end of each 12 hour shift, and it is also recommended that it be injected along with the mid-run CCV where used. This standard contains 13C12-4,4'-DDT, 13C12-endrin, and native endrin, endrin aldehyde and endrin ketone.

- For measurement of DDT breakdown, measure the concentration for 13C12-44'-DDE, 13C12-44'-DDD and 13C12-44'-DDT (the labelled DDT is part of the standard, and the labelled DDE and DDD are breakdown products). Calculate breakdown using the following formula:

13C12-44'-DDT % Breakdown =

$$\frac{(\text{concentration of 13C12-44'-DDD} + \text{concentration of 13C12-44'-DDE})}{\text{concentration of 13C12-44'-DDT}} \times 100\%$$

labelled DDT = part of standard; labelled DDE and DDD = breakdown products

- Additionally, measurement of endrin breakdown can be performed. For measurement of endrin breakdown, measure the concentration of endrin, endrin aldehyde, and endrin ketone (these natives are quantified by isotope dilution vs. the 13C12-endrin). Calculate breakdown using the following formula:

Endrin % Breakdown =

$$\frac{(\text{concentration of endrin aldehyde} + \text{concentration of endrin ketone})}{\text{concentration of endrin}} \times 100\%$$

If the breakdown of endrin and/or DDT exceeds 20% in a standard, the targets are decomposing on the inlet or column, and remedial action must be taken (inlet maintenance and trimming of the analytical column) before any valid sample data can be produced. If the breakdown of DDT or endrin in a sample exceeds 20% and there is that native in the sample above the MDL, that sample will have to be reanalyzed for that target (further cleanup or dilution of that sample is recommended before reanalysis).

Breakdown exceedences can be ignored under the following circumstances:

- Where the endrin breakdown fails but DDT breakdown passes and where DDT and/or it's metabolites are the only targets.
- Where the DDT breakdown fails but endrin breakdown passes and where endrin and/or it's metabolites are the only targets.
- For the determination of other pesticide targets (i.e. non-DDT and non-Endrin and metabolite targets) which have a corresponding labelled extraction/internal standard of exactly the same isomer.

c) Mass Resolution:

At the beginning of and just following the end of each 12 hour run sequence, the instrument must be checked to demonstrate a resolution of 10,000 for each quantification window.

The maximum time between scans within a descriptor is 1 second.

Lock mass deviations to the average response must be less than or equal 20%.

Laboratory Duplicates:

The % relative difference between duplicates should be less than or equal to 25% but only where the response is greater than the low calibration standard.

Analyte Identification Criteria:

Ion Ratio Criteria

For all compounds, a pair of ions with a specific isotopic ratio are being monitored. To have a confirmed positive response to a native or labelled OCP, that ratio must be within the theoretical limits in Table 1, or within 15% of the observed values on the most recent CS4 analysis.

Signal to Noise Criteria

The signal to noise ratio for each quantification and confirmation ion for labelled and non-labelled analytes must be greater than or equal to 10:1 for the initial calibration CS1 and for each calibration verification CS4. For positive identification of a native target in a sample, both ions must have a s/n ratio exceeding 2.5:1.

Matched RT on Peak Maxima

The retention time (RT) of the peak maxima for each pair of quantification ions must be no more than 2 seconds (i.e. 2 scans) difference.

Expected Retention Time (RT)

The peak must be at the expected RT

- within -1/+3 seconds of the labelled standard for natives with their own ¹³C labelled standard
- within +/- 0.008 RRT units of the RRT in the most recent CS4 analysis for targets with their own ²H labelled standard
- within +/- 0.010 RRT units of the RRT in the most recent CS4 analysis for targets without their own labelled standard

As per EPA 1699 Sections 16.5-16.6, it is possible that not all of the positive ID criteria are met. If a pesticide is deemed to be present in this case by the experienced spectroscopist, the result may be flagged as "this result is unconfirmed and must not be used for permitting or regulatory compliance purposes". If the ion abundance ratio criteria are not met, the result must also include an "R" flag.

