ALS Life Sciences
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: Whitney Davis
Submission ID(s): L2125039

Final Package Review by: 
Date Reviewed: 25-Jul-18

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

# SVOC DATA PACKAGE

## SECTION 1: PROJECT NARRATIVE

### ALSE Project Information

Project ID: AECOM100
Contact: Whitney Davis
Submission ID(s): L2125039

### Client Project Information

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

**Analytical Method:** Chlorinated Pesticides by EPA 1699 (modified)

| ALS Sample ID | Client Sample Descriptions | Matrix | Date Sampled | Date Received | Date Extracted | Date Analyzed |
|---|---|---|---|---|---|---|
| L2125039-1 | NIST 1944 | Sediment | n/a | n/a | 2018-Jul-06 | 2018-Jul-20 |
| L2125039-2 | NIST 1944 Duplicate | Sediment | n/a | n/a | 2018-Jul-06 | 2018-Jul-20 |
| WG2815918-1 | Laboratory Method Blank | QC | n/a | n/a | 2018-Jul-06 | 2018-Jul-20 |
| WG2815918-2 | Laboratory Control Sample | QC | n/a | n/a | 2018-Jul-06 | 2018-Jul-20 |

**Comments and Notes:**

**a) Sample Integrity:**
The NIST reference material has been refrigerated since receipt. It has not exceeded its expiry date of 31 March 2027.

**b) Extraction and Cleanup:**
The NIST material was extracted overnight by soxhlet extraction technique using dichloromethane as the extracting solvent. The extract was cleaned by silica gel column chromatography prior to instrumental analysis.

**c) Instrumental Analysis:**
Extraction standard recovery limits are listed herein have been derived from those recommended in EPA method 1699. The lower acceptance limit of 47% for 4,4'-DDE-13C12 has been found not to represent observed recoveries and this lower limit will soon be revised based upon historical data. On this data set, the 1st of the duplicates gave a recovery slightly below the listed acceptance limit. Due to isotope dilution technique, this apparent excedence is not expected to have a significant impact on data quality
Moisture content was assumed to be 1.3% as esimtated in NIST 1944 CoA.

No other criteria failures or exceedances.

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.

Reviewer and Title

2018-Jul-25
Date

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

# SVOC DATA PACKAGE

# SECTION 2: DATA SUMMARY REPORT

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

# Certificate of Analysis

**ALS Project Contact:** Whitney Davis
**ALS Project ID:** AECOM100
**ALS WO#:** L2125039
**Date of Report** 25-Jul-18

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

**COMMENTS:** **Chlorinated Pesticides by EPA 1699 (modified)**

Samples were analyzed by isotope dilution technique.

A high bias to results relative to the NIST reference values is expected due to the istope dilution technique employed.
Isotope dilution technique - where the samples are spiked with C-13 labelled targets prior to extraction - provides an inherent correction for losses during extraction and clean-up, a correction that is absent by other standard methods for environemental analysis.

Certified by:

Ron McLeod, PhD
Director, Air Toxics and Special Chemistries, Life Sciences

Results in this certificate relate only to the samples as submitted to the laboratory.
This report shall not be reproduced, except in full, without the written permission of ALS Canada Ltd.

# ALS Life sciences

## Sample Analysis summary Report

| **Sample Name** | **NIST 1944** | **% of Ref Value** | **NIST 1944 Duplicate** | **% of Ref Value** | **Ref Values** |
|---|---|---|---|---|---|
| ALS Sample ID | L2125039-1 | | L2125039-2 | | |
| Sample Size | 2.01 | | 2.02 | | |
| Sample size units | g | | g | | |
| Percent Solids | 98.7% | | 98.7% | | |
| Sample Matrix | Sediment | | Sediment | | |
| Sampling Date | n/a | | n/a | | |
| Extraction Date | 6-Jul-18 | | 6-Jul-18 | | |
| **Target Analytes** | **ng/g** | **%** | **ng/g** | **%** | **ng/g** |
| 2,4'-DDE | 23.0 | 121 | 23.6 | 124 | 19.0 |
| 4,4'-DDE | 83.4 | 97 | 80.9 | 94 | 86 |
| 2,4'-DDD | 77.8 | 205 | 57.8 | 152 | 38 |
| 4,4'-DDD | 139 | 129 | 117 | 108 | 108 |
| 4,4'-DDT | 189 | 111 | 199 | 117 | 170 |
| **Extraction Standards** | **% Rec** | | **% Rec** | | |
| 4,4'-DDE, 13C12- | 44 | | 62 | | |
| 4,4'-DDD, 13C12- | 37 | | 44 | | |
| 4,4'-DDT, 13C12- | 22 | | 26 | | |

