



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

SVOC DATA PACKAGE

Client Project Information

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

ALSE Project Information

Project ID: AECOM100
Contact: Claire Kocharakkal
Submission ID(s): L2159262

Final Package Review by:

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

Date Reviewed: 20-Sep-18



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SECTION 1: PROJECT NARRATIVE

ALSE Project Information

Project ID: AECOM100

Contact: Claire Kocharakkal

Submission ID(s): L2159262

Client Project Information

Project ID: 60566335

Project Description: Portland Harbor Pre- Remedial Design Investigation &

Contact: Amy Dahl

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

ALS Sample ID	Client Sample Descriptions	Matrix	Date Sampled	Date Received	Date Extracted	Date Analyzed
L2159262-1	NIST 1944	Sediment	n/a	n/a	06-Sep-18	17-Sep-18
WG2868044-1	Method Blank	QC	n/a	n/a	06-Sep-18	17-Sep-18
WG2868044-2	Laboratory Control Sample	QC	n/a	n/a	06-Sep-18	17-Sep-18

Comments and Notes:

a) Sample Integrity:

The sample consists of the NIST reference material SRM-1944

b) Instrumental Analysis:

All results have been reported on a dry weight basis.

The sample results have been calculated against the average daily relative response factors from the pre- and post-run continuing calibration verification (CCV) standards. CCV recoveries have been calculated against the initial calibration.

No criteria failures or exceedences.

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

20-Sep-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: Claire Kocharakkal
ALS Project ID: AECOM100
ALS WO#: L2159262
Date of Report: 18-Sep-18
Date of Sample Receipt: 6-Sep-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: 2,4'- and 4,4'- DDE, DDD and DDT by EPA 1699 (modified)

All results have been reported on a dry weight basis.

Certified by:

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

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 NIST 1944

ALS Sample ID L2159262-1

Sample Size 1.62
Sample size units g
Percent Solid 98.7%
Sample Matrix Sediment
Sampling Date n/a
Extraction Date 6-Sep-18

Target Analytes	ng/g	% Rec
2,4'-DDE	15.7	83%
4,4'-DDE	67.7	79%
2,4'-DDD	39.5	104%
4,4'-DDD	97.8	91%
2,4'-DDT	3.26	
4,4'-DDT	177	104%
Extraction Standards	% Rec	
4,4'-DDE, 13C12-	122	
4,4'-DDD, 13C12-	104	
4,4'-DDT, 13C12-	74	

ALS Life sciences

Quality Control Summary Report

Sample Name	Method Blank	Laboratory Control Sample
ALS Sample ID	WG2868044-1	WG2868044-2
Sample Size	1.60	1.00
Sample size units	g	g
Percent Solid	100.0%	49.7%
Sample Matrix	QC	QC
Sampling Date	n/a	n/a
Extraction Date	6-Sep-18	6-Sep-18
Target Analytes	ng/g	% Rec
2,4'-DDE	<0.10	97
4,4'-DDE	<0.13	89
2,4'-DDD	<0.17	101
4,4'-DDD	<0.19	93
2,4'-DDT	<0.18	95
4,4'-DDT	<0.31	95
Extraction Standards	% Rec	% Rec
4,4'-DDE, 13C12-	86	80
4,4'-DDD, 13C12-	82	79
4,4'-DDT, 13C12-	79	77

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Continuing Calibration Summary Report

Sample Name	CVS	CCV	CCV
ALS Sample ID	H6-18-RS1-079	H6-18-CCV-01013	H6-18-CCV-1015
Sample Size	1	1	1
Sample size units	n/a	n/a	n/a
Percent Solid	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	95	100	103
2,4'-DDD		99	102
4,4'-DDD	96	98	103
2,4'-DDT		101	99
4,4'-DDT	94	100	100
Extraction Standards	% Rec	% Rec	% Rec
4,4'-DDE, 13C12-	98	94	102
4,4'-DDD, 13C12-	105	102	105
4,4'-DDT, 13C12-	123	117	114