Table 3: Monitored Masses, Ion Abundance Ratios, and Quantitation/RT References

Entry	Native Standard	Quantification Method	Quantification vs. Entry #:	Quantitation Ion	Confirmation Ion	Theoretical Ion Abundance ratio	Ion Abundance Ratio Tolerance
1	Hexachlorobutadiene	rel_int	52	259.8102	261.8072	1.25	0.25
2	1,2,4,5-Tetrachlorobenzene	rel_int	52	215.8881	217.8852	2.08	0.25
3	1,2,3,4-Tetrachlorobenzene	rel_int	52	215.8881	217.8852	2.08	0.25
4	Pentachlorobenzene	rel_int	52	249.8491	251.8462	1.56	0.25
5	Hexachlorobenzene	rel_int	53	283.8102	285.8072	1.23	0.25
6	3,4,5,6-Tetrachloroveratrole	rel_int	54	275.9092	277.9063	2.08	0.25
7	Pentachloroanisole	rel_int	54	279.8597	281.8568	1.56	0.25
8	alpha-BHC	rel_int	54	218.9116	220.9086	2.1	0.25
9	beta-BHC	rel_int	55	218.9116	220.9086	2.1	0.25
10	gamma-BHC	rel_int	55	218.9116	220.9086	2.1	0.25
11	delta-BHC	rel_int	55	218.9116	220.9086	2.1	0.25
12	Pentachloronitrobenzene	rel_int	56	294.8342	296.8313	1.56	0.25
13	Heptachlor	rel_int	56	271.8102	273.8072	1.25	0.25
14	Aldrin	rel_int	57	262.857	264.854	1.56	0.25
15	4,4'-DDNU	rel_int	57	248.016	250.013	1.56	0.25
16	Dacthal	rel_int	57	331.8991	333.8961	2.08	0.25
17	Chlorpyrifos	rel_int	57	313.9574	315.9545	1.44	0.25
18	Octachlorostyrene	rel_int	57	342.779	344.7761	1.04	0.25
19	Heptachlor Epoxide B	rel_int	57	352.844	354.841	1.2	0.25
20	Heptachlor Epoxide A	rel_int	57	352.844	354.841	1.2	0.25
21	Oxychlorane	rel_int	57	386.805	388.802	1.02	0.25
22	4,4'-DDMU	rel_int	62	247.0081	249.0052	1.56	0.25
23	trans-Chlordane	rel_int	58	262.8571	264.8541	1.56	0.25
24	cis-Chlordane	rel_int	58	262.8571	264.8541	1.56	0.25
25	trans-Nonachlor	rel_int	58	262.8571	264.8541	1.56	0.25
26	Dieldrin	rel_int	59	262.8571	264.8541	1.56	0.25
27	Endrin	rel_int	60	262.8571	264.8541	1.56	0.25
28	cis-Nonachlor	rel_int	58	262.8571	264.8541	1.56	0.25
29	Endosulfan I	rel_int	61	276.8726	278.8697	1.56	0.25
30	Endosulfan II	rel_int	61	276.8726	278.8697	1.56	0.25
31	Endosulfan Sulfate	rel_int	61	276.8726	278.8697	1.56	0.25
32	24'-DDE	rel_int	62	246.0003	247.9974	1.56	0.25
33	44'-DDE	rel_int	62	246.0003	247.9974	1.56	0.25
34	24'-DDD	rel_int	63	235.0082	237.0053	1.56	0.25
35	44'-DDD	rel_int	63	235.0082	237.0053	1.56	0.25
36	24'-DDT	rel_int	63	235.0082	237.0053	1.56	0.25
37	44'-DDT	rel_int	64	235.0082	237.0053	1.56	0.25
38	Endrin Aldehyde	rel_int	60	247.8521	249.8492	0.64	0.35
39	Endrin Ketone	rel_int	60	247.8521	249.8492	0.64	0.35
40	Methoxychlor	rel_int	65	227.1072	228.1106	6.2	0.35
41	Dicofol	rel_int	65	251.003	253.0001	1.56	0.35
42	Mirex	rel_int	66	271.8103	273.8073	1.3	0.25
43	Parlar-26	rel_int	66	304.9039	306.901	1.56	0.35
44	Parlar-50	rel_int	66	338.8649	340.862	1.25	0.35
45	Parlar-62	rel_int	66	338.8649	340.862	1.25	0.35

Laboratory Surrogate							
46	1,3-Dibromobenzene	rel_int	67	233.868	235.8659	0.51	0.25
47	Endrin Ketone	rel_int	69	316.904	318.901	1.56	0.35
Field Surrogate							
48	1,3,5-Tribromobenzene	rel_int	67	313.7764	315.7744	1.03	0.25
49	1,2,4,5-Tetrabromobenzene	rel_int	68	312.7686	314.7666	1.03	0.25
50	delta-BHC	rel_int	69	218.9116	220.9086	2.1	0.25
GPC Recovery Standard							
51	13C12-PCB-133	rel_int	69	299.947	301.944	0.78	0.25
Extraction Standard							
52	13C6-Pentachlorobenzene	rel_int	67	255.8693	257.8663	1.56	0.25
53	13C6-Hexachlorobenzene	rel_int	67	289.8303	291.8273	1.23	0.25
54	13C6-alpha-BHC	rel_int	67	224.9317	226.9287	2.1	0.25
55	d6-gamma-BHC	rel_int	67	223.943	225.94	2.1	0.25
56	13C10-Heptachlor	rel_int	68	276.8269	278.824	1.25	0.25
57	13C10-Oxychlorane	rel_int	68	396.8385	398.8355	1.02	0.25
58	13C10-trans-Nonachlor	rel_int	69	269.8804	271.8775	1.56	0.25
59	13C12-Dieldrin	rel_int	69	269.8804	271.8775	1.56	0.25
60	13C12-Endrin	rel_int	69	269.8804	271.8775	1.56	0.25
61	13C9-Endosulfan-II	rel_int	69	284.8995	286.8965	1.56	0.25
62	13C12-44'-DDE	rel_int	69	258.0405	260.0376	1.56	0.25
63	13C12-44'-DDD	rel_int	69	247.0483	249.0454	1.56	0.25
64	13C12-44'-DDT	rel_int	69	247.0483	249.0454	1.56	0.25
65	d6-Methoxychlor	rel_int	69	233.145	234.1484	6.2	0.35
66	13C10-Mirex	rel_int	69	276.8269	278.824	1.3	0.35
Injection Standard							
67	13C12-PCB-9	abs_int	100	234.0406	236.0376	1.52	0.15
68	13C12-PCB-52	abs_int	100	301.9625	303.9597	0.77	0.15
69	13C12-PCB-101	abs_int	100	337.9206	339.9176	1.56	0.15

Data Calculations:

a) Analyte Concentrations:

The relative response factor of each target relative to the standard against which it is to be calculated is determined using the area responses of both quantification ions via equation 9.1.

In cases where a native target is calculated against an exact labelled analogue, the quantification will be considered to be by isotope dilution. In other cases, the quantification will be considered to be by internal standard.

$$\text{RRF} = \frac{(A_{1_t} + A_{2_t}) C_s}{(A_{1_s} + A_{2_s}) C_t} \quad \text{Equ. 9.1}$$

Where,

$A_{1_t} + A_{2_t}$ = The areas of the two quantification ions for the target analyte

$A_{1_s} + A_{2_s}$ = The areas of the two quantification ions for the labelled compound against which the target analyte will be calculated.

C_t = The concentration in the calibration standard of the target analyte.

C_s = The concentration in the calibration standard of the labelled compound against which the target will be calculated.