# ALS Life sciences

## Quality Control Summary Report

| **Sample Name** | **Method Blank** | **Laboratory Control Sample** |
|---|---|---|
| ALS Sample ID | WG2815918-1 | WG2815918-2 |
| Sample Size | 2.00 | 1.00 |
| Sample size units | g | n/a |
| Percent Solids | n/a | n/a |
| Sample Matrix | QC | QC |
| Sampling Date | n/a | n/a |
| Extraction Date | 6-Jul-18 | 6-Jul-18 |
| **Target Analytes** | **ng/g** | **% Rec** |
| 2,4'-DDE | <0.0049 | 101 |
| 4,4'-DDE | 0.0350 | 99 |
| 2,4'-DDD | <0.015 | 107 |
| 4,4'-DDD | <0.010 | 96 |
| 4,4'-DDT | <0.013 | 96 |
| **Extraction Standards** | **% Rec** | **% Rec** |
| 4,4'-DDE, 13C12- | 84 | 82 |
| 4,4'-DDD, 13C12- | 82 | 89 |
| 4,4'-DDT, 13C12- | 84 | 88 |

# ALS Life sciences

## Sample Analysis Report

| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | **NIST 1944** | Sampling Date | n/a | | |
| ALS Sample ID | L2125039-1 | Extraction Date | 6-Jul-18 | | Approved: |
| Analysis Method | EPA 1699 (mod) | Sample Size | 2.01 | g | *R. Bakthiari* |
| Analysis Type | Sample | Percent Solids | 98.7% | | --e-signature-- |
| Sample Matrix | Sediment | Split Ratio | 1 | | 20-Jul-2018 |

| **Run Information** | **Run 1** |
|---|---|
| Filename | 6-180719B44 |
| Run Date | 20-Jul-18 14:55 |
| Final Volume | 1020 uL |
| Dilution Factor | 1 |
| Analysis Units | ng/g |
| Instrument - Column | HRMS-6 HP5MSUSR160544H |

| **Target Analytes** | | **Ret. Time** | **Conc. ng/g** | **EDL ng/g** | **Flags** | **EMPC ng/g** | **LQL** |
|---|---|---|---|---|---|---|---|
| 2,4'-DDE | | 20.98 | 23.0 | 0.051 | | | 1.0 |
| 4,4'-DDE | | 21.91 | 83.4 | 0.033 | | | 1.0 |
| 2,4'-DDD | | 22.15 | 77.8 | 0.052 | | | 1.0 |
| 4,4'-DDD | | 23.11 | 139 | 0.038 | | | 1.0 |
| 4,4'-DDT | | 24.17 | 189 | 0.054 | | | 1.0 |
| **Extraction Standards** | **ng** | | | | | | |
| 4,4'-DDE, 13C12- | 125 | 21.90 | 44 | 47-160 | | | |
| 4,4'-DDD, 13C12- | 125 | 23.11 | 37 | 5-150 | | | |
| 4,4'-DDT, 13C12- | 125 | 24.17 | 22 | 5-120 | | | |

| | |
|---|---|
| EDL | Indicates the Estimated Detection Limit, based on the measured background noise for this target in this sample. |
| LQL | Lower Quantification Limit, based on the lowest calibration level corrected for sample size, splits and dilutions. |
| M | Indicates that a peak has been manually integrated. |
| U | Indicates that this compound was not detected above the EDL. |
| J | indicates that a target analyte was detected below the calibrated range. |
| R | Indicates that the ion abundance ratio for this compound did not meet the acceptance criterion. |
| B | Indicates that this target was detected in the blank at greater than 10% of the sample concentration. |
| EMPC | Estimated Maximum Possible Concentration – elevated detection limit due to interference or positive id criterion failure |