ALS Life sciences

Sample Analysis Report

Sample Name NIST 1944
 ALS Sample ID L2159262-1
 Analysis Method EPA 1699 (mod)
 Analysis Type Sample
 Sample Matrix Sediment

Sampling Date n/a
 Extraction Date 6-Sep-18
 Sample Size 1.62 g
 Percent Solid 98.7%
 Split Ratio 1

Approved:
R. Bakhtiari
 --e-signature--
 18-Sep-2018

Run Information Run 1
 Filename 6-180917A27
 Run Date 17-Sep-18 20:50
 Final Volume 1000 uL
 Dilution Factor 5
 Analysis Units ng/g
 Instrument - Column HRMS-6 HP5MSUSR280851H

Target Analytes	Ret.	Conc.	EDL	EMPC	NIST Reference (ng/g)				
	Time	ng/g	ng/g	Flags	ng/g	LQL	value	uncert.	% Rec
2,4'-DDE	10.85	15.7	0.063	M	6.2		19	3	83%
4,4'-DDE	11.38	67.7	0.083	M	6.2		86	12	79%
2,4'-DDD	11.55	39.5	0.12	M	6.2		38	8	104%
4,4'-DDD	12.06	97.8	0.14	M	6.2		108	16	91%
2,4'-DDT	12.11	3.26	0.13	M,J	6.2				
4,4'-DDT	12.55	177	0.32	M	6.2		170	32	104%
Extraction Standards	ng								
4,4'-DDE, 13C12-	125	11.37	122	21-125					
4,4'-DDD, 13C12-	125	12.05	104	5-150					
4,4'-DDT, 13C12-	125	12.55	74	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.
 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	24'-DDE	35	75-125	50-120	
	44'-DDE	20	75-125	50-120	
	24'-DDD	35	75-125	42-120	
	44'-DDD	20	75-125	42-120	
	24'-DDT	35	75-125	50-120	
	44'-DDT	20	75-125	50-120	
Extraction Standards	13C12-44'-DDE	35	70-130	21-125	21-125
	13C12-44'-DDD	35	70-130	13-200	5-150
	13C12-44'-DDT	35	70-130	13-200	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 =

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

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 =

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

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 its metabolites are the only targets.
- Where the DDT breakdown fails but endrin breakdown passes and where endrin and/or its 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	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.

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

Where,

$A1_t + A2_t$: 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.

$$\text{Amount in sample (ng)} = \frac{(A1_n + A2_n) Q_i}{(A1_l + A2_l) (\text{RRF}_{av})} \quad \text{Equ. 9.2}$$

$$\text{Concentration in sample (ng/g or ng/L)} = \frac{(A1_n + A2_n) Q_i}{(A1_l + A2_l) (\text{RRF}_{av}) (W_s)} \quad \text{Equ. 9.3}$$

Where,

Q_i = 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.

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

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	6-180917A12	17-Sep-2018 15:32
CS-2	6-180917A29	17-Sep-2018 21:31

Approved:	<i>R. Bakhtiari</i> --e-signature-- 18-Sep-2018
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Target Analytes	Relative Response Factors		Mean	% RSD
	CS-1	CS-2		
2,4'-DDE	1.290	1.304	1.297	1%
4,4'-DDE	0.967	0.992	0.980	2%
2,4'-DDD	1.034	1.062	1.048	2%
4,4'-DDD	0.976	1.026	1.001	4%
2,4'-DDT	1.061	1.039	1.050	1%
4,4'-DDT	0.893	0.897	0.895	0%
Extraction Standards				
4,4'-DDE, 13C12-	0.502	0.543	0.523	6%
4,4'-DDD, 13C12-	0.462	0.476	0.469	2%
4,4'-DDT, 13C12-	0.304	0.298	0.301	1%

ALS Life sciences

Calibration Report

ALS Sample ID **H6-18-CCV-01013**
 Analysis Method EPA 1699 (mod)
 Analysis Type Calibration

Filename 6-180917A12 Inst # HRMS-6 Column HP5MSUSR280851H Run Date 17-Sep-2018 15:32