For all analytes to be quantified and from the initial calibration series of standard injections, a table of RRFs is prepared. The relative standard deviation (%RSD, or the coefficient of variance) is checked to confirm that appropriate method criteria has been met as listed in Table 3. The average of the five or six levels of for each analyte, RRF_{av} is applied for quantification of samples according to Equations 9.2 and 9.3 below.

$$\text{Amount in sample (ng)} = \frac{(A_{1_n} + A_{2_n}) Q_l}{(A_{1_l} + A_{2_l}) (\text{RRF}_{av})} \quad \text{Equ. 9.2}$$

$$\text{Concentration in sample (ng/g or ng/L)} = \frac{(A_{1_n} + A_{2_n}) Q_l}{(A_{1_l} + A_{2_l}) (\text{RRF}_{av}) (W_s)} \quad \text{Equ. 9.3}$$

Where,

Q_l = The amount (pg) of labelled compound added to the sample

W_s = The weight (g) or volume (l) of sample

b) Extraction, Clean-up, and Sampling Standard Recovery Calculation:

The extraction, clean-up, and sampling standard recoveries are determined by Equation 9.4 below.

$$\% \text{ Recovery} = (\text{Amount in sample}) / (\text{Amount added to sample}) \times 100 \quad \text{Equ. 9.4}$$

c) Estimated Detection Limit

$$\text{EDL} = \frac{2.5 \times H_x \times Q_{es}}{H_{es} \times W \times \text{RFF}_{av}} \quad \text{Equ. 9.5}$$

Where,

EDL = estimated detection limit for native targets

H_x = sum of the height of the noise level for each quantification ions for the unlabeled target

H_{es} = Sum of the heights of responses of both quantification ions for the labelled extraction standard.

W = weight of volume of sample

RRF_{av} = average relative response factor

Q_{es} = Amount of extraction standard added

Chromatogram Annotation Codes

All manually integrated peaks are expanded and reprinted with the following annotations:

* Analyst Initials AA
 * Date YYMMDD
 * integration code CC

The Syntax is: Example:
 AAYYMMDDCC SK111220MB

Code	Mnemonic	Description
MB	Manual Baseline	The peak was manually integrated because the initial baseline was determined incorrectly by the software
MS	Manual Split	The peak was manually integrated because the peak was incorrectly or not split by the software
MJ/MC	Manual Join/Manual Combine	The peak was manually integrated because the peak was split by the software and the peak should be integrated as a single peak
MA	Manual Add	The peak was manually integrated because the signal:noise ratio was judged to be >2.5
MD	Manual Delete	The peak was excluded because the signal:noise ratio was judged to be <2.5
MX	Manual Exclude	The peak was excluded due to an interference
MT	Manual Time	The peak retention time was manually chosen

The following explanatory annotation codes may appear on the chromatograms of peaks that have been reviewed:

Code	Mnemonic	Description
+	Detected Peak	A peak was detected at this mass and retention time that was above 2.5:1 signal to noise
<	Below Detection Limit	The signal at this mass and retention time was below 2.5:1 signal to noise
EMPC	Estimated Maximum Possible Concentration	The signal at this mass and retention time is an interference such that the target compound could not be confirmed
X-RT	Not Detected due to Retention Time non-conformance	The signal at this retention time could not be used to positively identify the target compound because of retention time non-conformance (apex of quantification and confirmation ions do not maximize within the same two seconds, or the retention time of the peak does not fall within the expected range with respect to its labeled analogue)
X-LOC	Not Detected due to interference from a higher level of chlorination	The signal at this retention time is attributable to a fragment from a co-eluting compound at a higher level of chlorination, and cannot be used to positively identify the target. The result is expressed as an Estimated Maximum Possible Concentration (EMPC)
X-DPE	Not Detected due to diphenyl ether interference	The signal at this retention time is attributable to interference from a chlorinated diphenyl ether, and cannot be used to positively identify the target. The result is expressed as an Estimated Maximum Possible Concentration (EMPC)
X-IF	Not Detected due to interference	The signal at this retention time is attributable to a co-eluting interference, and cannot be used to positively identify the target. The result is expressed as an Estimated Maximum Possible Concentration (EMPC)

SVOC DATA PACKAGE

SECTION 4: CALIBRATION DATA

Including:

- for Multi-Point Calibration(s)
 - Multi-Point Calibration Tables
 - Individual Quantitation Reports

- for Continuing Calibration(s)
 - Individual Quantitation Reports

ALS Life sciences

Calibration Summary Report

Calibration Level	Filename	Run Date
CS-1	6-180809A02	09-Aug-2018 14:12
CS-2	6-180809A01	09-Aug-2018 13:43
CS-3	6-180809A07	09-Aug-2018 17:12
CS-4	6-180809A06	09-Aug-2018 16:39
CS-5	6-180809A05	09-Aug-2018 16:05
CS-6	6-180809A04	09-Aug-2018 15:32
CS-7	6-180809A03	09-Aug-2018 15:03

Approved:	<i>R. Bakhtiari</i>
	--e-signature--
	13-Aug-2018

Target Analytes	Relative Response Factors							Mean	% RSD
	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7		
2,4'-DDE	1.594	1.595	1.484	1.554	1.618	1.632		1.580	3%
4,4'-DDE	1.241	1.230	1.138	1.193	1.231	1.223		1.209	3%
2,4'-DDD	1.305	1.217	1.209	1.273	1.295	1.286		1.264	3%
4,4'-DDD	1.235	1.284	1.176	1.238	1.247	1.259		1.240	3%
2,4'-DDT	0.927	0.941	0.942	1.000	1.019	1.004	1.079	0.987	5%
4,4'-DDT	1.011	0.978	0.889	0.945	0.949	0.914	0.993	0.954	5%
Extraction Standards									
4,4'-DDE, 13C12-	2.672	2.597	3.040	3.126	2.657	2.509		2.767	9%
4,4'-DDD, 13C12-	2.580	2.574	2.911	3.036	2.630	2.622	2.727	2.726	7%
4,4'-DDT, 13C12-	2.219	2.209	2.636	2.740	2.341	2.362	2.424	2.419	8%

ALS Life sciences

Calibration Report

ALS Sample ID **H6-18-CS1-053**
 Analysis Method EPA 1699 (mod)
 Analysis Type Calibration

