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Proficiency Test Final Report AQA 26-06 Pesticides in Soil and Compost

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SUMMARY

This report presents the results of AQA 26-06 Pesticides in Soil and Compost.

Twenty-seven laboratories registered to participate, and all submitted results.

The sample set consisted of 2 soil samples and one compost sample, each fortified with a range of pesticide standard solutions.

The assigned values for all scored analytes were the robust averages of participants' results. The associated uncertainties were evaluated from the robust standard deviations of the participants' results.

The consensus of participants' results is not traceable to any external reference, so although expressed in SI units, metrological traceability has not been established.

The outcomes of the study were assessed against the aims as follows:

- *Assess participants' ability to correctly identify pesticides in soil and compost.*

Laboratory 6 reported results for all 14 analytes for which *z*-scores were calculated.

In this study, samples were fortified with pesticides at concentrations near typical laboratory reporting limits. There was a record number of false negative results (total of 46 results). Participants may need to review their limits of reporting.

Six participants reported additional analytes (total of 12 results).

- *Compare participants' performances and assess their capabilities in the measurement of pesticides in soil and compost.*

Of 206 *z*-scores, 186 (90%) returned $|z| \leq 2.0$, indicating an acceptable performance.

Of 194 E_n-scores, 159 (82%) returned $|E_n| < 1.0$, indicating agreement of the participant's result with the assigned value within their respective uncertainties.

- *Evaluate participants' methods for the measurement of pesticides in soil and compost.*

Most participants used the same procedure for the analysis of the soil samples as compared to the compost sample. However, for some analytes, there was poorer performance in compost as compared to soil, as demonstrated by lower analyte recoveries or greater variability between participants' results. This may indicate that the methods being used for soil may not be fully suitable for compost. Laboratories may need to review their analytical procedures and optimise them for pesticides in compost.

The most common methodology used in this study was solid-liquid extraction with mechanical agitation, dichloromethane/acetone as the extraction solvent, and with no additional clean-up step. Both GC and LC techniques were used, depending on the analyte.

- *develop the practical application of measurement uncertainty, and provide participants with information that will be useful in assessing their uncertainty evaluations;*

Of 307 numeric results reported for the analytes of interest, 296 (96%) were reported with an associated expanded measurement uncertainty. The magnitude of the reported relative uncertainties was within the range of 2.6% to 240%.

- *Produce materials that can be used in method validation and as control samples.*

The test samples produced for this study are homogeneous and are well characterised. Surplus of these samples is available for purchase and can be used for quality control and for method validation purposes.

1 INTRODUCTION

1.1 NMIA Proficiency Testing Program

The National Measurement Institute Australia (NMIA) is responsible for Australia's national measurement infrastructure, providing a range of services including a chemical proficiency testing program.

Proficiency testing (PT) is the 'evaluation of participant performance against pre-established criteria by means of interlaboratory comparisons'.[1] NMIA PT studies target chemical testing in areas of high public significance such as trade, environment, law enforcement and food safety. NMIA offers studies in:

- per- and polyfluoroalkyl substances in soil, biosolids, water, biota, food, and consumer goods;
- hydrocarbons, phenols, and other organic compounds in soil and water;
- pesticide residues in soil, water, fruit, vegetables and herbs;
- metals in soil, water, food, filters, and paint;
- nutrients, anions, and physical tests in water and soil;
- chlorophyll a in water; and
- controlled drug assay, drugs in wipes, and clandestine laboratory.

1.2 Study Aims

The aims of the study were to:

- assess participants' ability to correctly identify pesticides in soil and compost;
- compare participants' performances and assess their capabilities in the measurement of pesticides in soil and compost;
- evaluate participants' methods for the measurement of pesticides in soil and compost;
- develop the practical application of measurement uncertainty, and provide participants with information that will be useful in assessing their uncertainty evaluations; and
- produce materials that can be used in method validation and as control samples.

The choice of the test method was left to the participating laboratories.

1.3 Study Conduct

The conduct of NMIA PT studies is described in the NMIA Study Protocol for Proficiency Testing.[2] The statistical methods used are described in the NMIA Chemical Proficiency Testing Statistical Manual.[3] These documents have been prepared with reference to ISO/IEC 17043 and The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories.[1,4]

NMIA is accredited by the National Association of Testing Authorities, Australia (NATA) to ISO/IEC 17043 as a provider of proficiency testing schemes. This study falls within the scope of NMIA's accreditation (Accreditation No. 198; Site No. 14485).

2 STUDY INFORMATION

2.1 Study Timetable

The timetable of the study was:

Invitations sent 02/03/2026

Samples sent 23/03/2026

Results due 28/04/2026

Interim Report 01/05/2026

Preliminary Report 07/05/2026

2.2 Participation and Laboratory Code

Twenty-seven laboratories enrolled in this study, and all participants were assigned a confidential laboratory code number. All participants submitted results.

2.3 Selection of Pesticides and Test Material Preparation

The pesticides and spiked values used in this study were selected with consideration to:

- a variety of pesticides amenable to gas and/or liquid chromatography; and
- the National Environment Protection (Assessment of Site Contamination) Measure Schedule B1 Guideline on Investigation Levels for Soil and Groundwater.[5]

Three test samples were provided for analysis:

- Samples S1 and S2 each consisted of 50 g soil spiked with various pesticides.
- Sample S3 consisted of 45 g compost spiked with various pesticides.

Details of the spiked analytes and levels are presented in Table 1. Incurred values have not been included in the spiked values.

Table 1 Spiked Values of Test Samples

Sample	Analyte	Spiked Value (mg/kg)
S1	Total Chlordane	0.100
S1	Chlorpyrifos	0.0539
S1	p,p'-DDD	0.0152
S1	Dieldrin	0.00617
S1	Endosulfan sulfate	0.00199
S1	Ethion	0.0846
S1	Fenitrothion	0.0710
S1	Fenvalerate	0.152
S1	Lindane	0.0895
S1	Trifluralin	0.503
S2	Acephate	1.00
S2	Bromacil	0.707
S2	Carbendazim	0.804
S2	2,4-D	0.351

Sample	Analyte	Spiked Value (mg/kg)
S2	Fipronil	0.803
S2	Glyphosate	1.50
S2	Imidacloprid	0.604
S2	MCPA	0.201
S2	Metsulfuron-methyl	2.50
S2	Omethoate	0.707
S3	Acephate	1.51
S3	Total Chlordane	2.01
S3	Diazinon	1.01
S3	Dieldrin	0.502
S3	Endosulfan sulfate	0.706
S3	Ethion	0.202
S3	Glyphosate	1.51
S3	Imidacloprid	1.50
S3	Lindane	0.909
S3	Simazine	0.576

Further information on the preparation of the samples is given in Appendix 1.

2.4 Homogeneity and Stability of Test Materials

No homogeneity or stability testing was conducted for Sample S1 and S2. These samples were prepared, packaged, stored, and dispatched using a process that has been demonstrated to produce sufficiently homogeneous and stable samples in previous NMIA pesticides in soil PT studies.

Sample S3 compost was a new matrix introduced in this study. Therefore, homogeneity and stability testing was conducted for the pesticides of interest in this new matrix. The sample was demonstrated to be sufficiently homogenous and stable for the purposes of this PT study (Appendix 2).

To further assess possible instability, the results returned by participants were compared to the spiked values. Assigned values for scored analytes were within 58% to 94% of the spiked values (excluding Sample S2 glyphosate which had a significant incurred value not included in the spiked value). This is similar to ratios observed in previous NMIA pesticides in soil PT studies, for example, as presented in the report for PT study AQA 16-04.[6] Analytes were only scored when there was a reasonable consensus between participants' results.

Participants' results also gave no reason to question the homogeneity or transport stability of the samples (Appendix 2).

2.5 Sample Storage, Dispatch and Receipt

The test samples were refrigerated at 4°C prior to dispatch. Participants were sent 50 g of spiked soil for each of Samples S1 and S2, and 45 g of spiked compost for Sample S3. The samples were packed in polystyrene foam boxes with cooler bricks and dispatched by courier on 23 March 2026.

The following items were also packaged with the samples:

- a letter which included a description of the test samples and instructions for participants; and
- a form for participants to return to confirm the receipt and condition of the samples.

An Excel spreadsheet for the electronic reporting of results was emailed to participants.

2.6 Instructions to Participants

Participants were provided with a list of possible analytes spiked into the samples (Table 2).

Table 2 List of Possible Analytes

Sample S1 Soil	Aldrin	Cyprodinil	Ethion	Parathion
	Atrazine	p,p'-DDD	Fenitrothion	Parathion-methyl
	Bifenthrin	p,p'-DDE	Fenthion	Permethrin
	Buprofezin	p,p'-DDT	Fenvalerate	Propiconazole
	Total Chlordane	Total DDT	Heptachlor	Simazine
	Chlorpyrifos	Diazinon	Lindane	Trifluralin
	Cyfluthrin	Dieldrin	Maldison (Malathion)	
	Cypermethrin	Endosulfan sulfate	Mevinphos	
Sample S2 Soil	Acephate	2,4-D	MCPA	Picloram
	Aldicarb	Dicamba	Methamidophos	Propazine
	AMPA	Fipronil	Methomyl	Thiodicarb
	Bromacil	Glyphosate	Metsulfuron-methyl	Triadimefon
	Carbendazim	Imidacloprid	Omethoate	Trichlopyr
Sample S3 Compost	Acephate	p,p'-DDE	Fenitrothion	Omethoate
	Aldrin	p,p'-DDT	Glyphosate	Parathion
	AMPA	Total DDT	Heptachlor	Permethrin
	Bifenthrin	Diazinon	Imidacloprid	Picloram
	Total Chlordane	Dicamba	Lindane	Propiconazole
	Chlorpyrifos	Dieldrin	Maldison (Malathion)	Simazine
	2,4-D	Endosulfan sulfate	MCPA	Trichlopyr
	p,p'-DDD	Ethion	Methomyl	

Participants were instructed as follows:

- If samples cannot be analysed on the day of receipt, please store them refrigerated.
- Quantitatively analyse the samples using your routine test method. It is recommended to mix all samples well before subsampling.
- Participants need not test for all listed analytes.
- For each analyte in each sample, report a single result on as received basis in units of mg/kg. This figure will be used in all statistical analysis in the study report.

- Please report the result for the named analyte only - do not include values for metabolites or breakdown products.
- Report results as you would report to a client, i.e. corrected for recovery or not according to your standard procedure and applying the limit of reporting of the method used for analysis (no limit of reporting has been set for this study).
- For each analyte in each sample, report the associated expanded uncertainty in units of mg/kg (e.g. 0.50 ± 0.02 mg/kg).
- If determined, report your percentage recovery. This will be presented in the report for information only.
- Report any listed pesticide not tested as NT as the result.
- Report the basis of your uncertainty evaluations as requested in the results sheet (e.g. uncertainty budget, repeatability precision, long term result variability).
- Please complete the method details as requested in the Methodology sheet.
- Return the completed results sheet by email (proficiency@measurement.gov.au).
- Please return results by 20 April 2026. Late results may not be included in the study report.

The results due date was extended to 28 April 2026 due to public holidays.

2.7 Interim Report and Preliminary Report

An Interim Report was emailed to all participants on 1 May 2026.

A Preliminary Report was emailed to all participants on 7 May 2026. This report included a summary of the results reported by participants, assigned values, performance coefficients of variation (PCVs), *z*-scores and E_n-scores for each analyte in this study. No data from the Preliminary Report has been changed in the present Final Report, except for the spiked value and scoring of Sample S3 Simazine (Table 3).

A low recovery of the spiked value for Sample S3 Simazine was observed, and an investigation was conducted. The spiking standard solution was a lower concentration than the expected preparation value, potentially due to an inaccurate purity for the neat standard.

The spiked value has therefore been adjusted accordingly. Participants' results reported for this analyte were in good agreement with each other and with the adjusted spiked value. An assigned value was set for this analyte, and results have now been scored.

Table 3 Sample S3 Simazine Expected Spiked Value and Adjusted Spiked Value

Sample	Analyte	Expected Spiked Value (mg/kg)	Adjusted Spiked Value (mg/kg)
S3	Simazine	1.20	0.576

3 PARTICIPANT LABORATORY INFORMATION

3.1 Test Methods Reported by Participants

Participants were requested to provide information about their test methods. Responses received are presented in Appendix 4. Some responses may be modified so that the participant cannot be identified.

3.2 Basis of Participants' Measurement Uncertainty Evaluation

Participants were requested to provide information about their basis of measurement uncertainty (MU). Responses received are presented in Table 4. Some responses may be modified so that the participant cannot be identified.

Table 4 Basis of Uncertainty Evaluation

Lab. Code	Approach to Evaluating MU	Information Sources for MU Evaluation*		Guide Document for Evaluating MU
		Precision	Method Bias	
1	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - SS Duplicate analysis Instrument calibration	Instrument calibration Recoveries of SS Standard purity	ISO/GUM
2	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - SS Duplicate analysis Instrument calibration	Instrument calibration Recoveries of SS	Eurachem/ CITAC Guide
3	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - SS Duplicate analysis	CRM Instrument calibration Recoveries of SS	Eurachem/ CITAC Guide
4	Coverage factor not reported	Control samples - SS	Recoveries of SS	
5	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - SS	Recoveries of SS	Eurachem/ CITAC Guide
6	Top Down - precision and evaluations of the method and laboratory bias Coverage factor not reported	Control samples - SS Duplicate analysis	CRM Instrument calibration Recoveries of SS Standard purity	ISO/GUM
7	Coverage factor not reported			
8	Bottom Up (ISO/GUM, fish bone/cause and effect diagram) k = 2	Duplicate analysis	Recoveries of SS	Eurachem/ CITAC Guide
9	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples	CRM Laboratory bias from PT studies	Nordtest Report TR537
10	Standard deviation of replicate analyses multiplied by 2 or 3 k = 2			ISO/GUM

Lab. Code	Approach to Evaluating MU	Information Sources for MU Evaluation*		Guide Document for Evaluating MU
		Precision	Method Bias	
11	Top Down - precision and evaluations of the method and laboratory bias Coverage factor not reported	Duplicate analysis	Instrument calibration	
12	Top Down - precision and evaluations of the method and laboratory bias Coverage factor not reported	Control samples - SS Duplicate analysis	CRM Instrument calibration Recoveries of SS	Eurachem/ CITAC Guide
13	Top Down - precision and evaluations of the method and laboratory bias Coverage factor not reported	Control samples - CRM Duplicate analysis	CRM	Eurachem/ CITAC Guide
14	Top Down - precision and evaluations of the method and laboratory bias Coverage factor not reported	Duplicate analysis	Instrument calibration	ISO/GUM
15	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - SS	Recoveries of SS	Eurachem/ CITAC Guide
16	Top Down - precision and evaluations of the method and laboratory bias k = 2	Duplicate analysis	CRM	ISO/GUM
17	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - CRM Duplicate analysis Instrument calibration	CRM Instrument calibration	Eurachem/ CITAC Guide
18	Top Down - precision and evaluations of the method and laboratory bias Coverage factor not reported	Control samples - CRM Duplicate analysis		Eurachem/ CITAC Guide
19	Top Down - precision and evaluations of the method and laboratory bias Coverage factor not reported	Control samples - SS Duplicate analysis	CRM Recoveries of SS	Control Chart
20	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - CRM Duplicate analysis Instrument calibration	CRM Instrument calibration Recoveries of SS Standard purity	NMIA Uncertainty Course
21	Top Down - precision and evaluations of the method and laboratory bias k = 2	Duplicate analysis	Recoveries of SS	Eurachem/ CITAC Guide
22	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - CRM Duplicate analysis		ISO/GUM

Lab. Code	Approach to Evaluating MU	Information Sources for MU Evaluation*		Guide Document for Evaluating MU
		Precision	Method Bias	
23	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - SS Duplicate analysis Instrument calibration	Instrument calibration Recoveries of SS Standard purity	Eurachem/ CITAC Guide
24	Top Down - precision and evaluations of the method and laboratory bias Coverage factor not reported	Control samples - CRM Duplicate analysis Instrument calibration	CRM Instrument calibration Recoveries of SS Standard purity	NMIA Uncertainty Course
25	Standard uncertainty based on historical data k = 2	Duplicate analysis Instrument calibration	CRM Instrument calibration Standard purity	Eurachem/ CITAC Guide
26	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - SS Instrument calibration	Instrument calibration Recoveries of SS Standard purity	Eurachem/ CITAC Guide
27	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - SS		Eurachem/ CITAC Guide

* CRM = Certified Reference Material; SS = Spiked Samples

3.3 Participants' Comments

Participants were invited to make comments on the samples, study, or possible future studies. Such feedback may be useful in improving future studies. Participants' comments are presented in Table 5. Some comments may be modified so that the participant cannot be identified.

Table 5 Participants' Comments

Lab. Code	Participant's Comments	Study Coordinator's Response
9	Concentration range for OCP >0.1 mg/kg would be nice.	Thank you for your feedback. Based on previous suggestions from participants, this study was partially designed to give feedback on laboratories' performance when analysing pesticides at lower levels in soil. For future PT studies, we will ensure that analytes are included at a broader range of concentration levels.

4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

4.1 Results Summary

Participant results are listed in Tables 6 to 35 with summary statistics and other estimates of analyte mass fraction. Bar charts of results and performance scores are presented in Figures 2 to 31. An example chart with interpretation guide shown in Figure 1.

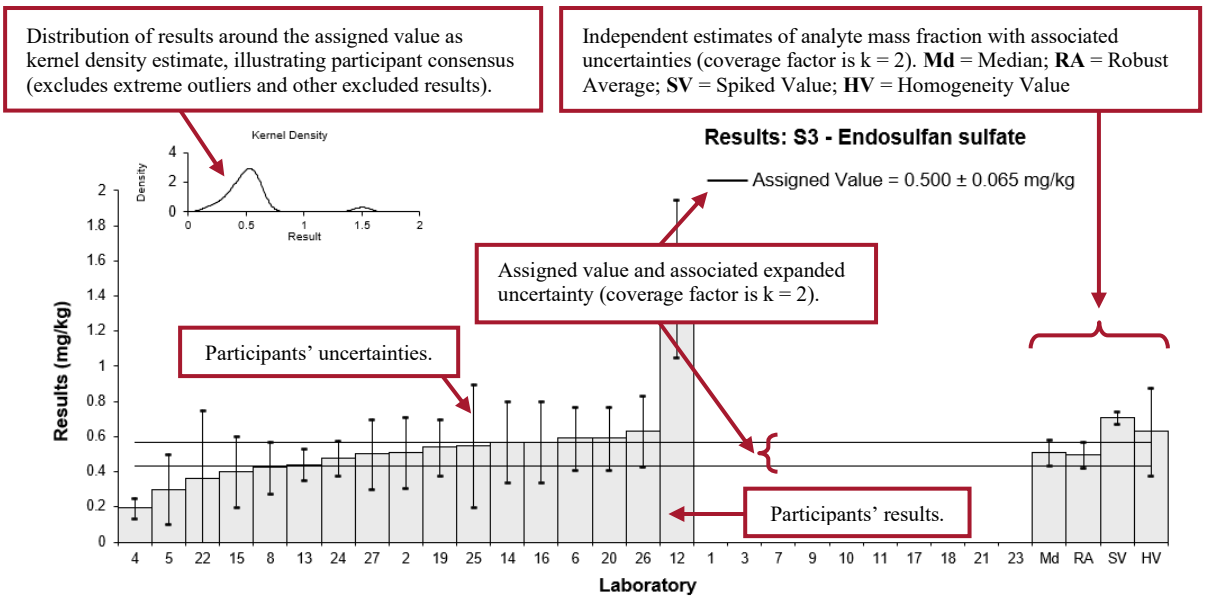


Figure 1 Guide to Presentation of Results

4.2 Outliers and Extreme Outliers

Outliers were results less than 50% and greater than 150% of the robust average, and these were removed before the calculation of the assigned value (when using the robust average).[3,4] Extreme outliers were obvious blunders, e.g. results reported with incorrect units, or for a different analyte, and such results were removed before the calculation of all statistics.[3]

4.3 Assigned Value

The assigned value is defined as the 'value attributed to a particular property or characteristic of a proficiency test item'.[1] In this PT study, the property is the mass fraction of the analytes in the samples. The assigned values for all scored analytes were the robust averages of participants' results, outliers removed; and the expanded uncertainties were evaluated from the associated robust standard deviations (robust SDs) (Appendix 3).

4.4 Robust Average and Robust Between-Laboratory Coefficient of Variation

The robust averages and associated expanded MUs, and robust coefficients of variation (robust CVs, a measure of the variability of participants' results) were calculated as described in ISO 13528.[7] The robust CVs as presented in the Section 5 statistics tables are the robust between-laboratory CVs including outliers but excluding extreme outliers. Everywhere else in the report, the robust between-laboratory CV refers to the robust CV calculated with both outliers and extreme outliers removed.

4.5 Performance Coefficient of Variation

The performance coefficient of variation (PCV) is a set measure of the between-laboratory variation that in the judgement of the study coordinator would be expected from participants, given the levels of analytes present. The PCV is not the CV of participants' results; it is set by the study coordinator and is based on the mass fraction of the analytes and experience from previous studies, and is supported by mathematical models such as the Thompson-Horwitz

equation.[8] By setting a realistic value for the PCV, a participant's performance does not depend on other participants' performance, and can be compared from study to study when the PCV remains the same.

4.6 Standard Deviation for Proficiency Assessment

The standard deviation for proficiency assessment (σ, SDPA) is the product of the assigned value (X) and the PCV, as presented in Equation 1.

$$\sigma = X \times PCV \qquad \textit{Equation 1}$$

4.7 z-Score

For each participant's result, a z-score is calculated according to Equation 2.

$$z = \frac{(\chi - X)}{\sigma} \qquad \textit{Equation 2}$$

where:

z is z-score

χ is a participant's result

X is the assigned value

σ is the SDPA from Equation 1

For the absolute value of a z-score: $|z| \leq 2.0$ is acceptable; $2.0 < |z| < 3.0$ is questionable; and $|z| \geq 3.0$ is not acceptable.

To account for potential low bias in consensus value due to inefficient methodologies, scores may be adjusted for a 'maximum acceptable result' (see Section 6.3 for more information).

4.8 E_n-Score

The E_n-score is complementary to the z-score in assessment of laboratory performance. The E_n-score includes expanded uncertainty and is calculated according to Equation 3.

$$E_n = \frac{(\chi - X)}{\sqrt{U_\chi{}^2 + U_X{}^2}} \qquad \textit{Equation 3}$$

where:

E_n is E_n-score

χ is a participant's result

X is the assigned value

U_χ is the expanded uncertainty of the participant's result

U_X is the expanded uncertainty of the assigned value

For the absolute value of an E_n-score: $|E_n| < 1.0$ is acceptable; and $|E_n| \geq 1.0$ is not acceptable.

4.9 Traceability and Measurement Uncertainty

Laboratories accredited to ISO/IEC 17025 must establish and demonstrate the traceability and measurement uncertainty associated with their test results.[9]

Guidelines for quantifying uncertainty in analytical measurement are described in the Eurachem/CITAC Guide.[10]

5 TABLES AND FIGURES

Table 6

Sample Details

Sample No.	S1
Matrix	Soil
Analyte	Total Chlordane
Unit	mg/kg

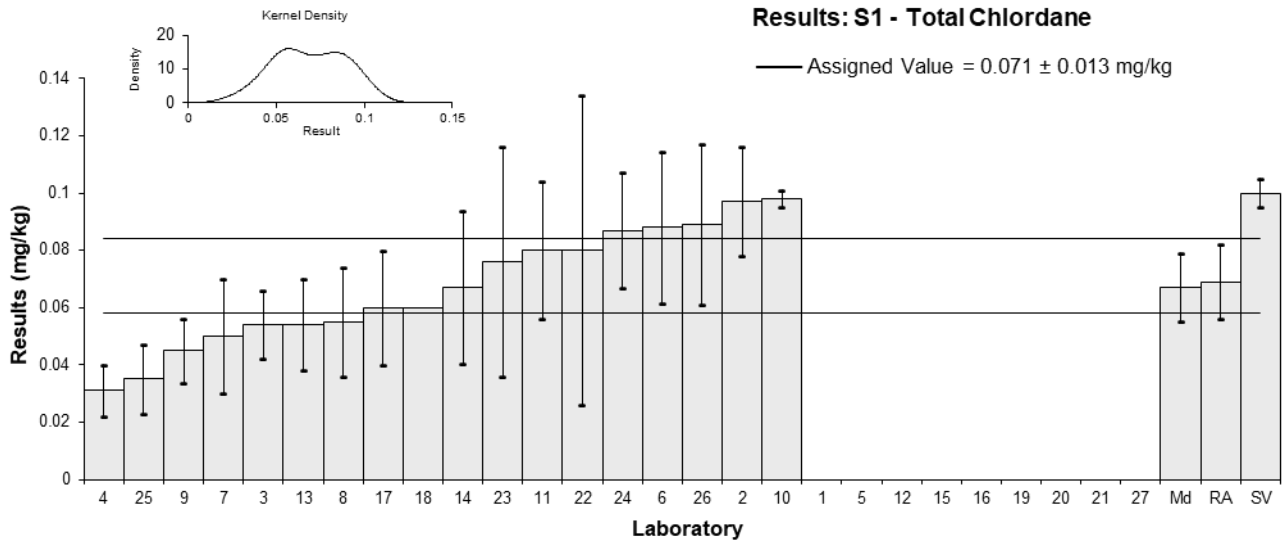
Participant Results

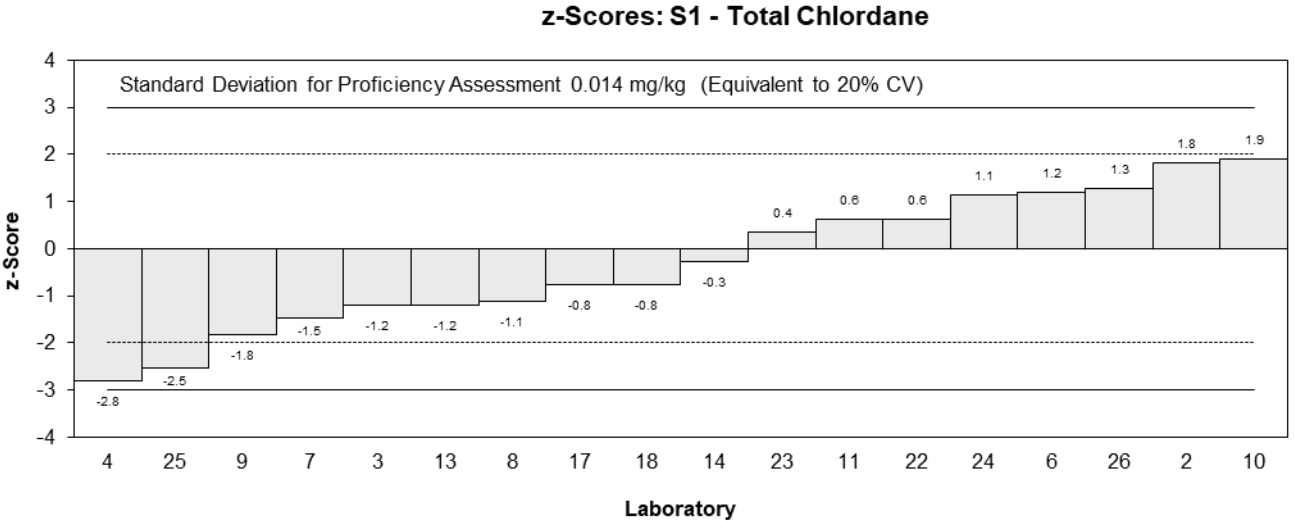
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	<0.5	NR	NR		
2	0.097	0.019	NR	1.83	1.13
3	0.054	0.01188	NR	-1.20	-0.97
4*	0.031	0.009	129	-2.82	-2.53
5	<0.1	NR	NR		
6	0.088	0.0264	93	1.20	0.58
7	0.05	0.02	NR	-1.48	-0.88
8	0.055	0.019	76	-1.13	-0.69
9	0.0450	0.0113	NR	-1.83	-1.51
10	0.098	0.003	NR	1.90	2.02
11	0.080	0.024	NR	0.63	0.33
12	<0.1	NR	NR		
13	0.054	0.016	NR	-1.20	-0.82
14	0.067	0.0268	141	-0.28	-0.13
15	<0.1	NR	80-120		
16	<0.2	0.2	NR		
17	0.06	0.02	117.67	-0.77	-0.46
18	0.06	NR	NR	-0.77	-0.85
19	< 0.1	NR	NR		
20	< 0.1	NR	NR		
21	<0.1	0.03	NR		
22	0.080	0.054	70	0.63	0.16
23	0.076	0.04	NR	0.35	0.12
24	0.087	0.02	NR	1.13	0.67
25**	0.035	0.012	NR	-2.54	-2.03
26	0.089	0.028	90	1.27	0.58
27	<0.1	NR	NR		

* Outlier, ** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	0.071	0.013
Spike Value	0.100	0.005
Robust Average	0.069	0.013
Median	0.067	0.012
Mean	0.0689	
N	17	
Max	0.098	
Min	0.031	
Robust SD	0.022	
Robust CV	31%	


Results: S1 - Total Chlordane
Assigned Value = 0.071 ± 0.013 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
4 25 9 7 3 13 8 17 18 14 23 11 22 24 6 26 2 10 1 5 12 15 16 19 20 21 27 Md RA SV


z-Scores: S1 - Total Chlordane
Standard Deviation for Proficiency Assessment 0.014 mg/kg (Equivalent to 20% CV)
z-Score
Laboratory
4 25 9 7 3 13 8 17 18 14 23 11 22 24 6 26 2 10
-2.8 -2.5 -1.8 -1.5 -1.2 -1.2 -1.1 -0.8 -0.8 -0.3 0.4 0.6 0.6 1.1 1.2 1.3 1.8 1.9

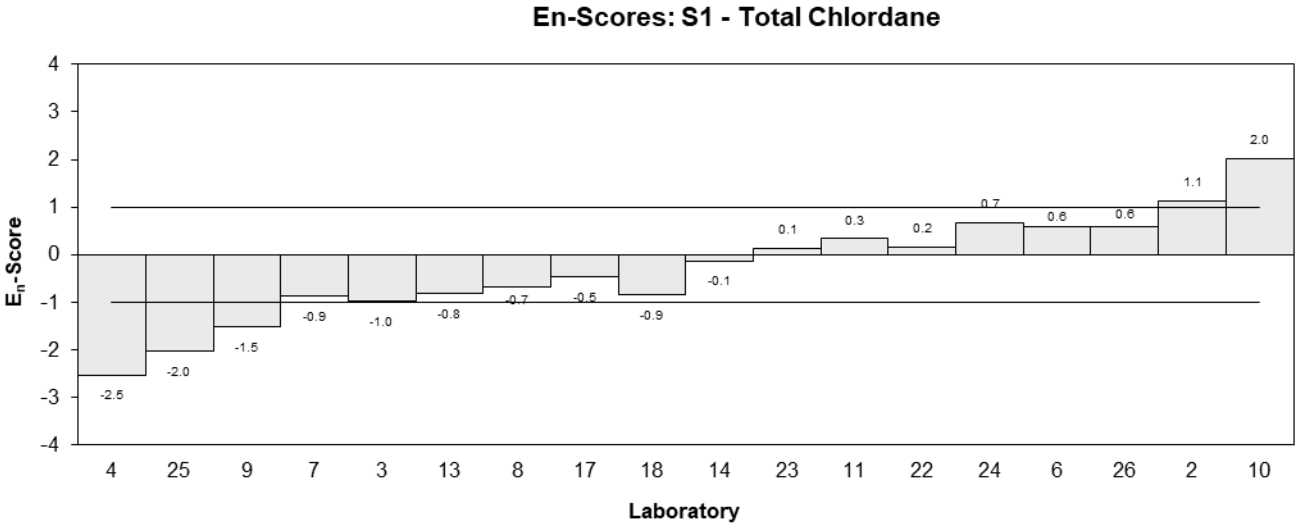

En-Scores: S1 - Total Chlordane
En-Score
Laboratory
4 25 9 7 3 13 8 17 18 14 23 11 22 24 6 26 2 10
-2.5 -2.0 -1.5 -0.9 -1.0 -0.8 -0.7 -0.5 -0.9 -0.1 0.1 0.3 0.2 0.7 0.6 0.6 1.1 2.0

Figure 2

Table 7

Sample Details

Sample No.	S1
Matrix	Soil
Analyte	Chlorpyrifos
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	<0.5	NR	NR
2	NR	NR	NR
3	<0.5	NR	NR
4	0.021	0.006	129
5	<0.1	NR	NR
6	0.039	0.0117	86
7	<0.05	0.6	NR
8	0.065	0.013	96
9	NR	NR	NR
10	0.014	0.001	NR
11	<0.050	NR	NR
12	<0.1	NR	NR
13	<0.05	NR	NR
14	<0.2	NR	NR
15	<0.1	NR	80-120
16	<0.1	0.1	NR
17	<0.05	NR	119.4
18	0.05	NR	NR
19	< 0.2	NR	NR
20	< 0.2	NR	NR
21	<0.1	0.03	NR
22	0.046	0.027	72
23	0.046	0.02	NR
24	0.022	0.01	NR
25	0.0278	0.0057	NR
26	0.05	0.015	90
27	<0.1	NR	NR

Statistics

Assigned Value	Not Set	
Spike Value	0.0539	0.0027
Robust Average	0.038	0.015
Median	0.043	0.013
Mean	0.038	
N	10	
Max	0.065	
Min	0.014	
Robust SD	0.018	
Robust CV	48%	

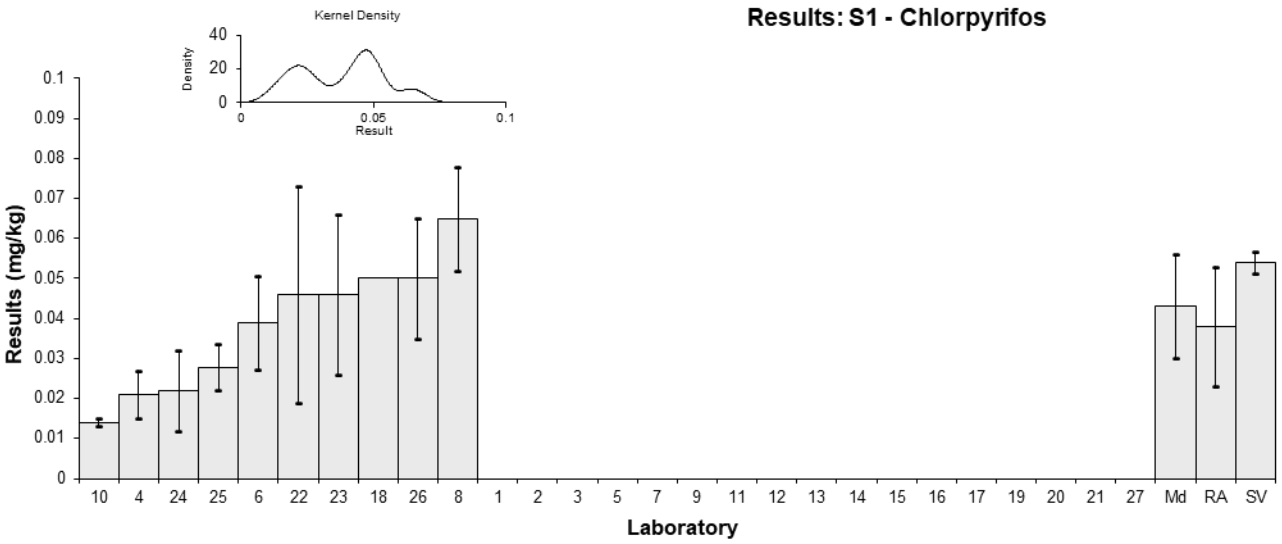

Results: S1 - Chlorpyrifos
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
10 4 24 25 6 22 23 18 26 8 1 2 3 5 7 9 11 12 13 14 15 16 17 19 20 21 27 Md RA SV

Figure 3

Table 8

Sample Details

Sample No.	S1
Matrix	Soil
Analyte	p,p'-DDD
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	<0.5	NR	NR
2	0.018	0.0035	NR
3	<0.05	NR	NR
4	<0.01	NR	NR
5	<0.1	NR	NR
6	0.0064	0.00192	80
7	<0.05	0.02	NR
8	0.010	0.004	83
9	0.0055	0.0014	NR
10	NR	NR	NR
11	0.013	0.0039	NR
12	<0.1	NR	NR
13	<0.05	NR	NR
14	<0.05	NR	141
15	<0.1	NR	80-120
16	<0.1	0.1	NR
17	<0.05	NR	117.67
18	<0.05	NR	NR
19	< 0.05	NR	NR
20	< 0.05	NR	NR
21	<0.1	0.03	NR
22	0.010	0.007	64
23	<0.02	0.02	NR
24	< 0.05	NR	NR
25	<0.010	0.0054	NR
26	0.0165	0.005	81
27	<0.1	NR	NR

