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

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Client Project Information

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

ALSE Project Information

Project ID:	AECOM100
Contact:	Claire Kocharakkal
Submission ID(s):	L2187236

Final Package Review by:

Date Reviewed: 16-Nov-18

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

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SECTION 1: PROJECT NARRATIVE

ALSE Project Information

Project ID: AECOM100

Contact: Claire Kocharakkal
Submission ID(s): L2187236

Client Project Information

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

Analytical Method: 4,4'- and 2,4'-DDD, DDE and DDT by EPA 1699 (modified)

ALS Sample ID	Client Sample Descriptions	Matrix	Date Sampled	Date Received	Date Extracted	Date Analyzed
L2187236-1	**PDI-SC-S154-4TO6**	**Sediment**	**24-Jul-18**	**25-Oct-18**	**29-Oct-18**	**12-Nov-18**
WG2914952-4	**Duplicate**	**QC**	**n/a**	**n/a**	**29-Oct-18**	**12-Nov-18**
L2187236-2	**PDI-SC-S185-5.5TO6.5**	**Sediment**	**26-Jul-18**	**25-Oct-18**	**29-Oct-18**	**12-Nov-18**
WG2914952-1	**Method Blank**	**QC**	**n/a**	**n/a**	**29-Oct-18**	**12-Nov-18**
WG2914952-2	**Laboratory Control Sample**	**QC**	**n/a**	**n/a**	**29-Oct-18**	**12-Nov-18**

Comments and Notes:
a) Sample Integrity:

The samples were received in good condition at 2.6 degrees C.

b) Instrumental Analysis:

All results have been reported on a dry weight basis.

For the continuing calibration verifications (CCVs) the recoveries of the labelled standards 4,4'-DDE, 13C12- and/or 4,4'-DDT, 13C12- were above the method control limit, likely due to changes in GC loading efficiency through the analytical sequence. As a result, the reported recoveries of these standards are elevated. The recoveries exceed the method control limits for the samples. Native target data are not expected to be biased.

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

16-Nov-18
Date

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SECTION 2: DATA SUMMARY REPORT

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

Certificate of Analysis

ALS Project Contact:	Claire Kocharakkal	**Client Name:**	AECOM
ALS Project ID:	AECOM100	**Client Address:**	1111 Third Avenue Suite 1600 Seattle, WA 98101
ALS WO#:	L2187236		
Date of Report	16-Nov-18		
Date of Sample Receipt	25-Oct-18	**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: SKennedy

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-S154-4TO6**	**Duplicate of PDI-SC-S154-4TO6**	**PDI-SC-S185-5.5TO6.5**
ALS Sample ID	L2187236-1	WG2914952-4	L2187236-2
Sample Size	7.43	7.44	7.24
Sample size units	g	g	g
Percent Solids	73.4%	73.3%	69.9%
Sample Matrix	Sediment	QC	Sediment
Sampling Date	24-Jul-18	n/a	26-Jul-18
Extraction Date	29-Oct-18	29-Oct-18	29-Oct-18
Target Analytes	**ng/g**	**ng/g**	**ng/g**
2,4'-DDE	<0.059	<0.036	<0.069
4,4'-DDE	<0.086	<0.053	<0.10
2,4'-DDD	<0.18	<0.12	<0.21
4,4'-DDD	<0.14	<0.15	<0.16
2,4'-DDT	<0.12	<0.10	<0.14
4,4'-DDT	<0.23	<0.21	<0.27
Extraction Standards	**% Rec**	**% Rec**	**% Rec**
4,4'-DDE, 13C12-	64	96	62
4,4'-DDD, 13C12-	103	123	96
4,4'-DDT, 13C12-	132	142	125

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Quality Control Summary Report

Sample Name	**Method Blank**	**Laboratory Control Sample**
ALS Sample ID	WG2914952-1	WG2914952-2
Sample Size	7.00	7.00
Sample size units	g	g
Percent Solids	n/a	n/a
Sample Matrix	QC	QC
Sampling Date	n/a	n/a
Extraction Date	29-Oct-18	29-Oct-18
Target Analytes	**ng/g**	**% Rec**
2,4'-DDE	<0.17	74
4,4'-DDE	<0.24	95
2,4'-DDD	<0.54	92
4,4'-DDD	<0.26	98
2,4'-DDT	<0.22	81
4,4'-DDT	<0.44	107
Extraction Standards	**% Rec**	**% Rec**
4,4'-DDE, 13C12-	25	44
4,4'-DDD, 13C12-	66	69
4,4'-DDT, 13C12-	79	80

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Continuing Calibration Summary Report

Sample Name	**CVS**	**CCV**	**CCV**
ALS Sample ID	H6-18-RS1-1196	H6-18-CCV-1198	H6-18-CCV-1200
Sample Size	1	1	1
Sample size units	n/a	n/a	n/a
Percent Solids	n/a	n/a	n/a
Sample Matrix	QC	QC	QC
Sampling Date	n/a	n/a	n/a
Extraction Date	n/a	n/a	n/a
Target Analytes	**% Rec**	**% Rec**	**% Rec**
2,4'-DDE		101	102
4,4'-DDE	100	101	101
2,4'-DDD		91	95
4,4'-DDD	100	100	101
2,4'-DDT		93	90
4,4'-DDT	97	96	100
Extraction Standards	**% Rec**	**% Rec**	**% Rec**
4,4'-DDE, 13C12-	105	108	108
4,4'-DDD, 13C12-	120	130	137
4,4'-DDT, 13C12-	138	155	176