Filename 6-180809A02 Inst # HRMS-6 Column HP5MSUSR163634H Run Date 09-Aug-2018 14:12

Approved: *R. Bakhtiari*
 --e-signature--
 13-Aug-2018

Target Analytes	Ret. Time	Ion Ratio	Concentration ng/mL	Response	RRF
2,4'-DDE	21.02	1.55	2.00	1.19E+05	1.594
4,4'-DDE	21.95	1.54	2.00	9.26E+04	1.241
2,4'-DDD	22.18	1.43	2.00	9.41E+04	1.305
4,4'-DDD	23.15	1.55	2.00	8.90E+04	1.235
2,4'-DDT	23.25	1.47	2.00	6.68E+04	0.927
4,4'-DDT	24.22	1.44	2.00	6.27E+04	1.011
Extraction Standards					
4,4'-DDE, 13C12-	21.94	1.54	250.00	9.33E+06	2.672
4,4'-DDD, 13C12-	23.15	1.59	250.00	9.01E+06	2.580
4,4'-DDT, 13C12-	24.2	1.55	250.00	7.75E+06	2.219
Labeled Injection Standards					
13C1-PCB-101 (IS)	21.07	1.65	100.00	1.40E+06	

ALS Life sciences

Calibration Report

ALS Sample ID **H6-18-CS2-053**
 Analysis Method EPA 1699 (mod)
 Analysis Type Calibration

Filename 6-180809A01 Inst # HRMS-6 Column HP5MSUSR163634H Run Date 09-Aug-2018 13:43

Approved: *R. Bakhtiari*
 --e-signature--
 13-Aug-2018

Target Analytes	Ret. Time	Ion Ratio	Concentration ng/mL	Response	RRF
2,4'-DDE	21	1.58	7.50	4.38E+05	1.595
4,4'-DDE	21.94	1.62	7.50	3.38E+05	1.230
2,4'-DDD	22.18	1.56	7.50	3.31E+05	1.217
4,4'-DDD	23.15	1.56	7.50	3.49E+05	1.284
2,4'-DDT	23.23	1.53	7.50	2.56E+05	0.941
4,4'-DDT	24.22	1.53	7.50	2.28E+05	0.978
Extraction Standards					
4,4'-DDE, 13C12-	21.92	1.57	250.00	9.15E+06	2.597
4,4'-DDD, 13C12-	23.14	1.59	250.00	9.07E+06	2.574
4,4'-DDT, 13C12-	24.2	1.55	250.00	7.78E+06	2.209
Labeled Injection Standards					
13C1-PCB-101 (IS)	21.06	1.67	100.00	1.41E+06	

ALS Life sciences

Calibration Report

ALS Sample ID **H6-18-CS3-053**
 Analysis Method EPA 1699 (mod)
 Analysis Type Calibration

Filename 6-180809A07 Inst # HRMS-6 Column HP5MSUSR163634H Run Date 09-Aug-2018 17:12

Approved: *R. Bakhtiari*
 --e-signature--
 13-Aug-2018

Target Analytes	Ret. Time	Ion Ratio	Concentration ng/mL	Response	RRF
2,4'-DDE	21	1.56	20.00	1.10E+06	1.484
4,4'-DDE	21.94	1.56	20.00	8.46E+05	1.138
2,4'-DDD	22.18	1.58	20.00	8.60E+05	1.209
4,4'-DDD	23.15	1.60	20.00	8.37E+05	1.176
2,4'-DDT	23.23	1.53	20.00	6.71E+05	0.942
4,4'-DDT	24.22	1.48	20.00	5.73E+05	0.889
Extraction Standards					
4,4'-DDE, 13C12-	21.92	1.55	250.00	9.29E+06	3.040
4,4'-DDD, 13C12-	23.14	1.60	250.00	8.89E+06	2.911
4,4'-DDT, 13C12-	24.2	1.55	250.00	8.05E+06	2.636
Labeled Injection Standards					
13C1-PCB-101 (IS)	21.06	1.66	100.00	1.22E+06	

ALS Life sciences

Calibration Report

ALS Sample ID **H6-18-CS4-053**
 Analysis Method EPA 1699 (mod)
 Analysis Type Calibration

Filename 6-180809A06 Inst # HRMS-6 Column HP5MSUSR163634H Run Date 09-Aug-2018 16:39

Approved: *R. Bakhtiari*
 --e-signature--
 13-Aug-2018

Target Analytes	Ret. Time	Ion Ratio	Concentration ng/mL	Response	RRF
2,4'-DDE	21	1.55	50.00	3.23E+06	1.554
4,4'-DDE	21.94	1.56	50.00	2.48E+06	1.193
2,4'-DDD	22.17	1.58	50.00	2.57E+06	1.273
4,4'-DDD	23.15	1.60	50.00	2.50E+06	1.238
2,4'-DDT	23.23	1.59	50.00	2.02E+06	1.000
4,4'-DDT	24.22	1.51	50.00	1.72E+06	0.945
Extraction Standards					
4,4'-DDE, 13C12-	21.92	1.56	250.00	1.04E+07	3.126
4,4'-DDD, 13C12-	23.14	1.59	250.00	1.01E+07	3.036
4,4'-DDT, 13C12-	24.2	1.53	250.00	9.11E+06	2.740
Labeled Injection Standards					
13C1-PCB-101 (IS)	21.06	1.70	100.00	1.33E+06	

ALS Life sciences

Calibration Report

ALS Sample ID **H6-18-CS5-053**
 Analysis Method EPA 1699 (mod)
 Analysis Type Calibration

Filename 6-180809A05 Inst # HRMS-6 Column HP5MSUSR163634H Run Date 09-Aug-2018 16:05