Statistics

Assigned Value	Not Set	
Spike Value	0.0152	0.0008
Robust Average	0.0113	0.0051
Median	0.0100	0.0050
Mean	0.0113	
N	7	
Max	0.018	
Min	0.0055	
Robust SD	0.0054	
Robust CV	48%	

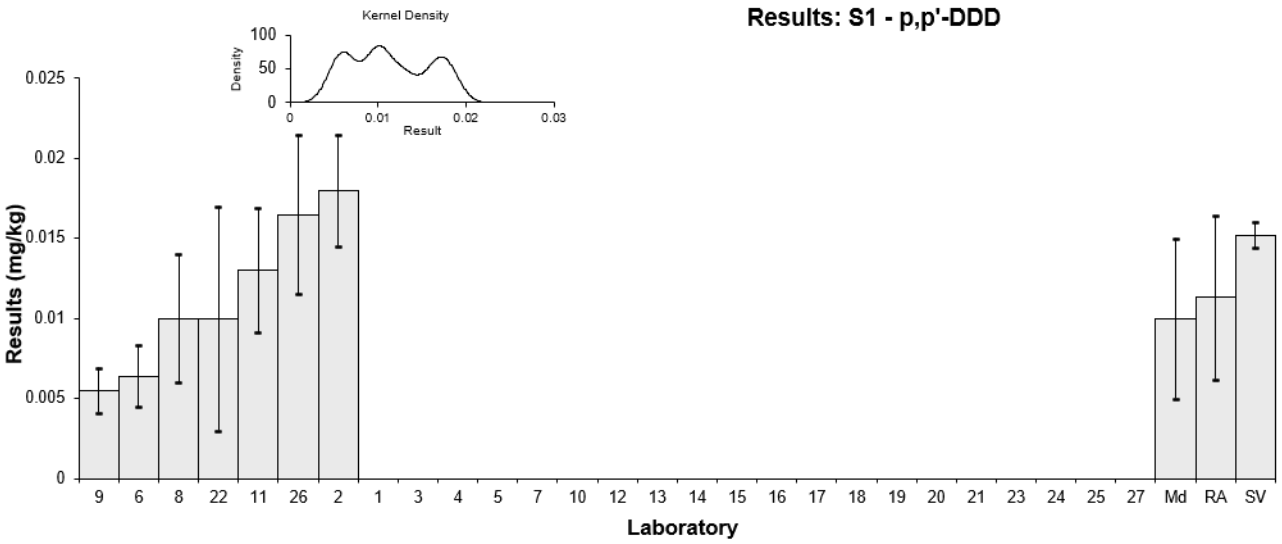

Results: S1 - p,p'-DDD
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
9 6 8 22 11 26 2 1 3 4 5 7 10 12 13 14 15 16 17 18 19 20 21 23 24 25 27 Md RA SV

Figure 4

Table 9

Sample Details

Sample No.	S1
Matrix	Soil
Analyte	Dieldrin
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	<0.5	NR	NR
2	NR	NR	NR
3	<0.05	NR	NR
4	<0.01	NR	NR
5	<0.1	NR	NR
6	0.007	0.0021	165
7	<0.05	0.02	NR
8	NR	NR	NR
9	<0.001	NR	NR
10	NR	NR	NR
11	<0.050	NR	NR
12	<0.1	NR	NR
13	<0.05	NR	NR
14	<0.05	NR	141
15	<0.1	NR	80-120
16	<0.1	0.1	NR
17	<0.05	NR	117.67
18	<0.05	NR	NR
19	< 0.05	NR	NR
20	< 0.05	NR	NR
21	<0.1	0.02	NR
22	0.008	0.004	78
23	<0.02	0.02	NR
24	< 0.05	NR	NR
25	<0.010	0.0051	NR
26	<0.01	NR	NR
27	<0.1	NR	NR

Statistics

Assigned Value	Not Set	
Spike Value	0.00617	0.00031
Robust Average	NA (N<6)	
Median	NA (N<3)	
Mean	0.0075	
N	2	
Max	0.008	
Min	0.007	
Robust SD	NA (N<6)	
Robust CV	NA (N<6)	

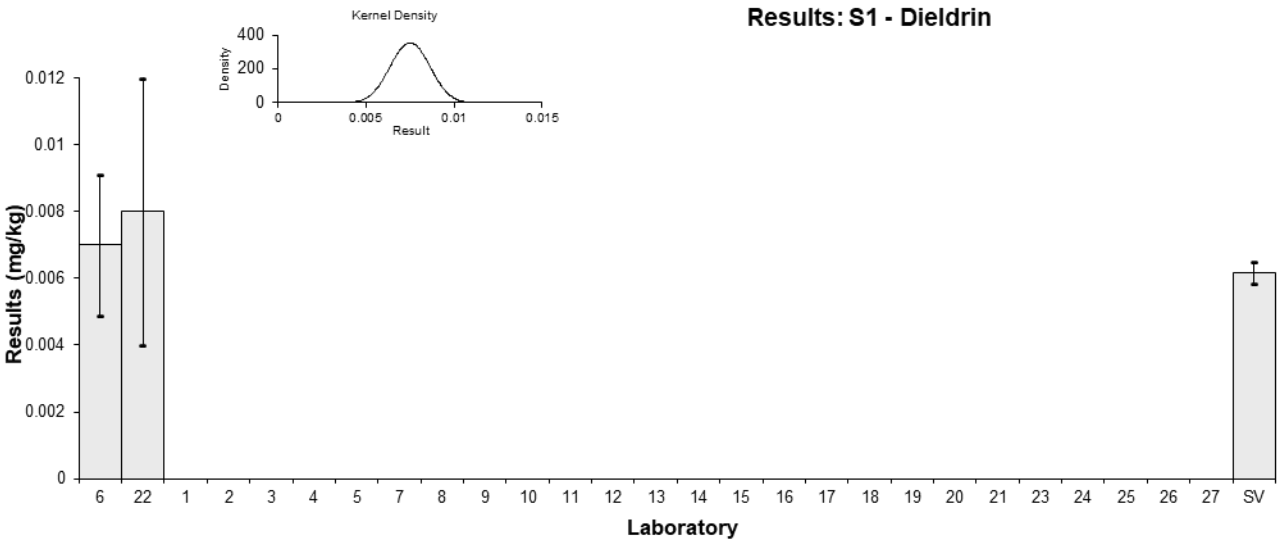

Results: S1 - Dieldrin
Kernel Density
Density
400
200
0
0
0.005
0.01
0.015
Result
Results (mg/kg)
0.012
0.01
0.008
0.006
0.004
0.002
0
6
22
1
2
3
4
5
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
23
24
25
26
27
SV
Laboratory

Figure 5

Table 10

Sample Details

Sample No.	S1
Matrix	Soil
Analyte	Ethion
Unit	mg/kg

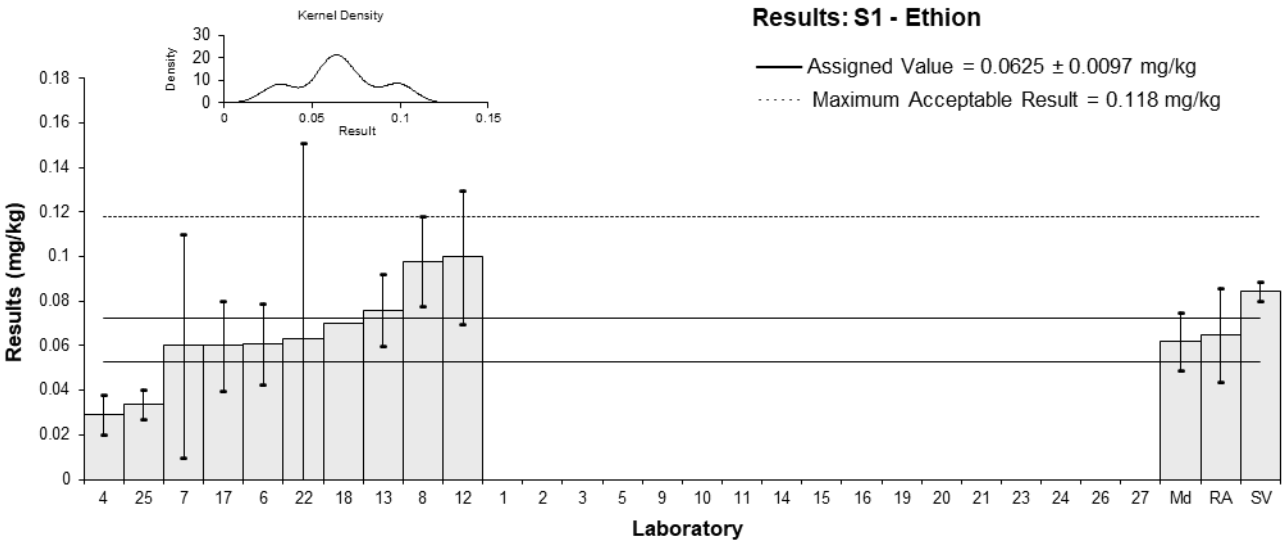
Participant Results

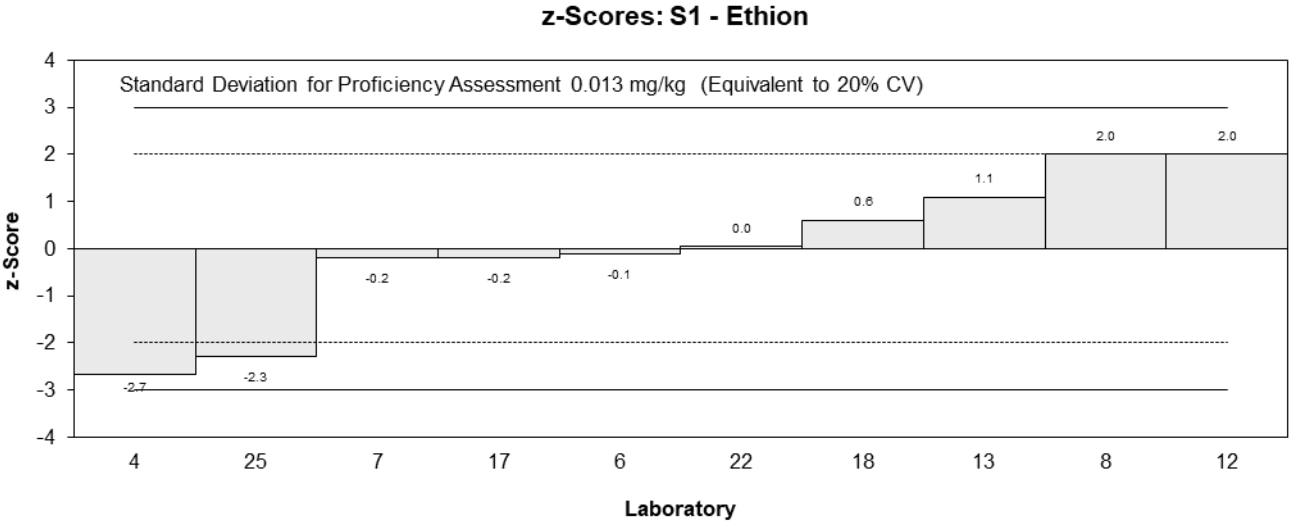
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	<0.5	NR	NR		
2	NR	NR	NR		
3	<0.5	NR	NR		
4*	0.029	0.009	125	-2.68	-2.53
5	<0.1	NR	NR		
6	0.061	0.0183	89.5	-0.12	-0.07
7	0.06	0.05	NR	-0.20	-0.05
8*	0.098	0.020	94	2.00▼	
9	NR	NR	NR		
10	NR	NR	NR		
11	NT	NT	NT		
12*	0.1	0.03	124	2.00▼	
13	0.076	0.016	NR	1.08	0.72
14	<0.2	NR	135		
15	<0.1	NR	80-120		
16	<0.2	0.2	NR		
17	0.06	0.02	117.67	-0.20	-0.11
18	0.07	NR	NR	0.60	0.77
19	< 0.2	NR	NR		
20	< 0.2	NR	NR		
21	NT	NT	NT		
22	0.063	0.088	45	0.04	0.01
23	<0.1	0.1	NR		
24	< 0.2	NR	NR		
25	0.0338	0.0067	NR	-2.30	-2.43
26	NT	NT	NT		
27	<0.1	NR	NR		

* Outlier, see Section 4.2; ▼ Adjusted Score, see Section 6.3

Statistics

Assigned Value	0.0625	0.0097
Spike Value	0.0846	0.0042
Robust Average	0.065	0.021
Max Acceptable Result	0.118	
Median	0.062	0.013
Mean	0.065	
N	10	
Max	0.1	
Min	0.029	
Robust SD	0.026	
Robust CV	40%	


Results: S1 - Ethion
Assigned Value = 0.0625 ± 0.0097 mg/kg
Maximum Acceptable Result = 0.118 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
4 25 7 17 6 22 18 13 8 12 1 2 3 5 9 10 11 14 15 16 19 20 21 23 24 26 27 Md RA SV


z-Scores: S1 - Ethion
Standard Deviation for Proficiency Assessment 0.013 mg/kg (Equivalent to 20% CV)
z-Score
Laboratory
4 25 7 17 6 22 18 13 8 12
-2.7
-2.3
-0.2
-0.2
-0.1
0.0
0.6
1.1
2.0
2.0

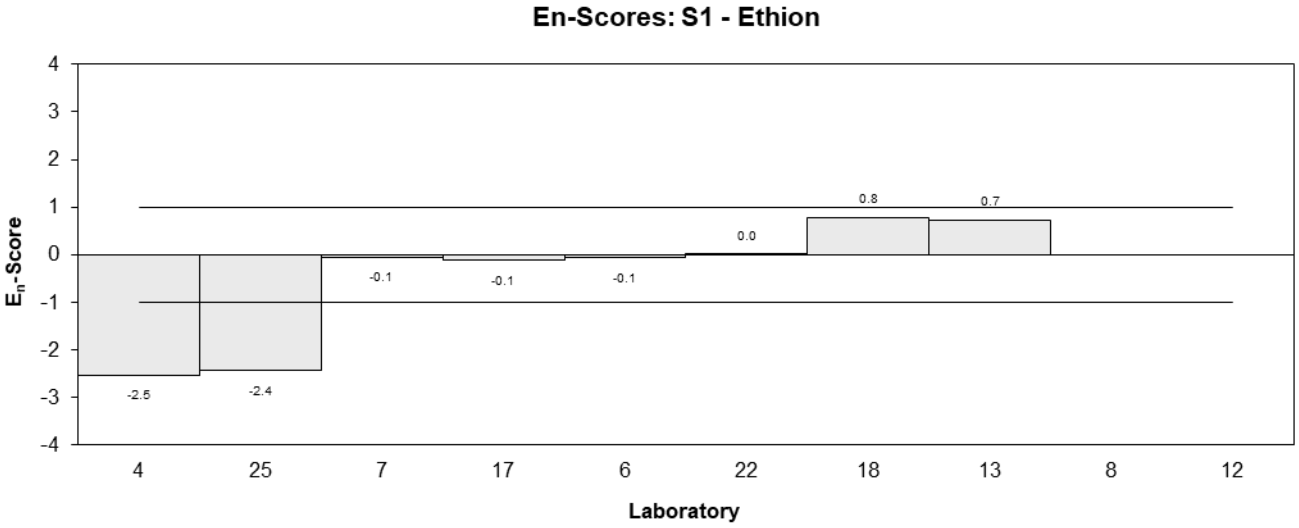

En-Scores: S1 - Ethion
En-Score
Laboratory
4 25 7 17 6 22 18 13 8 12
-2.5
-2.4
-0.1
-0.1
-0.1
0.0
0.8
0.7

Figure 6

Table 11

Sample Details

Sample No.	S1
Matrix	Soil
Analyte	Fenitrothion
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	<0.5	NR	NR
2	NR	NR	NR
3	NT	NT	NT
4	<0.05	NR	NR
5	<0.1	NR	NR
6	0.054	0.0162	41
7	NT	NT	NT
8	0.082	0.016	94
9	NR	NR	NR
10	0.046	0.0012	NR
11	NT	NT	NT
12	0.1	0.03	NR
13	NT	NT	NT
14	<0.2	NR	135
15	<0.1	NR	80-120
16	<0.2	0.2	NR
17	0.08	0.02	117.41
18	NR	NR	NR
19	< 0.2	NR	NR
20	< 0.2	NR	NR
21	<0.1	0.03	NR
22	0.050	0.12	41
23	0.06	0.03	NR
24	< 0.2	NR	NR
25	0.0334	0.006	NR
26	NT	NT	NT
27	<0.1	NR	NR

Statistics

Assigned Value	Not Set	
Spike Value	0.0710	0.0036
Robust Average	0.063	0.022
Median	0.057	0.022
Mean	0.063	
N	8	
Max	0.1	
Min	0.0334	
Robust SD	0.025	
Robust CV	40%	

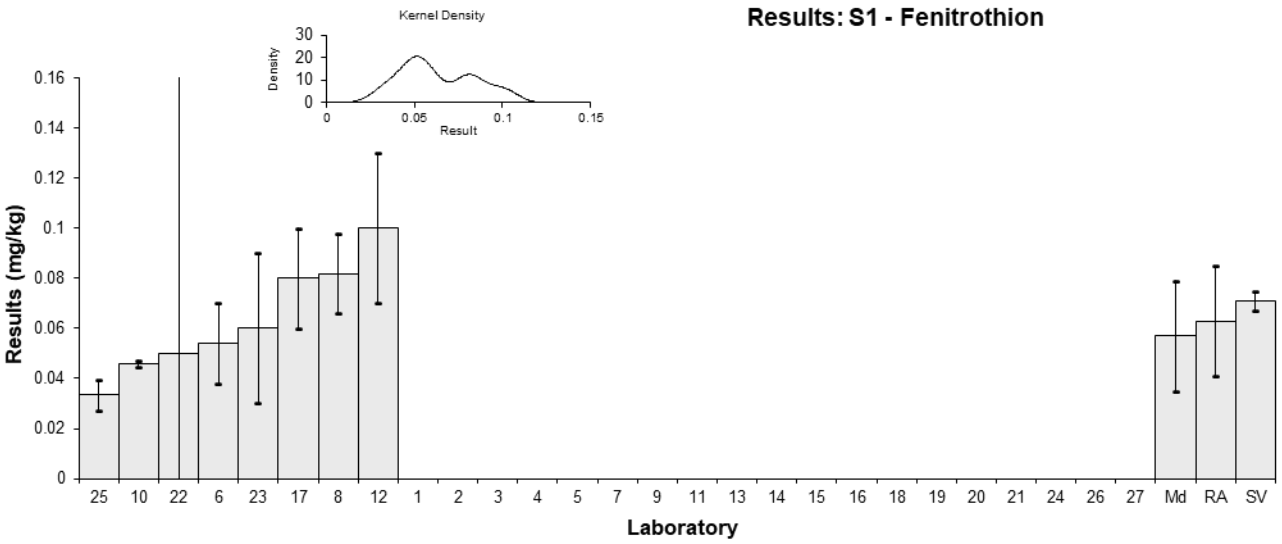

Results: S1 - Fenitrothion
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
25 10 22 6 23 17 8 12 1 2 3 4 5 7 9 11 13 14 15 16 18 19 20 21 24 26 27 Md RA SV

Figure 7

Table 12

Sample Details

Sample No.	S1
Matrix	Soil
Analyte	Fenvalerate
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	<0.5	NR	NR
2	NR	NR	NR
3	<0.5	NR	NR
4	0.049	0.015	90
5	NT	NT	NT
6	0.12	0.036	80
7	NT	NT	NT
8	0.099	0.035	114
9	NR	NR	NR
10	NR	NR	NR
11	0.096	0.0288	NR
12	NT	NT	NT
13	NT	NT	NT
14	NT	NT	NT
15	NT	NT	NT
16	NT	NT	NT
17	NT	NT	NT
18	NR	NR	NR
19	NT	NT	NT
20	< 2	NR	NR
21	NT	NT	NT
22	NT	NT	NT
23	0.13	0.1	NR
24	< 0.4	NR	NR
25	0.092	0.053	NR
26	NT	NT	NT
27	NT	NT	NT

Statistics

Assigned Value	Not Set	
Spike Value	0.152	0.008
Robust Average	0.098	0.031
Median	0.098	0.021
Mean	0.098	
N	6	
Max	0.13	
Min	0.049	
Robust SD	0.030	
Robust CV	31%	

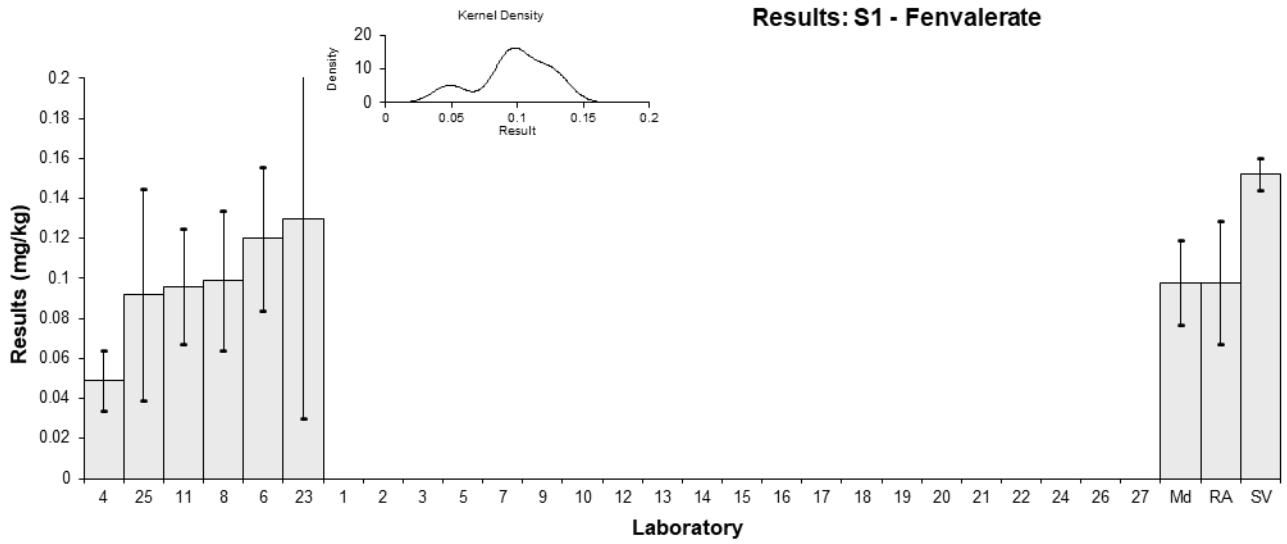

Results: S1 - Fenvalerate
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
4 25 11 8 6 23 1 2 3 5 7 9 10 12 13 14 15 16 17 18 19 20 21 22 24 26 27 Md RA SV

Figure 8

Table 13

Sample Details

Sample No.	S1
Matrix	Soil
Analyte	Lindane
Unit	mg/kg

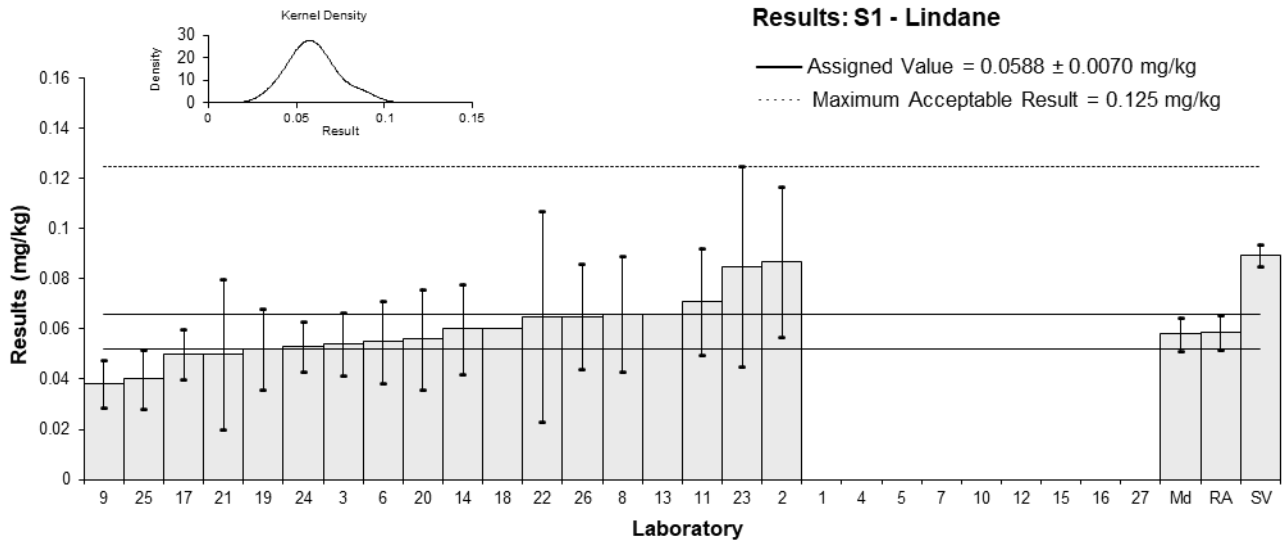
Participant Results

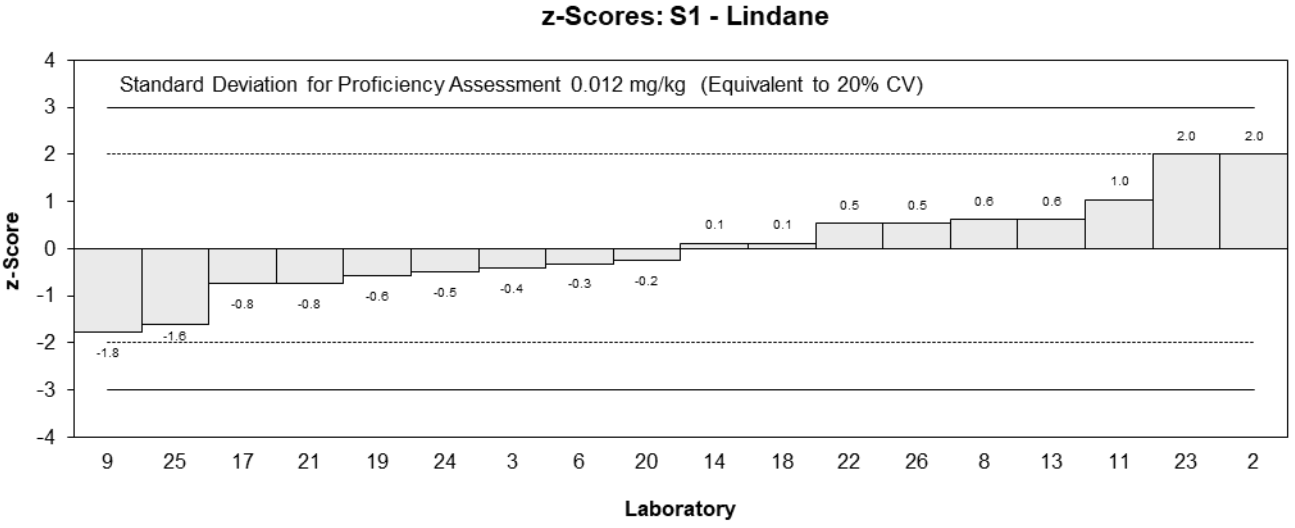
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	<0.5	NR	NR		
2	0.087	0.030	NR	2.00▼	
3	0.054	0.01242	NR	-0.41	-0.34
4	<0.01	NR	NR		
5	<0.1	NR	NR		
6	0.055	0.0165	101	-0.32	-0.21
7	<0.05	0.02	NR		
8	0.066	0.023	75	0.61	0.30
9	0.0380	0.0095	NR	-1.77	-1.76
10	NR	NR	NR		
11	0.071	0.0213	NR	1.04	0.54
12	<0.1	NR	NR		
13	0.066	NR	NR	0.61	1.03
14	0.06	0.018	141	0.10	0.06
15	<0.1	NR	80-120		
16	<0.1	0.1	NR		
17	0.05	0.01	119.4	-0.75	-0.72
18	0.06	NR	NR	0.10	0.17
19	0.052	0.016	NR	-0.58	-0.39
20	0.056	0.02	NR	-0.24	-0.13
21	0.05	0.03	NR	-0.75	-0.29
22	0.065	0.042	74	0.53	0.15
23	0.085	0.04	NR	2.00▼	
24	0.053	0.01	NR	-0.49	-0.48
25	0.04	0.012	NR	-1.60	-1.35
26	0.065	0.021	81	0.53	0.28
27	<0.1	NR	NR		

▼ Adjusted Score, see Section 6.3

Statistics

Assigned Value	0.0588	0.0070
Spike Value	0.0895	0.0045
Robust Average	0.0588	0.0070
Max Acceptable Result	0.125	
Median	0.0580	0.0066
Mean	0.0596	
N	18	
Max	0.087	
Min	0.0380	
Robust SD	0.012	
Robust CV	20%	


Kernel Density
Density
Result
Results: S1 - Lindane
Assigned Value = 0.0588 ± 0.0070 mg/kg
Maximum Acceptable Result = 0.125 mg/kg
Results (mg/kg)
Laboratory
9 25 17 21 19 24 3 6 20 14 18 22 26 8 13 11 23 2 1 4 5 7 10 12 15 16 27 Md RA SV


z-Scores: S1 - Lindane
Standard Deviation for Proficiency Assessment 0.012 mg/kg (Equivalent to 20% CV)
z-Score
Laboratory
9 25 17 21 19 24 3 6 20 14 18 22 26 8 13 11 23 2
-1.8 -1.6 -0.8 -0.8 -0.6 -0.5 -0.4 -0.3 -0.2 0.1 0.1 0.5 0.5 0.6 0.6 1.0 2.0 2.0

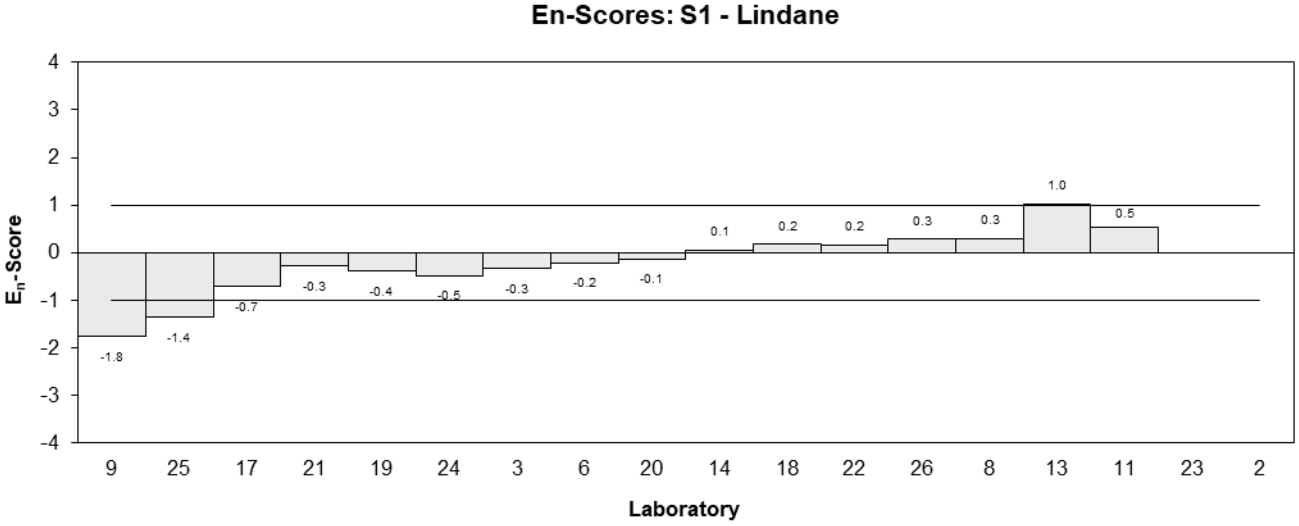

En-Scores: S1 - Lindane
En-Score
Laboratory
9 25 17 21 19 24 3 6 20 14 18 22 26 8 13 11 23 2
-1.8 -1.4 -0.7 -0.3 -0.4 -0.5 -0.3 -0.2 -0.1 0.1 0.2 0.2 0.3 0.3 1.0 0.5

Figure 9

Table 14

Sample Details

Sample No.	S1
Matrix	Soil
Analyte	Trifluralin
Unit	mg/kg

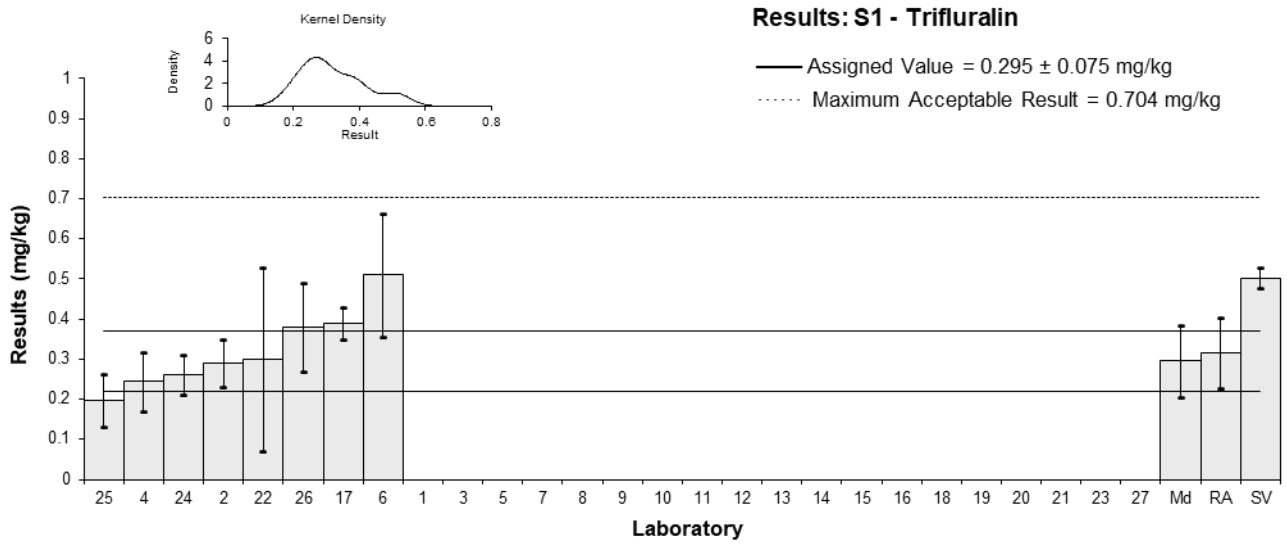
Participant Results

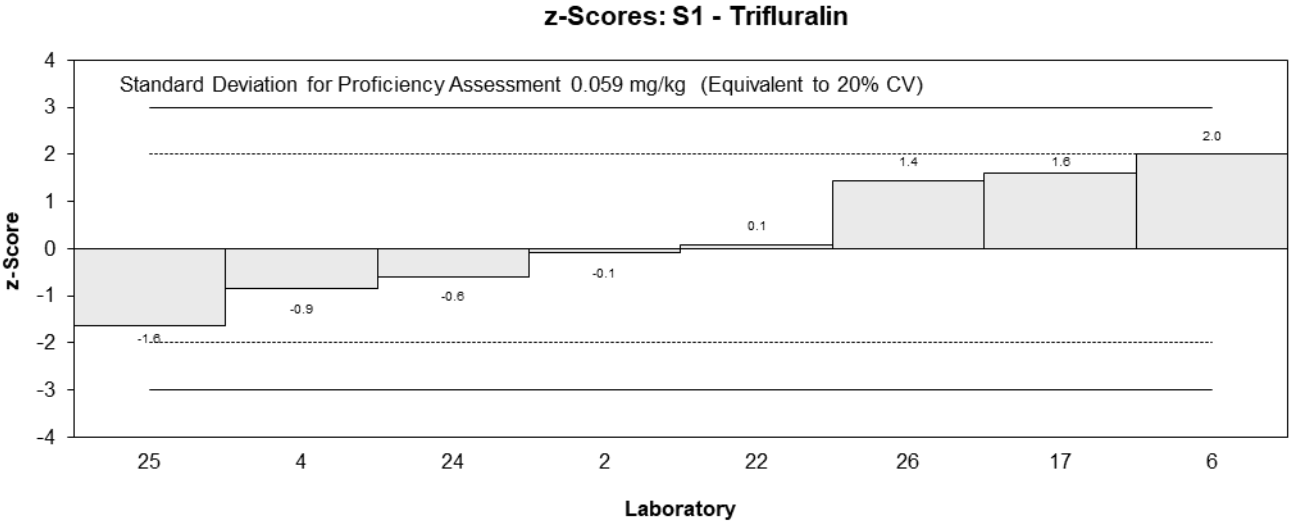
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	<0.5	NR	NR		
2	0.29	0.060	NR	-0.08	-0.05
3	NT	NT	NT		
4	0.244	0.073	108	-0.86	-0.49
5	<0.5	NR	NR		
6*	0.51	0.153	65	2.00▼	
7	NT	NT	NT		
8	NT	NT	NT		
9	NR	NR	NR		
10	NR	NR	NR		
11	NT	NT	NT		
12	NT	NT	NT		
13	NT	NT	NT		
14	NT	NT	NT		
15	<0.5	NR	80-120		
16	<0.5	0.5	NR		
17	0.39	0.04	117.41	1.61	1.12
18	NR	NR	NR		
19	NT	NT	NT		
20	< 0.5	NR	NR		
21	NT	NT	NT		
22	0.30	0.23	59	0.08	0.02
23	NT	NT	NT		
24	0.26	0.05	NR	-0.59	-0.39
25	0.198	0.066	NR	-1.64	-0.97
26	0.38	0.11	NR	1.44	0.64
27	<0.5	NR	NR		