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Sample Analysis Report

Sample Name	**PDI-SC-S154-4TO6**	Sampling Date	24-Jul-18	
ALS Sample ID	L2187236-1	Extraction Date	29-Oct-18	Approved:
Analysis Method	EPA 1699 (mod)	Sample Size	7.43 g	*Ella Gdyczynski*
Analysis Type	Sample	Percent Solids	73.4%	--e-signature--
Sample Matrix	Sediment	Split Ratio	1	13-Nov-2018

Run Information	**Run 1**
Filename	6-181112A27
Run Date	12-Nov-18 21:30
Final Volume	1000 uL
Dilution Factor	5
Analysis Units	ng/g
Instrument - Column	HRMS-6 HP5ms USR427822H

Target Analytes		**Ret. Time**	**Conc. ng/g**	**EDL ng/g**	**Flags**	**EMPC ng/g**	**LQL**
2,4'-DDE		NotFnd	<0.059	0.059	U		1.3
4,4'-DDE		NotFnd	<0.086	0.086	U		1.3
2,4'-DDD		NotFnd	<0.18	0.18	U		1.3
4,4'-DDD		NotFnd	<0.14	0.14	U		1.3
2,4'-DDT		NotFnd	<0.12	0.12	U		1.3
4,4'-DDT		NotFnd	<0.23	0.23	U		1.3
Extraction Standards	**ng**						
4,4'-DDE, 13C12-	125	11.80	64	21-125			
4,4'-DDD, 13C12-	125	12.39	103	5-150			
4,4'-DDT, 13C12-	125	12.86	132	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

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Sample Analysis Report

Sample Name	**Duplicate of PDI-SC-S154-4TO6**	Sampling Date	n/a	
ALS Sample ID	WG2914952-4	Extraction Date	29-Oct-18	Approved:
Analysis Method	EPA 1699 (mod)	Sample Size	7.44 g	*Ella Gdyczynski*
Analysis Type	Sample	Percent Solids	73.3%	--e-signature--
Sample Matrix	QC	Split Ratio	1	13-Nov-2018

Run Information	**Run 1**
Filename	6-181112A28
Run Date	12-Nov-18 21:50
Final Volume	1000 uL
Dilution Factor	5
Analysis Units	ng/g
Instrument - Column	HRMS-6 HP5ms USR427822H

Target Analytes		**Ret. Time**	**Conc. ng/g**	**EDL ng/g**	**Flags**	**EMPC ng/g**	**LQL**
2,4'-DDE		NotFnd	<0.036	0.036	U		1.3
4,4'-DDE		NotFnd	<0.053	0.053	U		1.3
2,4'-DDD		NotFnd	<0.12	0.12	U		1.3
4,4'-DDD		12.41	<0.15	0.12	J,R	0.15	1.3
2,4'-DDT		NotFnd	<0.10	0.10	U		1.3
4,4'-DDT		NotFnd	<0.21	0.21	U		1.3
Extraction Standards	**ng**						
4,4'-DDE, 13C12-	125	11.81	96	21-125			
4,4'-DDD, 13C12-	125	12.41	123	5-150			
4,4'-DDT, 13C12-	125	12.87	142	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.
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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Sample Analysis Report

Sample Name	**PDI-SC-S185-5.5TO6.5**	Sampling Date	26-Jul-18	
ALS Sample ID	L2187236-2	Extraction Date	29-Oct-18	Approved:
Analysis Method	EPA 1699 (mod)	Sample Size	7.24 g	*Ella Gdyczynski*
Analysis Type	Sample	Percent Solids	69.9%	--e-signature--
Sample Matrix	Sediment	Split Ratio	1	13-Nov-2018

Run Information	**Run 1**
Filename	6-181112A29
Run Date	12-Nov-18 22:10
Final Volume	1000 uL
Dilution Factor	5
Analysis Units	ng/g
Instrument - Column	HRMS-6 HP5ms USR427822H

Target Analytes		**Ret. Time**	**Conc. ng/g**	**EDL ng/g**	**Flags**	**EMPC ng/g**	**LQL**
2,4'-DDE		NotFnd	<0.069	0.069	U		1.4
4,4'-DDE		NotFnd	<0.10	0.10	U		1.4
2,4'-DDD		NotFnd	<0.21	0.21	U		1.4
4,4'-DDD		NotFnd	<0.16	0.16	U		1.4
2,4'-DDT		NotFnd	<0.14	0.14	U		1.4
4,4'-DDT		NotFnd	<0.27	0.27	U		1.4
Extraction Standards	**ng**						
4,4'-DDE, 13C12-	125	11.81	62	21-125			
4,4'-DDD, 13C12-	125	12.40	96	5-150			
4,4'-DDT, 13C12-	125	12.86	125	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