Approved: *R. Bakhtiari*
 --e-signature--
 18-Sep-2018

Target Analytes	Ret. Time	Ion Ratio	Concentration ng/mL	Response	RRF
2,4'-DDE	10.86	1.54	50.00	7.97E+05	1.290
4,4'-DDE	11.39	1.52	50.00	5.98E+05	0.967
2,4'-DDD	11.56	1.57	50.00	5.87E+05	1.034
4,4'-DDD	12.07	1.54	50.00	5.54E+05	0.976
2,4'-DDT	12.13	1.56	50.00	3.97E+05	1.061
4,4'-DDT	12.57	1.55	50.00	3.34E+05	0.893
Extraction Standards					
4,4'-DDE, 13C12-	11.38	1.49	250.00	3.09E+06	0.502
4,4'-DDD, 13C12-	12.06	1.57	250.00	2.84E+06	0.462
4,4'-DDT, 13C12-	12.56	1.52	250.00	1.87E+06	0.304
Labeled Injection Standards					
13C12-PCB-52 (IS)	9.52	0.79	100.00	2.46E+06	

ALS Life sciences

Calibration Report

ALS Sample ID **H6-18-CCV-1015**
 Analysis Method EPA 1699 (mod)
 Analysis Type Calibration

Filename 6-180917A29 Inst # HRMS-6 Column HP5MSUSR280851H Run Date 17-Sep-2018 21:31

Approved: *R. Bakhtiari*
 --e-signature--
 18-Sep-2018

Target Analytes	Ret. Time	Ion Ratio	Concentration ng/mL	Response	RRF
2,4'-DDE	10.84	1.54	50.00	1.38E+06	1.304
4,4'-DDE	11.37	1.55	50.00	1.05E+06	0.992
2,4'-DDD	11.55	1.56	50.00	9.82E+05	1.062
4,4'-DDD	12.06	1.57	50.00	9.49E+05	1.026
2,4'-DDT	12.11	1.57	50.00	6.00E+05	1.039
4,4'-DDT	12.56	1.56	50.00	5.18E+05	0.897
Extraction Standards					
4,4'-DDE, 13C12-	11.37	1.54	250.00	5.28E+06	0.543
4,4'-DDD, 13C12-	12.05	1.57	250.00	4.62E+06	0.476
4,4'-DDT, 13C12-	12.55	1.56	250.00	2.89E+06	0.298
Labeled Injection Standards					
13C12-PCB-52 (IS)	9.5	0.78	100.00	3.88E+06	

ALS Life sciences

Calibration Summary Report

Calibration Level	Filename	Run Date
CS-1	6-180917A04	17-Sep-2018 11:53
CS-2	6-180917A03	17-Sep-2018 11:37
CS-3	6-180917A08	17-Sep-2018 13:15
CS-4	6-180917A07	17-Sep-2018 12:55
CS-5	6-180917A06	17-Sep-2018 12:34
CS-6	6-180917A05	17-Sep-2018 12:17

Approved:	<i>R. Bakhtiari</i>
	--e-signature--
	18-Sep-2018

Target Analytes	Relative Response Factors						Mean	% RSD
	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6		
2,4'-DDE	1.235	1.290	1.164	1.299	1.338	1.329	1.276	5%
4,4'-DDE	0.910	1.023	0.894	0.976	1.003	0.974	0.963	5%
2,4'-DDD	0.988	1.082	0.982	1.045	1.080	1.058	1.039	4%
4,4'-DDD	1.019	1.073	0.940	0.976	1.011	0.986	1.001	5%
2,4'-DDT	1.048	1.066	0.928	1.083	1.105	1.069	1.050	6%
4,4'-DDT	0.931	0.952	0.828	0.875	0.907	0.888	0.897	5%
Extraction Standards								
4,4'-DDE, 13C12-	0.499	0.506	0.574	0.530	0.544	0.543	0.533	5%
4,4'-DDD, 13C12-	0.424	0.408	0.480	0.449	0.466	0.486	0.452	7%
4,4'-DDT, 13C12-	0.224	0.201	0.317	0.275	0.274	0.272	0.261	16%