* Outlier, see Section 4.2; ▼ Adjusted Score, see Section 6.3

Statistics

Assigned Value	0.295	0.075
Spike Value	0.503	0.025
Robust Average	0.316	0.089
Max Acceptable Result	0.704	
Median	0.295	0.089
Mean	0.322	
N	8	
Max	0.51	
Min	0.198	
Robust SD	0.10	
Robust CV	32%	


Kernel Density
Density
Result
Results: S1 - Trifluralin
Assigned Value = 0.295 ± 0.075 mg/kg
Maximum Acceptable Result = 0.704 mg/kg
Results (mg/kg)
Laboratory
25 4 24 2 22 26 17 6 1 3 5 7 8 9 10 11 12 13 14 15 16 18 19 20 21 23 27 Md RA SV


z-Scores: S1 - Trifluralin
Standard Deviation for Proficiency Assessment 0.059 mg/kg (Equivalent to 20% CV)
z-Score
Laboratory
25 4 24 2 22 26 17 6
-1.6
-0.9
-0.6
-0.1
0.1
1.4
1.6
2.0

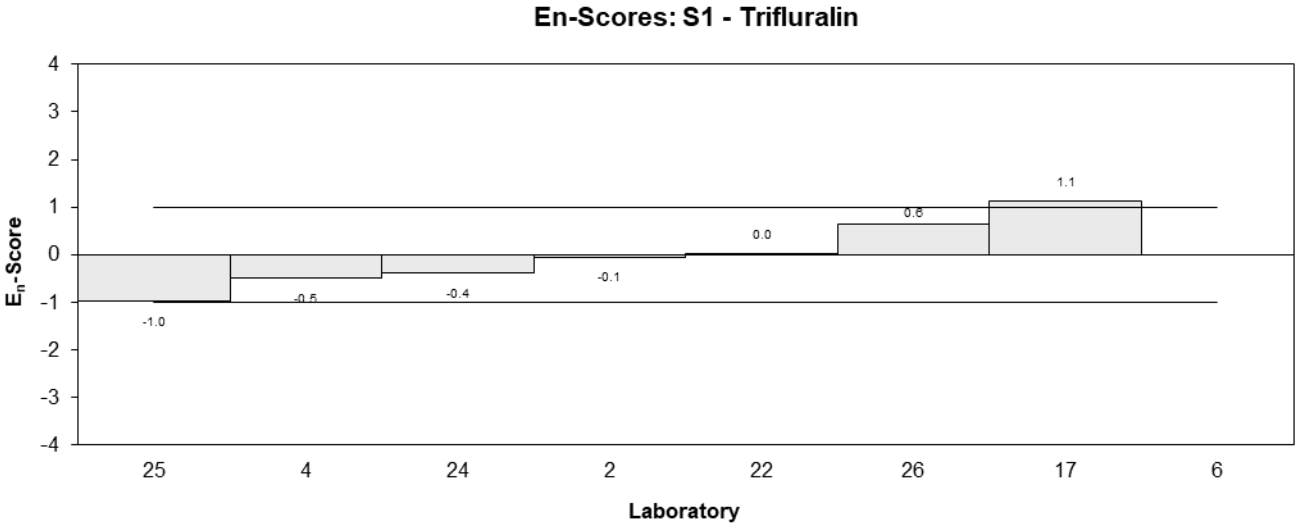

En-Scores: S1 - Trifluralin
En-Score
Laboratory
25 4 24 2 22 26 17 6
-1.0
-0.5
-0.4
-0.1
0.0
0.6
1.1

Figure 10

Table 15

Sample Details

Sample No.	S2
Matrix	Soil
Analyte	Acephate
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	<0.5	NR	NR
2	0.74	0.22	NR
3	NT	NT	NT
4	NT	NT	NT
5	NT	NT	NT
6	0.54	0.162	145
7	NT	NT	NT
8	0.800	0.160	77
9	NR	NR	NR
10	NS	NS	NS
11	0.714	0.2142	NR
12	NS	NS	NS
13	NT	NT	NT
14	NT	NT	NT
15	NT	NT	NT
16	NT	NT	NT
17	NT	NT	NT
18	NR	NR	NR
19	NT	NT	NT
20	NT	NT	NT
21	NS	NS	NS
22	NT	NT	NT
23	NT	NT	NT
24	NT	NT	NT
25	NT	NT	NT
26	NT	NT	NT
27	NT	NT	NT

Statistics

Assigned Value	Not Set	
Spike Value	1.00	0.05
Robust Average	NA (N<6)	
Median	0.727	0.080
Mean	0.70	
N	4	
Max	0.800	
Min	0.54	
Robust SD	NA (N<6)	
Robust CV	NA (N<6)	

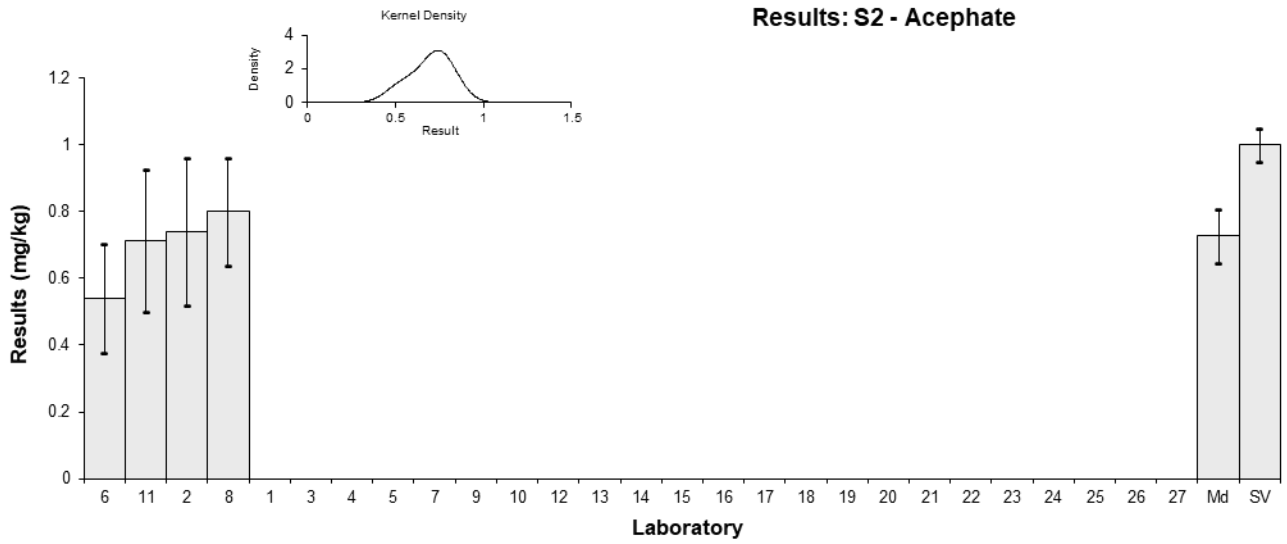

Results: S2 - Acephate
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
6 11 2 8 1 3 4 5 7 9 10 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 Md SV

Figure 11

Table 16

Sample Details

Sample No.	S2
Matrix	Soil
Analyte	Bromacil
Unit	mg/kg

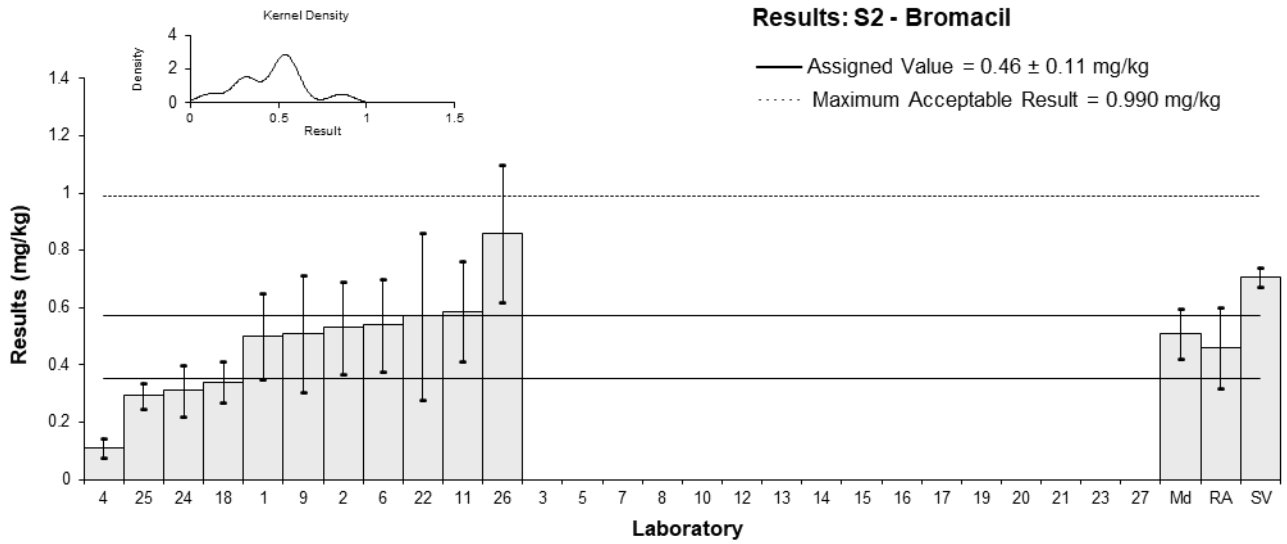
Participant Results

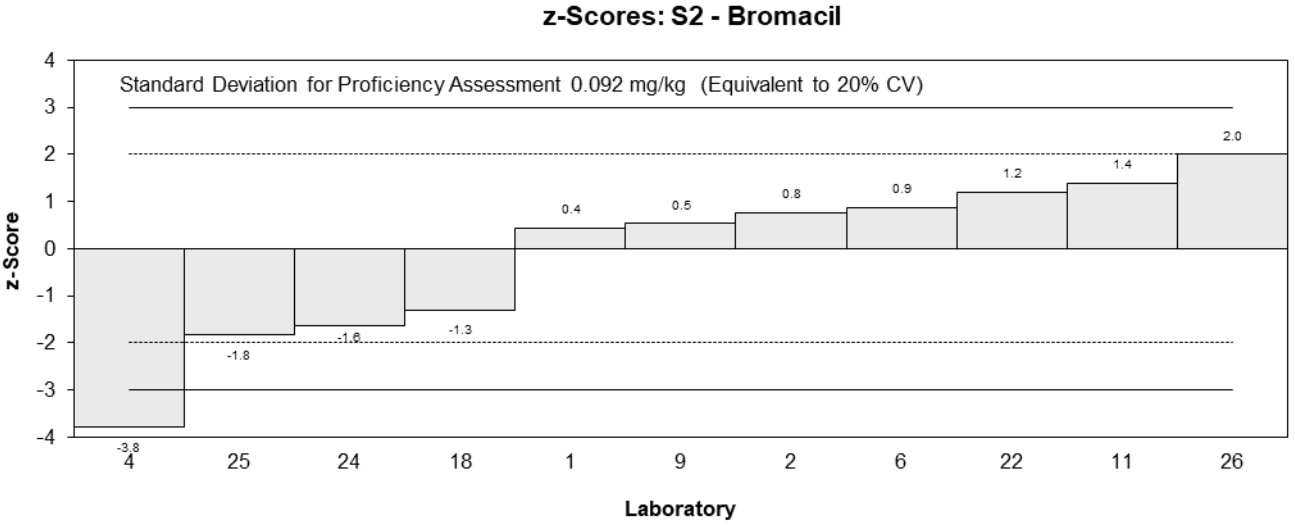
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	0.5	0.15	NR	0.43	0.22
2	0.53	0.16	NR	0.76	0.36
3	NT	NT	NT		
4*	0.111	0.033	108	-3.79	-3.04
5	NT	NT	NT		
6	0.54	0.162	110	0.87	0.41
7	NT	NT	NT		
8	NT	NT	NT		
9	0.509	0.204	NR	0.53	0.21
10	NS	NS	NS		
11	0.587	0.1761	NR	1.38	0.61
12	NS	NS	NS		
13	NT	NT	NT		
14	NT	NT	NT		
15	<2	NR	80-120		
16	NT	NT	NT		
17	NT	NT	NT		
18	0.34	0.071	NR	-1.30	-0.92
19	NT	NT	NT		
20	NT	NT	NT		
21	NS	NS	NS		
22	0.57	0.29	89	1.20	0.35
23	NT	NT	NT		
24	0.31	0.09	NR	-1.63	-1.06
25	0.293	0.045	NR	-1.82	-1.41
26*	0.86	0.24	98	2.00▼	
27	<2	NR	NR		

* Outlier, see Section 4.2; ▼ Adjusted Score, see Section 6.3

Statistics

Assigned Value	0.46	0.11
Spike Value	0.707	0.035
Robust Average	0.46	0.14
Max Acceptable Result	0.990	
Median	0.509	0.087
Mean	0.47	
N	11	
Max	0.86	
Min	0.111	
Robust SD	0.18	
Robust CV	39%	


Kernel Density
Density
Result
Results: S2 - Bromacil
Assigned Value = 0.46 ± 0.11 mg/kg
Maximum Acceptable Result = 0.990 mg/kg
Results (mg/kg)
Laboratory
4 25 24 18 1 9 2 6 22 11 26 3 5 7 8 10 12 13 14 15 16 17 19 20 21 23 27 Md RA SV


z-Scores: S2 - Bromacil
Standard Deviation for Proficiency Assessment 0.092 mg/kg (Equivalent to 20% CV)
z-Score
Laboratory
-3.8 -1.8 -1.6 -1.3 0.4 0.5 0.8 0.9 1.2 1.4 2.0
4 25 24 18 1 9 2 6 22 11 26

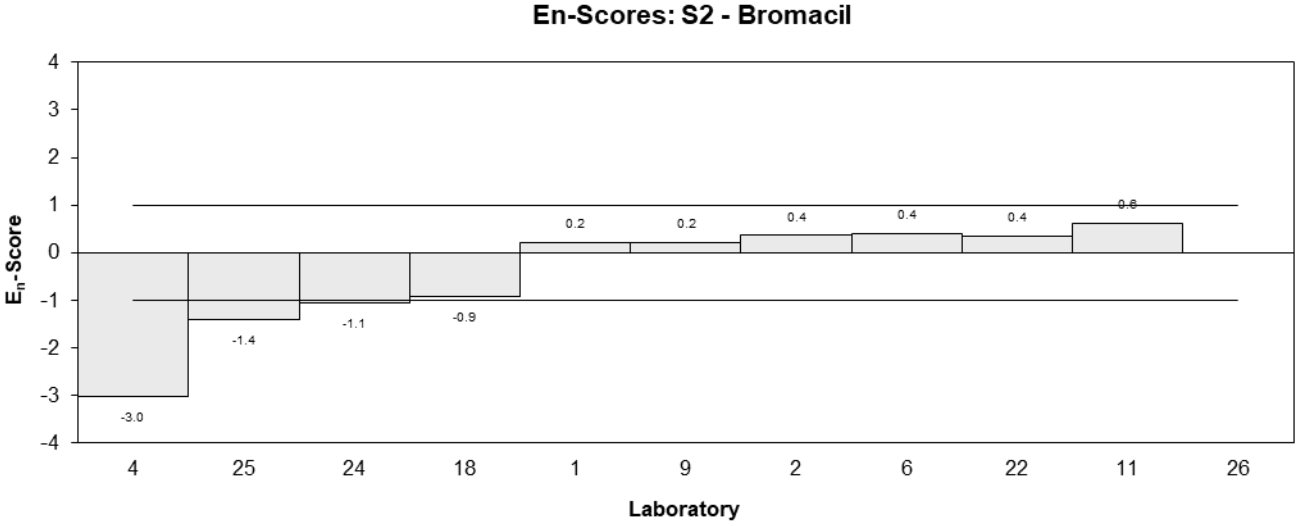

En-Scores: S2 - Bromacil
En-Score
Laboratory
-3.0 -1.4 -1.1 -0.9 0.2 0.2 0.4 0.4 0.4 0.6
4 25 24 18 1 9 2 6 22 11 26

Figure 12

Table 17

Sample Details

Sample No.	S2
Matrix	Soil
Analyte	Carbendazim
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	0.2	0.05	NR
2	NR	NR	NR
3	NT	NT	NT
4	0.069	0.021	77
5	0.8	0.3	NR
6	0.25	0.075	89
7	NT	NT	NT
8	NR	NR	NR
9	NR	NR	NR
10	NS	NS	NS
11	0.419	0.1257	NR
12	NS	NS	NS
13	NT	NT	NT
14	NT	NT	NT
15	NT	NT	NT
16	NT	NT	NT
17	NT	NT	NT
18	NR	NR	NR
19	NT	NT	NT
20	NT	NT	NT
21	NS	NS	NS
22	NT	NT	NT
23	NT	NT	NT
24	0.06	0.02	NR
25	NT	NT	NT
26	0.67	0.22	80
27	NT	NT	NT

Statistics

Assigned Value	Not Set	
Spike Value	0.804	0.040
Robust Average	0.35	0.31
Median	0.25	0.25
Mean	0.35	
N	7	
Max	0.8	
Min	0.06	
Robust SD	0.33	
Robust CV	93%	

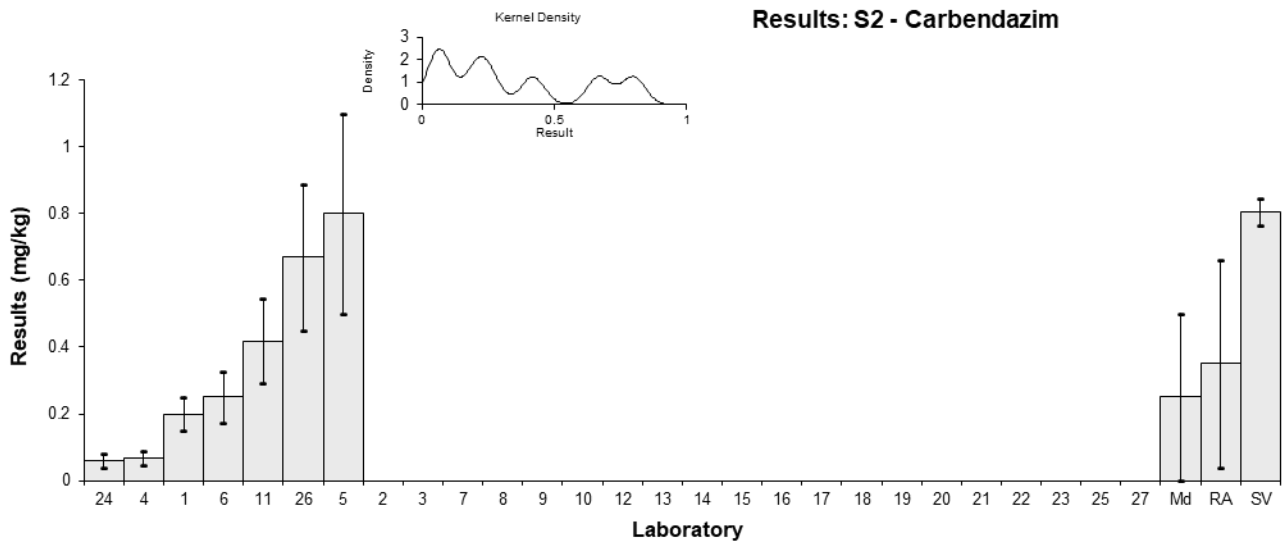

Results: S2 - Carbendazim
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
24 4 1 6 11 26 5 2 3 7 8 9 10 12 13 14 15 16 17 18 19 20 21 22 23 25 27 Md RA SV

Figure 13

Table 18

Sample Details

Sample No.	S2
Matrix	Soil
Analyte	2,4-D
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	0.09	0.03	99
2	NT	NT	NT
3	0.12	0.03	NR
4	NT	NT	NT
5	<0.5	NR	NR
6	0.1	0.03	96.5
7	NT	NT	NT
8	NT	NT	NT
9	NR	NR	NR
10	NS	NS	NS
11	0.129	0.0387	NR
12	NS	NS	NS
13	NT	NT	NT
14	NT	NT	NT
15	<0.5	NR	80-120
16	NT	NT	NT
17	NT	NT	NT
18	0.09	0.019	NR
19	NT	NT	NT
20	NT	NT	NT
21	NS	NS	NS
22	0.15	0.17	54
23	NT	NT	NT
24	0.09	0.03	NR
25	0.101	0.014	NR
26	0.082	0.025	80
27	<0.5	NR	NR

Statistics

Assigned Value	Not Set	
Spike Value	0.351	0.018
Robust Average	0.104	0.019
Median	0.100	0.012
Mean	0.106	
N	9	
Max	0.15	
Min	0.082	
Robust SD	0.022	
Robust CV	22%	

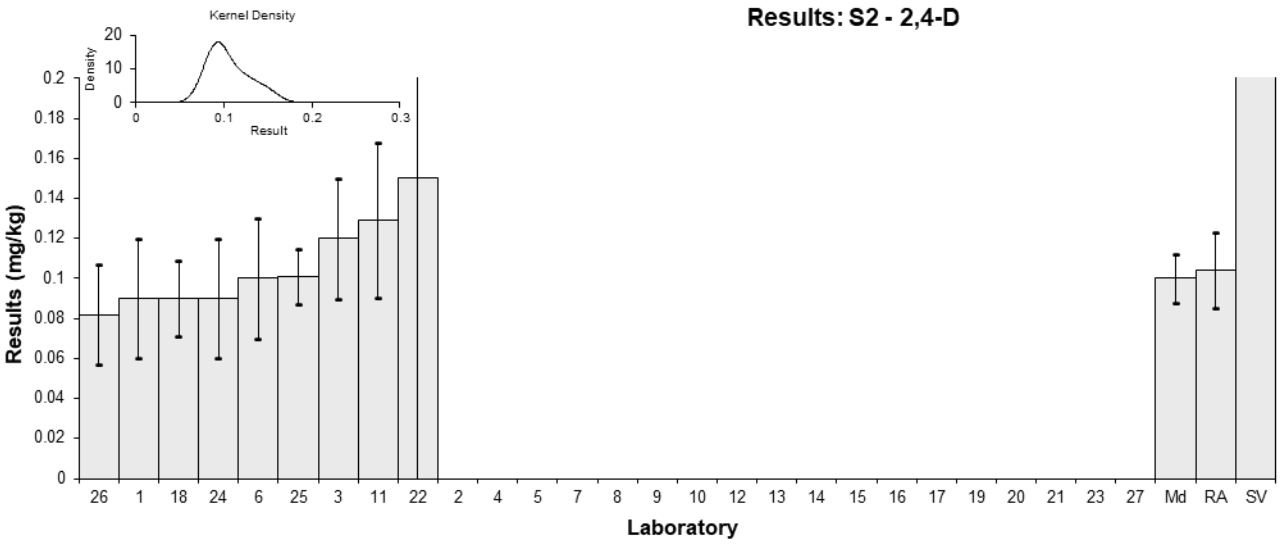

Results: S2 - 2,4-D
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
26 1 18 24 6 25 3 11 22 2 4 5 7 8 9 10 12 13 14 15 16 17 19 20 21 23 27 Md RA SV

Figure 14

Table 19

Sample Details

Sample No.	S2
Matrix	Soil
Analyte	Fipronil
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	0.67	0.17	NR
2	NR	NR	NR
3	NT	NT	NT
4	0.214	0.064	84
5	0.9	0.3	NR
6	0.66	0.198	101
7	NT	NT	NT
8	NT	NT	NT
9	NR	NR	NR
10	NS	NS	NS
11	NT	NT	NT
12	NS	NS	NS
13	NT	NT	NT
14	NT	NT	NT
15	NT	NT	NT
16	NT	NT	NT
17	0.72	0.14	116.52
18	NR	NR	NR
19	NT	NT	NT
20	NT	NT	NT
21	NS	NS	NS
22	NT	NT	NT
23	NT	NT	NT
24	0.95	0.29	NR
25	NT	NT	NT
26	NT	NT	NT
27	NT	NT	NT

Statistics

Assigned Value	Not Set	
Spike Value	0.803	0.040
Robust Average	0.71	0.25
Median	0.70	0.18
Mean	0.69	
N	6	
Max	0.95	
Min	0.214	
Robust SD	0.25	
Robust CV	35%	

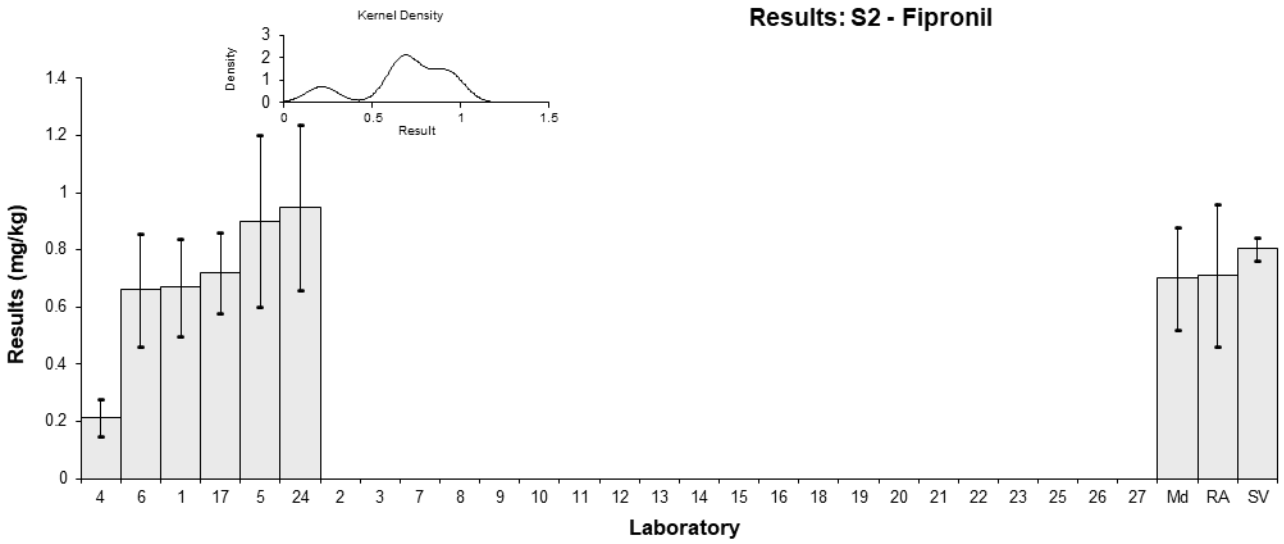

Results: S2 - Fipronil
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
4 6 1 17 5 24 2 3 7 8 9 10 11 12 13 14 15 16 18 19 20 21 22 23 25 26 27 Md RA SV

Figure 15

Table 20

Sample Details

Sample No.	S2
Matrix	Soil
Analyte	Glyphosate
Unit	mg/kg

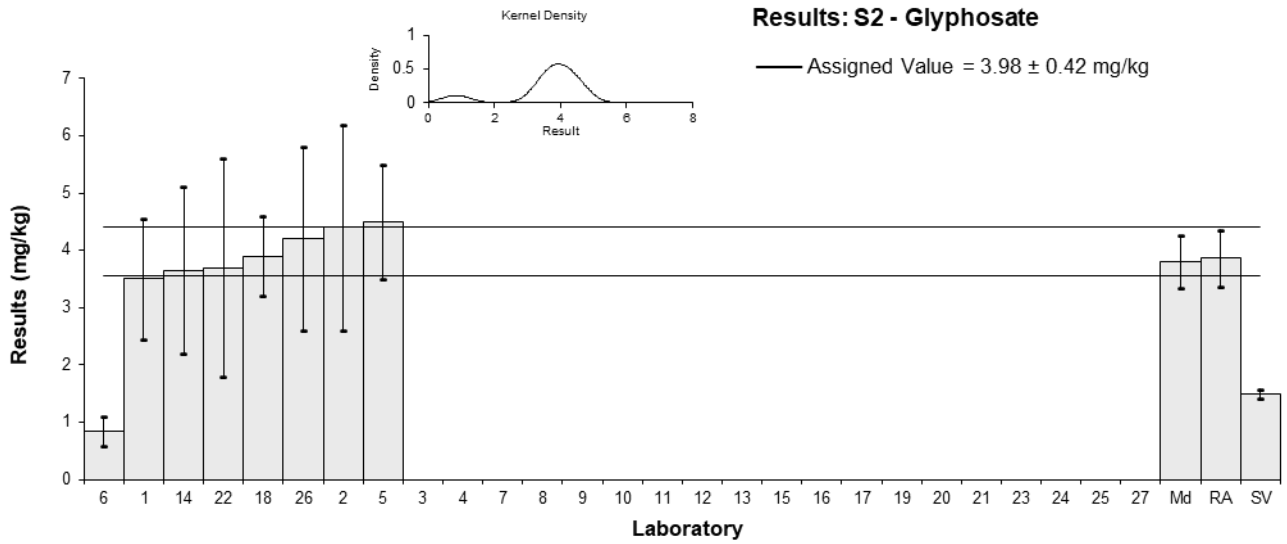
Participant Results

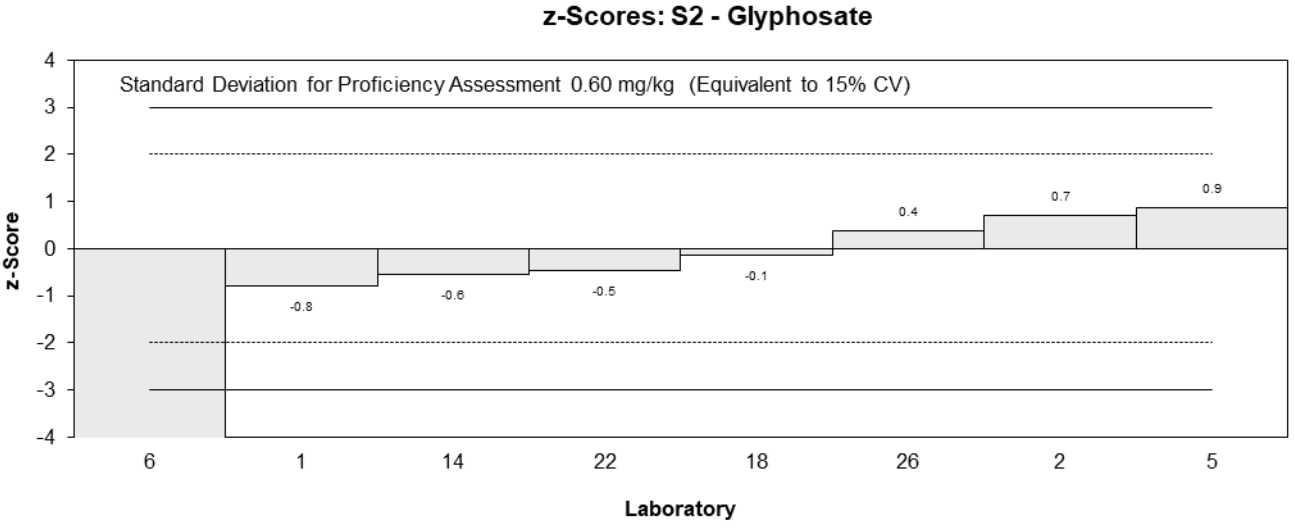
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	3.5	1.05	110	-0.80	-0.42
2	4.4	1.8	NR	0.70	0.23
3	NT	NT	NT		
4	NT	NT	NT		
5	4.5	1	NR	0.87	0.48
6*	0.84	0.252	119	-5.26	-6.41
7	NT	NT	NT		
8	NT	NT	NT		
9	NR	NR	NR		
10	NS	NS	NS		
11	NT	NT	NT		
12	NS	NS	NS		
13	NT	NT	NT		
14	3.65	1.46	90	-0.55	-0.22
15	NT	NT	NT		
16	NT	NT	NT		
17	NT	NT	NT		
18	3.9	0.7	NR	-0.13	-0.10
19	NT	NT	NT		
20	NT	NT	NT		
21	NS	NS	NS		
22	3.7	1.9	90	-0.47	-0.14
23	NT	NT	NT		
24	NT	NT	NT		
25	NT	NT	NT		
26	4.2	1.6	NR	0.37	0.13
27	NT	NT	NT		

* Outlier, see Section 4.2

Statistics

Assigned Value	3.98	0.42
Spike Value	1.50	0.08
Robust Average	3.86	0.50
Median	3.80	0.46
Mean	3.59	
N	8	
Max	4.5	
Min	0.84	
Robust SD	0.57	
Robust CV	15%	


Kernel Density
Density
Result
Results: S2 - Glyphosate
Assigned Value = 3.98 ± 0.42 mg/kg
Results (mg/kg)
Laboratory
6 1 14 22 18 26 2 5 3 4 7 8 9 10 11 12 13 15 16 17 19 20 21 23 24 25 27 Md RA SV


z-Scores: S2 - Glyphosate
Standard Deviation for Proficiency Assessment 0.60 mg/kg (Equivalent to 15% CV)
z-Score
-0.8
-0.6
-0.5
-0.1
0.4
0.7
0.9
Laboratory
6 1 14 22 18 26 2 5

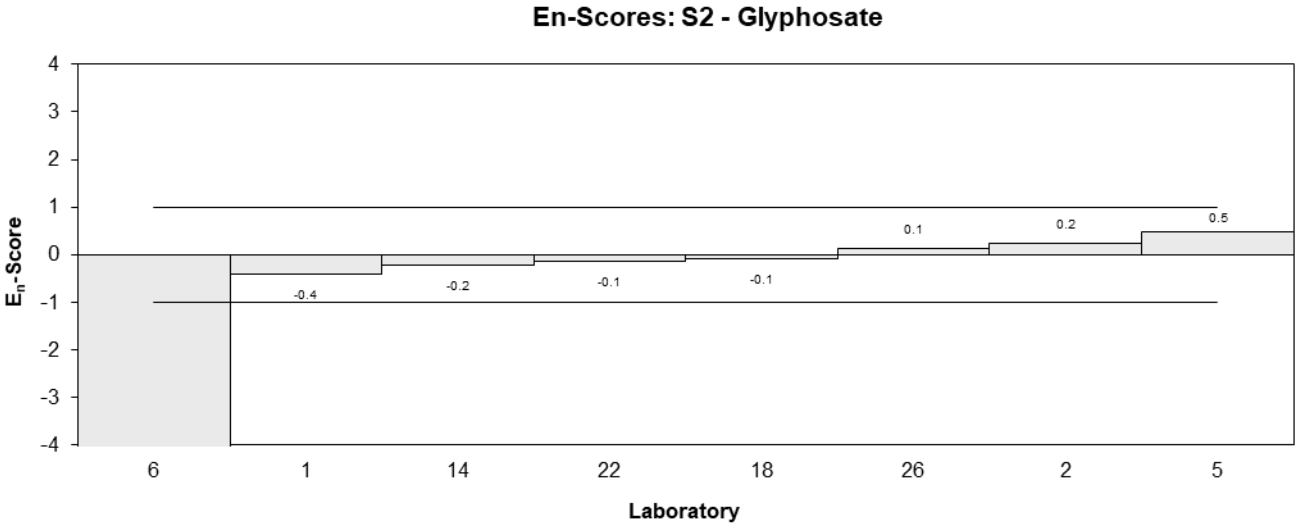

En-Scores: S2 - Glyphosate
En-Score
-0.4
-0.2
-0.1
-0.1
0.1
0.2
0.5
Laboratory
6 1 14 22 18 26 2 5