# ALS Life sciences

## Sample Analysis Report

| | | | | |
|---|---|---|---|---|
| **Sample Name** | **NIST 1944 (Duplicate)** | Sampling Date | n/a | |
| ALS Sample ID | L2125039-2 | Extraction Date | 6-Jul-18 | Approved: |
| Analysis Method | EPA 1699 (mod) | Sample Size | 2.02 g | *R. Bakthiari* |
| Analysis Type | Sample | Percent Solids | 98.7% | --e-signature-- |
| Sample Matrix | Sediment | Split Ratio | 1 | 20-Jul-2018 |

| **Run Information** | **Run 1** |
|---|---|
| Filename | 6-180719B45 |
| Run Date | 20-Jul-18 15:29 |
| Final Volume | 1020 uL |
| Dilution Factor | 1 |
| Analysis Units | ng/g |
| Instrument - Column | HRMS-6 HP5MSUSR160544H |

| **Target Analytes** | | **Ret. Time** | **Conc. ng/g** | **EDL ng/g** | **Flags** | **EMPC ng/g** | **LQL** |
|---|---|---|---|---|---|---|---|
| 2,4'-DDE | | 20.99 | 23.6 | 0.011 | | | 1.0 |
| 4,4'-DDE | | 21.91 | 80.9 | 0.035 | | | 1.0 |
| 2,4'-DDD | | 22.16 | 57.8 | 0.055 | | | 1.0 |
| 4,4'-DDD | | 23.12 | 117 | 0.070 | | | 1.0 |
| 4,4'-DDT | | 24.18 | 199 | 0.076 | | | 1.0 |
| **Extraction Standards** | **ng** | | | | | | |
| 4,4'-DDE, 13C12- | 125 | 21.91 | 62 | 47-160 | | | |
| 4,4'-DDD, 13C12- | 125 | 23.11 | 44 | 5-150 | | | |
| 4,4'-DDT, 13C12- | 125 | 24.17 | 26 | 5-120 | | | |

| | |
|---|---|
| EDL | Indicates the Estimated Detection Limit, based on the measured background noise for this target in this sample. |
| LQL | Lower Quantification Limit, based on the lowest calibration level corrected for sample size, splits and dilutions. |
| 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. |
| B | Indicates that this target was detected in the blank at greater than 10% of the sample concentration. |
| EMPC | Estimated Maximum Possible Concentration – elevated detection limit due to interference or positive id criterion failure |

# SVOC DATA PACKAGE

# 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 | 36-139 |
| | 13C12-Dieldrin | 35 | 70-130 | 19-161 | 40-151 |
| | 13C12-Endrin | 35 | 70-130 | 20-157 | 35-155 |
| | 13C9-Endosulfan-II | 35 | 70-130 | 5-144 | 15-148 |
| | 13C12-44'-DDE | 35 | 70-130 | 26-169 | 47-160 |
| | 13C12-44'-DDD | 35 | 70-130 | 13-200 | 5-150 |
| | 13C12-44'-DDT | 35 | 70-130 | 13-200 | 5-120 |
| | d6-Methoxychlor | 35 | 70-130 | 8-200 | 5-120 |
| | 13C10-Mirex | 35 | 70-130 | 5-138 | 5-120 |

***Additional Continuing Calibration Details:***

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

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

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

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 =

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

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

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

***Data Calculations:***

**a) Analyte Concentrations:**

The relative response factor of each target relative to the standard against which it is to be calculated is determined using the area responses of both quantification ions via equation 9.1.
In cases where a native target is calculated against an exact labelled analogue, the quantification will be considered to be by isotope dilution. In other cases, the quantification will be considered to be by internal standard.