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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-Oxychlordane	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-Oxychlordane	35	70-130	5-144	23-135
	13C10-trans-Nonachlor	35	70-130	17-154	14.136
	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	5-122
13C12-44'-DDE	35	70-130	26-169	21-125
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 that 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 CalibrationVerification:

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 that 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 that 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 =

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

  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 =

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

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 ^{13}C labelled standard
- within +/- 0.008 RRT units of the RRT in the most recent CS4 analysis for targets with their own ^{2}H 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 Abuncance 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	24'-DDE	rel_int	7	246.0003	247.9974	1.56	0.25
2	44'-DDE	rel_int	7	246.0003	247.9974	1.56	0.25
3	24'-DDD	rel_int	8	235.0082	237.0053	1.56	0.25
4	44'-DDD	rel_int	8	235.0082	237.0053	1.56	0.25
5	24'-DDT	rel_int	9	235.0082	237.0053	1.56	0.25
6	44'-DDT	rel_int	9	235.0082	237.0053	1.56	0.25
	Extraction Standard						
7	13C12-44'-DDE	rel_int	10	258.0405	260.0376	1.56	0.25
8	13C12-44'-DDD	rel_int	10	247.0483	249.0454	1.56	0.25
9	13C12-44'-DDT	rel_int	10	247.0483	249.0454	1.56	0.25
	Injection Standard						
10	13C12-PCB-52	abs_int	-	301.9625	303.9597	0.77	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.

$$RRF = \frac{(A1_t + A2_t)\ C_s}{(A1_s + A2_s)\ C_t} \qquad \text{Equ. 9.1}$$

Where,
$A1_t$ + A The areas of the two quantification ions for the target analyte
$A1_s + A2_s$ = 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.

$$\text{Amount in sample (ng)} = \frac{(A1_n + A2_n)\ Q_l}{(A1_l + A2_l)\ (RRF_{av})} \qquad \text{Equ. 9.2}$$

$$\text{Concentration in sample (ng/g or ng/L)} = \frac{(A1_n + A2_n)\ Q_l}{(A1_l + A2_l)\ (RRF_{av})\ (W_S)} \qquad \text{Equ. 9.3}$$

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.

% Recovery = (Amount in sample)/(Amount added to sample) X 100 Equ. 9.4

c) Estimated Detection Limit

$$EDL = \frac{2.5 \times H_X \times Q_{es}}{H_{es} \times W \times RFF_{av}} \qquad \text{Equ. 9.5}$$

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)

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

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	6-181112A02	12-Nov-2018 13:03
CS-2	6-181112A03	12-Nov-2018 13:31
CS-3	6-181112A08	12-Nov-2018 15:08
CS-4	6-181112A07	12-Nov-2018 14:48
CS-5	6-181112A06	12-Nov-2018 14:28
CS-6	6-181112A05	12-Nov-2018 14:08
CS-7	6-181112A04	12-Nov-2018 13:48

Approved: *Ella Gdyczynski*
--e-signature--
13-Nov-2018

Relative Response Factors

Target Analytes	**CS-1**	**CS-2**	**CS-3**	**CS-4**	**CS-5**	**CS-6**	**CS-7**	**Mean**	**% RSD**
2,4'-DDE	1.826	1.765	1.910	1.891	1.933	1.899	2.065	1.898	5%
4,4'-DDE	1.234	1.195	1.322	1.298	1.353	1.361	1.376	1.306	5%
2,4'-DDD	1.709	1.537	1.705	1.601	1.793	1.725	1.718	1.684	5%
4,4'-DDD	1.417	1.342	1.450	1.351	1.487	1.447	1.522	1.431	5%
2,4'-DDT	1.733	1.632	1.587	1.649	1.633	1.794	1.719	1.678	4%
4,4'-DDT	1.247	1.294	1.311	1.222	1.332	1.342	1.365	1.302	4%
Extraction Standards									
4,4'-DDE, 13C12-	0.732	0.665	0.734	0.709	0.799	0.756	0.759	0.736	6%
4,4'-DDD, 13C12-	0.569	0.568	0.744	0.745	0.758	0.789	0.856	0.718	15%
4,4'-DDT, 13C12-	0.217	0.239	0.422	0.394	0.428	0.380	0.410	0.356	25%

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Calibration Report

ALS Sample ID	**H6-18-CS1-1196**
Analysis Method	EPA 1699 (mod)
Analysis Type	Calibration

Filename	Inst #	Column	Run Date	Approved:	*Ella Gdyczynski* --e-signature-- 13-Nov-2018
6-181112A02	HRMS-6	COL#DB5ms-SN#USR427822H	12-Nov-2018 13:03		