Figure 16

Table 21

Sample Details

Sample No.	S2
Matrix	Soil
Analyte	Imidacloprid
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	0.3	0.08	NR
2	NR	NR	NR
3	<0.5	NR	NR
4	0.09	0.027	78
5	0.63	0.2	NR
6	0.52	0.156	83.5
7	NT	NT	NT
8	NT	NT	NT
9	NR	NR	NR
10	NS	NS	NS
11	0.491	0.1473	NR
12	NS	NS	NS
13	NT	NT	NT
14	NT	NT	NT
15	NT	NT	NT
16	NT	NT	NT
17	NT	NT	NT
18	NR	NR	NR
19	NT	NT	NT
20	NT	NT	NT
21	NS	NS	NS
22	0.58	0.29	93
23	NT	NT	NT
24	NT	NT	NT
25	NT	NT	NT
26	<0.01	NR	NR
27	NT	NT	NT

Statistics

Assigned Value	Not Set	
Spike Value	0.604	0.030
Robust Average	0.44	0.23
Median	0.51	0.15
Mean	0.44	
N	6	
Max	0.63	
Min	0.09	
Robust SD	0.23	
Robust CV	53%	

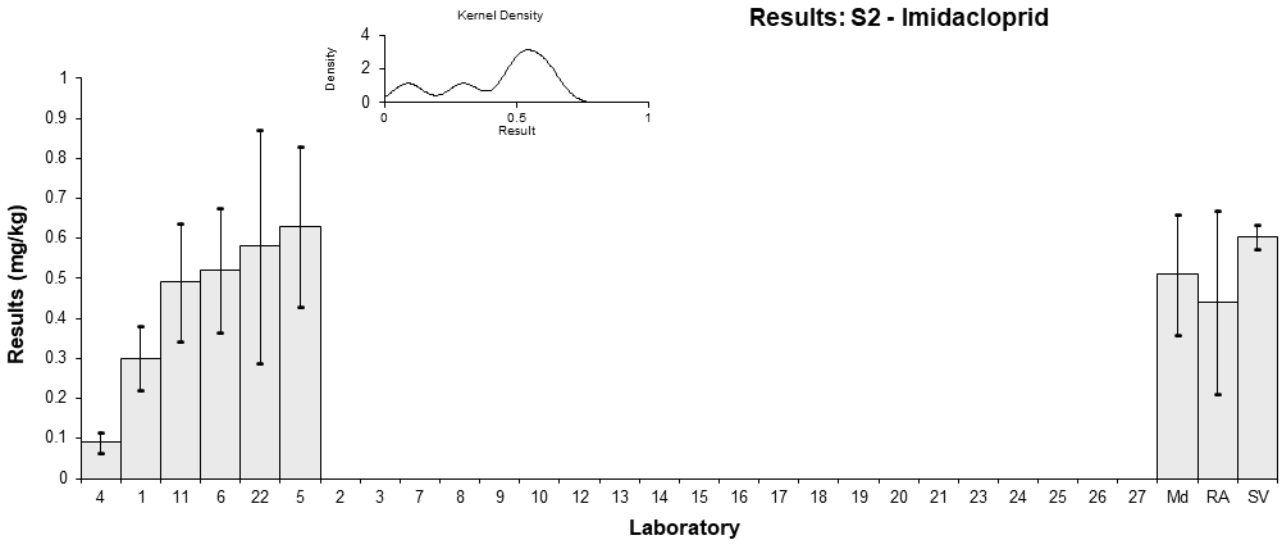

Results: S2 - Imidacloprid
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
4 1 11 6 22 5 2 3 7 8 9 10 12 13 14 15 16 17 18 19 20 21 23 24 25 26 27 Md RA SV

Figure 17

Table 22

Sample Details

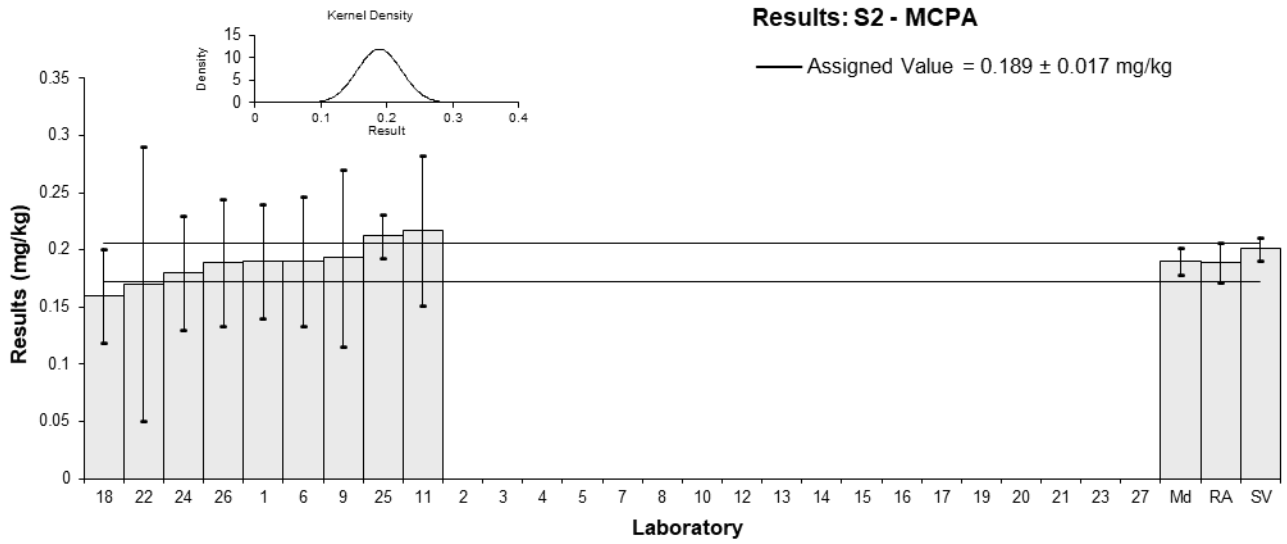
Sample No.	S2
Matrix	Soil
Analyte	MCPA
Unit	mg/kg

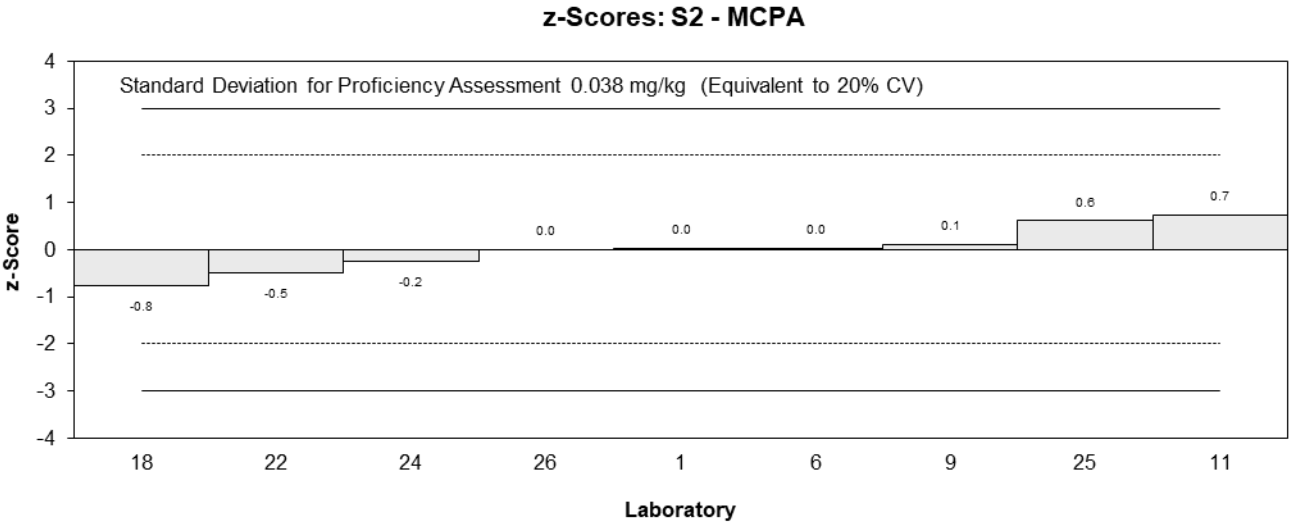
Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E_n
1	0.19	0.05	NR	0.03	0.02
2	NT	NT	NT		
3	<0.5	NR	NR		
4	NT	NT	NT		
5	<0.5	NR	NR		
6	0.19	0.057	96.5	0.03	0.02
7	NT	NT	NT		
8	NT	NT	NT		
9	0.193	0.077	NR	0.11	0.05
10	NS	NS	NS		
11	0.217	0.0651	NR	0.74	0.42
12	NS	NS	NS		
13	NT	NT	NT		
14	NT	NT	NT		
15	<0.5	NR	80-120		
16	NT	NT	NT		
17	NT	NT	NT		
18	0.16	0.041	NR	-0.77	-0.65
19	NT	NT	NT		
20	< 0.5	NR	NR		
21	NS	NS	NS		
22	0.17	0.12	66	-0.50	-0.16
23	NT	NT	NT		
24	0.18	0.05	NR	-0.24	-0.17
25	0.212	0.019	NR	0.61	0.90
26	0.189	0.055	85	0.00	0.00
27	<0.5	NR	NR		

Statistics

Assigned Value	0.189	0.017
Spike Value	0.201	0.010
Robust Average	0.189	0.017
Median	0.190	0.012
Mean	0.189	
N	9	
Max	0.217	
Min	0.16	
Robust SD	0.020	
Robust CV	11%	


Kernel Density
Density
Result
Results: S2 - MCPA
Assigned Value = 0.189 ± 0.017 mg/kg
Results (mg/kg)
Laboratory
18 22 24 26 1 6 9 25 11 2 3 4 5 7 8 10 12 13 14 15 16 17 19 20 21 23 27 Md RA SV


z-Scores: S2 - MCPA
Standard Deviation for Proficiency Assessment 0.038 mg/kg (Equivalent to 20% CV)
z-Score
-0.8 -0.5 -0.2 0.0 0.0 0.0 0.1 0.6 0.7
Laboratory
18 22 24 26 1 6 9 25 11

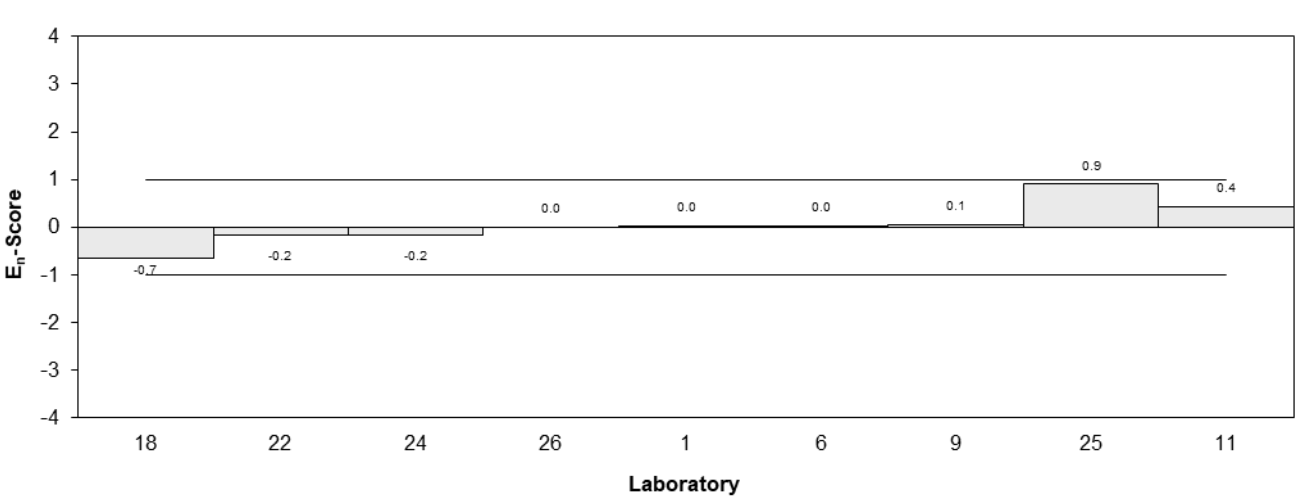

En-Scores: S2 - MCPA
En-Score
-0.7 -0.2 -0.2 0.0 0.0 0.0 0.1 0.9 0.4
Laboratory
18 22 24 26 1 6 9 25 11

Figure 18

Table 23

Sample Details

Sample No.	S2
Matrix	Soil
Analyte	Metsulfuron-methyl
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	3	0.75	NR
2	4.6	1.4	NR
3	NT	NT	NT
4	NT	NT	NT
5	4	1	NR
6	6.2	1.86	169.5
7	NT	NT	NT
8	NT	NT	NT
9	NR	NR	NR
10	NS	NS	NS
11	NT	NT	NT
12	NS	NS	NS
13	NT	NT	NT
14	NT	NT	NT
15	NT	NT	NT
16	NT	NT	NT
17	NT	NT	NT
18	NT	NT	NT
19	NT	NT	NT
20	1.8	0.54	NR
21	NS	NS	NS
22	3.6	0.18	82
23	NT	NT	NT
24	NT	NT	NT
25	NT	NT	NT
26	3.82	0.99	70
27	NT	NT	NT

Statistics

Assigned Value	Not Set	
Spike Value	2.50	0.13
Robust Average	3.8	1.4
Median	3.8	1.1
Mean	3.9	
N	7	
Max	6.2	
Min	1.8	
Robust SD	1.5	
Robust CV	39%	

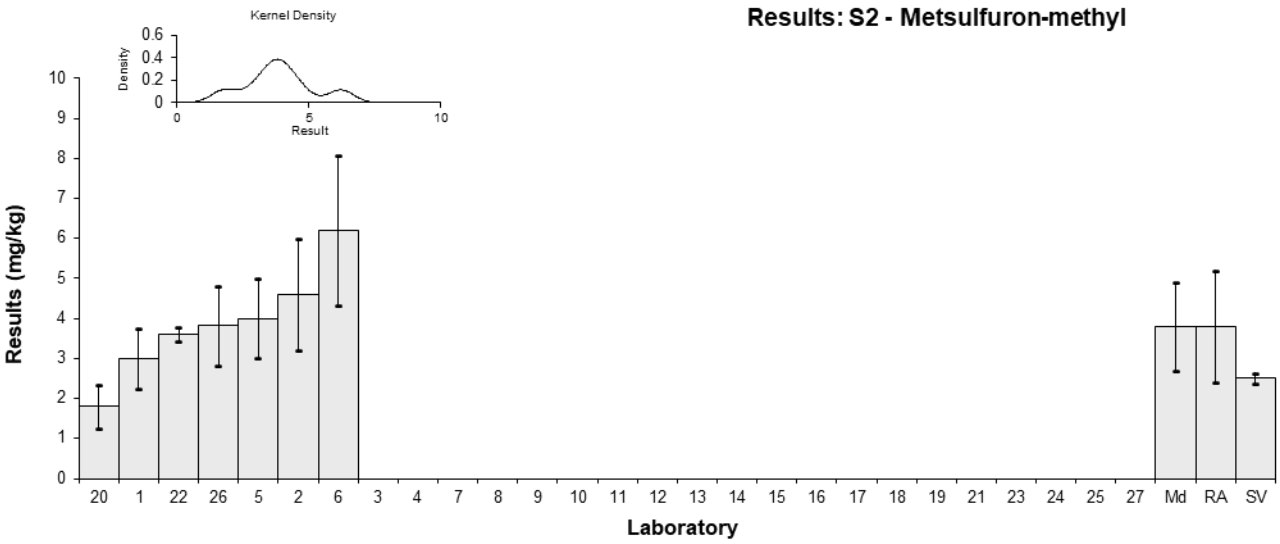

Results: S2 - Metsulfuron-methyl
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
20 1 22 26 5 2 6 3 4 7 8 9 10 11 12 13 14 15 16 17 18 19 21 23 24 25 27 Md RA SV

Figure 19

Table 24

Sample Details

Sample No.	S2
Matrix	Soil
Analyte	Omethoate
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	<0.5	NR	NR
2	0.51	0.15	NR
3	NT	NT	NT
4	NT	NT	NT
5	NT	NT	NT
6	0.36	0.108	64.5
7	NT	NT	NT
8	0.476	0.095	83
9	NR	NR	NR
10	NS	NS	NS
11	0.511	0.1533	NR
12	NS	NS	NS
13	NT	NT	NT
14	< 2	NR	130
15	NT	NT	NT
16	NT	NT	NT
17	NT	NT	NT
18	0.149	0.045	NR
19	< 2	NR	NR
20	< 2	NR	NR
21	NS	NS	NS
22	NT	NT	NT
23	NT	NT	NT
24	< 2	NR	NR
25	0.055	0.037	NR
26	NT	NT	NT
27	NT	NT	NT

Statistics

Assigned Value	Not Set	
Spike Value	0.707	0.035
Robust Average	0.34	0.23
Median	0.42	0.14
Mean	0.34	
N	6	
Max	0.511	
Min	0.055	
Robust SD	0.22	
Robust CV	65%	

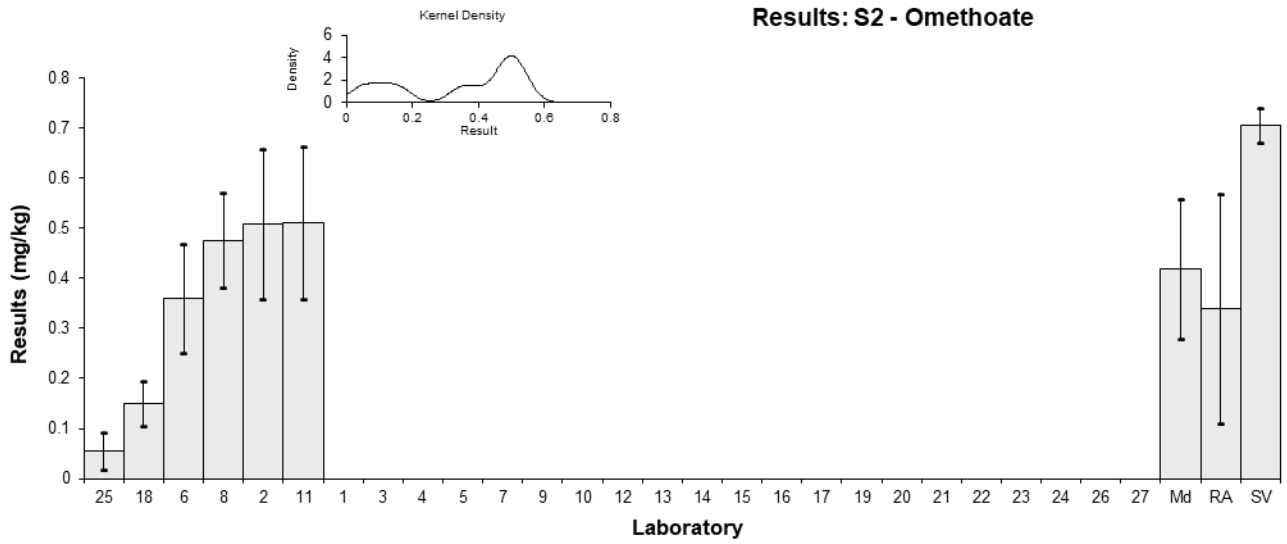

Results: S2 - Omethoate
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
25 18 6 8 2 11 1 3 4 5 7 9 10 12 13 14 15 16 17 19 20 21 22 23 24 26 27 Md RA SV

Figure 20

Table 25

Sample Details

Sample No.	S3
Matrix	Compost
Analyte	Acephate
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	NS	NS	NS
2	1.3	0.38	NR
3	NT	NT	NT
4	NT	NT	NT
5	NT	NT	NT
6	0.75	0.225	145
7	NT	NT	NT
8	0.903	0.181	97
9	NS	NS	NS
10	NS	NS	NS
11	NS	NS	NS
12	NT	NT	NT
13	NT	NT	NT
14	NT	NT	NT
15	NT	NT	NT
16	NT	NT	NT
17	NT	NT	NT
18	NR	NR	NR
19	NT	NT	NT
20	NT	NT	NT
21	NS	NS	NS
22	NT	NT	NT
23	NT	NT	NT
24	NT	NT	NT
25	NT	NT	NT
26	NT	NT	NT
27	NT	NT	NT

Statistics

Assigned Value	Not Set	
Spike Value	1.51	0.08
Homogeneity Value	1.08	0.54
Robust Average	NA (N<6)	
Median	0.90	0.33
Mean	0.98	
N	3	
Max	1.3	
Min	0.75	
Robust SD	NA (N<6)	
Robust CV	NA (N<6)	

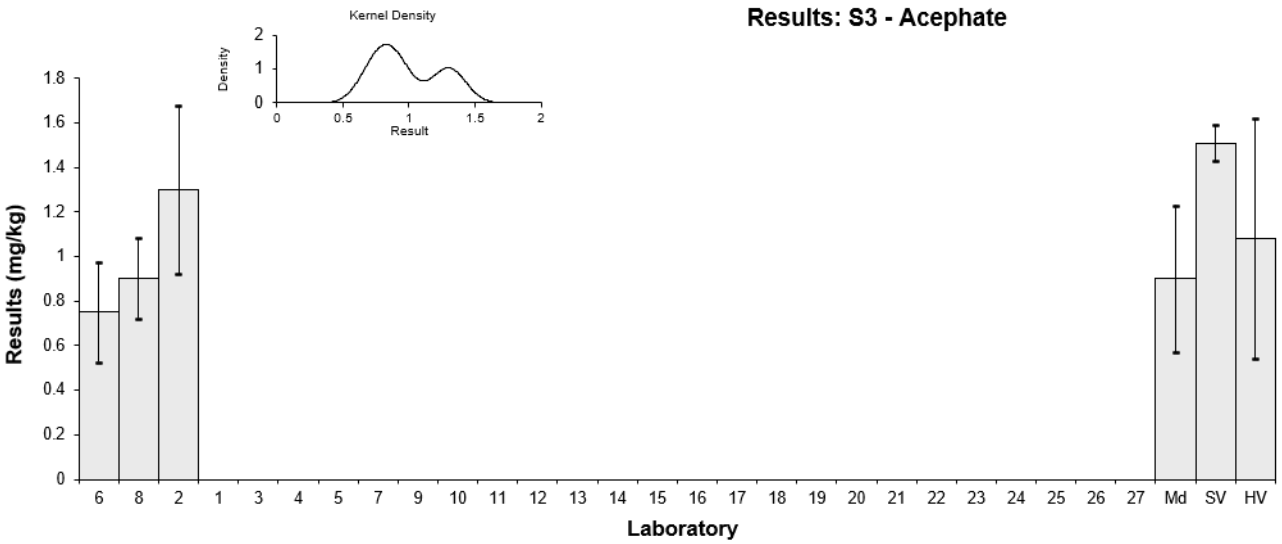

Results: S3 - Acephate
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
6 8 2 1 3 4 5 7 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 Md SV HV

Figure 21

Table 26

Sample Details

Sample No.	S3
Matrix	Compost
Analyte	Bifenthrin
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	NS	NS	NS
2	0.11	0.020	NR
3	<2	NR	NR
4	0.082	0.025	129
5	<3	NR	NR
6	0.39	0.117	100
7	NT	NT	NT
8	NR	NR	NR
9	NS	NS	NS
10	NS	NS	NS
11	NS	NS	NS
12	NT	NT	NT
13	<0.05	NR	NR
14	0.23	0.0805	134
15	<1	NR	80-120
16	<0.5	0.5	NR
17	<1.52	NR	110.94
18	NR	NR	NR
19	0.25	0.075	NR
20	0.21	0.06	NR
21	NS	NS	NS
22	0.081	0.097	49
23	NT	NT	NT
24	0.25	0.05	NR
25	<0.3	0.048	NR
26	NT	NT	NT
27	<1	NR	NR

Statistics

Assigned Value	Not Set	
Spike Value	Not Spiked	
Homogeneity Value	0.107	0.011
Robust Average	0.20	0.10
Median	0.220	0.092
Mean	0.200	
N	8	
Max	0.39	
Min	0.081	
Robust SD	0.11	
Robust CV	58%	

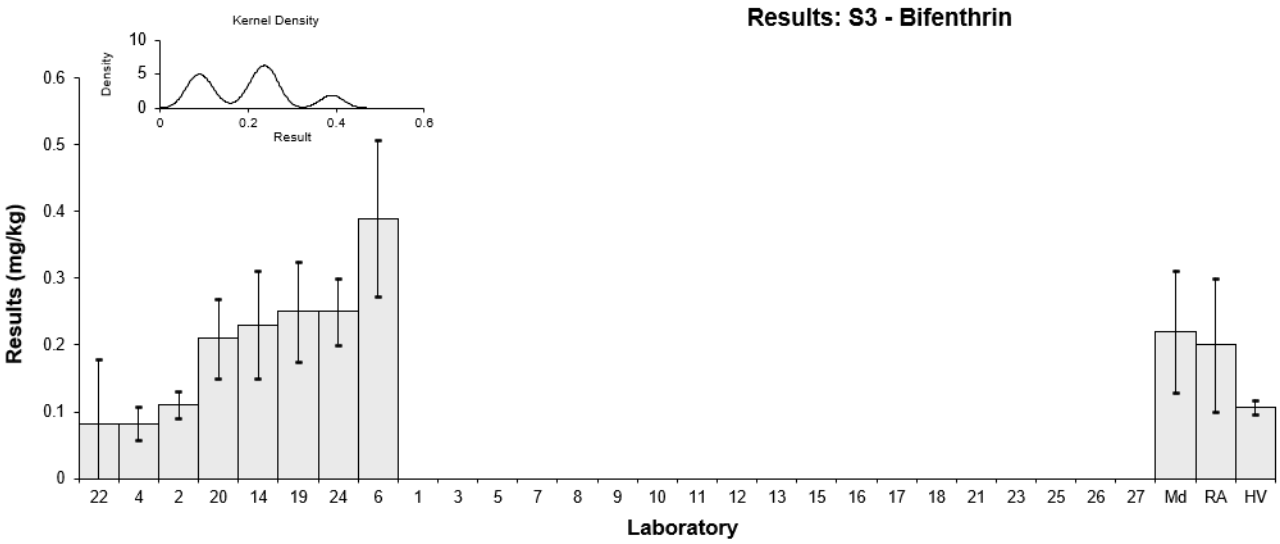

Results: S3 - Bifenthrin
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
22 4 2 20 14 19 24 6 1 3 5 7 8 9 10 11 12 13 15 16 17 18 21 23 25 26 27 Md RA HV

Figure 22

Table 27

Sample Details

Sample No.	S3
Matrix	Compost
Analyte	Total Chlordane
Unit	mg/kg

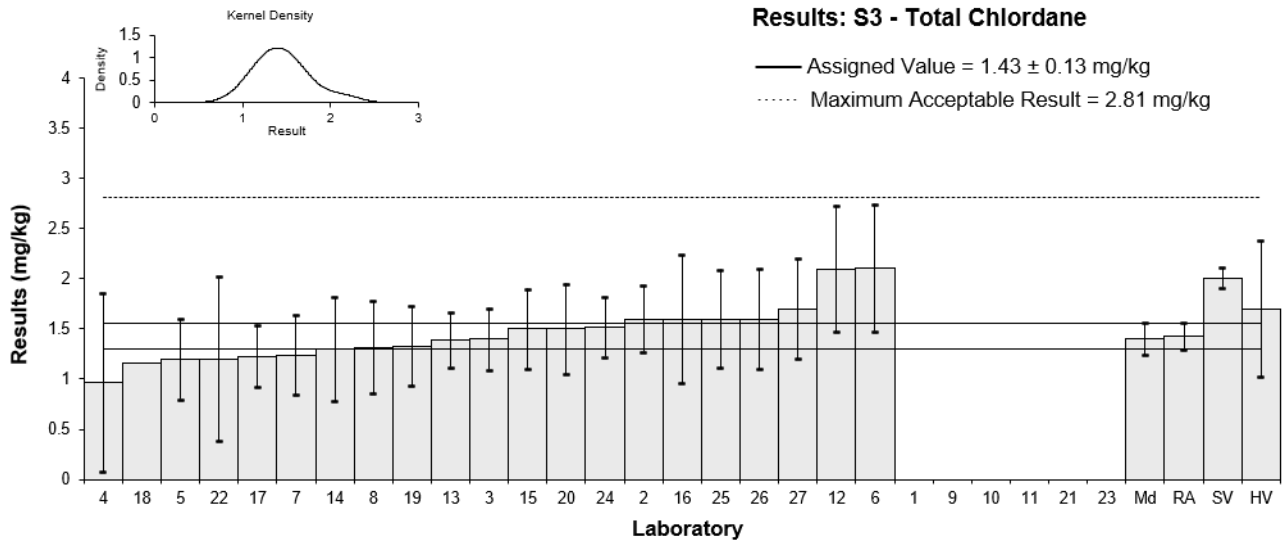
Participant Results

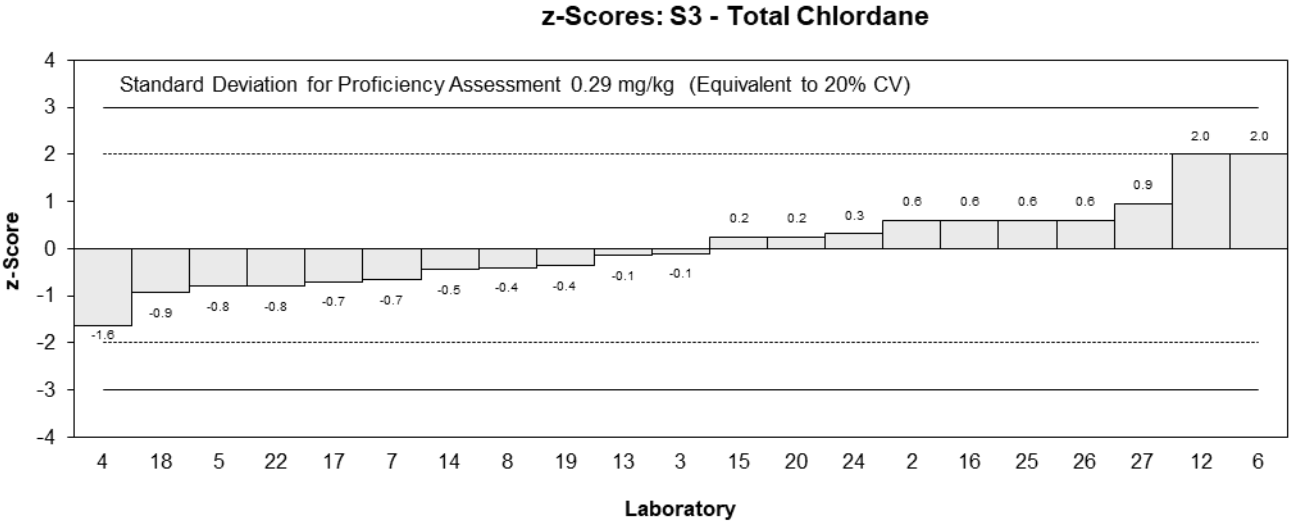
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	NS	NS	NS		
2	1.6	0.33	NR	0.59	0.48
3	1.4	0.308	NR	-0.10	-0.09
4	0.964	0.89	84	-1.63	-0.52
5	1.2	0.4	NR	-0.80	-0.55
6	2.11	0.633	NR	2.00▼	
7	1.24	0.4	NR	-0.66	-0.45
8	1.317	0.461	82	-0.40	-0.24
9	NS	NS	NS		
10	NS	NS	NS		
11	NS	NS	NS		
12	2.1	0.63	NR	2.00▼	
13	1.39	0.271	NR	-0.14	-0.13
14	1.3	0.52	134	-0.45	-0.24
15	1.5	0.4	80-120	0.24	0.17
16	1.6	0.64	98	0.59	0.26
17	1.23	0.31	110.94	-0.70	-0.59
18	1.16	NR	NR	-0.94	-2.08
19	1.33	0.4	NR	-0.35	-0.24
20	1.5	0.45	NR	0.24	0.15
21	NS	NS	NS		
22	1.2	0.82	70	-0.80	-0.28
23	NT	NT	NT		
24	1.52	0.30	NR	0.31	0.28
25	1.6	0.49	NR	0.59	0.34
26	1.6	0.5	100	0.59	0.33
27	1.7	0.5	NR	0.94	0.52

▼ Adjusted Score, see Section 6.3

Statistics

Assigned Value	1.43	0.13
Spike Value	2.01	0.10
Homogeneity Value	1.70	0.68
Robust Average	1.43	0.13
Max Acceptable Result	2.81	
Median	1.40	0.16
Mean	1.46	
N	21	
Max	2.11	
Min	0.964	
Robust SD	0.24	
Robust CV	17%	


Kernel Density
Density
Result
Results: S3 - Total Chlordane
Assigned Value = 1.43 ± 0.13 mg/kg
Maximum Acceptable Result = 2.81 mg/kg
Results (mg/kg)
4 18 5 22 17 7 14 8 19 13 3 15 20 24 2 16 25 26 27 12 6 1 9 10 11 21 23 Md RA SV HV
Laboratory


z-Scores: S3 - Total Chlordane
Standard Deviation for Proficiency Assessment 0.29 mg/kg (Equivalent to 20% CV)
z-Score
-1.6 -0.9 -0.8 -0.8 -0.7 -0.7 -0.5 -0.4 -0.4 -0.1 -0.1 0.2 0.2 0.3 0.6 0.6 0.6 0.6 0.9 2.0 2.0
4 18 5 22 17 7 14 8 19 13 3 15 20 24 2 16 25 26 27 12 6
Laboratory

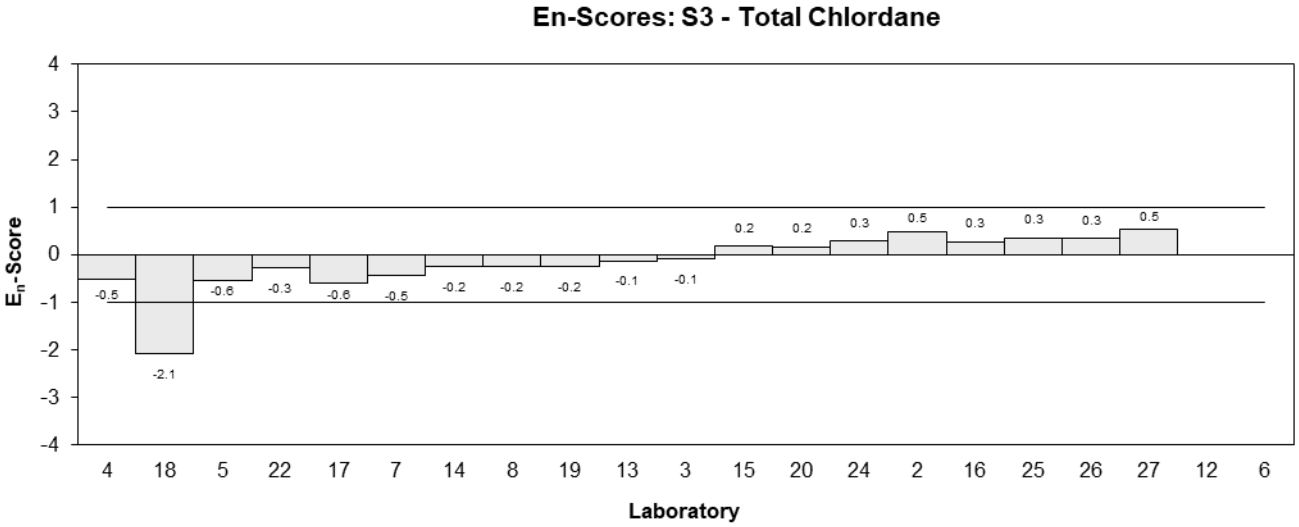

En-Scores: S3 - Total Chlordane
En-Score
-0.5 -2.1 -0.6 -0.3 -0.6 -0.5 -0.2 -0.2 -0.2 -0.1 -0.1 0.2 0.2 0.3 0.5 0.3 0.3 0.3 0.5
4 18 5 22 17 7 14 8 19 13 3 15 20 24 2 16 25 26 27 12 6
Laboratory