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

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**

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

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) |

ALS Life Sciences
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 | 6-180719B37 | 20-Jul-2018 10:51 |
| CS-2 | 6-180719B46 | 20-Jul-2018 16:02 |
| CS-3 | 6-180719B37 | 20-Jul-2018 10:51 |
| CS-4 | 6-180719B46 | 20-Jul-2018 16:02 |
| CS-5 | | |
| CS-6 | | |
| CS-7 | | |

Approved: *R. Bakthiari*
--e-signature--
20-Jul-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** | 1.423 | 1.479 | 1.423 | 1.479 | | | | 1.451 | 3% |
| **4,4'-DDE** | 1.142 | 1.135 | 1.142 | 1.135 | | | | 1.139 | 0% |
| **2,4'-DDD** | 1.055 | 0.957 | 1.055 | 0.957 | | | | 1.006 | 7% |
| **4,4'-DDD** | 1.079 | 1.180 | 1.079 | 1.180 | | | | 1.130 | 6% |
| **2,4'-DDT** | 0.965 | 0.832 | 0.965 | 0.832 | | | | 0.899 | 10% |
| **4,4'-DDT** | 0.899 | 0.917 | 0.899 | 0.917 | | | | 0.908 | 1% |
| **Extraction Standards** | | | | | | | | | |
| **4,4'-DDE, 13C12-** | 1.926 | 1.649 | 1.926 | 1.649 | | | | 1.788 | 11% |
| **4,4'-DDD, 13C12-** | 1.819 | 1.230 | 1.819 | 1.230 | | | | 1.525 | 27% |
| **4,4'-DDT, 13C12-** | 1.583 | 0.833 | 1.583 | 0.833 | | | | 1.208 | 44% |

# ALS Life sciences

## Calibration Report

| | |
|---|---|
| ALS Sample ID | **H6-18-CCV-0661** |
| Analysis Method | EPA 1699 (mod) |
| Analysis Type | Calibration |

| Filename | Inst # | Column | Run Date | Approved: | |
|---|---|---|---|---|---|
| 6-180719B37 | HRMS-6 | HP5MSUSR160544H | 20-Jul-2018 10:51 | | *R. Bakthiari*<br>--e-signature--<br>20-Jul-2018 |

| **Target Analytes** | **Ret. Time** | **Ion Ratio** | **Concentration ng/mL** | **Response** | **RRF** |
|---|---|---|---|---|---|
| **2,4'-DDE** | 20.95 | 1.56 | 50.00 | 3.25E+06 | 1.423 |
| **4,4'-DDE** | 21.89 | 1.54 | 50.00 | 2.61E+06 | 1.142 |
| **2,4'-DDD** | 22.12 | 1.63 | 50.00 | 2.41E+06 | 1.055 |
| **4,4'-DDD** | 23.1 | 1.61 | 50.00 | 2.33E+06 | 1.079 |
| **2,4'-DDT** | 23.19 | 1.64 | 50.00 | 2.08E+06 | 0.965 |
| **4,4'-DDT** | 24.17 | 1.56 | 50.00 | 1.69E+06 | 0.899 |
| **Extraction Standards** | | | | | |
| **4,4'-DDE, 13C12-** | 21.88 | 1.55 | 250.00 | 1.14E+07 | 1.926 |
| **4,4'-DDD, 13C12-** | 23.08 | 1.56 | 250.00 | 1.08E+07 | 1.819 |
| **4,4'-DDT, 13C12-** | 24.15 | 1.54 | 250.00 | 9.39E+06 | 1.583 |