Target Analytes	**Ret. Time**	**Ion Ratio**	**Concentration ng/mL**	**Response**	**RRF**
2,4'-DDE	11.31	1.71	2.00	1.68E+04	1.826
4,4'-DDE	11.82	1.47	2.00	1.14E+04	1.234
2,4'-DDD	11.96	1.41	2.00	1.22E+04	1.709
4,4'-DDD	12.41	1.32	2.00	1.01E+04	1.417
2,4'-DDT	12.46	1.46	2.00	4.72E+03	1.733
4,4'-DDT	12.87	1.68	2.00	3.40E+03	1.247
Extraction Standards					
4,4'-DDE, 13C12-	11.81	1.60	250.00	1.15E+06	0.732
4,4'-DDD, 13C12-	12.41	1.65	250.00	8.95E+05	0.569
4,4'-DDT, 13C12-	12.86	1.63	250.00	3.41E+05	0.217
Labeled Injection Standards					
13C12-PCB-52 (IS)	9.91	0.80	100.00	6.29E+05	

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Calibration Report

ALS Sample ID	**H6-18-CS2-1196**
Analysis Method	EPA 1699 (mod)
Analysis Type	Calibration

Filename	Inst #	Column	Run Date	Approved:	*Ella Gdyczynski* --e-signature-- 13-Nov-2018
6-181112A03	HRMS-6	COL#DB5ms-SN#USR427822H	12-Nov-2018 13:31		

Target Analytes	Ret. Time	Ion Ratio	Concentration ng/mL	Response	RRF
2,4'-DDE	11.32	1.57	7.50	7.75E+04	1.765
4,4'-DDE	11.83	1.58	7.50	5.25E+04	1.195
2,4'-DDD	11.97	1.58	7.50	5.76E+04	1.537
4,4'-DDD	12.42	1.62	7.50	5.03E+04	1.342
2,4'-DDT	12.48	1.41	7.50	2.57E+04	1.632
4,4'-DDT	12.88	1.50	7.50	2.04E+04	1.294
Extraction Standards					
4,4'-DDE, 13C12-	11.82	1.59	250.00	1.46E+06	0.665
4,4'-DDD, 13C12-	12.41	1.64	250.00	1.25E+06	0.568
4,4'-DDT, 13C12-	12.87	1.65	250.00	5.25E+05	0.239
Labeled Injection Standards					
13C12-PCB-52 (IS)	9.92	0.82	100.00	8.81E+05	

ALS Life sciences

Calibration Report

ALS Sample ID	**H6-18-CS3-1196**
Analysis Method	EPA 1699 (mod)
Analysis Type	Calibration

Filename	Inst #	Column	Run Date	Approved:	*Ella Gdyczynski* --e-signature-- 13-Nov-2018
6-181112A08	HRMS-6	COL#DB5ms-SN#USR427822H	12-Nov-2018 15:08		

Target Analytes	**Ret. Time**	**Ion Ratio**	**Concentration ng/mL**	**Response**	**RRF**
2,4'-DDE	11.3	1.64	20.00	2.15E+05	1.910
4,4'-DDE	11.81	1.64	20.00	1.48E+05	1.322
2,4'-DDD	11.96	1.58	20.00	1.94E+05	1.705
4,4'-DDD	12.41	1.62	20.00	1.65E+05	1.450
2,4'-DDT	12.46	1.57	20.00	1.02E+05	1.587
4,4'-DDT	12.87	1.51	20.00	8.47E+04	1.311
Extraction Standards					
4,4'-DDE, 13C12-	11.81	1.58	250.00	1.40E+06	0.734
4,4'-DDD, 13C12-	12.4	1.64	250.00	1.42E+06	0.744
4,4'-DDT, 13C12-	12.86	1.63	250.00	8.07E+05	0.422
Labeled Injection Standards					
13C12-PCB-52 (IS)	9.9	0.81	100.00	7.65E+05	

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Calibration Report

ALS Sample ID	**H6-18-CS4-1196**
Analysis Method	EPA 1699 (mod)
Analysis Type	Calibration

Filename	Inst #	Column	Run Date	Approved:	
6-181112A07	HRMS-6	COL#DB5ms-SN#USR427822H	12-Nov-2018 14:48		*Ella Gdyczynski* --e-signature-- 13-Nov-2018

Target Analytes	**Ret. Time**	**Ion Ratio**	**Concentration ng/mL**	**Response**	**RRF**
2,4'-DDE	11.3	1.57	50.00	5.55E+05	1.891
4,4'-DDE	11.81	1.61	50.00	3.81E+05	1.298
2,4'-DDD	11.95	1.62	50.00	4.94E+05	1.601
4,4'-DDD	12.4	1.63	50.00	4.17E+05	1.351
2,4'-DDT	12.46	1.64	50.00	2.69E+05	1.649
4,4'-DDT	12.86	1.59	50.00	2.00E+05	1.222
Extraction Standards					
4,4'-DDE, 13C12-	11.8	1.60	250.00	1.47E+06	0.709
4,4'-DDD, 13C12-	12.4	1.62	250.00	1.54E+06	0.745
4,4'-DDT, 13C12-	12.86	1.63	250.00	8.16E+05	0.394
Labeled Injection Standards					
13C12-PCB-52 (IS)	9.9	0.80	100.00	8.28E+05	

ALS Life sciences

Calibration Report

ALS Sample ID **H6-18-CS5-1196**
Analysis Method EPA 1699 (mod)
Analysis Type Calibration

Filename	Inst #	Column	Run Date	Approved:	
6-181112A06	HRMS-6	COL#DB5ms-SN#USR427822H	12-Nov-2018 14:28		*Ella Gdyczynski* --e-signature-- 13-Nov-2018