ALS Life sciences

Calibration Report

ALS Sample ID **H6-18-CS1-079**
 Analysis Method EPA 1699 (mod)
 Analysis Type Calibration

Filename	Inst #	Column	Run Date
6-180917A04	HRMS-6	HP5MSUSR280851H	17-Sep-2018 11:53

Approved: *R. Bakhtiari*
 --e-signature--
 18-Sep-2018

Target Analytes	Ret. Time	Ion Ratio	Concentration ng/mL	Response	RRF
2,4'-DDE	10.85	1.52	2.00	4.18E+04	1.235
4,4'-DDE	11.38	1.61	2.00	3.08E+04	0.910
2,4'-DDD	11.55	1.46	2.00	2.84E+04	0.988
4,4'-DDD	12.07	1.43	2.00	2.93E+04	1.019
2,4'-DDT	12.12	1.38	2.00	1.59E+04	1.048
4,4'-DDT	12.57	1.24	2.00	1.42E+04	0.931

Extraction Standards

4,4'-DDE, 13C12-	11.38	1.53	250.00	4.23E+06	0.499
4,4'-DDD, 13C12-	12.06	1.55	250.00	3.59E+06	0.424
4,4'-DDT, 13C12-	12.56	1.57	250.00	1.90E+06	0.224

Labeled Injection Standards

13C12-PCB-52 (IS)	9.52	0.78	100.00	3.39E+06	
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ALS Life sciences

Calibration Report

ALS Sample ID **H6-18-CS2-079**
 Analysis Method EPA 1699 (mod)
 Analysis Type Calibration

Filename 6-180917A03 Inst # HRMS-6 Column HP5MSUSR280851H Run Date 17-Sep-2018 11:37

Approved: *R. Bakhtiari*
 --e-signature--
 18-Sep-2018

Target Analytes	Ret. Time	Ion Ratio	Concentration ng/mL	Response	RRF
2,4'-DDE	10.86	1.54	7.50	1.80E+05	1.290
4,4'-DDE	11.39	1.53	7.50	1.43E+05	1.023
2,4'-DDD	11.56	1.58	7.50	1.22E+05	1.082
4,4'-DDD	12.07	1.47	7.50	1.21E+05	1.073
2,4'-DDT	12.13	1.41	7.50	5.92E+04	1.066
4,4'-DDT	12.57	1.35	7.50	5.28E+04	0.952
Extraction Standards					
4,4'-DDE, 13C12-	11.38	1.53	250.00	4.65E+06	0.506
4,4'-DDD, 13C12-	12.06	1.57	250.00	3.75E+06	0.408
4,4'-DDT, 13C12-	12.56	1.53	250.00	1.85E+06	0.201
Labeled Injection Standards					
13C12-PCB-52 (IS)	9.52	0.80	100.00	3.68E+06	

ALS Life sciences

Calibration Report

ALS Sample ID **H6-18-CS3-079**
 Analysis Method EPA 1699 (mod)
 Analysis Type Calibration

Filename 6-180917A08 Inst # HRMS-6 Column HP5MSUSR280851H Run Date 17-Sep-2018 13:15

Approved: *R. Bakhtiari*
 --e-signature--
 18-Sep-2018

Target Analytes	Ret. Time	Ion Ratio	Concentration ng/mL	Response	RRF
2,4'-DDE	10.84	1.53	20.00	5.03E+05	1.164
4,4'-DDE	11.37	1.58	20.00	3.87E+05	0.894
2,4'-DDD	11.54	1.58	20.00	3.56E+05	0.982
4,4'-DDD	12.06	1.64	20.00	3.40E+05	0.940
2,4'-DDT	12.11	1.41	20.00	2.21E+05	0.928
4,4'-DDT	12.55	1.56	20.00	1.98E+05	0.828
Extraction Standards					
4,4'-DDE, 13C12-	11.37	1.52	250.00	5.41E+06	0.574
4,4'-DDD, 13C12-	12.05	1.55	250.00	4.53E+06	0.480
4,4'-DDT, 13C12-	12.55	1.56	250.00	2.98E+06	0.317
Labeled Injection Standards					
13C12-PCB-52 (IS)	9.5	0.77	100.00	3.77E+06	