# ALS Life sciences

## Calibration Report

| | |
|---|---|
| ALS Sample ID | **H6-18-CCV-0662** |
| Analysis Method | EPA 1699 (mod) |
| Analysis Type | Calibration |

| Filename | Inst # | Column | Run Date | Approved: | *R. Bakthiari* |
|---|---|---|---|---|---|
| 6-180719B46 | HRMS-6 | HP5MSUSR160544H | 20-Jul-2018 16:02 | | --e-signature--<br>20-Jul-2018 |

| **Target Analytes** | **Ret. Time** | **Ion Ratio** | **Concentration ng/mL** | **Response** | **RRF** |
|---|---|---|---|---|---|
| **2,4'-DDE** | 20.95 | 1.56 | 50.00 | 3.86E+06 | 1.479 |
| **4,4'-DDE** | 21.88 | 1.56 | 50.00 | 2.96E+06 | 1.135 |
| **2,4'-DDD** | 22.12 | 1.63 | 50.00 | 2.50E+06 | 0.957 |
| **4,4'-DDD** | 23.1 | 1.61 | 50.00 | 2.30E+06 | 1.180 |
| **2,4'-DDT** | 23.18 | 1.62 | 50.00 | 1.62E+06 | 0.832 |
| **4,4'-DDT** | 24.15 | 1.56 | 50.00 | 1.21E+06 | 0.917 |
| **Extraction Standards** | | | | | |
| **4,4'-DDE, 13C12-** | 21.88 | 1.54 | 250.00 | 1.30E+07 | 1.649 |
| **4,4'-DDD, 13C12-** | 23.08 | 1.57 | 250.00 | 9.73E+06 | 1.230 |
| **4,4'-DDT, 13C12-** | 24.15 | 1.55 | 250.00 | 6.59E+06 | 0.833 |

# ALS Life sciences

## Calibration Report

ALS Sample ID **H6-18-CCV-0661**
Analysis Method EPA 1699 (mod)
Analysis Type Calibration

| Filename | Inst # | Column | Run Date | Approved: | *R. Bakthiari*<br>--e-signature--<br>20-Jul-2018 |
|---|---|---|---|---|---|
| 6-180719B37 | HRMS-6 | HP5MSUSR160544H | 20-Jul-2018 10:51 | | |

| Target Analytes | Ret. Time | Ion Ratio | Concentration ng/mL | Response | RRF |
|---|---|---|---|---|---|
| **2,4'-DDE** | 20.95 | 1.56 | 50.00 | 3.25E+06 | 1.423 |
| **4,4'-DDE** | 21.89 | 1.54 | 50.00 | 2.61E+06 | 1.142 |
| **2,4'-DDD** | 22.12 | 1.63 | 50.00 | 2.41E+06 | 1.055 |
| **4,4'-DDD** | 23.1 | 1.61 | 50.00 | 2.33E+06 | 1.079 |
| **2,4'-DDT** | 23.19 | 1.64 | 50.00 | 2.08E+06 | 0.965 |
| **4,4'-DDT** | 24.17 | 1.56 | 50.00 | 1.69E+06 | 0.899 |
| **Extraction Standards** | | | | | |
| **4,4'-DDE, 13C12-** | 21.88 | 1.55 | 250.00 | 1.14E+07 | 1.926 |
| **4,4'-DDD, 13C12-** | 23.08 | 1.56 | 250.00 | 1.08E+07 | 1.819 |
| **4,4'-DDT, 13C12-** | 24.15 | 1.54 | 250.00 | 9.39E+06 | 1.583 |

# ALS Life sciences

## Calibration Report

ALS Sample ID **H6-18-CCV-0662**
Analysis Method EPA 1699 (mod)
Analysis Type Calibration

| Filename | Inst # | Column | Run Date | Approved: | |
|---|---|---|---|---|---|
| 6-180719B46 | HRMS-6 | HP5MSUSR160544H | 20-Jul-2018 16:02 | | *R. Bakthiari*<br>--e-signature--<br>20-Jul-2018 |