Target Analytes	**Ret. Time**	**Ion Ratio**	**Concentration ng/mL**	**Response**	**RRF**
2,4'-DDE	11.3	1.60	150.00	2.20E+06	1.933
4,4'-DDE	11.81	1.59	150.00	1.54E+06	1.353
2,4'-DDD	11.95	1.64	150.00	1.93E+06	1.793
4,4'-DDD	12.41	1.61	150.00	1.60E+06	1.487
2,4'-DDT	12.46	1.59	150.00	9.95E+05	1.633
4,4'-DDT	12.87	1.66	150.00	8.12E+05	1.332
Extraction Standards					
4,4'-DDE, 13C12-	11.8	1.58	250.00	1.90E+06	0.799
4,4'-DDD, 13C12-	12.4	1.62	250.00	1.80E+06	0.758
4,4'-DDT, 13C12-	12.86	1.62	250.00	1.02E+06	0.428
Labeled Injection Standards					
13C12-PCB-52 (IS)	9.9	0.81	100.00	9.49E+05	

ALS Life sciences

Calibration Report

ALS Sample ID	**H6-18-CS6-1196**
Analysis Method	EPA 1699 (mod)
Analysis Type	Calibration

Filename	Inst #	Column	Run Date	Approved:	*Ella Gdyczynski* --e-signature-- 13-Nov-2018
6-181112A05	HRMS-6	COL#DB5ms-SN#USR427822H	12-Nov-2018 14:08		

Target Analytes	**Ret. Time**	**Ion Ratio**	**Concentration ng/mL**	**Response**	**RRF**
2,4'-DDE	11.3	1.61	400.00	4.75E+06	1.899
4,4'-DDE	11.81	1.60	400.00	3.40E+06	1.361
2,4'-DDD	11.95	1.65	400.00	4.50E+06	1.725
4,4'-DDD	12.41	1.68	400.00	3.78E+06	1.447
2,4'-DDT	12.46	1.67	400.00	2.25E+06	1.794
4,4'-DDT	12.87	1.64	400.00	1.69E+06	1.342
Extraction Standards					
4,4'-DDE, 13C12-	11.8	1.57	250.00	1.56E+06	0.756
4,4'-DDD, 13C12-	12.4	1.62	250.00	1.63E+06	0.789
4,4'-DDT, 13C12-	12.86	1.63	250.00	7.85E+05	0.380
Labeled Injection Standards					
13C12-PCB-52 (IS)	9.9	0.80	100.00	8.27E+05	

ALS Life sciences

Calibration Report

ALS Sample ID	**H6-18-CS7-1196**
Analysis Method	EPA 1699 (mod)
Analysis Type	Calibration

Filename	Inst #	Column	Run Date	Approved:	*Ella Gdyczynski* --e-signature-- 13-Nov-2018
6-181112A04	HRMS-6	COL#DB5ms-SN#USR427822H	12-Nov-2018 13:48		

Target Analytes	**Ret. Time**	**Ion Ratio**	**Concentration ng/mL**	**Response**	**RRF**
2,4'-DDE	11.3	1.58	1200.00	1.57E+07	2.065
4,4'-DDE	11.81	1.59	1200.00	1.05E+07	1.376
2,4'-DDD	11.95	1.67	1200.00	1.48E+07	1.718
4,4'-DDD	12.41	1.66	1200.00	1.31E+07	1.522
2,4'-DDT	12.46	1.66	1200.00	7.08E+06	1.719
4,4'-DDT	12.87	1.67	1200.00	5.62E+06	1.365
Extraction Standards					
4,4'-DDE, 13C12-	11.81	1.55	250.00	1.59E+06	0.759
4,4'-DDD, 13C12-	12.4	1.58	250.00	1.79E+06	0.856
4,4'-DDT, 13C12-	12.86	1.67	250.00	8.58E+05	0.410
Labeled Injection Standards					
13C12-PCB-52 (IS)	9.9	0.80	100.00	8.37E+05	

ALS Life sciences

Second Source Calibration Verification Report

Sample Name	**CVS**	Sampling Date	n/a	
ALS Sample ID	H6-18-RS1-1196	Extraction Date	n/a	
Analysis Method	EPA 1699 (mod)	Sample Size	1	n/a
Analysis Type	CCV	Percent Moisture	n/a	
Sample Matrix	QC	Split Ratio	1	

Approved:
Ella Gdyczynski
--e-signature--
13-Nov-2018

Run Information	**Run 1**
Filename	6-181112A09
Run Date	12-Nov-18 15:28
Final Volume	1000 uL
Dilution Factor	1
Analysis Units	%
Instrument - Column	HRMS-6 HP5ms USR427822H

Target Analytes	**ng/mL**	**Ret. Time**	**% Rec**	**Limits**	**Flags**
2,4'-DDE	0				
4,4'-DDE	150	11.81	100	75-125	
2,4'-DDD	0				
4,4'-DDD	150	12.41	100	75-125	
2,4'-DDT	0				
4,4'-DDT	150	12.87	97	75-125	
Extraction Standards	**ng/mL**				
4,4'-DDE, 13C12-	250	11.81	105	70-130	
4,4'-DDD, 13C12-	250	12.40	120	70-130	
4,4'-DDT, 13C12-	250	12.86	138	70-130	