Approved: *R. Bakhtiari*
 --e-signature--
 13-Aug-2018

Target Analytes	Ret. Time	Ion Ratio	Concentration ng/mL	Response	RRF
2,4'-DDE	21	1.56	150.00	9.67E+06	1.618
4,4'-DDE	21.94	1.58	150.00	7.36E+06	1.231
2,4'-DDD	22.18	1.66	150.00	7.67E+06	1.295
4,4'-DDD	23.15	1.65	150.00	7.38E+06	1.247
2,4'-DDT	23.25	1.62	150.00	6.03E+06	1.019
4,4'-DDT	24.22	1.59	150.00	5.00E+06	0.949
Extraction Standards					
4,4'-DDE, 13C12-	21.94	1.55	250.00	9.97E+06	2.657
4,4'-DDD, 13C12-	23.14	1.59	250.00	9.86E+06	2.630
4,4'-DDT, 13C12-	24.2	1.56	250.00	8.78E+06	2.341
Labeled Injection Standards					
13C1-PCB-101 (IS)	21.06	1.63	100.00	1.50E+06	

ALS Life sciences

Calibration Report

ALS Sample ID **H6-18-CS6-053**
 Analysis Method EPA 1699 (mod)
 Analysis Type Calibration

Filename 6-180809A04 Inst # HRMS-6 Column HP5MSUSR163634H Run Date 09-Aug-2018 15:32

Approved: *R. Bakhtiari*
 --e-signature--
 13-Aug-2018

Target Analytes	Ret. Time	Ion Ratio	Concentration ng/mL	Response	RRF
2,4'-DDE	21.02	1.56	400.00	2.54E+07	1.632
4,4'-DDE	21.95	1.59	400.00	1.90E+07	1.223
2,4'-DDD	22.18	1.63	400.00	2.09E+07	1.286
4,4'-DDD	23.15	1.63	400.00	2.05E+07	1.259
2,4'-DDT	23.25	1.64	400.00	1.63E+07	1.004
4,4'-DDT	24.22	1.57	400.00	1.34E+07	0.914
Extraction Standards					
4,4'-DDE, 13C12-	21.94	1.54	250.00	9.72E+06	2.509
4,4'-DDD, 13C12-	23.15	1.57	250.00	1.02E+07	2.622
4,4'-DDT, 13C12-	24.2	1.56	250.00	9.15E+06	2.362
Labeled Injection Standards					
13C1-PCB-101 (IS)	21.07	1.62	100.00	1.55E+06	

ALS Life sciences

Calibration Report

ALS Sample ID **H6-18-CS7-053**
 Analysis Method EPA 1699 (mod)
 Analysis Type Calibration

Filename	Inst #	Column	Run Date
6-180809A03	HRMS-6	HP5MSUSR163634H	09-Aug-2018 15:03

Approved: *R. Bakhtiari*
 --e-signature--
 13-Aug-2018

Target Analytes	Ret. Time	Ion Ratio	Concentration ng/mL	Response	RRF
2,4'-DDE					
4,4'-DDE					
2,4'-DDD					
4,4'-DDD					
2,4'-DDT	23.25	1.63	1200.00	5.60E+07	1.079
4,4'-DDT	24.22	1.59	1200.00	4.58E+07	0.993
Extraction Standards					
4,4'-DDE, 13C12-	0				
4,4'-DDD, 13C12-	23.14	1.60	250.00	1.08E+07	2.727
4,4'-DDT, 13C12-	24.2	1.56	250.00	9.60E+06	2.424
Labeled Injection Standards					
13C1-PCB-101 (IS)	21.06	1.64	100.00	1.58E+06	

ALS Life sciences

Continuing Calibration Report

Sample Name	CCV	Sampling Date	n/a	
ALS Sample ID	H6-18-RS1-053	Extraction Date	n/a	
Analysis Method	EPA 1699 (mod)	Sample Size	1	n/a
Analysis Type	CCV	Percent Solid	n/a	
Sample Matrix	QC	Split Ratio	1	

Approved:
R. Bakhtiari
 --e-signature--
 13-Aug-2018

Run Information	Run 1
Filename	6-180809A08
Run Date	09-Aug-18 17:45
Final Volume	1020 uL
Dilution Factor	1
Analysis Units	%
Instrument - Column	HRMS-6 HP5MSUSR163634H

Target Analytes	ng/mL	Ret. Time	% Rec	Limits	Flags
2,4'-DDE	0				
4,4'-DDE	150	21.94	92	75-125	
2,4'-DDD	0				
4,4'-DDD	150	23.14	95	75-125	
2,4'-DDT	0				
4,4'-DDT	150	24.20	89	75-125	
Extraction Standards					
	ng/mL				
4,4'-DDE, 13C12-	250	21.92	111	70-130	
4,4'-DDD, 13C12-	250	23.14	111	70-130	
4,4'-DDT, 13C12-	250	24.20	115	70-130	

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Continuing Calibration Report

Sample Name	CCV	Sampling Date	n/a	
ALS Sample ID	H6-18-CCV-0745	Extraction Date	n/a	
Analysis Method	EPA 1699 (mod)	Sample Size	1	n/a
Analysis Type	CCV	Percent Solid	n/a	
Sample Matrix	QC	Split Ratio	1	

Approved:
R. Bakhtiari
 --e-signature--
 13-Aug-2018

Run Information	Run 1
Filename	6-180809A09
Run Date	09-Aug-18 18:35
Final Volume	1020 uL
Dilution Factor	1
Analysis Units	%
Instrument - Column	HRMS-6 HP5MSUSR163634H

Target Analytes	ng/mL	Ret.		Limits	
		Time	% Rec	Flags	Flags
2,4'-DDE	50	21.02	94	75-125	
4,4'-DDE	50	21.95	98	75-125	
2,4'-DDD	50	22.18	95	75-125	
4,4'-DDD	50	23.15	100	75-125	
2,4'-DDT	50	23.25	98	75-125	
4,4'-DDT	50	24.22	98	75-125	
Extraction Standards					
	ng/mL				
4,4'-DDE, 13C12-	250	21.94	110	70-130	
4,4'-DDD, 13C12-	250	23.14	110	70-130	
4,4'-DDT, 13C12-	250	24.20	114	70-130	