| Target Analytes | Ret. Time | Ion Ratio | Concentration ng/mL | Response | RRF |
|---|---|---|---|---|---|
| **2,4'-DDE** | 20.95 | 1.56 | 50.00 | 3.86E+06 | 1.479 |
| **4,4'-DDE** | 21.88 | 1.56 | 50.00 | 2.96E+06 | 1.135 |
| **2,4'-DDD** | 22.12 | 1.63 | 50.00 | 2.50E+06 | 0.957 |
| **4,4'-DDD** | 23.1 | 1.61 | 50.00 | 2.30E+06 | 1.180 |
| **2,4'-DDT** | 23.18 | 1.62 | 50.00 | 1.62E+06 | 0.832 |
| **4,4'-DDT** | 24.15 | 1.56 | 50.00 | 1.21E+06 | 0.917 |
| **Extraction Standards** | | | | | |
| **4,4'-DDE, 13C12-** | 21.88 | 1.54 | 250.00 | 1.30E+07 | 1.649 |
| **4,4'-DDD, 13C12-** | 23.08 | 1.57 | 250.00 | 9.73E+06 | 1.230 |
| **4,4'-DDT, 13C12-** | 24.15 | 1.55 | 250.00 | 6.59E+06 | 0.833 |

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

# 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 | WG2815918-1 | Extraction Date | 6-Jul-18 | |
| Analysis Method | EPA 1699 (mod) | Sample Size | 2 g | |
| Analysis Type | Blank | Percent Solids | n/a | |
| Sample Matrix | QC | Split Ratio | 1 | |

Approved:
*R. Bakthiari*
--e-signature--
20-Jul-2018

| **Run Information** | **Run 1** |
|---|---|
| Filename | 6-180719B43 |
| Run Date | 20-Jul-18 14:22 |
| Final Volume | 1020 uL |
| Dilution Factor | 1 |
| Analysis Units | ng/g |
| Instrument - Column | HRMS-6 HP5MSUSR160544H |

| **Target Analytes** | | **Ret. Time** | **Conc. ng/g** | **EDL ng/g** | **Flags** | **EMPC ng/g** | **LQL** |
|---|---|---|---|---|---|---|---|
| 2,4'-DDE | | NotFnd | <0.0049 | 0.0049 | U | | 1.0 |
| 4,4'-DDE | | 21.89 | 0.0350 | 0.0086 | J | | 1.0 |
| 2,4'-DDD | | NotFnd | <0.015 | 0.015 | U | | 1.0 |
| 4,4'-DDD | | NotFnd | <0.010 | 0.010 | U | | 1.0 |
| 4,4'-DDT | | NotFnd | <0.013 | 0.013 | U | | 1.0 |
| **Extraction Standards** | **ng** | | | | | | |
| 4,4'-DDE, 13C12- | 125 | 21.88 | 84 | 47-160 | | | |
| 4,4'-DDD, 13C12- | 125 | 23.08 | 82 | 5-150 | | | |
| 4,4'-DDT, 13C12- | 125 | 24.15 | 84 | 5-120 | | | |

| | |
|---|---|
| EDL | Indicates the Estimated Detection Limit, based on the measured background noise for this target in this sample. |
| LQL | Lower Quantification Limit, based on the lowest calibration level corrected for sample size, splits and dilutions. |
| M | Indicates that a peak has been manually integrated. |
| U | Indicates that this compound was not detected above the EDL. |
| J | indicates that a target analyte was detected below the calibrated range. |
| R | Indicates that the ion abundance ratio for this compound did not meet the acceptance criterion. |
| EMPC | Estimated Maximum Possible Concentration – elevated detection limit due to interference or positive id criterion failure |

# ALS Life sciences

## Laboratory Control Sample Analysis Report

| | | | | |
|---|---|---|---|---|
| **Sample Name** | **Laboratory Control Sample** | Sampling Date | n/a | |
| ALS Sample ID | WG2815918-2 | Extraction Date | 6-Jul-18 | |
| Analysis Method | EPA 1699 (mod) | Sample Size | 1 | n/a |
| Analysis Type | LCS | Percent Solids | n/a | |
| Sample Matrix | QC | Split Ratio | 1 | |