Figure 23

Table 28

Sample Details

Sample No.	S3
Matrix	Compost
Analyte	Diazinon
Unit	mg/kg

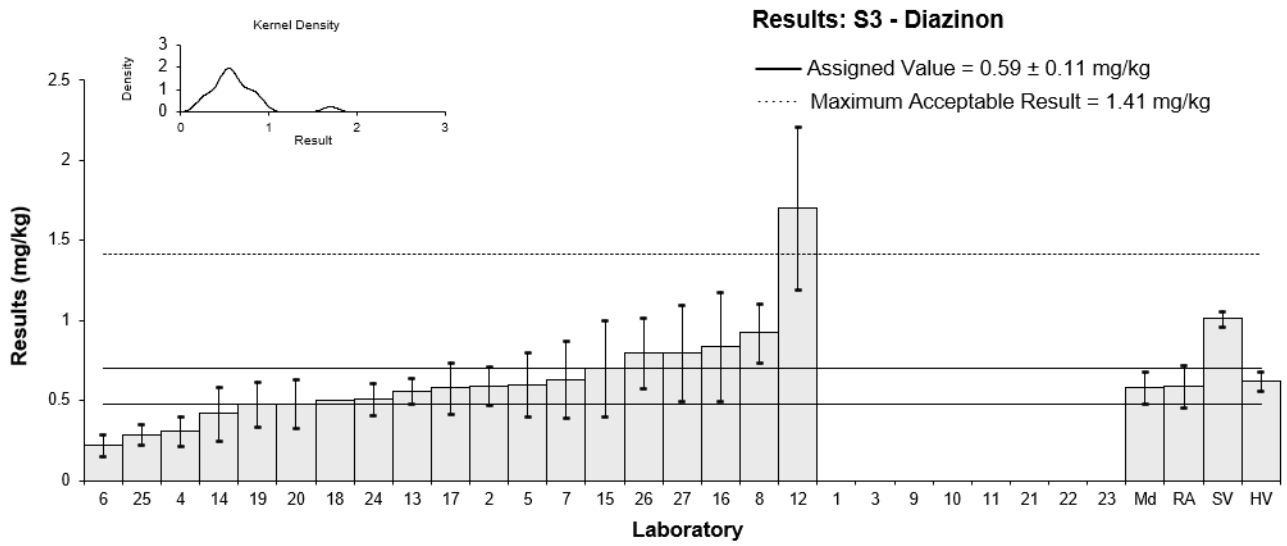
Participant Results

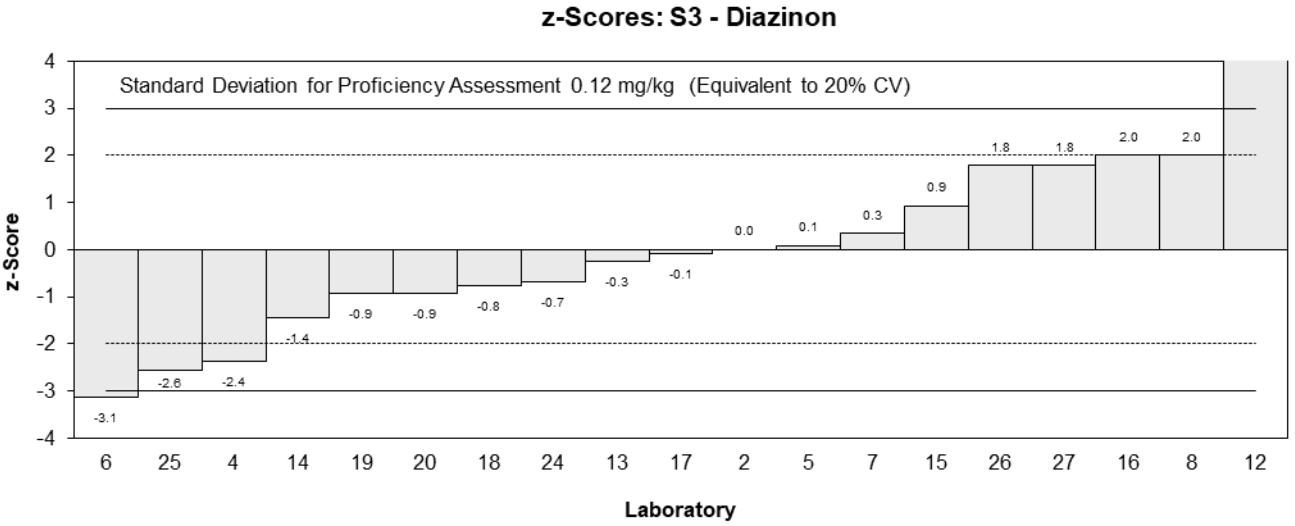
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	NS	NS	NS		
2	0.59	0.12	NR	0.00	0.00
3	<0.7	NR	NR		
4	0.31	0.093	129	-2.37	-1.94
5	0.6	0.2	NR	0.08	0.04
6*	0.22	0.066	72.5	-3.14	-2.88
7	0.63	0.24	NR	0.34	0.15
8*	0.922	0.184	94	2.00▼	
9	NS	NS	NS		
10	NS	NS	NS		
11	NS	NS	NS		
12*	1.7	0.51	127	9.41	2.13
13	0.56	0.077	NR	-0.25	-0.22
14	0.42	0.168	134	-1.44	-0.85
15	0.7	0.3	80-120	0.93	0.34
16	0.84	0.34	84	2.00▼	
17	0.58	0.16	101.15	-0.08	-0.05
18	0.5	NR	NR	-0.76	-0.82
19	0.48	0.14	NR	-0.93	-0.62
20	0.48	0.15	NR	-0.93	-0.59
21	NS	NS	NS		
22	NT	NT	NT		
23	NT	NT	NT		
24	0.51	0.10	NR	-0.68	-0.54
25*	0.288	0.062	NR	-2.56	-2.39
26	0.8	0.22	NR	1.78	0.85
27	0.8	0.3	NR	1.78	0.66

* Outlier, see Section 4.2; ▼ Adjusted Score, see Section 6.3

Statistics

Assigned Value	0.59	0.11
Spike Value	1.01	0.05
Homogeneity Value	0.619	0.062
Robust Average	0.59	0.13
Max Acceptable Result	1.41	
Median	0.58	0.10
Mean	0.63	
N	19	
Max	1.7	
Min	0.22	
Robust SD	0.23	
Robust CV	39%	


Results: S3 - Diazinon
Kernel Density
Density
Result
Assigned Value = 0.59 ± 0.11 mg/kg
Maximum Acceptable Result = 1.41 mg/kg
Results (mg/kg)
Laboratory
6 25 4 14 19 20 18 24 13 17 2 5 7 15 26 27 16 8 12 1 3 9 10 11 21 22 23 Md RA SV HV


z-Scores: S3 - Diazinon
Standard Deviation for Proficiency Assessment 0.12 mg/kg (Equivalent to 20% CV)
z-Score
Laboratory
6 25 4 14 19 20 18 24 13 17 2 5 7 15 26 27 16 8 12
-3.1 -2.6 -2.4 -1.4 -0.9 -0.9 -0.8 -0.7 -0.3 -0.1 0.0 0.1 0.3 0.9 1.8 1.8 2.0 2.0

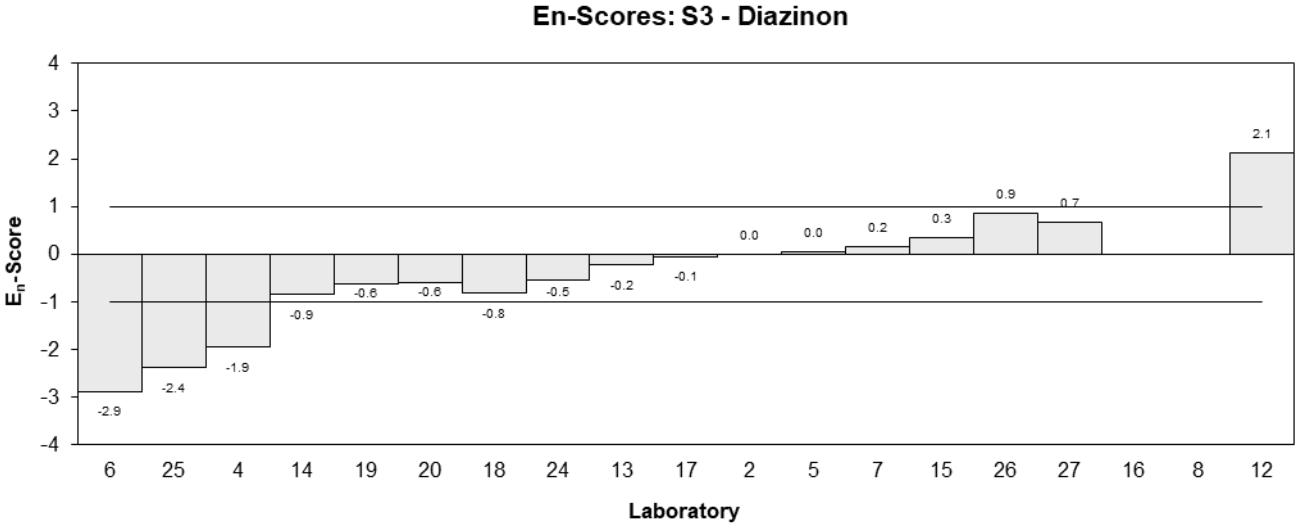

En-Scores: S3 - Diazinon
En-Score
Laboratory
6 25 4 14 19 20 18 24 13 17 2 5 7 15 26 27 16 8 12
-2.9 -2.4 -1.9 -0.9 -0.6 -0.6 -0.8 -0.5 -0.2 -0.1 0.0 0.0 0.2 0.3 0.9 0.7 2.1

Figure 24

Table 29

Sample Details

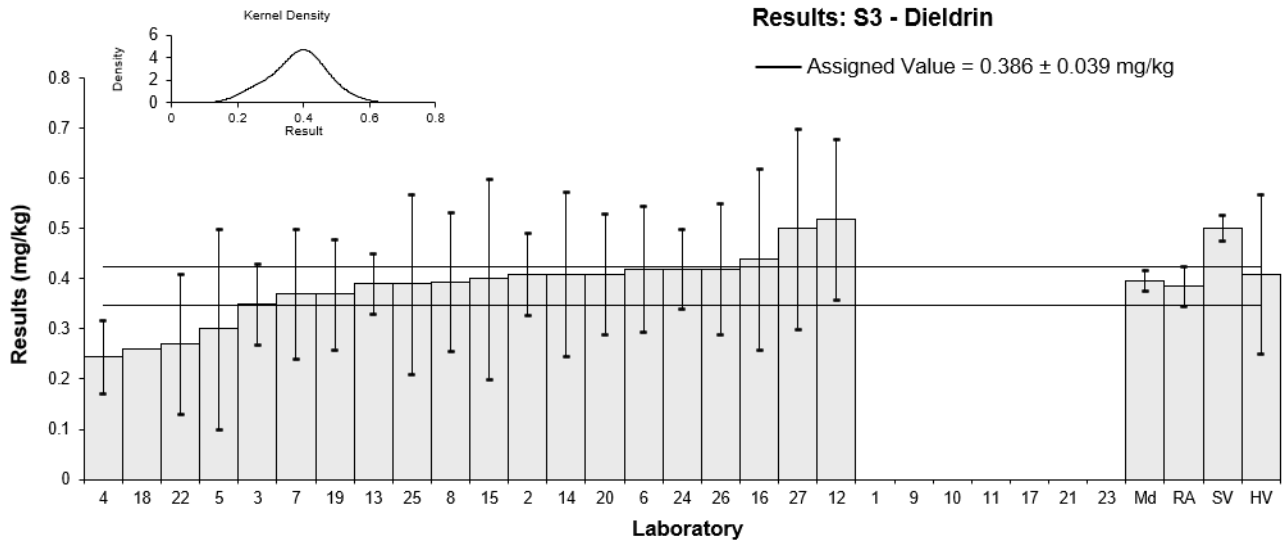
Sample No.	S3
Matrix	Compost
Analyte	Dieldrin
Unit	mg/kg

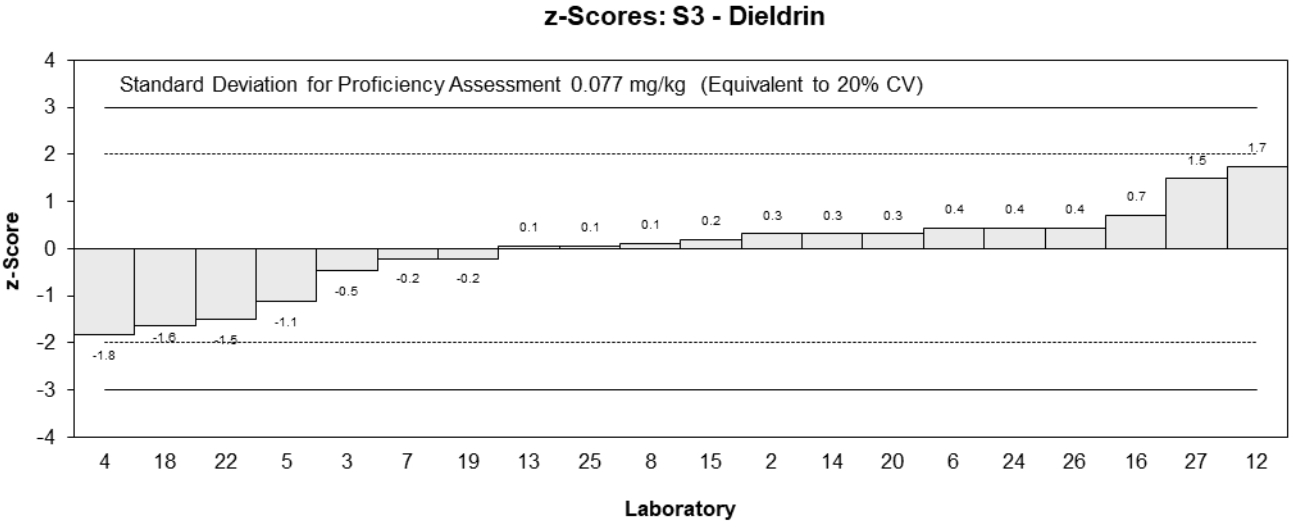
Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E_n
1	NS	NS	NS		
2	0.41	0.082	NR	0.31	0.26
3	0.35	0.0805	NR	-0.47	-0.40
4	0.244	0.073	101	-1.84	-1.72
5	0.3	0.2	NR	-1.11	-0.42
6	0.42	0.126	87	0.44	0.26
7	0.37	0.13	NR	-0.21	-0.12
8	0.394	0.138	100	0.10	0.06
9	NS	NS	NS		
10	NS	NS	NS		
11	NS	NS	NS		
12	0.52	0.16	138	1.74	0.81
13	0.39	0.06	NR	0.05	0.06
14	0.41	0.164	134	0.31	0.14
15	0.4	0.2	80-120	0.18	0.07
16	0.44	0.18	98	0.70	0.29
17	<0.48	NR	110.94		
18	0.26	NR	NR	-1.63	-3.23
19	0.37	0.11	NR	-0.21	-0.14
20	0.41	0.12	NR	0.31	0.19
21	NS	NS	NS		
22	0.27	0.14	78	-1.50	-0.80
23	NT	NT	NT		
24	0.42	0.08	NR	0.44	0.38
25	0.39	0.18	NR	0.05	0.02
26	0.42	0.13	81	0.44	0.25
27	0.5	0.2	NR	1.48	0.56

Statistics

Assigned Value	0.386	0.039
Spike Value	0.502	0.025
Homogeneity Value	0.41	0.16
Robust Average	0.386	0.039
Median	0.397	0.021
Mean	0.384	
N	20	
Max	0.52	
Min	0.244	
Robust SD	0.071	
Robust CV	18%	


Results: S3 - Dieldrin
Assigned Value = 0.386 ± 0.039 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
4 18 22 5 3 7 19 13 25 8 15 2 14 20 6 24 26 16 27 12 1 9 10 11 17 21 23 Md RA SV HV


z-Scores: S3 - Dieldrin
Standard Deviation for Proficiency Assessment 0.077 mg/kg (Equivalent to 20% CV)
z-Score
Laboratory
4 18 22 5 3 7 19 13 25 8 15 2 14 20 6 24 26 16 27 12

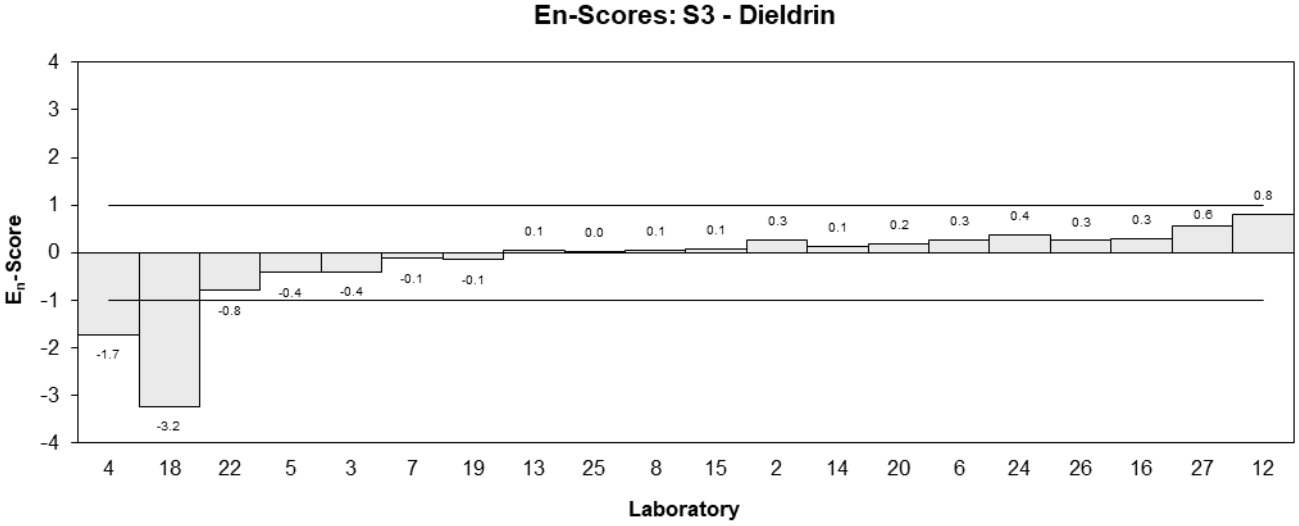

En-Scores: S3 - Dieldrin
En-Score
Laboratory
4 18 22 5 3 7 19 13 25 8 15 2 14 20 6 24 26 16 27 12

Figure 25

Table 30

Sample Details

Sample No.	S3
Matrix	Compost
Analyte	Endosulfan sulfate
Unit	mg/kg

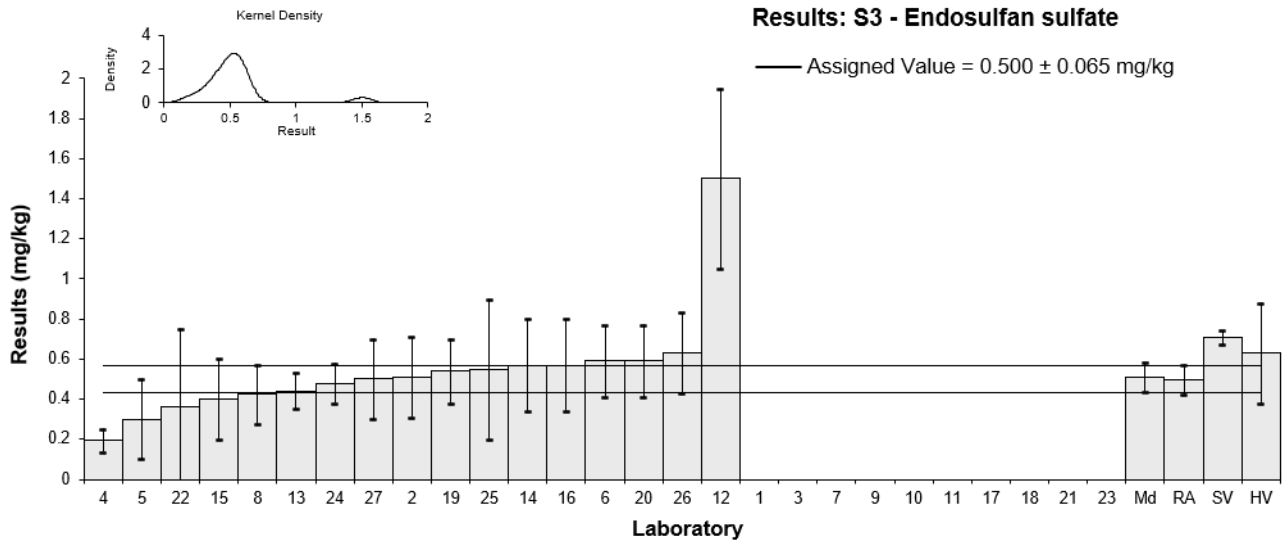
Participant Results

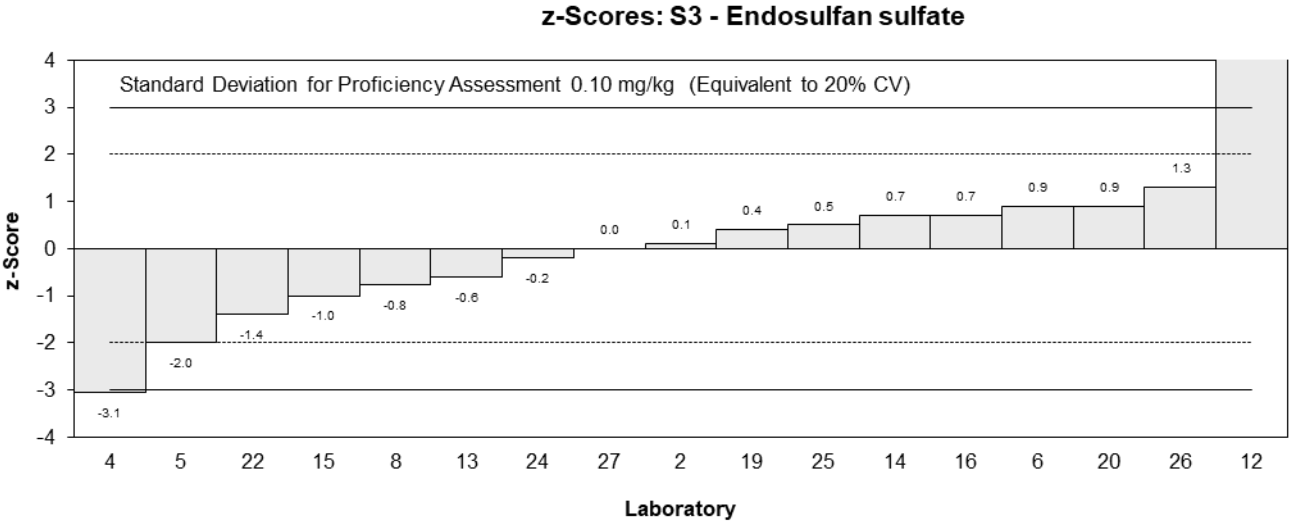
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	NS	NS	NS		
2	0.51	0.20	NR	0.10	0.05
3	<0.4	NR	NR		
4*	0.194	0.058	100	-3.06	-3.51
5	0.3	0.2	NR	-2.00	-0.95
6	0.59	0.177	101	0.90	0.48
7	<0.45	0.22	NR		
8	0.424	0.148	83	-0.76	-0.47
9	NS	NS	NS		
10	NS	NS	NS		
11	NS	NS	NS		
12*	1.5	0.45	NR	10.00	2.20
13	0.44	0.089	NR	-0.60	-0.54
14	0.57	0.228	134	0.70	0.30
15	0.4	0.2	80-120	-1.00	-0.48
16	0.57	0.23	98	0.70	0.29
17	<0.48	NR	110.94		
18	<0.25	NR	NR		
19	0.54	0.16	NR	0.40	0.23
20	0.59	0.18	NR	0.90	0.47
21	NS	NS	NS		
22	0.36	0.39	66	-1.40	-0.35
23	NT	NT	NT		
24	0.48	0.10	NR	-0.20	-0.17
25	0.55	0.35	NR	0.50	0.14
26	0.63	0.2	78	1.30	0.62
27	0.5	0.2	NR	0.00	0.00

* Outlier, see Section 4.2

Statistics

Assigned Value	0.500	0.065
Spike Value	0.706	0.035
Homogeneity Value	0.63	0.25
Robust Average	0.498	0.075
Median	0.510	0.072
Mean	0.54	
N	17	
Max	1.5	
Min	0.194	
Robust SD	0.12	
Robust CV	25%	


Kernel Density
Density
Result
Results: S3 - Endosulfan sulfate
Assigned Value = 0.500 ± 0.065 mg/kg
Results (mg/kg)
Laboratory
4 5 22 15 8 13 24 27 2 19 25 14 16 6 20 26 12 1 3 7 9 10 11 17 18 21 23 Md RA SV HV


z-Scores: S3 - Endosulfan sulfate
Standard Deviation for Proficiency Assessment 0.10 mg/kg (Equivalent to 20% CV)
z-Score
Laboratory
4 5 22 15 8 13 24 27 2 19 25 14 16 6 20 26 12
-3.1 -2.0 -1.4 -1.0 -0.8 -0.6 -0.2 0.0 0.1 0.4 0.5 0.7 0.7 0.9 0.9 1.3

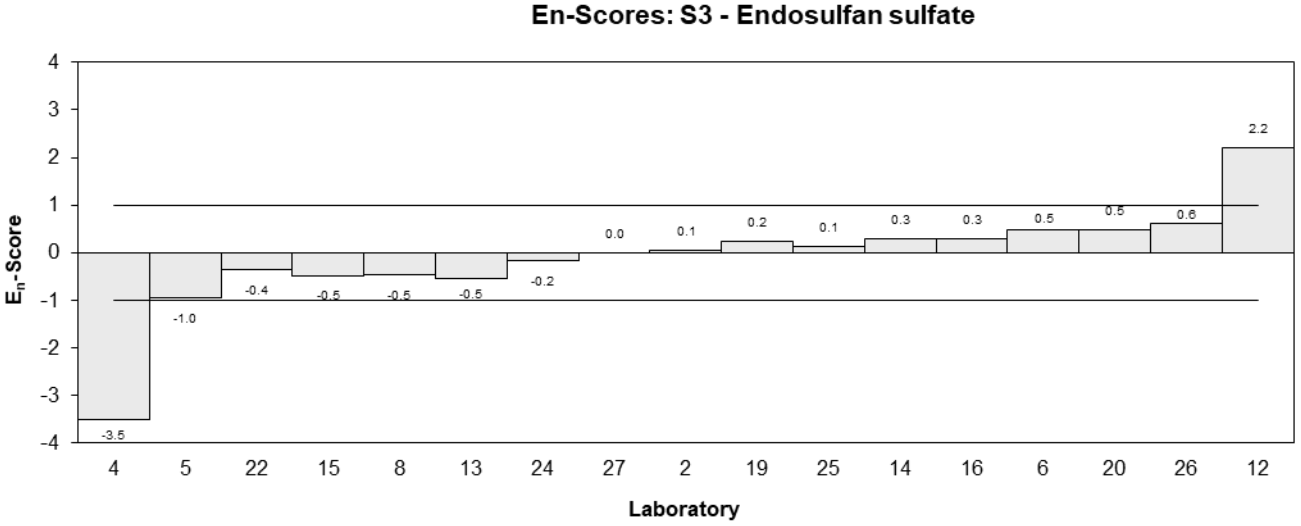

En-Scores: S3 - Endosulfan sulfate
En-Score
Laboratory
4 5 22 15 8 13 24 27 2 19 25 14 16 6 20 26 12
-3.5 -1.0 -0.4 -0.5 -0.5 -0.5 -0.2 0.0 0.1 0.2 0.1 0.3 0.3 0.5 0.5 0.6 2.2

Figure 26

Table 31

Sample Details

Sample No.	S3
Matrix	Compost
Analyte	Ethion
Unit	mg/kg

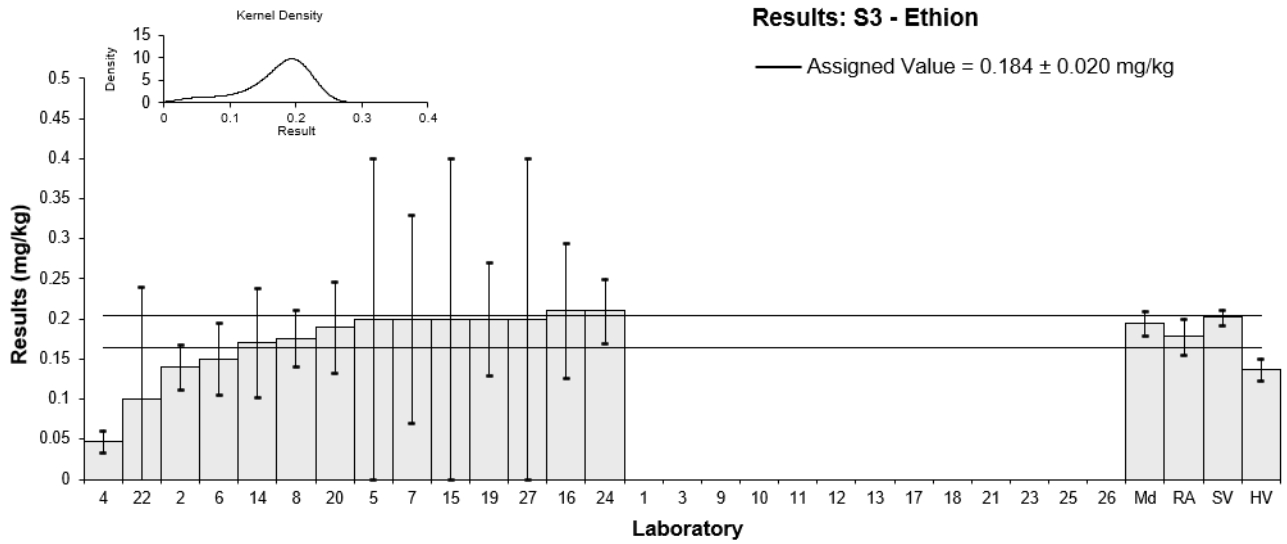
Participant Results

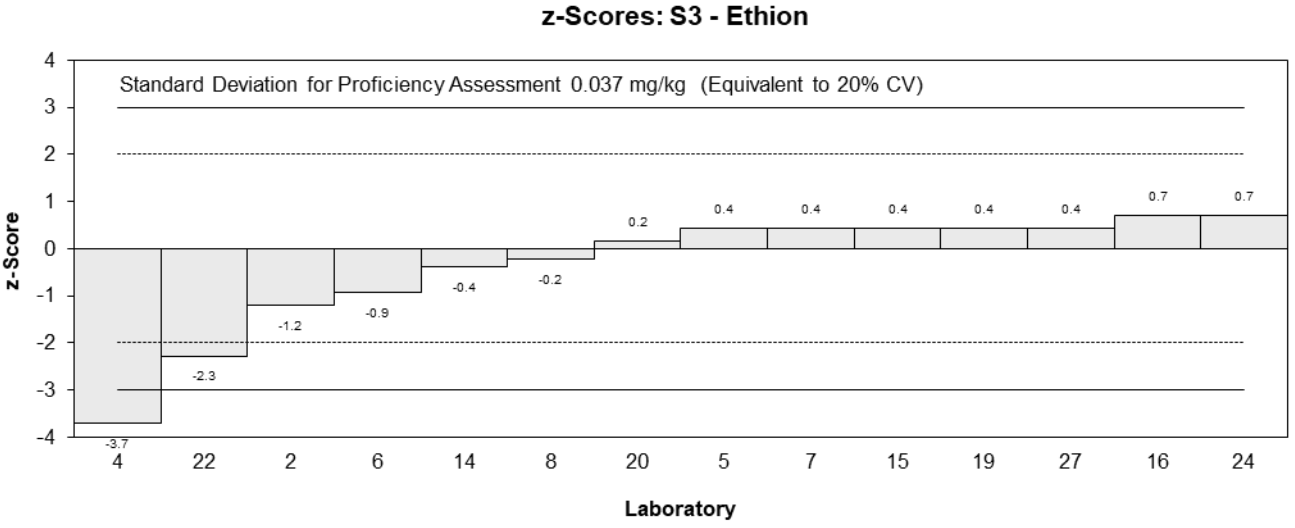
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	NS	NS	NS		
2	0.14	0.028	NR	-1.20	-1.28
3	<0.5	NR	NR		
4*	0.047	0.014	125	-3.72	-5.61
5	0.2	0.2	NR	0.43	0.08
6	0.15	0.045	89.5	-0.92	-0.69
7	0.2	0.13	NR	0.43	0.12
8	0.176	0.035	89	-0.22	-0.20
9	NS	NS	NS		
10	NS	NS	NS		
11	NS	NS	NS		
12	<0.5	NR	NR		
13	<0.05	NR	NR		
14	0.17	0.068	134	-0.38	-0.20
15	0.2	0.2	80-120	0.43	0.08
16	0.21	0.084	98	0.71	0.30
17	<0.48	NR	110.94		
18	<0.25	NR	NR		
19	0.2	0.07	NR	0.43	0.22
20	0.19	0.057	NR	0.16	0.10
21	NS	NS	NS		
22	0.10	0.14	45	-2.28	-0.59
23	NT	NT	NT		
24	0.21	0.04	NR	0.71	0.58
25	<0.6	0.093	NR		
26	NT	NT	NT		
27	0.2	0.2	NR	0.43	0.08

* Outlier, see Section 4.2

Statistics

Assigned Value	0.184	0.020
Spike Value	0.202	0.010
Homogeneity Value	0.137	0.014
Robust Average	0.178	0.023
Median	0.195	0.015
Mean	0.171	
N	14	
Max	0.21	
Min	0.047	
Robust SD	0.035	
Robust CV	19%	


Kernel Density
Density
Result
Results: S3 - Ethion
Assigned Value = 0.184 ± 0.020 mg/kg
Results (mg/kg)
Laboratory
4 22 2 6 14 8 20 5 7 15 19 27 16 24 1 3 9 10 11 12 13 17 18 21 23 25 26 Md RA SV HV


z-Scores: S3 - Ethion
Standard Deviation for Proficiency Assessment 0.037 mg/kg (Equivalent to 20% CV)
z-Score
Laboratory
4 22 2 6 14 8 20 5 7 15 19 27 16 24
-3.7 -2.3 -1.2 -0.9 -0.4 -0.2 0.2 0.4 0.4 0.4 0.4 0.4 0.7 0.7

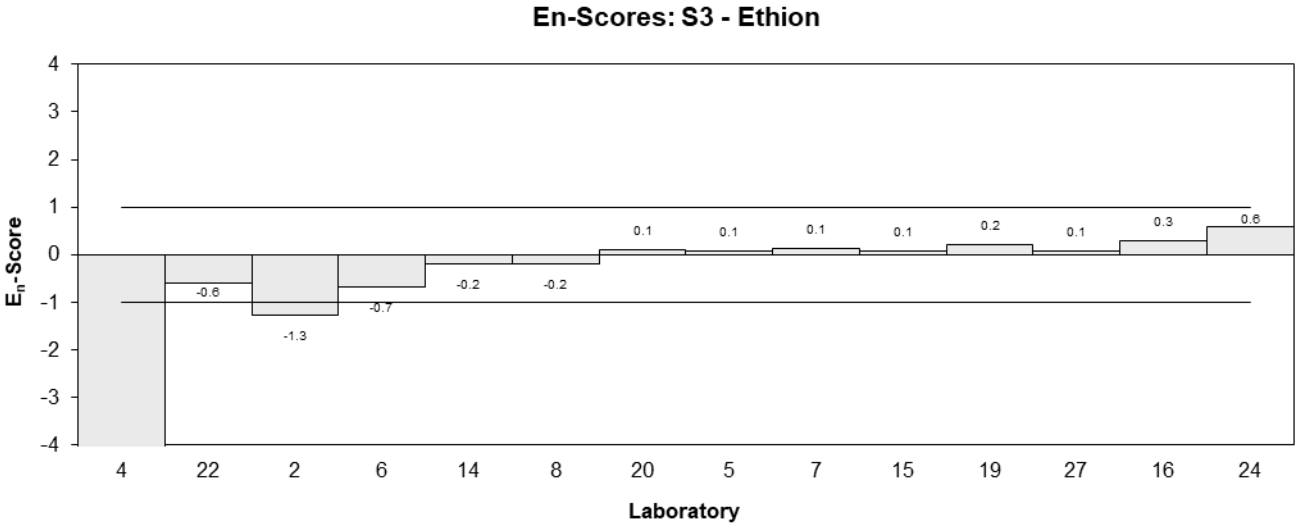

En-Scores: S3 - Ethion
En-Score
Laboratory
4 22 2 6 14 8 20 5 7 15 19 27 16 24
-0.6 -1.3 -0.7 -0.2 -0.2 0.1 0.1 0.1 0.1 0.2 0.1 0.3 0.6