ALS Life sciences

Calibration Report

ALS Sample ID **H6-18-CS4-079**
 Analysis Method EPA 1699 (mod)
 Analysis Type Calibration

Filename 6-180917A07 Inst # HRMS-6 Column HP5MSUSR280851H Run Date 17-Sep-2018 12:55

Approved: *R. Bakhtiari*
 --e-signature--
 18-Sep-2018

Target Analytes	Ret. Time	Ion Ratio	Concentration ng/mL	Response	RRF
2,4'-DDE	10.85	1.53	50.00	1.37E+06	1.299
4,4'-DDE	11.38	1.55	50.00	1.03E+06	0.976
2,4'-DDD	11.55	1.58	50.00	9.35E+05	1.045
4,4'-DDD	12.07	1.57	50.00	8.74E+05	0.976
2,4'-DDT	12.12	1.53	50.00	5.94E+05	1.083
4,4'-DDT	12.56	1.59	50.00	4.80E+05	0.875
Extraction Standards					
4,4'-DDE, 13C12-	11.38	1.51	250.00	5.29E+06	0.530
4,4'-DDD, 13C12-	12.06	1.54	250.00	4.48E+06	0.449
4,4'-DDT, 13C12-	12.56	1.59	250.00	2.74E+06	0.275
Labeled Injection Standards					
13C12-PCB-52 (IS)	9.5	0.78	100.00	3.99E+06	

ALS Life sciences

Calibration Report

ALS Sample ID **H6-18-CS5-079**
 Analysis Method EPA 1699 (mod)
 Analysis Type Calibration

Filename 6-180917A06 Inst # HRMS-6 Column HP5MSUSR280851H Run Date 17-Sep-2018 12:34

Approved: *R. Bakhtiari*
 --e-signature--
 18-Sep-2018

Target Analytes	Ret. Time	Ion Ratio	Concentration ng/mL	Response	RRF
2,4'-DDE	10.85	1.53	150.00	4.21E+06	1.338
4,4'-DDE	11.38	1.51	150.00	3.15E+06	1.003
2,4'-DDD	11.55	1.56	150.00	2.91E+06	1.080
4,4'-DDD	12.06	1.57	150.00	2.73E+06	1.011
2,4'-DDT	12.12	1.55	150.00	1.75E+06	1.105
4,4'-DDT	12.56	1.57	150.00	1.44E+06	0.907
Extraction Standards					
4,4'-DDE, 13C12-	11.37	1.51	250.00	5.24E+06	0.544
4,4'-DDD, 13C12-	12.06	1.51	250.00	4.49E+06	0.466
4,4'-DDT, 13C12-	12.55	1.53	250.00	2.64E+06	0.274
Labeled Injection Standards					
13C12-PCB-52 (IS)	9.5	0.83	100.00	3.86E+06	

ALS Life sciences

Calibration Report

ALS Sample ID **H6-18-CS6-079**
 Analysis Method EPA 1699 (mod)
 Analysis Type Calibration

Filename 6-180917A05 Inst # HRMS-6 Column HP5MSUSR280851H Run Date 17-Sep-2018 12:17

Approved: *R. Bakhtiari*
 --e-signature--
 18-Sep-2018

Target Analytes	Ret. Time	Ion Ratio	Concentration ng/mL	Response	RRF
2,4'-DDE	10.85	1.51	400.00	1.25E+07	1.329
4,4'-DDE	11.38	1.52	400.00	9.15E+06	0.974
2,4'-DDD	11.55	1.58	400.00	8.88E+06	1.058
4,4'-DDD	12.06	1.59	400.00	8.28E+06	0.986
2,4'-DDT	12.12	1.61	400.00	5.03E+06	1.069
4,4'-DDT	12.56	1.57	400.00	4.18E+06	0.888
Extraction Standards					
4,4'-DDE, 13C12-	11.37	1.52	250.00	5.87E+06	0.543
4,4'-DDD, 13C12-	12.05	1.56	250.00	5.25E+06	0.486
4,4'-DDT, 13C12-	12.55	1.54	250.00	2.94E+06	0.272
Labeled Injection Standards					
13C12-PCB-52 (IS)	9.5	0.79	100.00	4.32E+06	