ALS Life sciences

Continuing Calibration Report

Sample Name	CCV	Sampling Date	n/a	
ALS Sample ID	H6-18-CCV-0747	Extraction Date	n/a	
Analysis Method	EPA 1699 (mod)	Sample Size	1	n/a
Analysis Type	CCV	Percent Solid	n/a	
Sample Matrix	QC	Split Ratio	1	

Approved:
R. Bakhtiari
 --e-signature--
 13-Aug-2018

Run Information		Run 1	
Filename	6-180809A20	Run Date	10-Aug-18 00:35
Final Volume	1020 uL	Dilution Factor	1
Analysis Units	%	Instrument - Column	HRMS-6 HP5MSUSR163634H

Target Analytes	ng/mL	Ret.		Limits	
		Time	% Rec	Flags	
2,4'-DDE	50	21.00	111	75-125	
4,4'-DDE	50	21.94	95	75-125	
2,4'-DDD	50	22.17	138	75-125	
4,4'-DDD	50	23.14	101	75-125	
2,4'-DDT	50	23.23	85	75-125	
4,4'-DDT	50	24.20	98	75-125	
Extraction Standards					
4,4'-DDE, 13C12-	250	21.92	97	70-130	
4,4'-DDD, 13C12-	250	23.14	47	70-130	
4,4'-DDT, 13C12-	250	24.20	31	70-130	

ALS Life sciences

Continuing Calibration Report

Sample Name	CCV	Sampling Date	n/a	
ALS Sample ID	H6-18-CCV-0749	Extraction Date	n/a	
Analysis Method	EPA 1699 (mod)	Sample Size	1	n/a
Analysis Type	CCV	Percent Solid	n/a	
Sample Matrix	QC	Split Ratio	1	

Approved:
R. Bakhtiari
 --e-signature--
 13-Aug-2018

Run Information	Run 1
Filename	6-180809A30
Run Date	10-Aug-18 06:19
Final Volume	1020 uL
Dilution Factor	1
Analysis Units	%
Instrument - Column	HRMS-6 HP5MSUSR163634H

Target Analytes	ng/mL	Ret.		Limits	
		Time	% Rec	Flags	Flags
2,4'-DDE	50	21.00	101	75-125	
4,4'-DDE	50	21.94	92	75-125	
2,4'-DDD	50	22.18	120	75-125	
4,4'-DDD	50	23.15	94	75-125	
2,4'-DDT	50	23.23	93	75-125	
4,4'-DDT	50	24.22	94	75-125	
Extraction Standards					
	ng/mL				
4,4'-DDE, 13C12-	250	21.94	117	70-130	
4,4'-DDD, 13C12-	250	23.14	71	70-130	
4,4'-DDT, 13C12-	250	24.20	51	70-130	

SVOC DATA PACKAGE

SECTION 5: QC SAMPLE DATA

Including:

- Laboratory Method Blank Analysis Reports
- Laboratory Control Sample Analysis Reports
- Matrix Spike Analysis Reports
- Other QC Sample Analysis Reports (where applicable)

ALS Life sciences

Laboratory Method Blank Analysis Report

Sample Name	Method Blank	Sampling Date	n/a		
ALS Sample ID	WG2831102-1	Extraction Date	25-Jul-18		
Analysis Method	EPA 1699 (mod)	Sample Size	5.79	g	
Analysis Type	Blank	Percent Solid	100.0%		
Sample Matrix	QC	Split Ratio	1		
					Approved: <i>R. Bakhtiari</i> --e-signature-- 13-Aug-2018

Run Information	Run 1
Filename	6-180809A15
Run Date	09-Aug-18 21:50
Final Volume	1020 uL
Dilution Factor	1
Analysis Units	ng/g
Instrument - Column	HRMS-6 HP5MSUSR163634H

Target Analytes	Ret. Time	Conc. ng/g	EDL ng/g	Flags	EMPC ng/g	LQL
2,4'-DDE	NotFnd	<0.010	0.010	U	0.35	
4,4'-DDE	NotFnd	<0.011	0.011	U	0.35	
2,4'-DDD	NotFnd	<0.018	0.018	U	0.35	
4,4'-DDD	23.14	<0.013	0.013	M,U	0.0024	0.35
2,4'-DDT	NotFnd	<0.017	0.017	U	0.35	
4,4'-DDT	NotFnd	<0.041	0.041	U	0.35	
Extraction Standards						
4,4'-DDE, 13C12-	125	21.92	82	21-125	M	
4,4'-DDD, 13C12-	125	23.14	98	5-150		
4,4'-DDT, 13C12-	125	24.20	92	5-120	M	

EDL Indicates the Estimated Detection Limit, based on the measured background noise for this target in this sample.
 LQL Lower Quantification Limit, based on the lowest calibration level corrected for sample size, splits and dilutions.
 M Indicates that a peak has been manually integrated.
 U Indicates that this compound was not detected above the EDL.

EMPC Estimated Maximum Possible Concentration – elevated detection limit due to interference or positive id criterion failure

ALS Life sciences

Laboratory Control Sample Analysis Report

Sample Name	Laboratory Control Sample	Sampling Date	n/a		
ALS Sample ID	WG2831102-2	Extraction Date	25-Jul-18		
Analysis Method	EPA 1699 (mod)	Sample Size	1	n/a	
Analysis Type	LCS	Percent Solid	50.2%		
Sample Matrix	QC	Split Ratio	1		
					Approved: <i>R. Bakhtiari</i> --e-signature-- 13-Aug-2018

Run Information	Run 1
Filename	6-180809A12
Run Date	09-Aug-18 20:10
Final Volume	1020 uL
Dilution Factor	1
Analysis Units	%
Instrument - Column	HRMS-6 HP5MSUSR163634H