Approved:
*R. Bakthiari*
--e-signature--
20-Jul-2018

| **Run Information** | **Run 1** |
|---|---|
| Filename | 6-180719B40 |
| Run Date | 20-Jul-18 12:43 |
| Final Volume | 1020 uL |
| Dilution Factor | 1 |
| Analysis Units | % |
| Instrument - Column | HRMS-6 HP5MSUSR160544H |

| **Target Analytes** | **ng** | **Ret. Time** | **% Rec** | **Limits** | **Flags** |
|---|---|---|---|---|---|
| 2,4'-DDE | 25 | 20.95 | 101 | 24-123 | |
| 4,4'-DDE | 25 | 21.88 | 99 | 50-120 | |
| 2,4'-DDD | 25 | 22.12 | 107 | 50-120 | |
| 4,4'-DDD | 25 | 23.10 | 96 | 42-120 | |
| 4,4'-DDT | 25 | 24.17 | 96 | 50-120 | |
| **Extraction Standards** | **ng** | | | | |
| 4,4'-DDE, 13C12- | 125 | 21.88 | 82 | 26-169 | |
| 4,4'-DDD, 13C12- | 125 | 23.08 | 89 | 13-200 | |
| 4,4'-DDT, 13C12- | 125 | 24.15 | 88 | 13-200 | |

# SVOC DATA PACKAGE

# SECTION 6: INTERNAL RECORDS

Including:

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

# Extraction Workup Sheet

**Batch ID:** WG2815918

**Analysis:** NIST - OCP (HR)

Prep Procedure: BU-TM-1110/BU-TP-2103

**Analyst:** Radhika Menon

**Date:** 6-July-2018

## SUBSAMPLING

| Sample I.D. | Client I.D. | Subsample Size (g) |
|---|---|---|
| **WG2815918-1** | Method Blank | 2.00 |
| **WG2815918-2** | Laboratory Control Sample | 2.07 |
| **WG2815918-3** | Extraction and Injection STD. | — |
| **L2125039-1** | NIST 1944 (A) | 2.04 |
| **L2125039-2** | NIST 1944 (B) | 2.06 |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |

## BATCH TRACKING

| | Date/Time/Initials |
|---|---|
| **Subsampling:** | RM 6-July-18 3:30pm |
| **Client Labels Checked:** | RM 6-July-18 3:30pm |
| **Balance ID:** | 3955 |
| **Samples Spiked** | MSM |
| **Soxhlet Start Time:** | 6-Jul-18 4:20PM/MSM |
| **Soxhlets Reflux Properly:** | MSM |
| **Soxhlet End Time:** | 7-Jul-18 09:30 |
| **Rotovap Reduction:** | MSM |
| **Rotovap Temp Verified:** | MSM |
| **Split:** | — |
| **GPC - run set up:** | — |
| **Rotovap - post GPC:** | — |
| **Silicarb Column:** | JA2 10-July-2018 |
| **Rotovap - post silicarb:** | 10-July-2018 AP |
| **N-Evap Temp Verified:** | 10-July-2018 AP |
| **Micro/Robo Vial:** | 10-July-2018 AP |
| | ← |
| **Lipid Analysis:** | — |

**Batch ID:** WG2815918

**OCP Extraction Standard:**

| Sample I.D. | Volume (ul) | (Checkmark)<br>Spiked |
|---|---|---|
| WG2815918-1 | 20 | ✓ |
| WG2815918-2 | 20 | ✓ |
| WG2815918-3 | 20 | ✓ |
| L2125039-1 | 20 | ✓ |
| L2125039-2 | 20 | ✓ |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |

| | |
|---|---|
| **Syringe ID:** | 137 |
| **Standard:** | **HROCP-ES#1**-023A |
| **Date & Initials:** | 6-July-2018 |

**Spike Witnessing**

| | |
|---|---|
| **Chemist:** | Chemist's Initials<br>MSM |
| **Witness:** | Witness's Initials<br>RM |
| **Correct Syringe Obtained:** | Witness's Initials<br>RM |
| **Correct Standard Obtained:** | Witness's Initials<br>RM |
| **Correct Technique Followed:** | Witness's Initials<br>RM |