ALS Life sciences

Second Source Calibration Verification Report

Sample Name	CVS	Sampling Date	n/a	
ALS Sample ID	H6-18-RS1-079	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--
 18-Sep-2018

Run Information	Run 1
Filename	6-180917A09
Run Date	17-Sep-18 13:35
Final Volume	1000 uL
Dilution Factor	1
Analysis Units	%
Instrument - Column	HRMS-6 HP5MSUSR280851H

Target Analytes	ng/mL	Ret.		Limits	
		Time	% Rec	Flags	Flags
2,4'-DDE					
4,4'-DDE	150	11.38	95	75-125	
2,4'-DDD					
4,4'-DDD	150	12.06	96	75-125	
2,4'-DDT					
4,4'-DDT	150	12.56	94	75-125	
Extraction Standards	ng/mL				
4,4'-DDE, 13C12-	250	11.37	98	70-130	
4,4'-DDD, 13C12-	250	12.06	105	70-130	
4,4'-DDT, 13C12-	250	12.56	123	70-130	

ALS Life sciences

Continuing Calibration Report

Sample Name	CCV	Sampling Date	n/a	
ALS Sample ID	H6-18-CCV-01013	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--
 18-Sep-2018

Run Information	Run 1
Filename	6-180917A12
Run Date	17-Sep-18 15:32
Final Volume	1000 uL
Dilution Factor	1
Analysis Units	%
Instrument - Column	HRMS-6 HP5MSUSR280851H

Target Analytes	ng/mL	Ret.		Limits	
		Time	% Rec	Time	Flags
2,4'-DDE	50	10.86	101	75-125	
4,4'-DDE	50	11.39	100	75-125	
2,4'-DDD	50	11.56	99	75-125	
4,4'-DDD	50	12.07	98	75-125	
2,4'-DDT	50	12.13	101	75-125	
4,4'-DDT	50	12.57	100	75-125	
Extraction Standards					
	ng/mL				
4,4'-DDE, 13C12-	250	11.38	94	70-130	
4,4'-DDD, 13C12-	250	12.06	102	70-130	
4,4'-DDT, 13C12-	250	12.56	117	70-130	

ALS Life sciences

Continuing Calibration Report

Sample Name	CCV	Sampling Date	n/a	
ALS Sample ID	H6-18-CCV-1015	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--
 18-Sep-2018

Run Information	Run 1
Filename	6-180917A29
Run Date	17-Sep-18 21:31
Final Volume	1000 uL
Dilution Factor	1
Analysis Units	%
Instrument - Column	HRMS-6 HP5MSUSR280851H

Target Analytes	ng/mL	Ret.		Limits	
		Time	% Rec	Flags	Flags
2,4'-DDE	50	10.84	102	75-125	
4,4'-DDE	50	11.37	103	75-125	
2,4'-DDD	50	11.55	102	75-125	
4,4'-DDD	50	12.06	103	75-125	
2,4'-DDT	50	12.11	99	75-125	
4,4'-DDT	50	12.56	100	75-125	
Extraction Standards					
	ng/mL				
4,4'-DDE, 13C12-	250	11.37	102	70-130	
4,4'-DDD, 13C12-	250	12.05	105	70-130	
4,4'-DDT, 13C12-	250	12.55	114	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	WG2868044-1	Extraction Date	6-Sep-18		
Analysis Method	EPA 1699 (mod)	Sample Size	1.6	g	
Analysis Type	Blank	Percent Solid	100.0%		
Sample Matrix	QC	Split Ratio	1		
					Approved: <i>R. Bakhtiari</i> --e-signature-- 18-Sep-2018