Figure 27

Table 32

Sample Details

Sample No.	S3
Matrix	Compost
Analyte	Glyphosate
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	NS	NS	NS
2	2.1	0.40	NR
3	NT	NT	NT
4	NT	NT	NT
5	<1	NR	NR
6	0.44	0.132	119
7	NT	NT	NT
8	NT	NT	NT
9	NS	NS	NS
10	NS	NS	NS
11	NS	NS	NS
12	NT	NT	NT
13	NT	NT	NT
14	1.4	0.56	91
15	NT	NT	NT
16	NT	NT	NT
17	NT	NT	NT
18	1	0.2	NR
19	NT	NT	NT
20	NT	NT	NT
21	NS	NS	NS
22	2.4	1.2	90
23	NT	NT	NT
24	NT	NT	NT
25	NT	NT	NT
26	1.6	0.6	NR
27	NT	NT	NT

Statistics

Assigned Value	Not Set	
Spike Value	1.51	0.08
Homogeneity Value	1.98	0.81
Robust Average	1.49	0.83
Median	1.50	0.83
Mean	1.49	
N	6	
Max	2.4	
Min	0.44	
Robust SD	0.81	
Robust CV	54%	

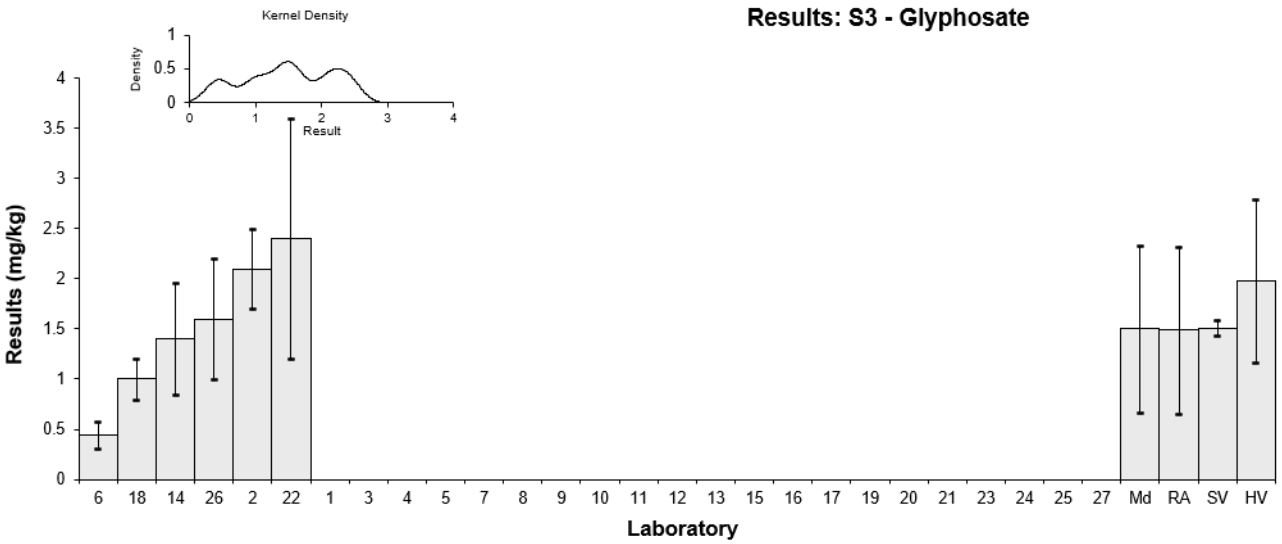

Results: S3 - Glyphosate
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
6 18 14 26 2 22 1 3 4 5 7 8 9 10 11 12 13 15 16 17 19 20 21 23 24 25 27 Md RA SV HV

Figure 28

Table 33

Sample Details

Sample No.	S3
Matrix	Compost
Analyte	Imidacloprid
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	NS	NS	NS
2	NR	NR	NR
3	<1	NR	NR
4	0.178	0.053	78
5	1.1	0.4	NR
6	0.74	0.222	83.5
7	NT	NT	NT
8	NT	NT	NT
9	NS	NS	NS
10	NS	NS	NS
11	NS	NS	NS
12	NT	NT	NT
13	NT	NT	NT
14	NT	NT	NT
15	NT	NT	NT
16	NT	NT	NT
17	NT	NT	NT
18	NR	NR	NR
19	NT	NT	NT
20	NT	NT	NT
21	NS	NS	NS
22	1.1	0.55	93
23	NT	NT	NT
24	NT	NT	NT
25	NT	NT	NT
26	0.8	0.26	80
27	NT	NT	NT

Statistics

Assigned Value	Not Set	
Spike Value	1.50	0.08
Homogeneity Value	0.48	0.24
Robust Average	NA (N<6)	
Median	0.80	0.50
Mean	0.78	
N	5	
Max	1.1	
Min	0.178	
Robust SD	NA (N<6)	
Robust CV	NA (N<6)	

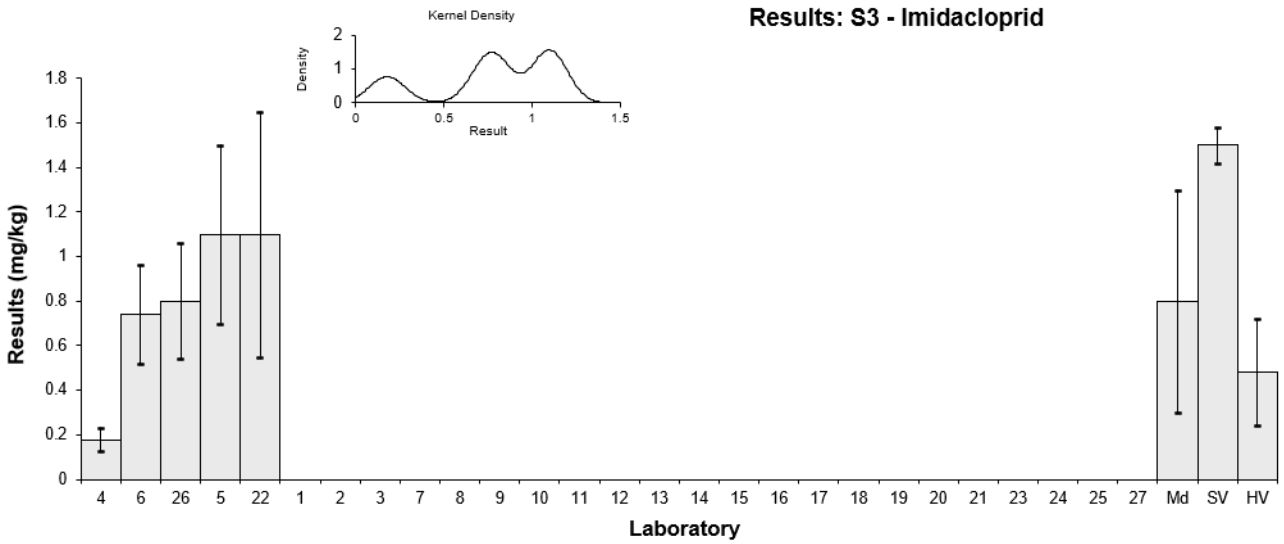

Results: S3 - Imidacloprid
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
4 6 26 5 22 1 2 3 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 23 24 25 27 Md SV HV

Figure 29

Table 34

Sample Details

Sample No.	S3
Matrix	Compost
Analyte	Lindane
Unit	mg/kg

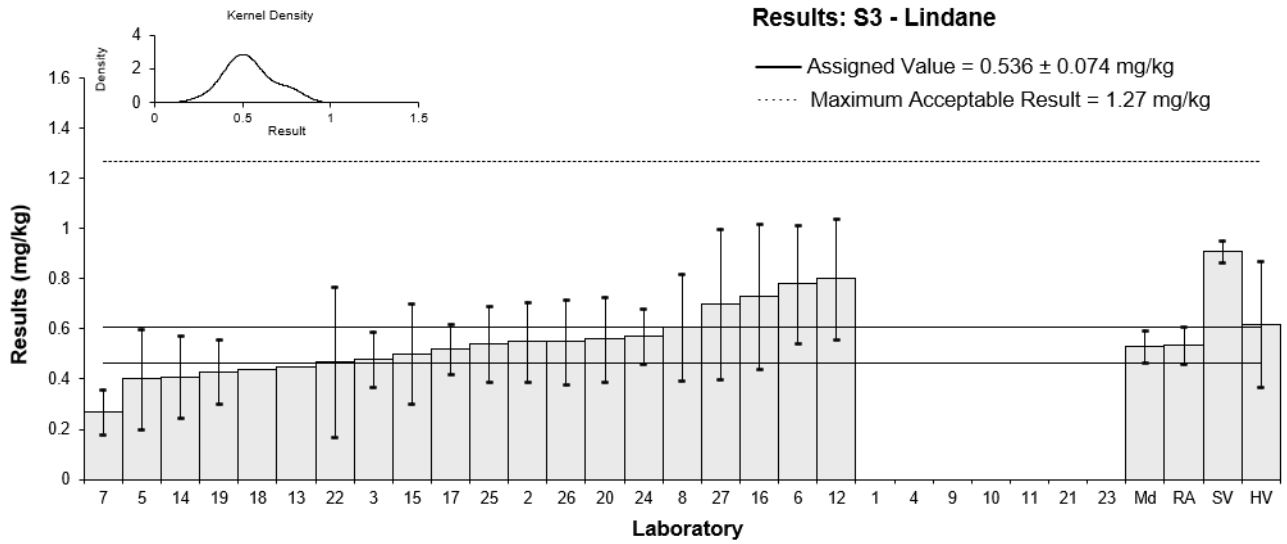
Participant Results

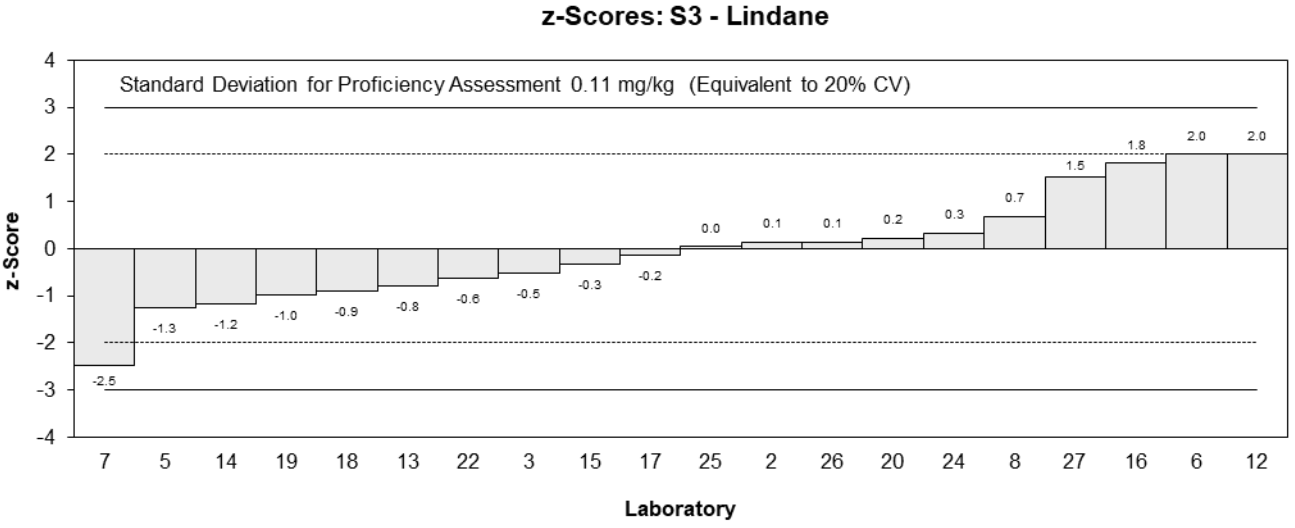
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	NS	NS	NS		
2	0.55	0.16	NR	0.13	0.08
3	0.48	0.1104	NR	-0.52	-0.42
4	<0.01	NR	NR		
5	0.4	0.2	NR	-1.27	-0.64
6	0.78	0.234	101	2.00▼	
7	0.27	0.09	NR	-2.48	-2.28
8	0.608	0.213	89	0.67	0.32
9	NS	NS	NS		
10	NS	NS	NS		
11	NS	NS	NS		
12	0.8	0.24	108	2.00▼	
13	0.45	NR	NR	-0.80	-1.16
14	0.41	0.164	134	-1.18	-0.70
15	0.5	0.2	80-120	-0.34	-0.17
16	0.73	0.29	93	1.81	0.65
17	0.52	0.1	101.15	-0.15	-0.13
18	0.44	NR	NR	-0.90	-1.30
19	0.43	0.13	NR	-0.99	-0.71
20	0.56	0.17	NR	0.22	0.13
21	NS	NS	NS		
22	0.47	0.30	74	-0.62	-0.21
23	NT	NT	NT		
24	0.57	0.11	NR	0.32	0.26
25	0.54	0.15	NR	0.04	0.02
26	0.55	0.17	81	0.13	0.08
27	0.7	0.3	NR	1.53	0.53

▼ Adjusted Score, see Section 6.3

Statistics

Assigned Value	0.536	0.074
Spike Value	0.909	0.045
Homogeneity Value	0.62	0.25
Robust Average	0.536	0.074
Max Acceptable Result	1.27	
Median	0.530	0.065
Mean	0.538	
N	20	
Max	0.8	
Min	0.27	
Robust SD	0.13	
Robust CV	25%	


Kernel Density
Density
Result
Results: S3 - Lindane
Assigned Value = 0.536 ± 0.074 mg/kg
Maximum Acceptable Result = 1.27 mg/kg
Results (mg/kg)
7 5 14 19 18 13 22 3 15 17 25 2 26 20 24 8 27 16 6 12 1 4 9 10 11 21 23 Md RA SV HV
Laboratory


z-Scores: S3 - Lindane
Standard Deviation for Proficiency Assessment 0.11 mg/kg (Equivalent to 20% CV)
z-Score
-2.5 -1.3 -1.2 -1.0 -0.9 -0.8 -0.6 -0.5 -0.3 -0.2 0.0 0.1 0.1 0.2 0.3 0.7 1.5 1.8 2.0 2.0
7 5 14 19 18 13 22 3 15 17 25 2 26 20 24 8 27 16 6 12
Laboratory

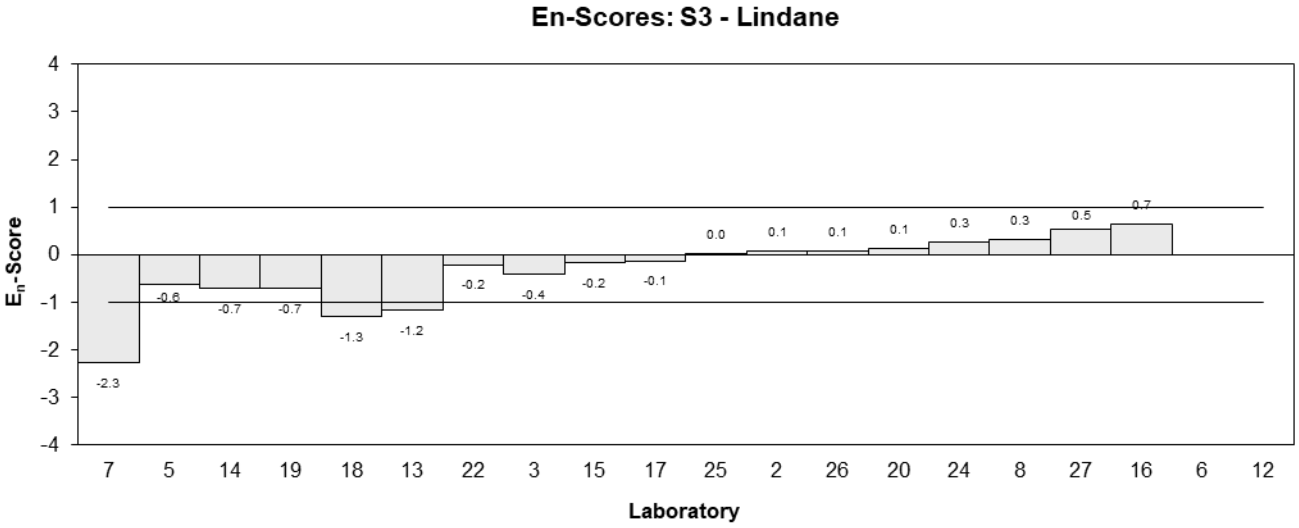

En-Scores: S3 - Lindane
En-Score
-2.3 -0.6 -0.7 -0.7 -1.3 -1.2 -0.2 -0.4 -0.2 -0.1 0.0 0.1 0.1 0.1 0.3 0.3 0.5 0.7
7 5 14 19 18 13 22 3 15 17 25 2 26 20 24 8 27 16 6 12
Laboratory

Figure 30

Table 35

Sample Details

Sample No.	S3
Matrix	Compost
Analyte	Simazine
Unit	mg/kg

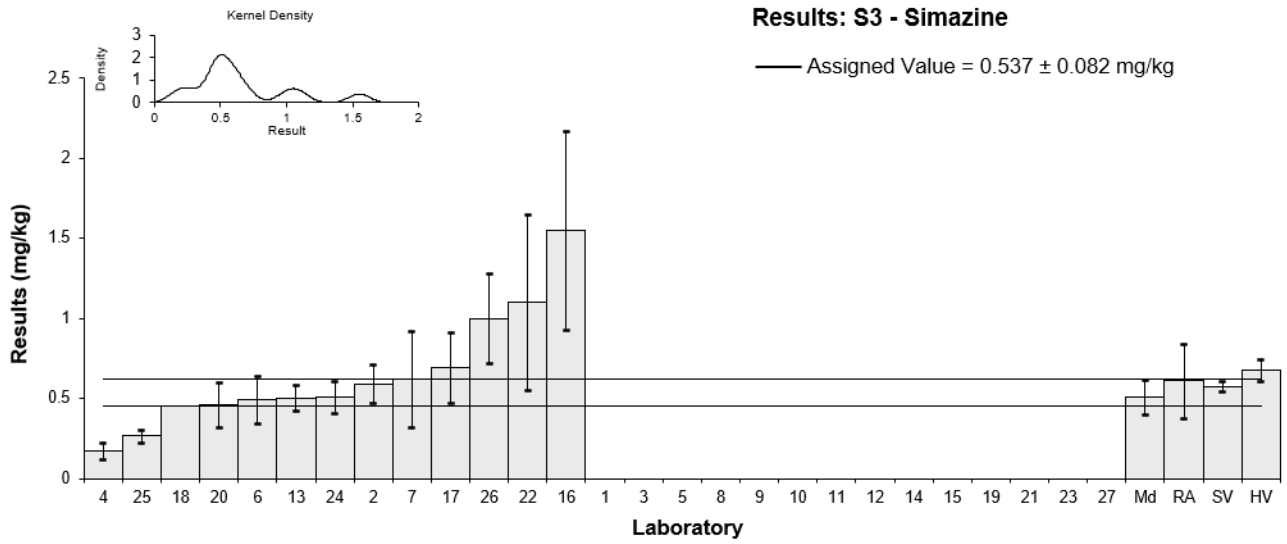
Participant Results

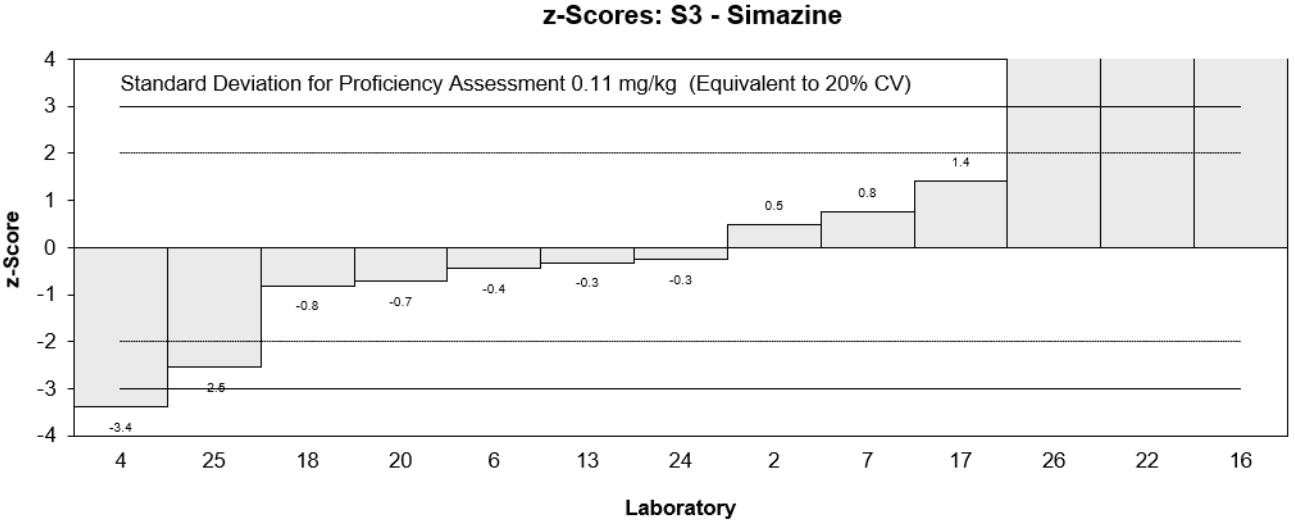
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	NS	NS	NS		
2	0.59	0.12	NR	0.49	0.36
3	<1	NR	NR		
4*	0.174	0.052	95	-3.38	-3.74
5	<1	NR	NR		
6	0.49	0.147	96.5	-0.44	-0.28
7	0.62	0.3	NR	0.77	0.27
8	NT	NT	NT		
9	NS	NS	NS		
10	NS	NS	NS		
11	NS	NS	NS		
12	NT	NT	NT		
13	0.503	0.079	NR	-0.32	-0.30
14	NT	NT	NT		
15	<1	NR	80-120		
16*	1.55	0.62	108	9.43	1.62
17	0.69	0.22	101.15	1.42	0.65
18	0.45	NR	NR	-0.81	-1.06
19	NT	NT	NT		
20	0.46	0.14	NR	-0.72	-0.47
21	NS	NS	NS		
22*	1.1	0.55	70	5.24	1.01
23	NT	NT	NT		
24	0.51	0.10	NR	-0.25	-0.21
25*	0.265	0.042	NR	-2.53	-2.95
26*	1	0.28	90	4.31	1.59
27	<1	NR	NR		

* Outlier, see Section 4.2

Statistics

Assigned Value	0.537	0.082
Spike Value	0.576	0.029
Homogeneity Value	0.680	0.068
Robust Average	0.61	0.23
Median	0.51	0.11
Mean	0.65	
N	13	
Max	1.55	
Min	0.174	
Robust SD	0.33	
Robust CV	55%	


Kernel Density
Density
Result
Results: S3 - Simazine
Assigned Value = 0.537 ± 0.082 mg/kg
Results (mg/kg)
Laboratory
4 25 18 20 6 13 24 2 7 17 26 22 16 1 3 5 8 9 10 11 12 14 15 19 21 23 27 Md RA SV HV


z-Scores: S3 - Simazine
Standard Deviation for Proficiency Assessment 0.11 mg/kg (Equivalent to 20% CV)
z-Score
Laboratory
4 25 18 20 6 13 24 2 7 17 26 22 16
-3.4
-2.6
-0.8
-0.7
-0.4
-0.3
-0.3
0.5
0.8
1.4

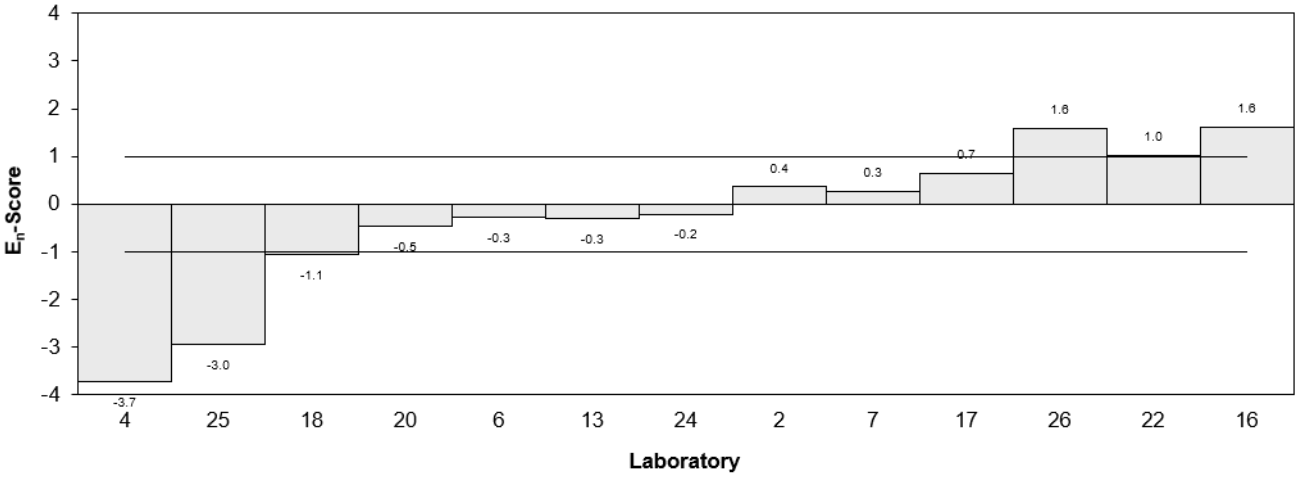

En-Scores: S3 - Simazine
En-Score
Laboratory
4 25 18 20 6 13 24 2 7 17 26 22 16
-3.7
-3.0
-1.1
-0.5
-0.3
-0.3
-0.2
0.4
0.3
0.7
1.6
1.0
1.6

Figure 31

6 DISCUSSION OF RESULTS

6.1 Assigned Value

Assigned values for all scored analytes were the robust averages of participants' results. Results less than 50% or greater than 150% of the robust average were excluded from the calculation of each assigned value.[3,4] The robust averages and associated expanded uncertainties were calculated using the procedure described in ISO 13528.[7] The calculation of the expanded uncertainty for robust averages is presented in Appendix 3, using Sample S1 lindane as an example.

Thirty-one analytes were included in this study across the three samples. No assigned values were established for chlorpyrifos, p,p'-DDD, dieldrin, fenitrothion, and fenvalerate in Sample S1; acephate, carbendazim, 2,4-D, fipronil, imidacloprid, metsulfuron-methyl, and omethoate in Sample S2; and acephate, bifenthrin, glyphosate, and imidacloprid in Sample S3. This was due to an insufficient number of numeric results, excessive variability of the reported results, or poor recovery of the spiked analyte. For these analytes, participants may still compare their results with the descriptive statistics and spiked values as presented in Section 5.

No assigned value was set for Sample S1 endosulfan sulfate either. This analyte was omitted from Section 5 because only one participant reported a numeric result for this test. The spiked value for endosulfan sulfate was 0.00199 ± 0.00010 mg/kg, and the result reported by Laboratory 6 was 0.003 ± 0.0009 mg/kg.

Laboratory 25 reported for trans-chlordane in Sample S1, rather than total chlordane. To avoid unfair scoring, this result was excluded from the robust average calculation as it would bias the assigned value; it was also excluded from the calculation of all summary statistics.

The ratios of the assigned values to spiked values for scored tests are presented in Table 36. Incurred values have not been included in the spiked values.

For all scored analytes, the assigned values were within the range of 58% to 94% of the spiked values (excluding Sample S2 glyphosate which had a significant incurred value not included in the spiked value). Similar ratios have been observed in previous NMIA pesticides in soil PT studies.[6] An assigned value was set only if there was a reasonable consensus of results.

Traceability: The consensus of participants' results is not traceable to any external reference, so although expressed in SI units, the metrological traceability of the assigned values has not been established.

Table 36 Ratio of Assigned Value to Spiked Value

Sample	Analyte	Assigned Value / Spiked Value (%)
S1	Total Chlordane	71
S1	Ethion	74
S1	Lindane	66
S1	Trifluralin	59
S2	Bromacil	65
S2	Glyphosate	265
S2	MCPA	94
S3	Total Chlordane	71
S3	Diazinon	58
S3	Dieldrin	77

Sample	Analyte	Assigned Value / Spiked Value (%)
S3	Endosulfan sulfate	71
S3	Ethion	91
S3	Lindane	59
S3	Simazine	93

*Calculated using the robust average or median value as applicable (assigned value not set).

6.2 Measurement Uncertainty Reported by Participants

Participants were asked to report an evaluation of the expanded MU associated with their results and the basis of this evaluation. It is a requirement of ISO/IEC 17025 that laboratories have procedures to evaluate the uncertainty of chemical measurements and to report this uncertainty in specific circumstances, including when the client's instruction so requires.[9]

Of 307 numeric results, 296 (96%) were reported with an associated expanded MU. Participants used a wide variety of procedures to evaluate their uncertainties (Table 4).

Laboratories 13 and 18 did not report uncertainties for some of their results, however both participants reported being accredited to ISO/IEC 17025.

The magnitude of the reported expanded uncertainties was within the range 2.6% to 240% of the reported value. In general, an expanded uncertainty of less than 15% may be unrealistically small for the routine measurement of pesticides, while over 50% may be too large to be fit-for-purpose. In this study, 257 (87%) relative uncertainties were between 15% and 50%; 8 relative uncertainties were less than 15%, and 31 relative uncertainties were greater than 50%.

The following participants may need to review the procedure they use to evaluate and report uncertainties, or review if an error was made when reporting their uncertainties:

- Laboratory 4 reported relative uncertainties of around 30% for all analytes, except for Sample S3 total chlordane where they reported a relative uncertainty of 92%.
- Laboratory 5 reported relative uncertainties ranging from 22% to 67% for all analytes, except for Sample S3 ethion where they reported a relative uncertainty of 100%.
- Laboratory 10 reported unrealistically small uncertainties for all analytes, ranging from 3% to 7%.
- Laboratory 15 reported relative uncertainties ranging from 27% to 50% for all analytes, except for Sample S3 ethion where they reported a relative uncertainty of 100%.
- Laboratory 22 reported a wide range of relative uncertainties, ranging from 5% to 240%.
- Laboratory 27 reported relative uncertainties ranging from 29% to 43% for all analytes, except for Sample S3 ethion where they reported a relative uncertainty of 100%.

Participants were also requested to report the coverage factor associated with their uncertainties (Table 4). All participants reporting their coverage factor reported k = 2.

Uncertainties associated with results returning an acceptable *z*-score but an unacceptable E_n-score may have been underestimated.

Laboratories 7, 16, 21, 23, and 25 attached evaluations of expanded MU for results reported as less than their limit of reporting (LOR). An evaluation of uncertainty expressed as a value cannot be attached to a result expressed as a range.[10]

In some cases, results were reported with an inappropriate number of significant figures. Including too many significant figures may inaccurately reflect the precision of measurements.

The recommended format is to write the uncertainty to no more than two significant figures, and then to write the result with the corresponding number of decimal places. For example, instead of 0.054 ± 0.01188 mg/kg, report this as 0.054 ± 0.012 mg/kg.[10]

6.3 z-Score

The *z*-score compares the participant's deviation from the assigned value based on the standard deviation for proficiency assessment (SDPA). The SDPA defines acceptable performance in a PT study. SDPAs equivalent to 15% and 20% were used to calculate *z*-scores in this study. The SDPAs were assigned with consideration of the challenges associated with analysing pesticides in soil at low concentrations for several analytes in Samples S1 and S2, as well as the introduction of the new Sample S3 compost matrix in this study. Unlike the standard deviation based on between-laboratory CV, setting the SDPA as a realistic value enables *z*-scores to be used as fixed reference value points for assessment of individual laboratory performance, independent of the group's performance.

The CVs predicted by the Thompson-Horwitz equation,[8] between-laboratory CVs in this study, and the SDPAs (as PCV), are presented for comparison in Table 37 (Sample S1 dieldrin and endosulfan sulfate have not been included as too few participants reported numeric results).

Table 37 Comparison of Between-Laboratory CV, Thompson-Horwitz CV, and SDPA

Sample	Analyte	Numeric Results Reported*	Assigned Value (mg/kg)	Between-Laboratory CV* (%)	Thompson-Horwitz CV (%)	SDPA (as PCV, %)
S1	Total Chlordane	16	0.071	28	22	20
S1	Chlorpyrifos	10	Not Set	48	22**	Not Set
S1	p,p'-DDD	7	Not Set	48	22**	Not Set
S1	Ethion	7	0.0625	16	22	20
S1	Fenitrothion	8	Not Set	40	22**	Not Set
S1	Fenvalerate	6	Not Set	31	22**	Not Set
S1	Lindane	18	0.0588	20	22	20
S1	Trifluralin	7	0.295	27	19	20
S2	Acephate	4	Not Set	8.8	17**	Not Set
S2	Bromacil	9	0.46	28	18	20
S2	Carbendazim	7	Not Set	93	19**	Not Set
S2	2,4-D	9	Not Set	22	22**	Not Set
S2	Fipronil	6	Not Set	35	17**	Not Set
S2	Glyphosate	7	3.98	11	13	15
S2	Imidacloprid	6	Not Set	53	18**	Not Set
S2	MCPA	9	0.189	11	21	20
S2	Metsulfuron-methyl	7	Not Set	39	13**	Not Set
S2	Omethoate	6	Not Set	65	19**	Not Set
S3	Acephate	3	Not Set	25	16**	Not Set
S3	Bifenthrin	8	Not Set	58	20**	Not Set
S3	Total Chlordane	21	1.43	17	15	20

Sample	Analyte	Numeric Results Reported*	Assigned Value (mg/kg)	Between-Laboratory CV* (%)	Thompson-Horwitz CV (%)	SDPA (as PCV, %)
S3	Diazinon	15	0.59	28	17	20
S3	Dieldrin	20	0.386	18	18	20
S3	Endosulfan sulfate	15	0.500	20	18	20
S3	Ethion	13	0.184	16	21	20
S3	Glyphosate	6	Not Set	54	15**	Not Set
S3	Imidacloprid	5	Not Set	56	17**	Not Set
S3	Lindane	20	0.536	25	18	20
S3	Simazine	8	0.537	17	17	20

*Values exclude outliers if applicable. Shaded cells represent between-laboratory CVs higher than both the SDPA and the Thompson-Horwitz CV for scored analytes. **Calculated using the robust average or median value as applicable (assigned value not set).

To account for possible low bias in consensus values due to participants using inefficient extraction or analytical techniques, a total of 12 *z*-scores were adjusted across the following analytes: Sample S1 ethion, lindane, trifluralin, Sample S2 bromacil, and Sample S3 total chlordane, diazinon, and lindane. A maximum acceptable result was set as the spiked value plus two SDPAs of the spiked value. Results lower than the maximum acceptable result but with a *z*-score greater than 2.0 had their *z*-score adjusted to 2.0. This ensured that participants reporting results close to the spiked value were not penalised. *z*-Scores for results higher than the maximum acceptable result and *z*-scores less than 2.0 were left unaltered.

Of 206 results for which *z*-scores were calculated, 186 (90%) returned $|z| \leq 2.0$, indicating an acceptable performance.

Laboratory 6 reported results for all 14 analytes for which *z*-scores were calculated.

Nineteen participants received acceptable *z*-scores for all analytes they reported results for: Laboratories 2 (12), 24 (12), 18 (11), 8 (9), 13 (9), 14 (9), 17 (8), 20 (8), 5 (7), 19 (7), 15 (6), 27 (6), 3 (5), 9 (4), 11 (4), 1 (3), 23 (2), 10 (1), and 21 (1).

The dispersal of participants' *z*-scores is presented graphically by laboratory in Figure 32 and by analyte in Figure 33.