**OCP Native Standard:**

| Sample I.D. | Volume (ul) | (Checkmark)<br>Spiked |
|---|---|---|
| WG2815918-2 | 20 | ✓ |
| WG2815918-3 | 20 | ✓ |
| | | |

| | |
|---|---|
| **Syringe ID:** | 202 |
| **Standard:** | **HROCP-NS#1-** 016B |
| **Date & Initials:** | 6-July-2018 MSM |

| | |
|---|---|
| **Batch ID:** | WG2815918 |

## OCP Injection Standard:

| Sample I.D. | Volume (ul) | (Checkmark)<br>Spiked |
|---|---|---|
| WG2815918-1 | 20 | ✓ |
| WG2815918-2 | 20 | ✓ |
| WG2815918-3 | 20 | ✓ |
| L2125039-1 | 20 | ✓ |
| L2125039-2 | 20 | ✓ |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |

| | |
|---|---|
| **Syringe ID:** | 196 |
| **Standard:** | HROCP-IS#1-014B |
| **Date & Initials:** | 10-July-2018 AP |

| | Chemist's Initials |
|---|---|
| **Correct Syringe Obtained:** | AP |
| **Correct Standard Obtained:** | AP |
| **Correct Technique Followed:** | AP |

## Reagent Lot Numbers:

| Reagent | Lot# | Manufacturer |
|---|---|---|
| Acetone | 182822 | |
| Hexane | 182082 | |
| DCM | 183227 | |
| Toluene | 103253 | |
| Nonane | ORG-WAKONON- — | |
| 1:1 DCM:HEX | ORG-DH2- — | |
| Sodium Sulphate | ORG-SSU- 1872 | |
| Acid Silica | ORG-ASI- — | |
| Neutral Silica | ORG-NSI- — | |
| Alumina | ORG-ALU- — | |
| 2% Deactivated Silica | ORG-2%DAS- 193 | |
| Chromacarb | ORG-CC- 225 | |

## Comments:

**NOTE: Label and Save All Columns.**

**Batch ID:** WG2815918

**Procedure:**

**Extraction:**

- Subsample size = 2g
- Mix with sodium sulphate (Use enough sodium sulphate and mixing to create a free-flowing mixture.)
- Spike Extraction and Native Standards.
- Soxhlet extract in DCM.
- Roto-vap down to ~2ml

- 1mL Pass through OCP Silicarb clean up

-Sili Carb Column - Load sample with 3x1mL hexane rinses

    - **F1** = 25 mL of Hexane
    - **F2** =250mL of 1:1 DCM:Hexane

-Reduce F2 to 1mL, vortex and transfer to robovial

- Spike injection standard and submit FV=1020ul

Approval of Deviation from Standard Method

☐ Procedure does not deviate from Standard Method. **(Batch Writer):** ____________________

☐ Procedure does deviate from Standard Method. **Approved (Supervisor/Manager):** ____________________

| 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: *R. Bakthiari*
--e-signature--
20-Jul-2018

**CS3 RRF Check**

$$\text{RRF} = \frac{\text{Response of 4,4'-DDE}}{\text{Response of 13C12-4,4'DDE}} \times \frac{\text{Concentration of 13C2-4,4'DDE}}{\text{Concentration of DDE}}$$

Calculated Value | Value from TargetLynx

$$\textbf{RRF} = \frac{2609863.40}{11429690.50} \times \frac{250.00}{50} = 1.14$$

Calculated Value: **1.14** Value from TargetLynx: **1.14**

**Calculation of 4,4'-DDE amount in L2125039-1**

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

$$\textbf{ng/g} = \frac{2832138.5}{1855507.9} \times \frac{125}{1.14 * 2.01} = 83.374$$

Calculated Value: **83.374** Value from TargetLynx: **83.4**

**Calculation of 13C12-4,4'-DDE Recovery in L2125039-1**

$$\text{% Recovery} = \frac{\text{Response of 13C12-4,4'-DDE}}{\text{Response of 13C12-PCB-101}} \times \frac{\text{ng of 13C12-PCB-101} * 100}{\text{Mean RRF} * \text{Amount Spiked}}$$

$$\textbf{% Recovery} = \frac{1855507.9}{933448.2} \times \frac{50 * 100}{1.79 * 125} = 44$$

Calculated Value: **44** Value from TargetLynx: **44 %**