Run Information	Run 1
Filename	6-180917A21
Run Date	17-Sep-18 18:53
Final Volume	1000 uL
Dilution Factor	5
Analysis Units	ng/g
Instrument - Column	HRMS-6 HP5MSUSR280851H

Target Analytes	Ret. Time	Conc. ng/g	EDL ng/g	Flags	EMPC ng/g	LQL
2,4'-DDE	NotFnd	<0.10	0.10	U	6.3	
4,4'-DDE	NotFnd	<0.13	0.13	U	6.3	
2,4'-DDD	NotFnd	<0.17	0.17	U	6.3	
4,4'-DDD	NotFnd	<0.19	0.19	U	6.3	
2,4'-DDT	NotFnd	<0.18	0.18	U	6.3	
4,4'-DDT	NotFnd	<0.31	0.31	U	6.3	
Extraction Standards ng						
4,4'-DDE, 13C12-	125	11.37	86	21-125		
4,4'-DDD, 13C12-	125	12.05	82	5-150		
4,4'-DDT, 13C12-	125	12.55	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	WG2868044-2	Extraction Date	6-Sep-18		
Analysis Method	EPA 1699 (mod)	Sample Size	1	g	
Analysis Type	LCS	Percent Solid	49.7%		
Sample Matrix	QC	Split Ratio	1		

Approved:
R. Bakhtiari
 --e-signature--
 18-Sep-2018

Run Information	Run 1
Filename	6-180917A17
Run Date	17-Sep-18 17:27
Final Volume	1000 uL
Dilution Factor	5
Analysis Units	%
Instrument - Column	HRMS-6 HP5MSUSR280851H

Target Analytes	ng	Ret.		Limits	
		Time	% Rec	Flags	Limits
2,4'-DDE	25	10.85	97	50-120	
4,4'-DDE	25	11.38	89	50-120	
2,4'-DDD	25	11.56	101	42-120	
4,4'-DDD	25	12.07	93	42-120	
2,4'-DDT	25	12.12	95	50-120	
4,4'-DDT	25	12.56	95	50-120	
Extraction Standards					
4,4'-DDE, 13C12-	125	11.38	80	21-125	
4,4'-DDD, 13C12-	125	12.06	79	13-200	
4,4'-DDT, 13C12-	125	12.56	77	13-200	

SVOC DATA PACKAGE

SECTION 6: INTERNAL RECORDS

Including:

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

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

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

CS3 RRF Check

Approved:	<i>R. Bakhtiari</i> --e-signature-- 18-Sep-2018
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$$\text{RRF} = \frac{\text{Response of 4,4'-DDE}}{\text{Response of 13C12-4,4'DDE}} \times \frac{\text{Concentration of 13C2-4,4'DDE}}{\text{Concentration of DDE}}$$

$$\text{RRF} = \frac{597751.20}{3089483.40} \times \frac{250.00}{50}$$

Calculated Value	Value from TargetLynx
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=	0.97	0.97
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Calculation of 4,4'-DDE amount in L2159262-1

$$\text{ng} = \frac{\text{Response of 4,4'-DDE}}{\text{Response of 13C12-4,4'DDE}} \times \frac{\text{ng of 13C12-4,4'-DDE spiked}}{\text{Mean RRF} \times \text{Sample Size}}$$

$$\text{ng/g} = \frac{781460.2}{909412} \times \frac{125}{0.98 \times 1.62} = 67.7 \quad \text{67.7}$$

Calculation of 13C12-4,4'-DDE Recovery in L2159262-1

$$\% \text{ Recovery} = \frac{\text{Response of 13C12-4,4'-DDE}}{\text{Response of 13C12-PCB-52}} \times \frac{\text{ng of 13C12-PCB-52} \times 100}{\text{Mean RRF} \times \text{Amount Spiked}}$$

$$\% \text{ Recovery} = \frac{909412}{568165.7} \times \frac{50 \times 100}{0.523 \times 125} = 122.5 \quad \text{122.0 \%}$$