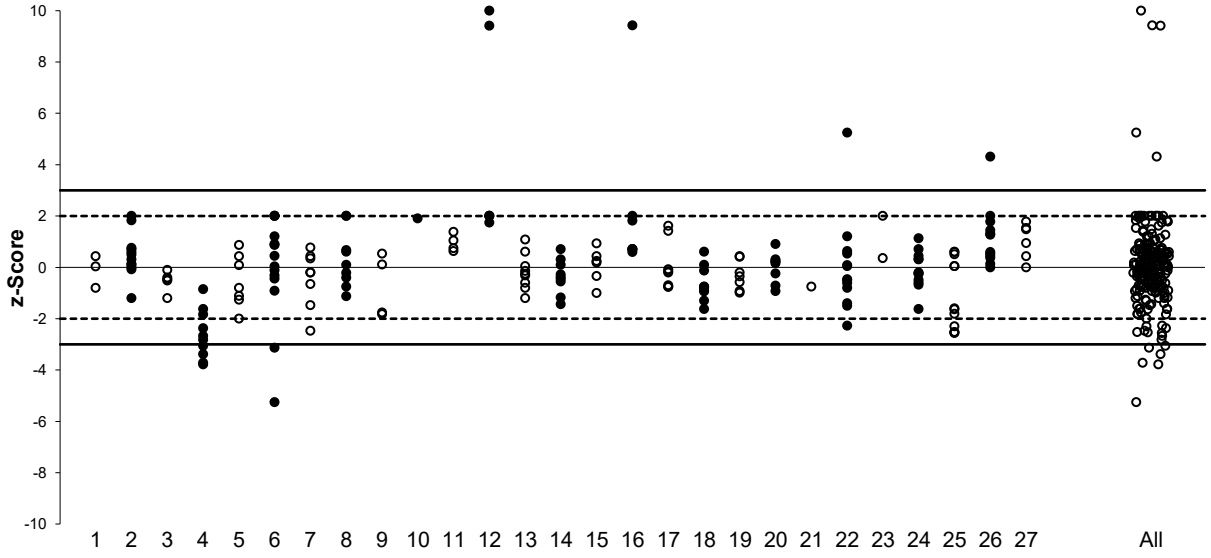


Figure 32 *z*-Score Dispersal by Laboratory

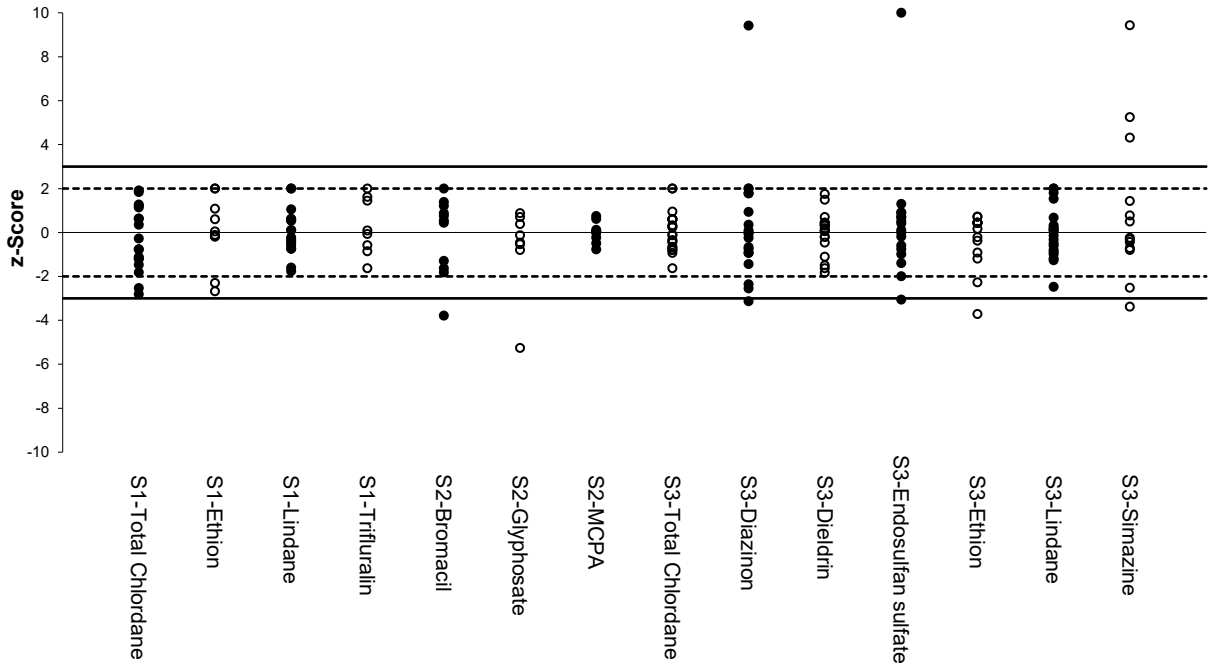


Figure 33 *z*-Score Dispersal by Analyte

6.4 E_n-Score

The E_n-score indicates how closely a result agrees with the assigned value taking into consideration the respective uncertainties. E_n-scores can be interpreted in conjunction with *z*-scores, as an unacceptable E_n-score can either be caused by issues with measurement, an inappropriate evaluation of measurement uncertainty, or both.

Where a laboratory did not report an uncertainty with a result, an expanded uncertainty of zero (0) was used to calculate the E_n-score. For results whose *z*-scores were adjusted as discussed in Section 6.3, no E_n-score has been reported.

Of 194 results for which E_n-scores were calculated, 159 (82%) were acceptable with $|E_n| < 1.0$, indicating agreement of the participant's result with the assigned value within their respective uncertainties.

Ten participants received acceptable E_n-scores for all analytes they reported results for: Laboratories 14 (9), 20 (8), 5 (7), 19 (7), 15 (6), 27 (6), 3 (5), 11 (4), 1 (3), and 21 (1).

Some participants had results where the *z*-score was adjusted as described above, and so E_n-scores were only calculated for some of their results. Of these participants, 2 participants received acceptable E_n-scores for all scored analytes that they reported results for: Laboratories 8 (7) and 23 (1).

The dispersal of participants' E_n-scores is presented graphically by laboratory in Figure 34.

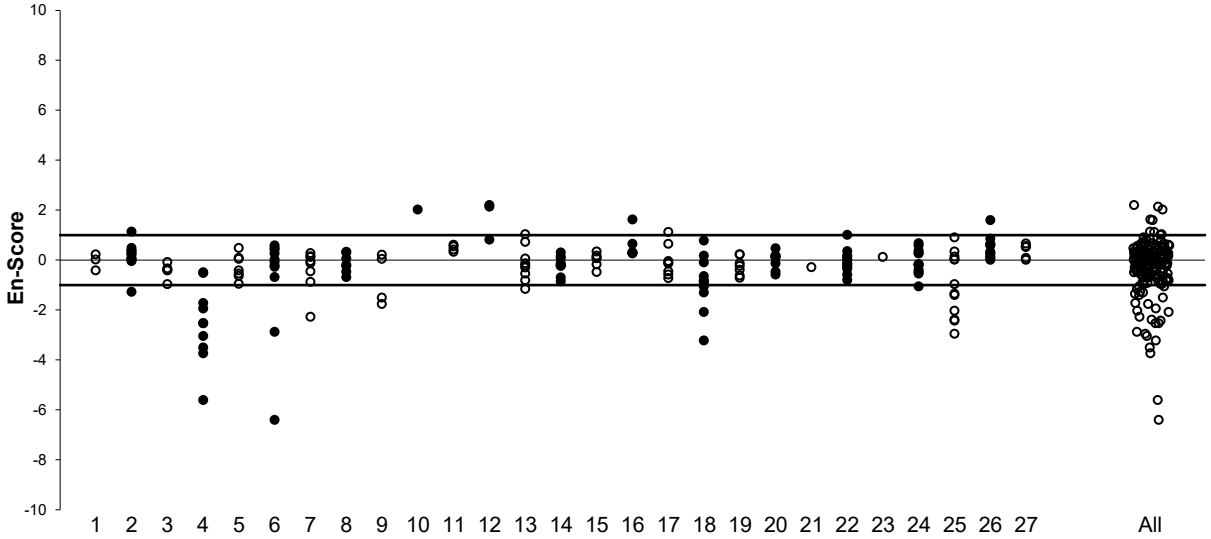


Figure 34 E_n-Score Dispersal by Laboratory

6.5 False Negatives

False negatives are presented in Table 38. These are analytes present in the samples which a participant tested for but did not report a numeric result; for example, participants reporting a 'less than' result (< *x*) when the assigned value was higher than their limit of reporting (LOR), or participants that did not report anything. For analytes where no assigned value was set, results have only been considered to be false negatives where the consensus value and spiked value were significantly higher than the participants' LOR (i.e. the consensus value minus the expanded uncertainty, and the spiked value minus the expanded uncertainty, were both greater than the LOR), or if no value was reported.

The analytes in Samples S1 and S2 were spiked at low concentrations. One of the purposes of this study was to provide laboratories with feedback on the suitability of their LORs for pesticides in soil, and their ability to detect and report low-level analytes without generating false negative results.

A large number of false negative results were reported in the present study, with these analytes having the largest number (4 each): fenvalerate in Sample S1; carbendazim and imidacloprid in Sample S2; and endosulfan sulfate in Sample S3.

Table 38 False Negatives

Lab. Code	Sample	Analyte	Assigned Value (mg/kg)	Consensus Value* (mg/kg)	Spiked Value (mg/kg)	Result** (mg/kg)
1	S2	Acephate	Not Set	0.727	1.00	<0.5
2	S1	Chlorpyrifos	Not Set	0.38	0.0539	NR
2	S1	Ethion	0.0625	N/A	0.0846	NR
2	S1	Fenitrothion	Not Set	0.063	0.0710	NR
2	S1	Fenvalerate	Not Set	0.098	0.152	NR
2	S2	Carbendazim	Not Set	0.35	0.804	NR
2	S2	Fipronil	Not Set	0.71	0.803	NR
2	S2	Imidacloprid	Not Set	0.44	0.604	NR
2	S3	Imidacloprid	Not Set	0.80	1.50	NR
3	S3	Endosulfan sulfate	0.500	N/A	0.706	<0.4

Lab. Code	Sample	Analyte	Assigned Value (mg/kg)	Consensus Value* (mg/kg)	Spiked Value (mg/kg)	Result** (mg/kg)
4	S1	Lindane	0.0588	N/A	0.0895	<0.01
4	S3	Lindane	0.536	N/A	0.909	<0.01
7	S1	Lindane	0.0588	N/A	0.0895	<0.05
7	S3	Endosulfan sulfate	0.500	N/A	0.706	<0.45
8	S2	Carbendazim	Not Set	0.35	0.804	NR
9	S1	Chlorpyrifos	Not Set	0.38	0.0539	NR
9	S1	Ethion	0.0625	N/A	0.0846	NR
9	S1	Fenitrothion	Not Set	0.063	0.0710	NR
9	S1	Fenvalerate	Not Set	0.098	0.152	NR
9	S1	Trifluralin	0.295	N/A	0.503	NR
9	S2	Acephate	Not Set	0.727	1.00	NR
9	S2	Carbendazim	Not Set	0.35	0.804	NR
9	S2	2,4-D	Not Set	0.104	0.351	NR
9	S2	Fipronil	Not Set	0.71	0.803	NR
9	S2	Glyphosate	3.98	N/A	1.50	NR
9	S2	Imidacloprid	Not Set	0.44	0.604	NR
9	S2	Metsulfuron-methyl	Not Set	3.8	2.50	NR
9	S2	Omethoate	Not Set	0.34	0.707	NR
10	S1	Ethion	0.0625	N/A	0.0846	NR
10	S1	Fenvalerate	Not Set	0.098	0.152	NR
10	S1	Lindane	0.0588	N/A	0.0895	NR
10	S1	p,p'-DDD	Not Set	0.113	0.0152	NR
10	S1	Trifluralin	0.295	N/A	0.503	NR
13	S3	Ethion	0.184	N/A	0.202	<0.05
17	S3	Endosulfan sulfate	0.500	N/A	0.706	<0.48
18	S1	Fenitrothion	Not Set	0.063	0.0710	NR
18	S1	Fenvalerate	Not Set	0.098	0.152	NR
18	S1	Trifluralin	0.295	N/A	0.503	NR
18	S2	Acephate	Not Set	0.727	1.00	NR
18	S2	Carbendazim	Not Set	0.35	0.804	NR
18	S2	Fipronil	Not Set	0.71	0.803	NR
18	S2	Imidacloprid	Not Set	0.44	0.604	NR
18	S3	Acephate	Not Set	0.90	1.51	NR
18	S3	Endosulfan sulfate	0.500	N/A	0.706	<0.25
18	S3	Imidacloprid	Not Set	0.80	1.50	NR
26	S2	Imidacloprid	Not Set	0.44	0.604	<0.01

*Robust average or median value as applicable (assigned value not set). **Results reported as NR may or may not be false negatives, depending on the participant's actual LOR.

6.6 Reporting of Additional Analytes

Additional analytes reported by participants are presented in Table 39. These were analytes not spiked into the sample by the study coordinator and not identified as incurred analytes.

Table 39 Additional Analytes Reported by Participants

Lab. Code	Sample	Analyte	Result (mg/kg)	Uncertainty (mg/kg)	Recovery (%)
6	S3	Aldrin	0.01	0.003	30
6	S3	Chlorpyrifos	0.002	0.0006	85
6	S3	MCPA	0.017	0.0051	96.5
14	S2	AMPA	0.12	0.05	51
21	S1	p,p'-DDT	0.09	0.03	NT
22	S2	AMPA	0.10	0.05	93
22	S3	AMPA	0.025	0.013	93
22	S3	MCPA	0.008	0.006	66
25	S3	MCPA	0.03	NR	NR
26	S3	MCPA	0.013	0.004	85
26	S3	Propiconazole	0.012	0.048	NR
26	S3	Trichlopyr	0.008	0.003	NR

Participants were asked to report results for p,p'-DDD, in Sample S1. Several participants also reported a total DDT value, which was not scored. These results are presented in Table 40 for information only.

Table 40 Reported Results for Sample S1 Total DDT

Lab. Code	Result (mg/kg)	Uncertainty (mg/kg)	Recovery (%)
2	0.018	0.0035	NR
22	0.010	0.007	64
26	0.017	0.006	NR

6.7 Summary of Participants' Results and Performances

Summaries of participants' results and performances in this PT study are presented in Tables 41 to 43, and Figure 35.

Laboratory 4 reported consistently low-biased results and was therefore excluded from the discussion of analytical methods in Section 6.8, except when examining the effect of extraction time.

Carbendazim, omethoate, and imidacloprid were the tests that most challenged participants, with high variability between participants' results, and low recovery of the spiked analyte.

Participants were also challenged more generally by the analytes spiked at low levels close to their limits of reporting (LOR). Participants may need to review whether their LORs are appropriate so as to not produce false negative results.

Participants were requested to analyse the samples using their routine test method and to report a single result as they would to a client; that is, corrected for recovery or not, according to their standard procedure. Results reported in this way reflect the true variability of results reported by laboratories to clients. Laboratories 1, 4, 6, 8, 12, 14, 15, 16, 17, 22, and 26. reported recoveries for at least one analyte considered in this study, and the recoveries reported were in the range of 41% to 170%. Laboratory 16 reported that they corrected results for recovery.

Table 41 Summary of Participants' Sample S1 Results*

Lab. Code	Total Chlordane	Chlorpyrifos	p,p'-DDD	Dieldrin	Endosulfan sulfate	Ethion	Fenitrothion	Fenvalerate	Lindane	Trifluralin
AV	0.071	Not Set	Not Set	Not Set	Not Set	0.0625	Not Set	Not Set	0.0588	0.295
1	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
2	0.097	NR	0.018	NR	NR	NR	NR	NR	0.087	0.29
3	0.054	<0.5	<0.05	<0.05	<0.05	<0.5	NT	<0.5	0.054	NT
4	0.031	0.021	<0.01	<0.01	<0.01	0.029	<0.05	0.049	<0.01	0.244
5	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	NT	<0.1	<0.5
6	0.088	0.039	0.0064	0.007	0.003	0.061	0.054	0.12	0.055	0.51
7	0.05	<0.05	<0.05	<0.05	<0.05	0.06	NT	NT	<0.05	NT
8	0.055	0.065	0.010	NR	NR	0.098	0.082	0.099	0.066	NT
9	0.0450	NR	0.0055	<0.001	NR	NR	NR	NR	0.0380	NR
10	0.098	0.014	NR	NR	NR	NR	0.046	NR	NR	NR
11	0.080	<0.050	0.013	<0.050	<0.010	NT	NT	0.096	0.071	NT
12	<0.1	<0.1	<0.1	<0.1	<0.1	0.1	0.1	NT	<0.1	NT
13	0.054	<0.05	<0.05	<0.05	<0.05	0.076	NT	NT	0.066	NT
14	0.067	<0.2	<0.05	<0.05	<0.05	<0.2	<0.2	NT	0.06	NT
15	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	NT	<0.1	<0.5
16	<0.2	<0.1	<0.1	<0.1	<0.1	<0.2	<0.2	NT	<0.1	<0.5
17	0.06	<0.05	<0.05	<0.05	<0.05	0.06	0.08	NT	0.05	0.39
18	0.06	0.05	<0.05	<0.05	<0.05	0.07	NR	NR	0.06	NR
19	< 0.1	< 0.2	< 0.05	< 0.05	< 0.05	< 0.2	< 0.2	NT	0.052	NT
20	< 0.1	< 0.2	< 0.05	< 0.05	< 0.05	< 0.2	< 0.2	< 2	0.056	< 0.5
21	<0.1	<0.1	<0.1	<0.1	<0.1	NT	<0.1	NT	0.05	NT
22	0.080	0.046	0.010	0.008	< 0.008	0.063	0.050	NT	0.065	0.30
23	0.076	0.046	<0.02	<0.02	<0.04	<0.1	0.06	0.13	0.085	NT
24	0.087	0.022	< 0.05	< 0.05	< 0.05	< 0.2	< 0.2	< 0.4	0.053	0.26
25	0.035	0.0278	<0.010	<0.010	<0.010	0.0338	0.0334	0.092	0.04	0.198
26	0.089	0.05	0.0165	<0.01	<0.01	NT	NT	NT	0.065	0.38
27	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	NT	<0.1	<0.5

*All results are given in mg/kg. Shaded cells are results which returned a questionable or not acceptable *z*-score for scored analytes. AV = Assigned Value; NT = Not Tested; NR = Not Reported; NS = Not Supplied.

Table 42 Summary of Participants' Sample S2 Results*

Lab. Code	Acephate	Bromacil	Carbendazim	2,4-D	Fipronil	Glyphosate	Imidacloprid	MCPA	Metsulfuron-methyl	Omethoate
AV	Not Set	0.46	Not Set	Not Set	Not Set	3.98	Not Set	0.189	Not Set	Not Set
1	<0.5	0.5	0.2	0.09	0.67	3.5	0.3	0.19	3	<0.5
2	0.74	0.53	NR	NT	NR	4.4	NR	NT	4.6	0.51
3	NT	NT	NT	0.12	NT	NT	<0.5	<0.5	NT	NT
4	NT	0.111	0.069	NT	0.214	NT	0.09	NT	NT	NT
5	NT	NT	0.8	<0.5	0.9	4.5	0.63	<0.5	4	NT
6	0.54	0.54	0.25	0.1	0.66	0.84	0.52	0.19	6.2	0.36
7	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
8	0.800	NT	NR	NT	NT	NT	NT	NT	NT	0.476
9	NR	0.509	NR	NR	NR	NR	NR	0.193	NR	NR
10	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
11	0.714	0.587	0.419	0.129	NT	NT	0.491	0.217	NT	0.511
12	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
13	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
14	NT	NT	NT	NT	NT	3.65	NT	NT	NT	< 2
15	NT	<2	NT	<0.5	NT	NT	NT	<0.5	NT	NT
16	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
17	NT	NT	NT	NT	0.72	NT	NT	NT	NT	NT
18	NR	0.34	NR	0.09	NR	3.9	NR	0.16	NT	0.149
19	NT	NT	NT	NT	NT	NT	NT	NT	NT	< 2
20	NT	NT	NT	NT	NT	NT	NT	< 0.5	1.8	< 2
21	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
22	NT	0.57	NT	0.15	NT	3.7	0.58	0.17	3.6	NT
23	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
24	NT	0.31	0.06	0.09	0.95	NT	NT	0.18	NT	< 2
25	NT	0.293	NT	0.101	NT	NT	NT	0.212	NT	0.055
26	NT	0.86	0.67	0.082	NT	4.2	<0.01	0.189	3.82	NT
27	NT	<2	NT	<0.5	NT	NT	NT	<0.5	NT	NT

*All results are given in mg/kg. Shaded cells are results which returned a questionable or not acceptable *z*-score for scored analytes. AV = Assigned Value; NT = Not Tested; NR = Not Reported; NS = Not Supplied.

Table 43 Summary of Participants' Sample S3 Results*

Lab. Code	Acephate	Bifenthrin	Total Chlordane	Diazinon	Dieldrin	Endosulfan sulfate	Ethion	Glyphosate	Imidacloprid	Lindane	Simazine
AV	Not Set	Not Set	1.43	0.59	0.386	0.500	0.184	Not Set	Not Set	0.536	0.537
1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
2	1.3	0.11	1.6	0.59	0.41	0.51	0.14	2.1	NR	0.55	0.59
3	NT	<2	1.4	<0.7	0.35	<0.4	<0.5	NT	<1	0.48	<1
4	NT	0.082	0.964	0.31	0.244	0.194	0.047	NT	0.178	<0.01	0.174
5	NT	<3	1.2	0.6	0.3	0.3	0.2	<1	1.1	0.4	<1
6	0.75	0.39	2.11	0.22	0.42	0.59	0.15	0.44	0.74	0.78	0.49
7	NT	NT	1.24	0.63	0.37	<0.45	0.2	NT	NT	0.27	0.62
8	0.903	NR	1.317	0.922	0.394	0.424	0.176	NT	NT	0.608	NT
9	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
10	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
11	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
12	NT	NT	2.1	1.7	0.52	1.5	<0.5	NT	NT	0.8	NT
13	NT	<0.05	1.39	0.56	0.39	0.44	<0.05	NT	NT	0.45	0.503
14	NT	0.23	1.3	0.42	0.41	0.57	0.17	1.4	NT	0.41	NT
15	NT	<1	1.5	0.7	0.4	0.4	0.2	NT	NT	0.5	<1
16	NT	<0.5	1.6	0.84	0.44	0.57	0.21	NT	NT	0.73	1.55
17	NT	<1.52	1.23	0.58	<0.48	<0.48	<0.48	NT	NT	0.52	0.69
18	NR	NR	1.16	0.5	0.26	<0.25	<0.25	1	NR	0.44	0.45
19	NT	0.25	1.33	0.48	0.37	0.54	0.2	NT	NT	0.43	NT
20	NT	0.21	1.5	0.48	0.41	0.59	0.19	NT	NT	0.56	0.46
21	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
22	NT	0.081	1.2	NT	0.27	0.36	0.10	2.4	1.1	0.47	1.1
23	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
24	NT	0.25	1.52	0.51	0.42	0.48	0.21	NT	NT	0.57	0.51
25	NT	<0.3	1.6	0.288	0.39	0.55	<0.6	NT	NT	0.54	0.265
26	NT	NT	1.6	0.8	0.42	0.63	NT	1.6	0.8	0.55	1
27	NT	<1	1.7	0.8	0.5	0.5	0.2	NT	NT	0.7	<1

* All results are given in mg/kg. Shaded cells are results which returned a questionable or not acceptable *z*-score for scored analytes. AV = Assigned Value; NT = Not Tested; NR = Not Reported; NS = Not Supplied.

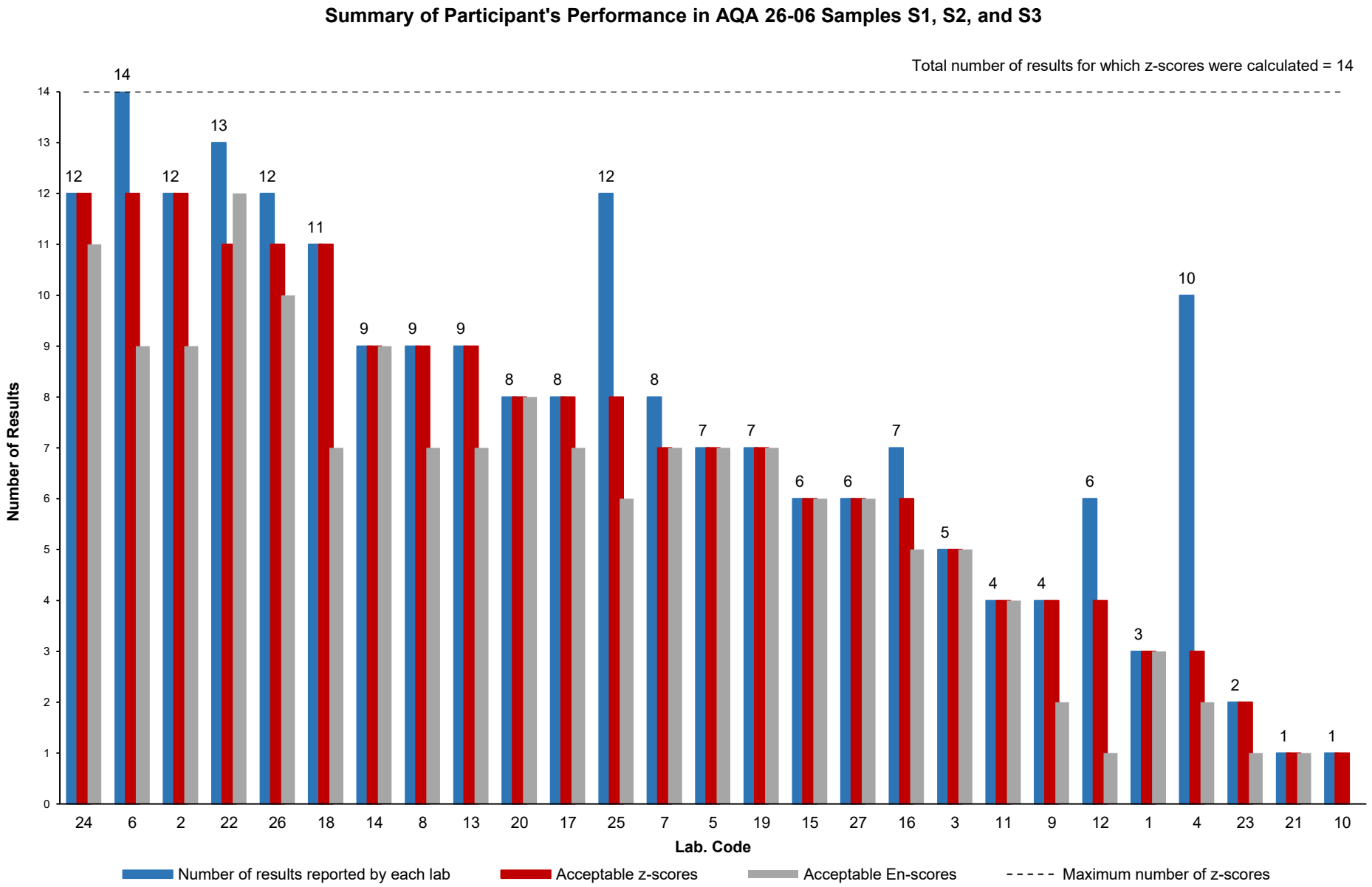


Figure 35 Summary of Participants' Performance

6.8 Participants' Analytical Methods

Samples S1 and S2 were soil samples, while Sample S3 was a compost sample. The soil samples were fortified with pesticides at concentrations near typical laboratory reporting limits. One of the purposes of this study was to provide participants with feedback on the suitability of their established reporting limits and their ability to accurately detect and quantify pesticides at low concentrations.

A variety of analytical methods were used for the determination of the different analytes in these samples (Appendix 4).

Most participants who analysed the soil and compost samples applied the same analytical methodology across both matrices. Some participants had a few adjustments between their methods, for example, used different sample mass, instrument detector and/or internal standard. Laboratory 25 was the only participant that reported using different methods across the soil and compost matrices, for some analytes only.

Extraction times reported by participants ranged from one minute up to 4 hours (Figure 36). Laboratory 4 reported an extraction time of only one minute. Their results were consistently biased low for both the soil and compost samples. The short extraction time employed may have resulted in low extraction efficiency and this may explain their low biased results. For the remainder of this section, results from Laboratory 4 have not been included in the charts so as to not attribute their biased results to other methodology parameters.

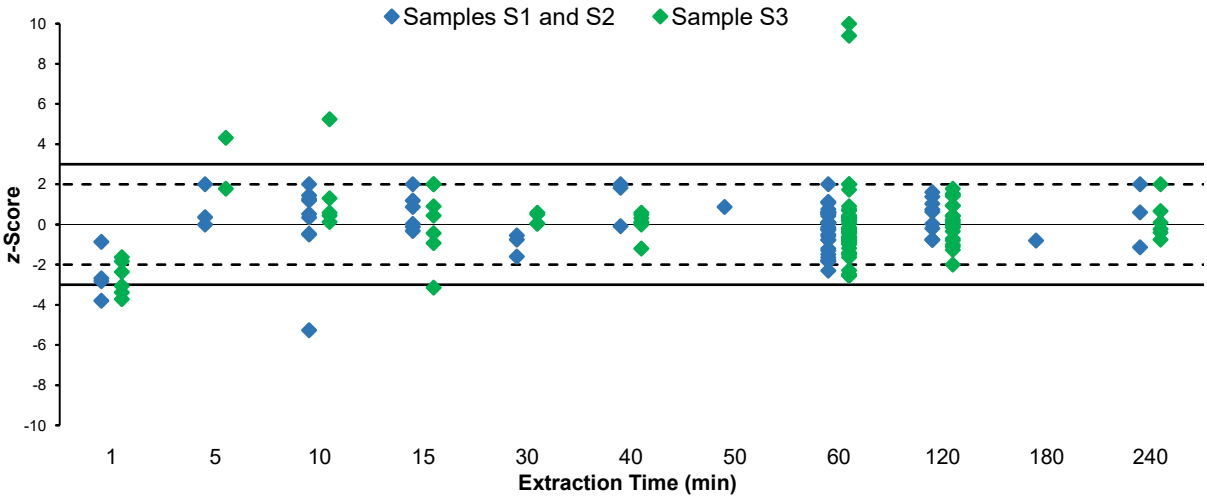


Figure 36 z-Scores vs Extraction Time

Participants reported using a wide range of sample sizes from 0.5 g to 20 g per analysis) with 10 g being most commonly used (Figure 37). Caution should be exercised when a small sample size is taken for analysis as this may not be representative of the whole sample. PT samples are prepared to be sufficiently homogeneous, and so the homogeneity of these PT samples may not reflect the homogeneity of routine samples encountered by laboratories. Participants using larger sample masses should also verify that their extraction methodology achieves adequate extraction efficiency.

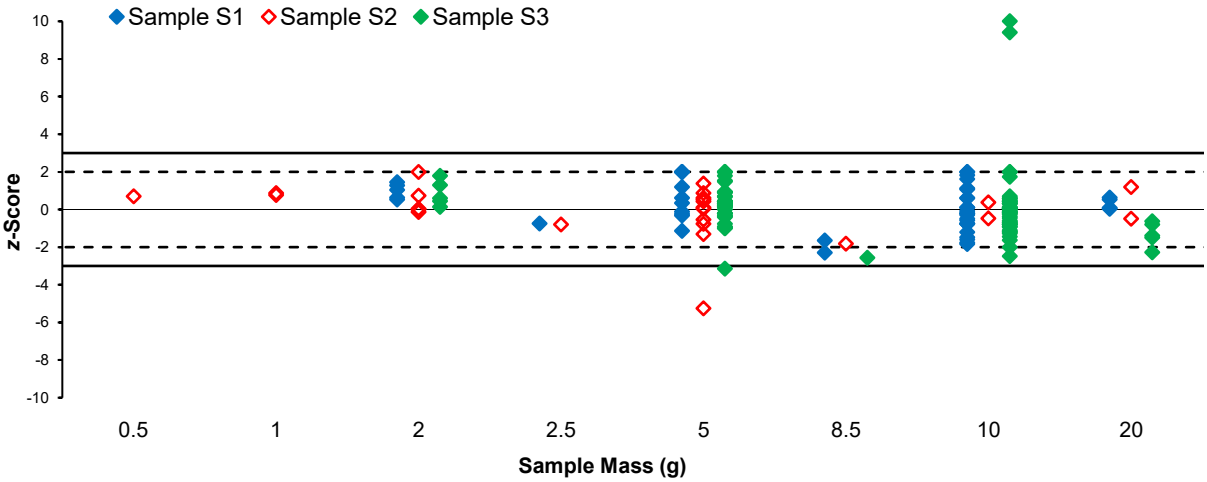


Figure 37 *z*-Scores vs Sample Mass Used for Analysis

Participants employed a range of extraction techniques, including solid-liquid extraction (SLE) with mechanical agitation (MA), sonication (S), and accelerated solvent extraction (ASE). Extraction solvents used included acetone (ACE), acetonitrile (ACN), dichloromethane (DCM), ethyl acetate (EtOAc), hexane (HEX), methanol (MeOH), water, and combinations thereof. Some participants also applied clean-up procedures prior to analysis, such as QuEChERS, Florisil, silica, and solid-phase extraction (SPE).

The most common methodology used by participants in this study involved SLE (MA), using DCM/ACE as the extraction solvent, with no additional clean-up step.

The analytical detection method of choice varied depending on the analyte, including gas chromatography (GC) coupled with mass spectrometry (MS), tandem mass spectrometry (MS/MS), electron capture detection (ECD), or flame photometric detection (FPD), and liquid chromatography (LC) coupled with MS, MS/MS, or UV.

Individual Analyte Commentary

Total Chlordane Sample S1 was spiked with both cis-chlordane and trans-chlordane in a ratio of approximately 1:5, and participants were requested to report for total chlordane present. Laboratory 25 was biased low, however they noted that they only reported for trans-chlordane. Otherwise, different methodologies generally produced compatible results (Figure 38).

Sample S3 was also spiked with both cis-chlordane and trans-chlordane in a ratio of approximately 1:3. All participants reported for total chlordane for this sample, and results from different methodologies were compatible with each other (Figure 39).

There was better agreement between participants' results for chlordane in Sample S3 compost as compared to Sample S1 soil, however, this is likely due to the higher levels of chlordane in Sample S3, rather than matrix effects. Participants' recoveries of the spiked analyte were similar across both samples.

p,p'-DDD No assigned value could be set for this analyte in Sample S1 as the results reported by participants were too varied (Figure 40).

Participants who employed SLE (ASE) or SLE (S) generally reported results closer to the spiked value, which may reflect higher extraction efficiency when these techniques are employed as compared to SLE (MA) only.

The use of GC-ECD with no clean-up step may also have led to an overestimate of the analyte due to matrix interference.