ALS Life sciences

Continuing Calibration Report

Sample Name	**CCV**	Sampling Date	n/a
ALS Sample ID	H6-18-CCV-1198	Extraction Date	n/a
Analysis Method	EPA 1699 (mod)	Sample Size	1 n/a
Analysis Type	CCV	Percent Moisture	n/a
Sample Matrix	QC	Split Ratio	1

Approved:
Ella Gdyczynski
--e-signature--
13-Nov-2018

Run Information	**Run 1**
Filename	6-181112A19
Run Date	12-Nov-18 18:49
Final Volume	1000 uL
Dilution Factor	1
Analysis Units	%
Instrument - Column	HRMS-6 HP5ms USR427822H

Target Analytes	**ng/mL**	**Ret. Time**	**% Rec**	**Limits**	**Flags**
2,4'-DDE	50	11.30	101	75-125	
4,4'-DDE	50	11.81	101	75-125	
2,4'-DDD	50	11.95	91	75-125	
4,4'-DDD	50	12.40	100	75-125	
2,4'-DDT	50	12.46	93	75-125	
4,4'-DDT	50	12.86	96	75-125	
Extraction Standards	**ng/mL**				
4,4'-DDE, 13C12-	250	11.80	108	70-130	
4,4'-DDD, 13C12-	250	12.40	130	70-130	
4,4'-DDT, 13C12-	250	12.86	155	70-130	

ALS Life sciences

Continuing Calibration Report

Sample Name	**CCV**	Sampling Date	n/a	
ALS Sample ID	H6-18-CCV-1200	Extraction Date	n/a	
Analysis Method	EPA 1699 (mod)	Sample Size	1	n/a
Analysis Type	CCV	Percent Moisture	n/a	
Sample Matrix	QC	Split Ratio	1	

Approved:
Ella Gdyczynski
--e-signature--
13-Nov-2018

Run Information	**Run 1**
Filename	6-181112A30
Run Date	12-Nov-18 22:30
Final Volume	1000 uL
Dilution Factor	1
Analysis Units	%
Instrument - Column	HRMS-6 HP5ms USR427822H

Target Analytes	**ng/mL**	**Ret. Time**	**% Rec**	**Limits**	**Flags**
2,4'-DDE	50	11.31	102	75-125	
4,4'-DDE	50	11.82	101	75-125	
2,4'-DDD	50	11.96	95	75-125	
4,4'-DDD	50	12.42	101	75-125	
2,4'-DDT	50	12.48	90	75-125	
4,4'-DDT	50	12.88	100	75-125	
Extraction Standards	**ng/mL**				
4,4'-DDE, 13C12-	250	11.81	108	70-130	
4,4'-DDD, 13C12-	250	12.41	137	70-130	
4,4'-DDT, 13C12-	250	12.87	176	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	WG2914952-1	Extraction Date	29-Oct-18	Approved:
Analysis Method	EPA 1699 (mod)	Sample Size	7 g	*Ella Gdyczynski*
Analysis Type	Blank	Percent Moisture	n/a	--e-signature--
Sample Matrix	QC	Split Ratio	1	13-Nov-2018

Run Information	**Run 1**
Filename	6-181112A23
Run Date	12-Nov-18 20:09
Final Volume	1000 uL
Dilution Factor	5
Analysis Units	ng/g
Instrument - Column	HRMS-6 HP5ms USR427822H

Target Analytes		**Ret. Time**	**Conc. ng/g**	**EDL ng/g**	**Flags**	**EMPC ng/g**	**LQL**
2,4'-DDE		NotFnd	<0.17	0.17	U		1.4
4,4'-DDE		NotFnd	<0.24	0.24	U		1.4
2,4'-DDD		NotFnd	<0.54	0.54	U		1.4
4,4'-DDD		NotFnd	<0.26	0.26	U		1.4
2,4'-DDT		NotFnd	<0.22	0.22	U		1.4
4,4'-DDT		NotFnd	<0.44	0.44	U		1.4
Extraction Standards	**ng**						
4,4'-DDE, 13C12-	125	11.80	25	21-125			
4,4'-DDD, 13C12-	125	12.39	66	5-150			
4,4'-DDT, 13C12-	125	12.86	79	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

Laboratory Control Sample Analysis Report

Sample Name	**Laboratory Control Sample**	Sampling Date	n/a	
ALS Sample ID	WG2914952-2	Extraction Date	29-Oct-18	Approved:
Analysis Method	EPA 1699 (mod)	Sample Size	7 g	*Ella Gdyczynski*
Analysis Type	LCS	Percent Moisture	n/a	--e-signature--
Sample Matrix	QC	Split Ratio	1	13-Nov-2018

Run Information	**Run 1**
Filename	6-181112A11
Run Date	12-Nov-18 16:08
Final Volume	1000 uL
Dilution Factor	5
Analysis Units	%
Instrument - Column	HRMS-6 HP5ms USR427822H