Target Analytes	ng	Ret. Time	% Rec	Limits	Flags
2,4'-DDE	25	21.00	112	50-120	
4,4'-DDE	25	21.94	96	50-120	
2,4'-DDD	25	22.17	102	42-120	
4,4'-DDD	25	23.14	98	42-120	
2,4'-DDT	25	23.23	79	50-120	
4,4'-DDT	25	24.20	96	50-120	
Extraction Standards					
4,4'-DDE, 13C12-	125	21.92	58	21-125	
4,4'-DDD, 13C12-	125	23.14	78	13-200	
4,4'-DDT, 13C12-	125	24.20	66	13-200	

SVOC DATA PACKAGE

SECTION 6: INTERNAL RECORDS

Including:

- Prep Logs
- Independent calculation checks
- Others as listed below:

Extraction Workup Sheet

Batch ID: WG2831102

Analysis: Sediments - OCP

BU-TM-1110 Overall HR Prep

Analyst: Marco Micketts

Date: 25-Jul-2018

SUBSAMPLING		
Sample I.D.	Client I.D.	Subsample Size (g)
WG2831102-1	Method Blank	10.11
WG2831102-2	Laboratory Control Sample	10.30
WG2831102-3	Extraction and Injection STD.	—
L2133758-1	PDI-SC-S033-0TO2	10.19
WG2831102-4	Duplicate(L2133758-1)	10.43
L2133758-2	PDI-SC-S033-2TO3	10.07
L2133758-3	PDI-SC-S033-3TO4	10.28
L2134269-1	PDI-SC-S036-0 TO 1.4	10.15
L2134269-2	PDI-SC-S036-1.4 TO 3.4	10.30
L2134269-3	PDI-SC-S036-3.4 TO 5.2	10.48
L2134269-4	PDI-SC-S022-0 TO 2	10.05
L2134269-5	PDI-SC-S022-2 TO 4	10.13
L2134269-6	PDI-SC-S022-4 TO 6	10.32
Samples may be processed on the rotovaps and N-Evaps at 50 degrees		

BATCH TRACKING

	Date/Time/Initials
Subsampling:	MM 25-Jul-2018 11:00pm
Balance ID	3985
Client Labels Checked:	MM
Samples Spiked	RM 3:15 PM 25-Jul-18
Soxhlet start time:	RM 3:30 PM 25-Jul-18
Soxhlet reflux properly:	RM 3:30 PM 25-Jul-18
Soxhlet end time:	26-Jul-2018 @ 8:00 AM
Rotovap + temp check:	JAZ 27-July-2018
Sili Column:	JAZ 27-July-2018
Mini Acid	CA 27-July-2018 @ 8:00 AM
Robo-Vialing:	CA 27-Jul-2018 @ 11:22 AM
Update to LIMS:	CA 27-Jul-2018 @ 2:00 PM

* See comments.

Batch ID: WG2831102

DX Extraction Standard:

Sample I.D.	Volume (ul)	(Checkmark) Spiked
WG2831102-1	20	✓
WG2831102-2	20	✓
WG2831102-3	20	✓
L2133758-1	20	✓
WG2831102-4	20	✓
L2133758-2	20	✓
L2133758-3	20	✓
L2134269-1	20	✓
L2134269-2	20	✓
L2134269-3	20	✓
L2134269-4	20	✓
L2134269-5	20	✓
L2134269-6	20	✓
	20	
	20	
	20	
	20	
	20	
	20	

Syringe ID: 137

Standard: HROCP-ES#1-023D

Spike Date: 25-July-2018

Spike Witnessing

Chemist's Initials: RM

Witness's Initials: EF

Correct Syringe Obtained: EF

Correct Standard Obtained: EF

Correct Technique Followed: EF

Batch ID: WG2831102

Syringe ID: 202
 Standard: HROCP-NS#1-01B
 Date & Initials: 25-July-2018 RM

25-July-2018
 RM

DX Native Standard:

(Checkmark)

Sample I.D.	Volume (ul)	Spiked
WG2831102-2	20	✓
WG2831102-3	20	✓

DX Injection Standard:

(Checkmark)

Sample I.D.	Volume (ul)	Spiked
WG2831102-1	20	✓
WG2831102-2	20	✓
WG2831102-3	20	✓
L2133758-1	20	✓
WG2831102-4	20	✓
L2133758-2	20	✓
L2133758-3	20	✓
L2134269-1	20	✓
L2134269-2	20	✓
L2134269-3	20	✓
L2134269-4	20	✓
L2134269-5	20	✓
L2134269-6	20	✓

Syringe ID: 196
 Standard: HROCP-IS#1-014C
 Date & Initials: 27 July-2018 RM

Correct Syringe Obtained: Chemist's Initials: [Signature]
 Correct Standard Obtained: Chemist's Initials: [Signature]
 Correct Technique Followed: Chemist's Initials: [Signature]

Procedure:

This batchsheet is a guideline only. Please see test procedure for complete set of instructions.

SubSampling

-
- Subsample 10g weight wet (5g dry weight)
- Spike the samples with Extraction/Native Standards.
- Soxhlet extract in DCM for 16 hours.
- Rotovap down to ~4ml. Transfer with hexane rinses to ctube.
- Reduce gently to 1mL

Sili Carb Column

- Load sample with 3x1mL hexane rinses
 - F1 = 25 mL of Hexane
 - F2 = 50mL of 1:1 DCM:Hexane
- Reduce silicarb F2 to 1mL.