Target Analytes	**ng**	**Ret. Time**	**% Rec**	**Limits**	**Flags**
2,4'-DDE	25	11.30	74	24-123	
4,4'-DDE	25	11.81	95	50-120	
2,4'-DDD	25	11.96	92	50-120	
4,4'-DDD	25	12.41	98	42-120	
2,4'-DDT	25	12.46	81	50-120	
4,4'-DDT	25	12.87	107	50-120	
Extraction Standards	**ng**				
4,4'-DDE, 13C12-	125	11.81	44	26-169	
4,4'-DDD, 13C12-	125	12.40	69	13-200	
4,4'-DDT, 13C12-	125	12.86	80	13-200	

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SVOC DATA PACKAGE

SECTION 6: INTERNAL RECORDS

Including:

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

Extraction Workup Sheet

Batch ID:	WG2914952	**Analysis:**	**Sediments - OCP**
Analyst:	Jackson Pegg	**Date:**	29-Oct-2018

BU-TM-1110 Overall HR Prep

SUBSAMPLING

Sample I.D.	Client I.D.	Subsample Size (g)
WG2914952-1	Method Blank	10.48
WG2914952-2	Laboratory Control Sample	10.25
WG2914952-3	Extraction and Injection STD.	—
L2187236-1	PDI-SC-S154-4TO6	10.13
WG2914952-4	Duplicate(L2187236-1)	10.15
L2187236-2	PDI-SC-S185-5.5TO6.5	10.36
	See Special submission instructions	

BATCH TRACKING

	Date/Time/Initials	
Subsampling:	29-Oct-2018 10:00AM JP	
Balance ID	3955	
Client Labels Checked:	JP	
Samples Spiked	JP	
Soxhlet start time:	29-Oct-2018 2:30PM JP	
Soxhlet reflux properly:	JP	
Soxhlet end time:	30-Oct-2018 8:00AM JP	
	—	
Rotovap + temp check:	30-Oct-18 AR	JA2
	—	
Sili Column:	30-Oct-18 AR	
Mini Acid	MM 31-Oct-2018	
	—	
	—	

	—
	—
Robo-Vialing:	MM 31-Oct-2018
Update to LIMS:	MM 31-Oct-2018

OCP Sediments- AecomPREP
30-Apr-18/ JP,MK
Page 1 of 6

Batch ID: WG2914952

DX Extraction Standard:

Sample I.D.	Volume (ul)	(Checkmark) Spiked
WG2914952-1	20	✓
WG2914952-2	20	✓
WG2914952-3	20	✓
L2187236-1	20	✓
WG2914952-4	20	✓
L2187236-2	20	✓
	20	
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	20	

Syringe ID 137

Standard: HROCP_DDX-ES#1- 001C

Spike Date: 29 - Oct - 2018

Spike Witnessing

Chemist: Chemist's Initials JP

Witness: Witness's Initials SP

Correct Syringe Obtained: Witness's Initials SP

Correct Standard Obtained: Witness's Initials SP

Correct Technique Followed: Witness's Initials SP

Batch ID:	WG2914952

Syringe ID	202
Standard:	**HROCP-NS#2-** 002A
Date & Initials:	29-Oct-2018 JP

DX Native Standard:

Sample I.D.	**Volume (ul)**	(Checkmark) **Spiked**
WG2914952-2	20	✓
WG2914952-3	20	✓
	20	
	20	

DX Injection Standard:

Sample I.D.	**Volume (ul)**	(Checkmark) **Spiked**
WG2914952-1	20	✓
WG2914952-2	20	✓
WG2914952-3	20	✓
L2187236-1	20	✓
WG2914952-4	20	✓
L2187236-2	20	✓
	20	
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	20	

Syringe ID	218 rinsed 10x Tol 10x Hex
Standard:	**HROCP-IS#1-** 016D
Date & Initials:	31-Oct-2018 MM

MM 31-Oct-2018

	Chemist's Initials
Correct Syringe Obtained:	MM
Correct Standard Obtained:	MM
Correct Technique Followed:	MM

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 Column (Column does not contain carbon)

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

Mini Acid Silica Column

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

Robo-vial

- Reduce to 5mL

-Spike IS standard and mix thoroughly

-Remove 1ml from C-tube and transfer to a robovial and submit FV=1000ul

- Transfer remaining 4ml to a supelco vial and archive

Reagent Lot Numbers:

Reagent	Lot#	Manufacturer
Acetone	103647	
Hexane	103734	
DCM	103681	
Toluene	103255	
Nonane	ORG-WAKONON-045	
1:1 DCM:HEX	ORG-DH2- 517	
Sodium Sulphate	ORG-SSU-1982	
Acid Silica	ORG-ASI- 8047	
Neutral Silica	ORG-NSI- 1771	
Alumina	ORG-ALU- 403	
Chromacarb	ORG-CC- 232	
Corn Oil	ORG-CO-	

Comments:

All samples went low on N-evap, ~500mL. Proceeded as normal. MK 31-Oct-2018

WG:	Prep Analyst:
Analysis:	Date:

	Very Good	Meets Method Req	Some Outliers	Very Poor	Comments / Was spl/batch sent for rework? Why?
MB					
LCS					
DUP					
ES rec					

ALS Life sciences

Sample Calculation Report

Approved: *Ella Gdyczynski*
--e-signature--
13-Nov-2018

CS3 RRF Check

$$\text{RRF} = \frac{\text{Response of 44'-DDT}}{\text{Response of 13C12-44'-DDT}} \times \frac{\text{Concentration of 13C2-44'-DDT}}{\text{Concentration of 44'-DDT}}$$

$$\textbf{RRF} = \frac{84665.10}{807007.60} \times \frac{250.00}{20} =$$

Calculated Value	Value from TargetLynx
1.31	**1.31**

Calculation of 44'-DDT amount in WG2914952-2

$$\% = \frac{\text{Response of 44'-DDT}}{\text{Response of 13C2-44'-DDT}} \times \frac{\text{ng of 13C12-44'-DDT spiked} * 100.00}{\text{Mean RRF} * \text{amount spiked}}$$

$$\% = \frac{16853.5}{60729.1} \times \frac{125}{1.30 * 25.00} =$$

Calculated Value	Value from TargetLynx
107	**107**

Calculation of 13C12-44'-DDT Recovery in L2187236-1

$$\text{\% Recovery} = \frac{\text{Response of 13C12-44'-DDT}}{\text{Response of 13C12-PCB-52}} \times \frac{\text{ng of 13C12-PCB-52} * 100}{\text{Mean RRF} * \text{Amount Spiked}}$$

$$\textbf{\% Recovery} = \frac{111652.3}{95446} \times \frac{50 * 100}{0.36 * 125} =$$

Calculated Value	Value from TargetLynx
132	**132 %**

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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:

L2187236

ALS-Burlington
1435 Norjohn Court Unit 1
~~Burlington, Ontario Canada~~ L7L 0E6
Ph: 1-905-331-3111 Fax: 0

SUBSURFACE SEDIMENT
CHAIN OF CUSTODY

Client Contact	**Project Contact: Amy Dahl / Chelsey Cook**	**Site Contact: Nicky Moody**	**Date: 10/24/18**	COC No. 1
AECOM	**Tel: (206) 438-2261 / (206) 438-2010**	**Laboratory Contact: Whitney Davis**	**Carrier: FedEx**	1 of 1 pages
1111 3rd Ave Suite 1600	**Analysis Turnaround Time**			
Seattle, WA 98101	Calendar (C) or Work Days (W) W			
Phone: (206) 438-2700 Fax: 1+(866) 495-5288 Project Name: Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling Portland, OR	☐ 21 days ☒ Other ASAP			
Project #: 60566335 Study: Subsurface Sediment				
Sample Type:				

	Sample Identification	Sample Date	Sample Time	Matrix	QC Sample	Sampler's Initials	Total No. of Cont.	Fraction	Pesticides, DDx only	Sample Specific Notes:
1	PDI-SC-S154 - 4 to 6	7/24/2018	15:45	SC		ED	1		x	Frozen 7/26/2018
2	PDI-SC-S185 - 5 to 6.5	7/26/2018	16:05	SC		ED	1		x	Frozen 7/26/2018

Container Type: WMG=Wide Mouth Glass Jar, P=HDPE, PP=Polypropylene, AG=amber glass, G=glass, RC=Resin Column AG AG WMG WMG AG

Preservative: HCl = Hydrochloric Acid, H3PO4 = Phosphoric Acid, HNO3 = Nitric Acid

Fraction: D = Dissolved, PRT = Particulate, T = Total (unfiltered)

Sample Disposal
☐ *Return To Client* ☒ *posal By Lab* ☒ *chive For 12 Months*

Special Instructions/QC Requirements & Comments:

2.6°C

Relinquished by: Nicky Moody	Company: AECOM	Date/Time: 10/24/2018 @ 1245	Received by: FEDEX	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received by: Aaron Burton	Company: ALS	Date/Time: 25-Oct-2018 14:30
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:

ORIGIN ID:MRIA (503) 969-6310
NUCKY MOODY
AELOM
111 SW COLUMBIA ST STE 1500

PORTLAND, OR 97201
UNITED STATES US

SHIP DATE: 24OCT18
ACTWGT: 23.30 LB
CAD: 6991797/SSF01922
DIMS: 15x11x16 IN

BILL THIRD PARTY

TO **CLAIRE KOCHARAKKAL ALS ENVIRON**
C/O FEDERAL EXPRESS DEPOT
299 CAYUGA RD

BUFFALO NY 14225
(905) 331-3111 REF:
INV:
PO: DEPT:

Part # 156297
09/18

FedEx
Express

E

J1S2112081501uv

THU - 25 OCT 10:30A
PRIORITY OVERNIGHT

TRK# 0200 **8124 0475 7526**

AHS
14225
NY-US **BUF**

XX BUFA

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
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
25-oct-2018 14:30	Aecom	2x jars of sediment	2.6°C	Good FedEx / ALS Driver 8124 0475 7526	Nz	25-oct-2018 15:50	L2187236	-1,2

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

☐ Other (specify): ____________________