Mini Acid Silica Column

- Load sample with 3x1mL hexane rinses
 - Elute with 15 ml of DCM

Robo-vial

- Reduce to 1mL
- Vortex well and transfer to robo-vial without rinses.
- Spike with Injection standard. Mark level and submit. **FV=1020uL**

Reagent Lot Numbers:

Reagent	Lot#	Manufacturer
Acetone	151822	FISW
Hexane	174496	FISW
DCM	103221	CAL
Toluene	103253	CAL
Nonane	ORG-WAKONON- /	
1:1 DCM:HEX	ORG-DH2- 500	CAL
Sodium Sulphate	ORG-SSU- 874	CAL
Acid Silica	ORG-ASI- 7767	CAL
Neutral Silica	ORG-NSI- 1732	CAL
Alumina	ORG-ALU- /	
Chromacarb	ORG-CC- /	
Corn Oil	ORG-CO- /	

Comments:

WG2769307 - OCP fish mealPREP
 30-Apr-18/JP,MK

WG:	Prep Analyst:
Analysis:	Date:

27 July 2018 - L2133758-1,2,3 } were yellow in colour
W4 } ~~were~~ when submitted.
L2134269-2,3 } L2133758-3 was the darkest
in colour. *AS*

	Very Good	meets Method Req.	Some Outliers	very Poor	Comments / was sp/patch sent for rework? Why?
MB					
LCS					
DUP					
ES rec					

ALS Life sciences

Sample Calculation Report

CS3 RRF Check

Approved: *R. Bakhtiari*
--e-signature--
13-Aug-2018

$$\begin{aligned}
 \text{RRF} &= \frac{\text{Response of 4,4'-DDE}}{\text{Response of 13C12-4,4'DDE}} \times \frac{\text{Concentration of 13C2-4,4'DDE}}{\text{Concentration of DDE}} \\
 \text{RRF} &= \frac{845652.10}{9287261.50} \times \frac{250.00}{20} = 1.14 \quad \text{Value from TargetLyn x } 1.14
 \end{aligned}$$

Calculation of 4,4'-DDE amount in L2134269-1

$$\begin{aligned}
 \text{ng} &= \frac{\text{Response of 4,4'-DDE}}{\text{Response of 13C12-4,4'DDE}} \times \frac{\text{ng of 13C12-4,4'-DDE spiked}}{\text{Mean RRF} * \text{Sample Size}} \\
 \text{ng/g} &= \frac{1102124.1}{3333622.1} \times \frac{125}{1.21 * 5.29} = 6.460 \quad \text{6.46}
 \end{aligned}$$

Calculation of 13C12-4,4'-DDE Recovery in L2134269-1

$$\begin{aligned}
 \% \text{ Recovery} &= \frac{\text{Response of 13C12-4,4'-DDE}}{\text{Response of 13C12-PCB-101}} \times \frac{\text{ng of 13C12-PCB-101} * 100}{\text{Mean RRF} * \text{Amount Spiked}} \\
 \% \text{ Recovery} &= \frac{3333622.1}{678255.9} \times \frac{50 * 100}{2.77 * 125} = 71 \quad \text{71 \%}
 \end{aligned}$$

SVOC DATA PACKAGE

SECTION 7: SHIPPING/RECEIVING DOCUMENTS

Including:

- Airbills
- Chain-of-Custody Records
- Sample Log-in Sheet(s) - where applicable
- Others as listed below:

L2134269

ALS Burlington 1435 Norjohn Court Unit 1 Burlington, Ontario Canada L7R 0P6 Tel: 1-905-331-3111 Fax: 0		SUBSURFACE SEDIMENT CHAIN OF CUSTODY						COC No: _____ of _____ pages	
1111 3rd Ave Suite 1600 Seattle, WA 98101 Phone (206) 438-2700 Fax: 1-(866) 495-5288 Project Name: Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling Portland, OR Project # 60566335 Study: Subsurface Sediment Sample Type:		Analysis Turnaround Time Calendar (C) or Work Days (W) <u>W</u> <input checked="" type="checkbox"/> 21 days <input type="checkbox"/> Other _____		Date: 7/23/18 Carrier: FedEx					
Sample Identification	Sample Date	Sample Time	Matrix	QC Sample	Sampler's Initials	Total No. of Cont.	Fraction	Particulates, DDs only	Sample Specific Notes:
1 PDI-SC-S036 - 0 to 1.4	7/20/2018	15:50	SC		ED	1		x	
2 PDI-SC-S036 - 1.4 to 3.4	7/20/2018	15:55	SC		ED	1		x	
3 PDI-SC-S036 - 3.4 to 5.2	7/20/2018	16:00	SC		ED	1		x	
4 PDI-SC-S022 - 0 to 2	7/20/2018	13:30	SC		ED	1		x	
5 PDI-SC-S022 - 2 to 4	7/20/2018	13:35	SC		ED	1		x	
6 PDI-SC-S022 - 4 to 6	7/20/2018	13:40	SC		ED	1		x	
Container Type: WMG=Wide Mouth Glass Jar, P=HDPE, PP=Polypropylene, AG=amber glass, G=glass, RC=Resin Collar Preservative: HCl = Hydrochloric Acid, H3PO4 = Phosphoric Acid, HNO3 = Nitric Acid Fraction: D = Dissolved, PRT = Particulate, T = Total (unfiltered)							Sample Disposal <input type="checkbox"/> Return To Client <input checked="" type="checkbox"/> Shipped By Lab <input checked="" type="checkbox"/> Archive For 12 Months		
Special Instructions/QC Requirements & Comments: Separate reports for each lab									
Relinquished by: <i>Michael Mc Coy</i> Relinquished by:		Company: <i>HECOM</i> Company:		Date/Time: <i>7-23-18 1500</i> Date/Time:		Received by: <i>FedEx</i> Received by: <i>AARON PORTER</i>		Company: <i>ALS</i> Company:	
Relinquished by:		Company:		Date/Time:		Received by:		Date/Time: <i>24 July 2018 14:45</i>	

4.2°C

Sample Receiving Log

Date/Time Received	Client ID	Number/Description of Containers	Temp. on Receipt*	Condition of Samples, Courier & Tracking Information	Receiver's Initials	Date/Time Login Completed	Submission ID	Sample ID Range
24-July-2018 14:45	Aecom	6x jars of soil	4.2°C	Hand FedEx 7819 6460 6920	MS	24-July-2018 15:17	L2134269	-1-6

*Temperatures were recorded using: 'Oakton infraPro' dedicated I.R. gun (serial #97800270)
 Other (specify): _____