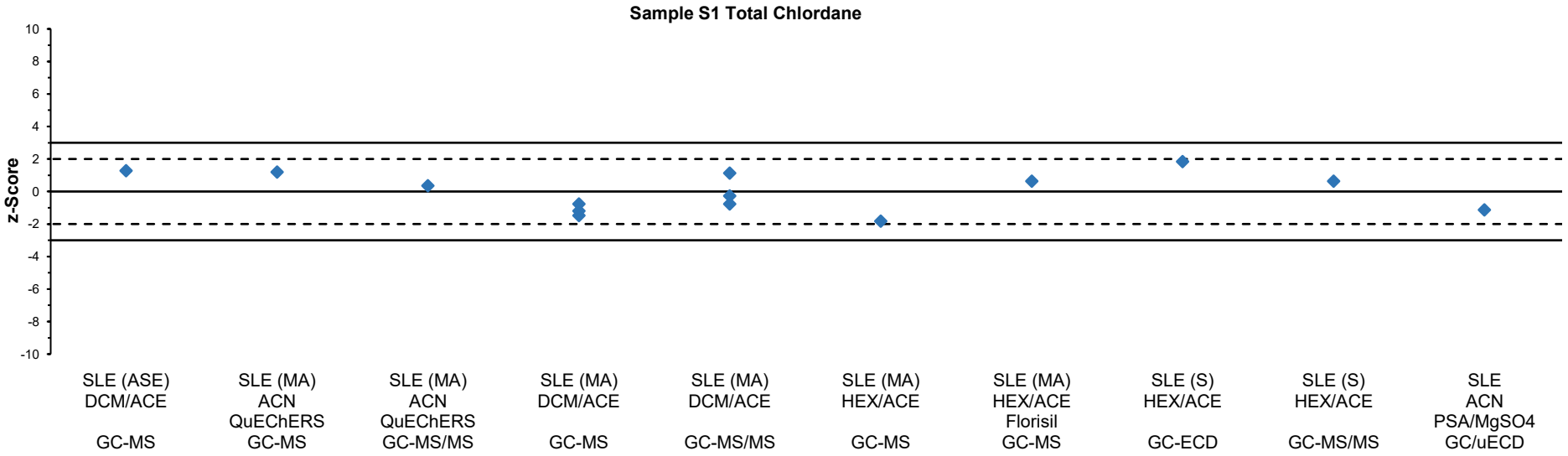


Figure 38 Sample S1 Total Chlordane *z*-Scores vs Methodology

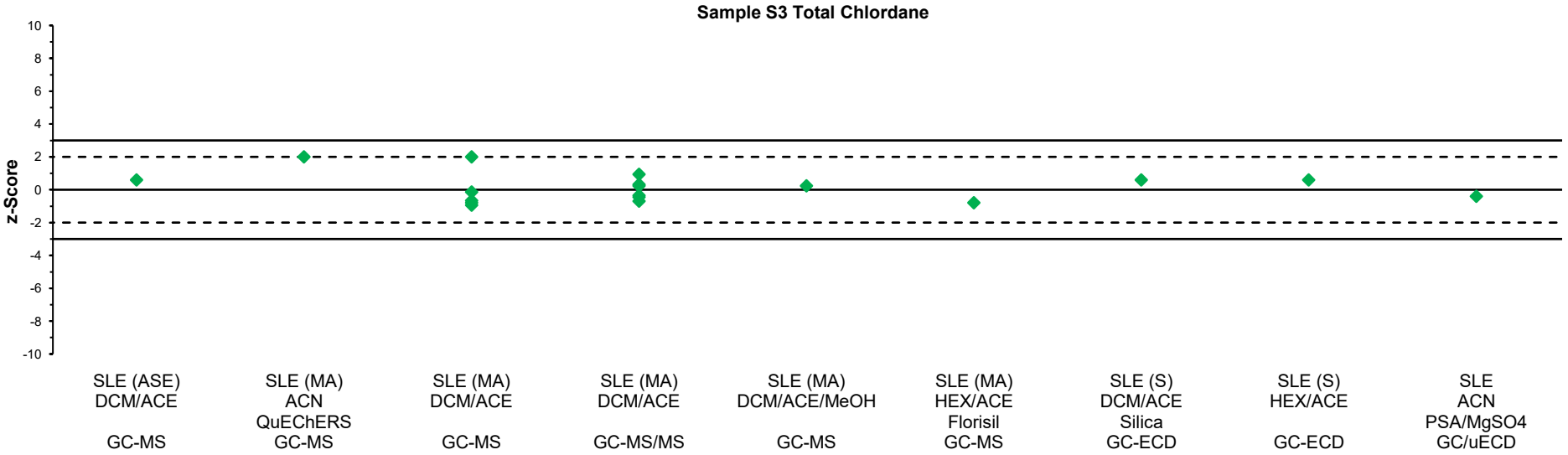


Figure 39 Sample S3 Total Chlordane *z*-Scores vs Methodology

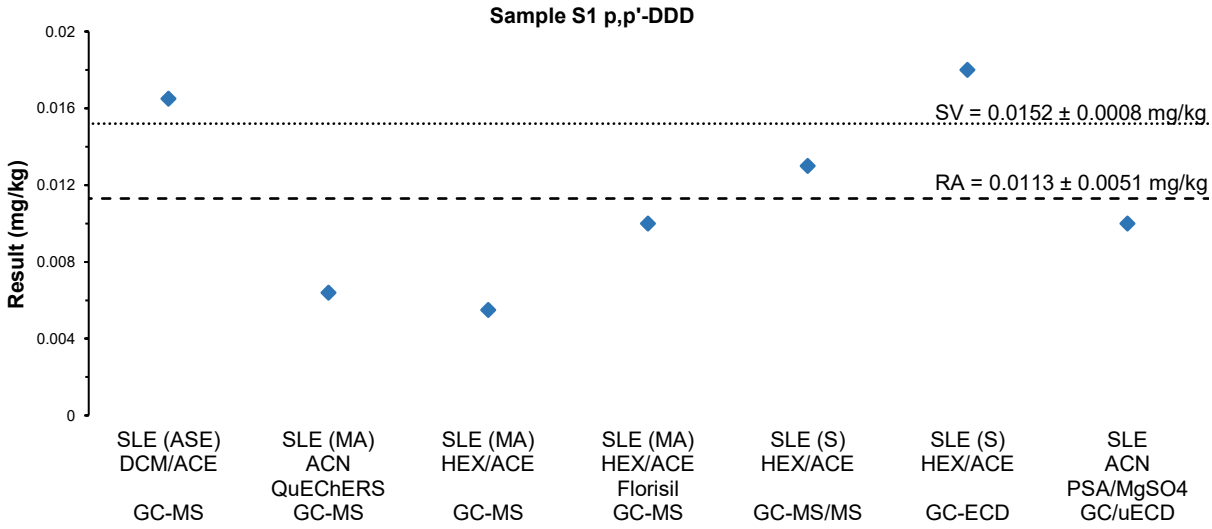


Figure 40 Sample S1 p,p'-DDD Results vs Methodology

Dieldrin was spiked at 0.00617 mg/kg in Sample S1 and 0.502 mg/kg in Sample S3. The low concentration of dieldrin in Sample S1 challenged participants' analytical capabilities. Most participants reported that this analyte was below their reporting limit, and only two participants reported quantitative results. These results were in good agreement with both each other and the spiked concentration.

By comparison, all results reported for Sample S3 achieved acceptable z-scores, regardless of the analytical methodology used (Figure 41).

Endosulfan sulfate in Sample S1 was spiked at 0.00199 mg/kg, and most laboratories reported this analyte as being below their reporting limit. Laboratory 6 reported a result of 0.003 mg/kg, which was in reasonable agreement with the spiked value.

Sample S3 was spiked with endosulfan sulfate at 0.706 mg/kg, and of the 17 numeric results reported, 15 returned an acceptable *z*-score (Figure 42). Several participants reported false negatives for this analyte, with reported limits of reporting (LORs) ranging from 0.25 mg/kg to 0.48 mg/kg; these laboratories should review the suitability of their LORs for this analyte.

Lindane concentration was 0.0588 mg/kg in the soil Sample S1, and 0.536 mg/kg in the compost Sample S3. Despite the difference in concentration and matrix, participants reported results that were in excellent agreement with each other, irrespective of the analytical methodology used (Figure 43 and Figure 44 respectively). Two participants reported false negatives for this analyte across the two samples; these laboratories should review the suitability of their LORs for this analyte.

Acephate This analyte is not commonly included in the analytical scope of many laboratories. Nevertheless, the 4 participants that reported results for this analyte in soil Sample S2 demonstrated good analytical capability, with results in good agreement with both each other and the assigned value. The methodologies used are presented in Figure 45. Of these 4 laboratories, 3 also reported results for Sample S3 (Figure 46).

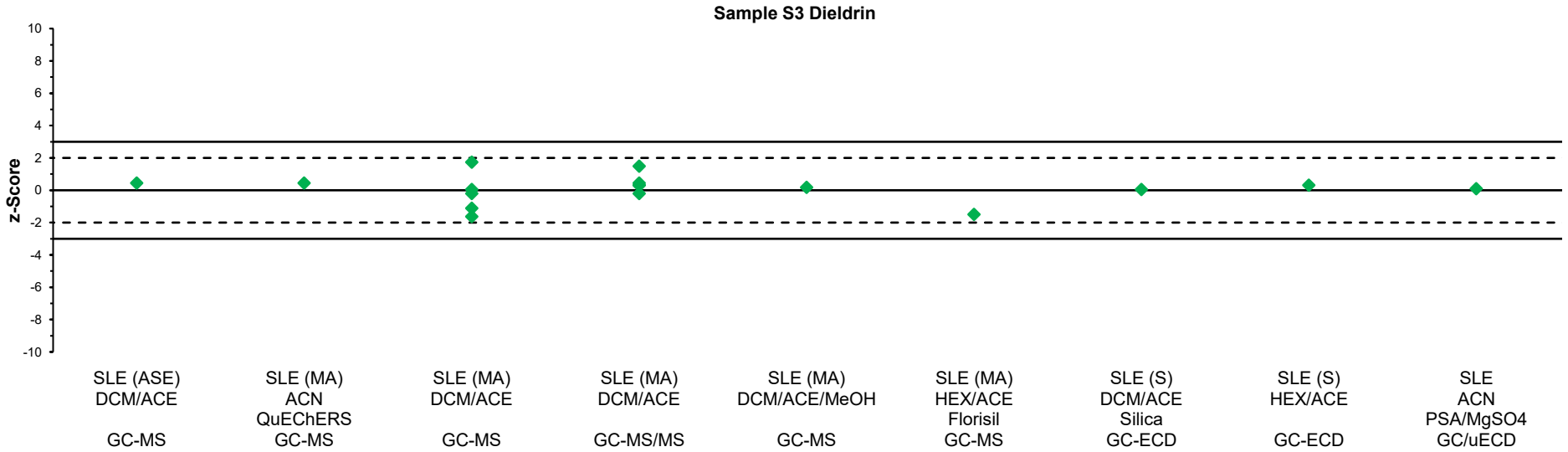


Figure 41 Sample S3 Dieldrin *z*-Scores vs Methodology

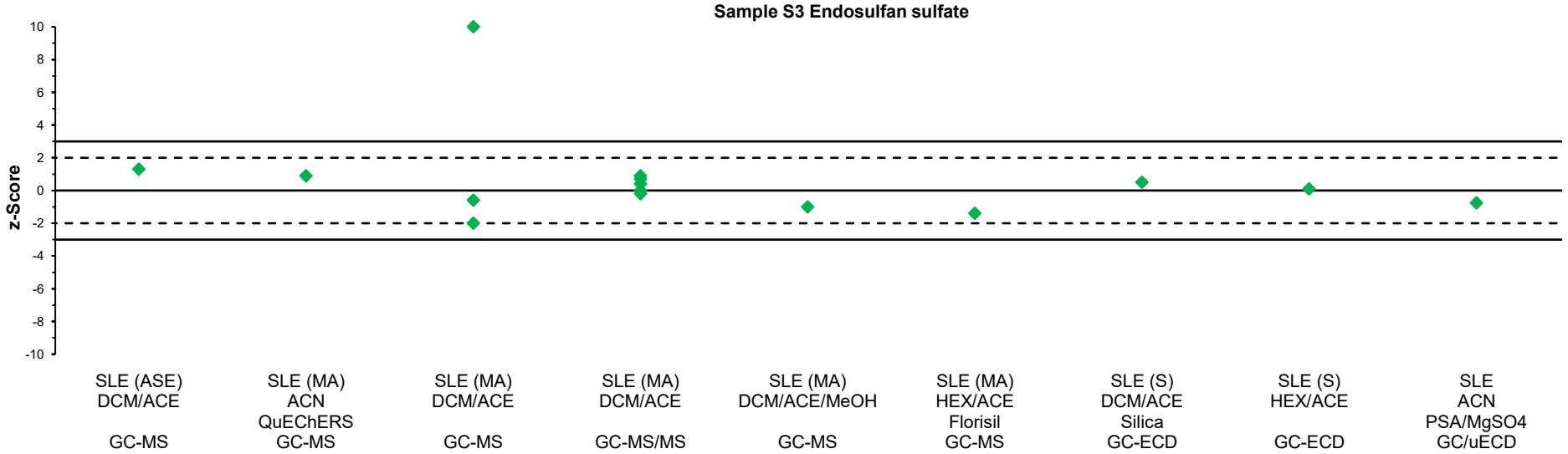


Figure 42 Sample S3 Endosulfan Sulfate *z*-Scores vs Methodology

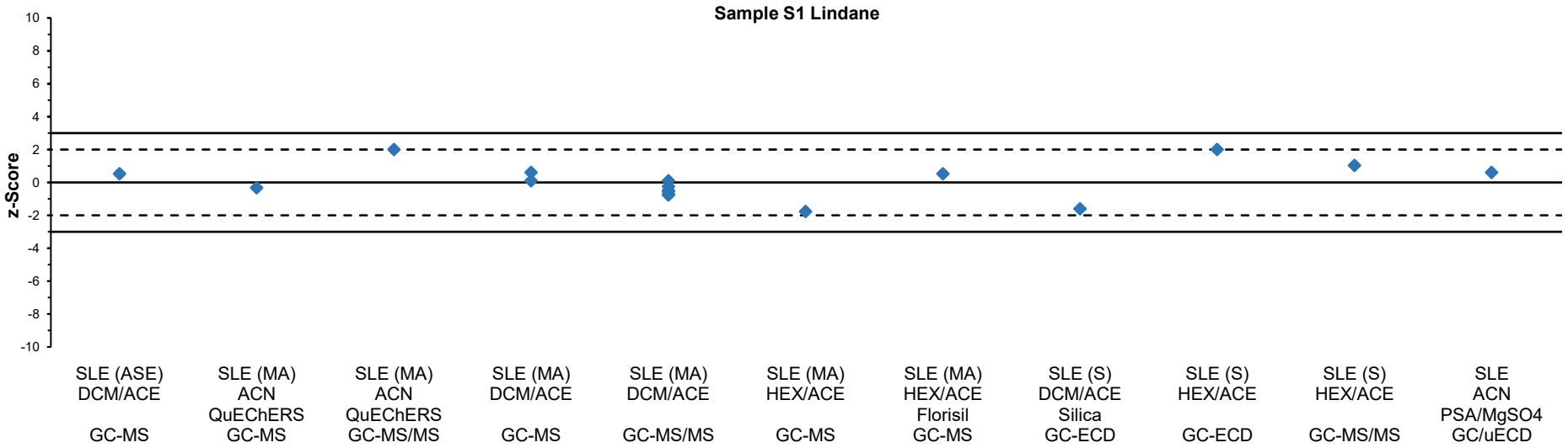


Figure 43 Sample S1 Lindane *z*-Scores vs Methodology

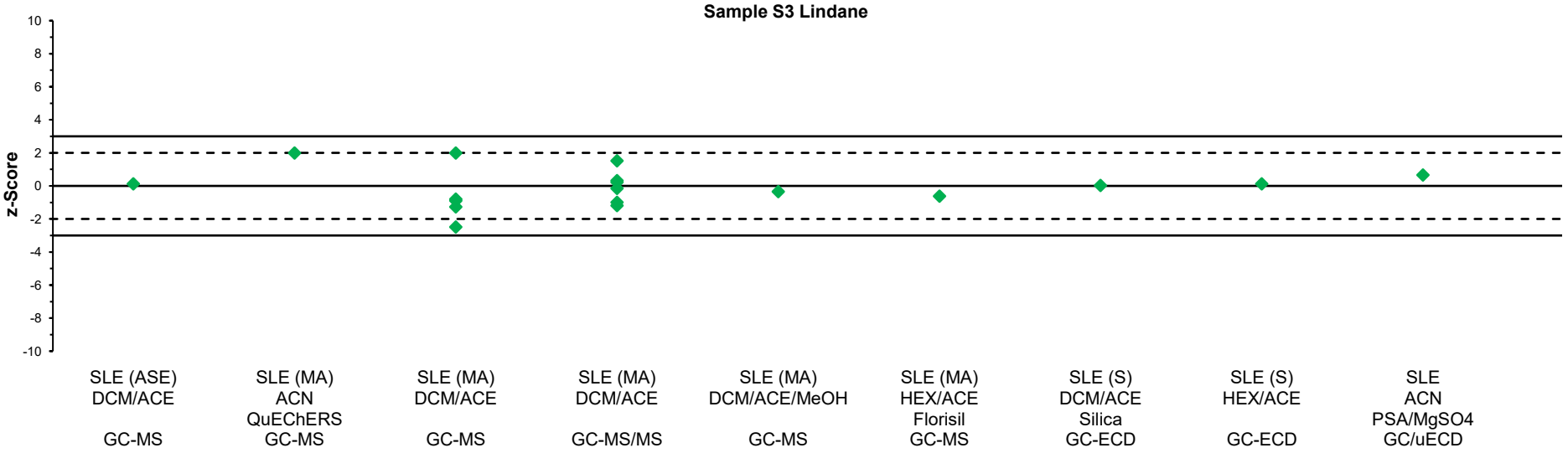


Figure 44 Sample S3 Lindane *z*-Scores vs Methodology

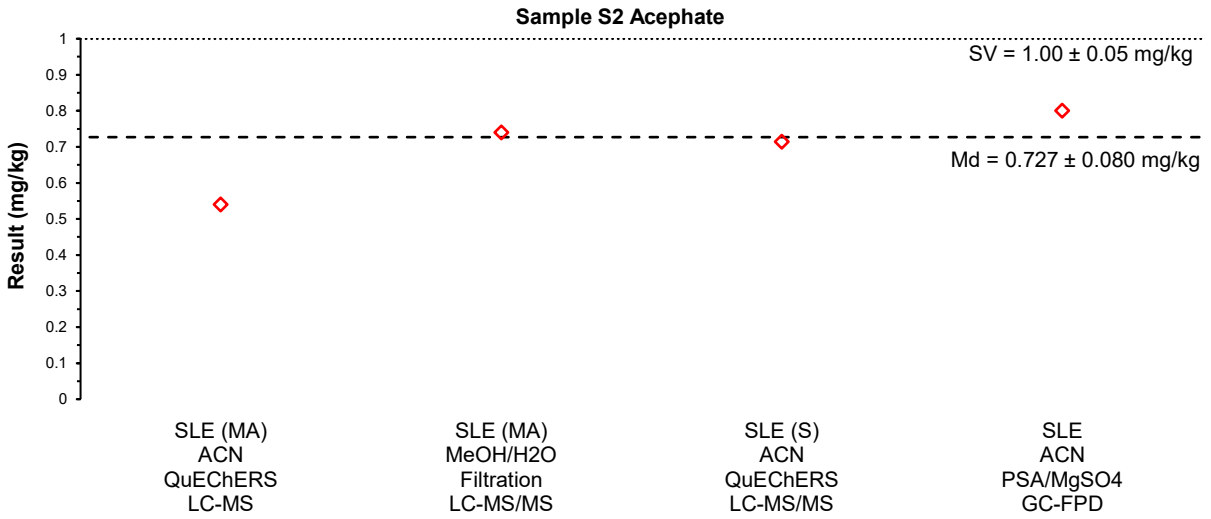


Figure 45 Sample S2 Acephate Results vs Methodology

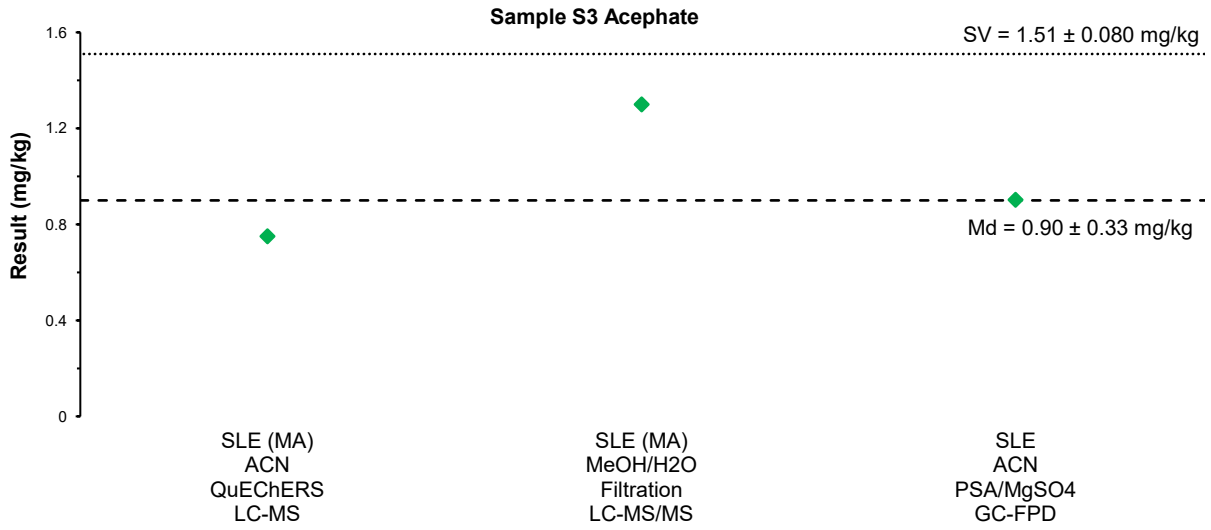


Figure 46 Sample S3 Acephate Results vs Methodology

Chlorpyrifos Results for Sample S1 chlorpyrifos were varied and no assigned value could be set (Figure 47).

Laboratory 25 reported using ethyl acetate (EtOAc) as their extraction solvent and was biased low. More generally, this participant used EtOAc as their extraction solvent for several organophosphate pesticides (diazinon, ethion, fenitrothion, omethoate), and those results were also biased low. Stability issues have been reported for organophosphate pesticides in ethyl acetate.[11] This participant may wish to review their use of EtOAc as an extraction solvent for this class of compounds.

The results reported by Laboratory 8 were biased high. The laboratory reported using a PSA/$MgSO_4$ clean-up step followed by GC-FPD determination. Caution should be exercised when GC-FPD is used in conjunction with PSA/$MgSO_4$ clean-up, as sulfur- and/or phosphorus-containing compounds may still be present in the extract and contribute to the overestimation of chlorpyrifos.

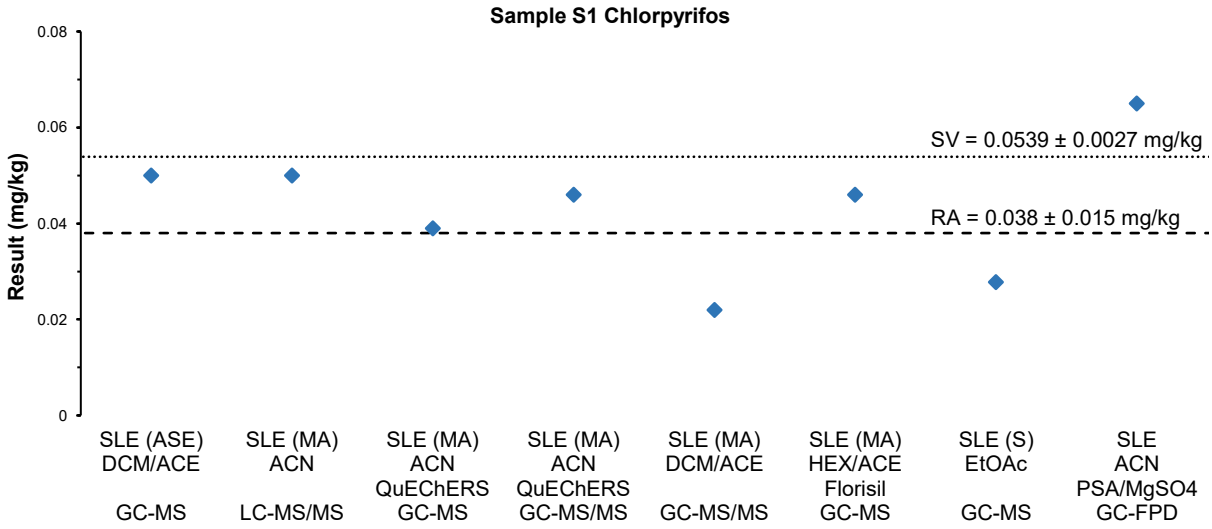


Figure 47 Sample S1 Chlorpyrifos Results vs Methodology

Diazinon Most results for Sample S3 diazinon were compatible with each other regardless of the method used (Figure 48).

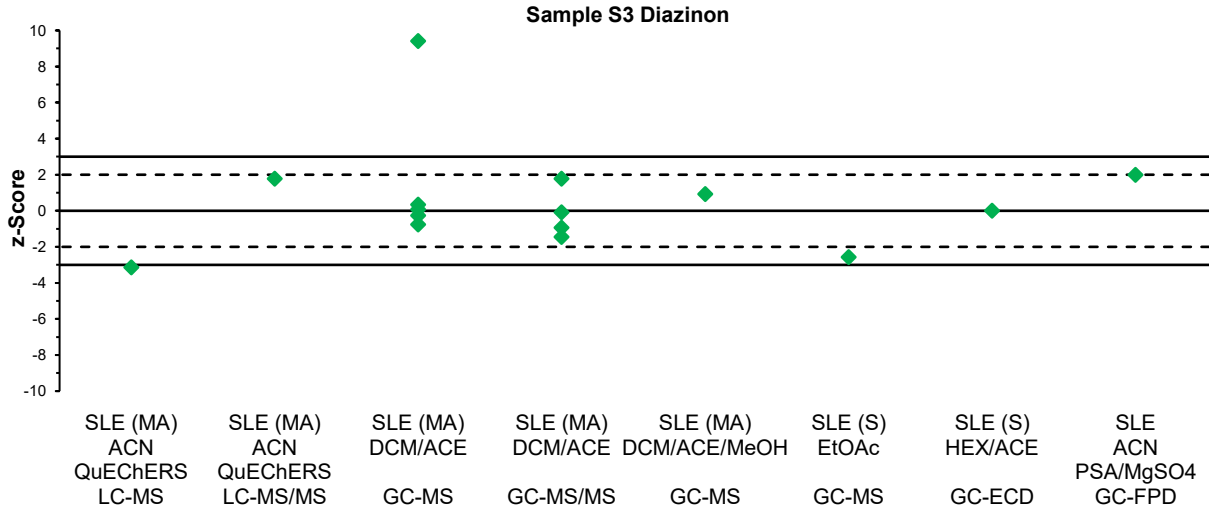


Figure 48 Sample S3 Diazinon *z*-Scores vs Methodology

Ethion measurements in Samples S1 and S3 did not challenge participants analytical techniques regardless of the matrix or analyte level. Most reported results returned acceptable *z*-scores (Figure 49 and Figure 50).

Fenitrothion Only eight laboratories reported numeric results for fenitrothion in Sample S1. The reported results showed considerable variability, and an assigned value could not be established (Figure 51). Fenitrothion was also associated with a high number of false negative results in this study, suggesting that participating laboratories should review the suitability of their limits of reporting for this analyte.

Omethoate is not routinely included in the analytical suite of many participating laboratories, with only 6 laboratories reporting numeric results. The reported results showed poor agreement between laboratories, and overall recoveries were generally low relative to the spiked concentration (Figure 52). These findings suggest that the determination of omethoate remains challenging and may require further method optimisation.

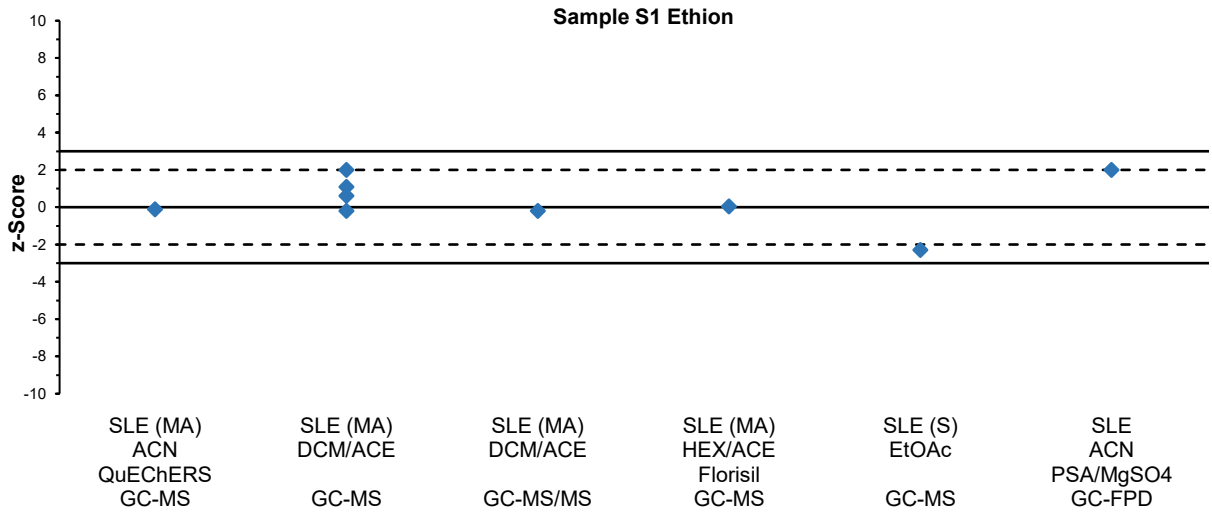


Figure 49 Sample S1 Ethion *z*-Scores vs Methodology

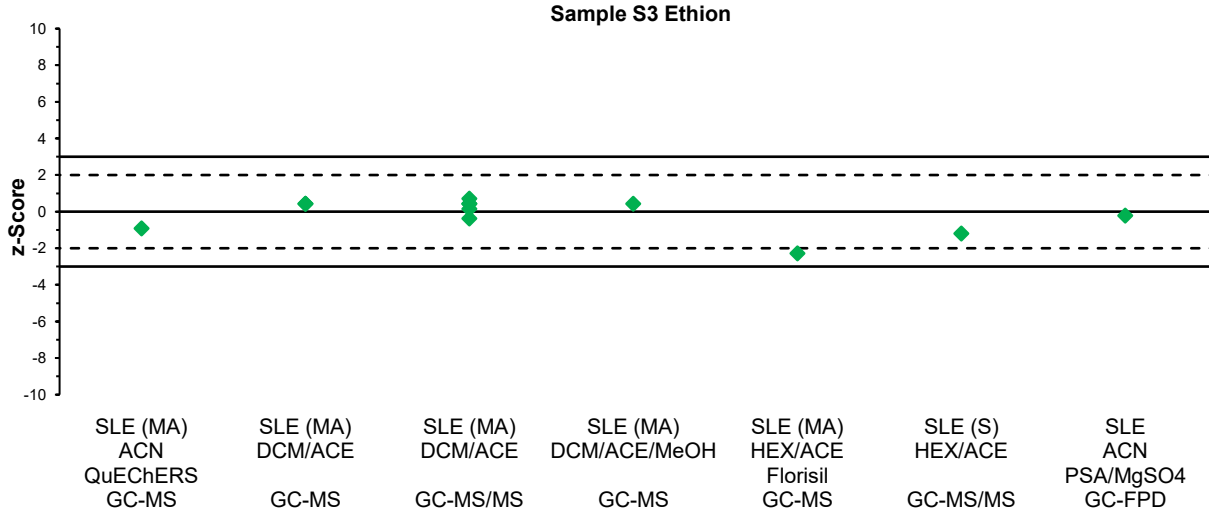


Figure 50 Sample S3 Ethion *z*-Scores vs Methodology

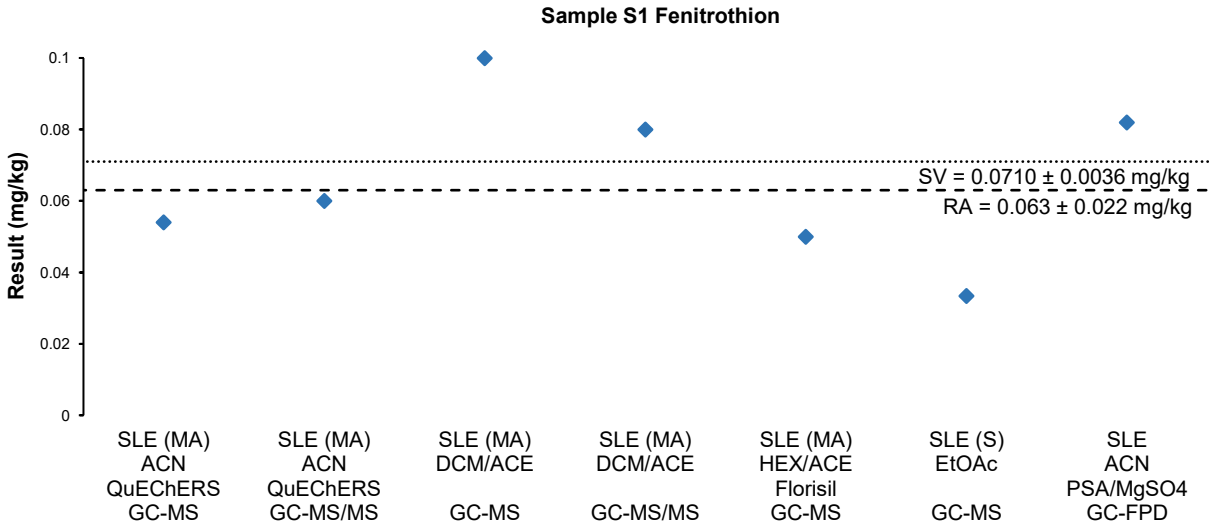


Figure 51 Sample S1 Fenitrothion Results vs Methodology

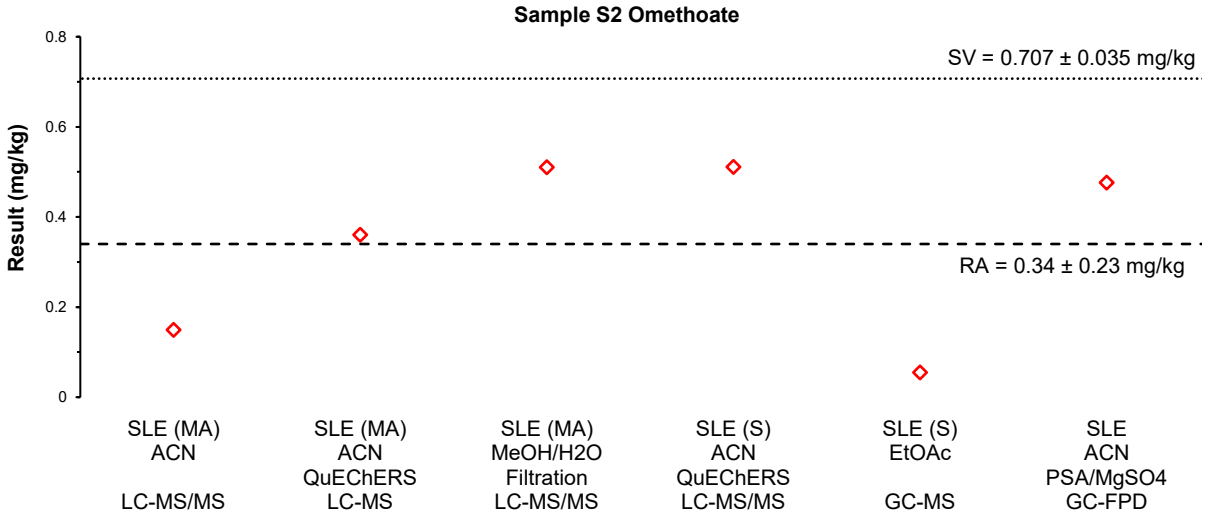


Figure 52 Sample S2 Omethoate Results vs Methodology

Glyphosate in Sample S2 did not present a significant analytical challenge to participating laboratories. Of the 8 laboratories that reported numeric results for this analyte, 7 achieved acceptable performance.

In contrast, the results reported for the compost sample (Sample S3) were highly variable, and no assigned value could be established. The difference in performance between the soil and compost matrices suggests that matrix effects may have influenced the analysis of glyphosate in compost. Laboratories should review and, where necessary, optimise their methods for the determination of glyphosate in compost samples.

Due to the high polarity of glyphosate, most participants used aqueous extraction solvents for this analyte. Laboratory 6 reported using organic solvents for extraction (methanol / acetonitrile), and their result was biased low for both samples (Figure 53 and Figure 54).

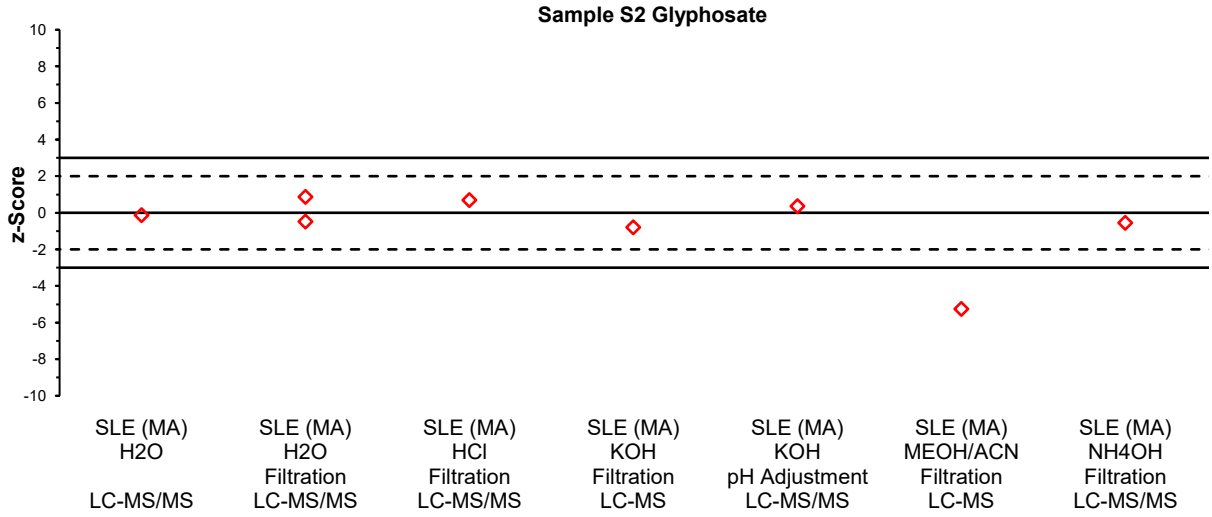


Figure 53 Sample S2 Glyphosate *z*-Scores vs Methodology

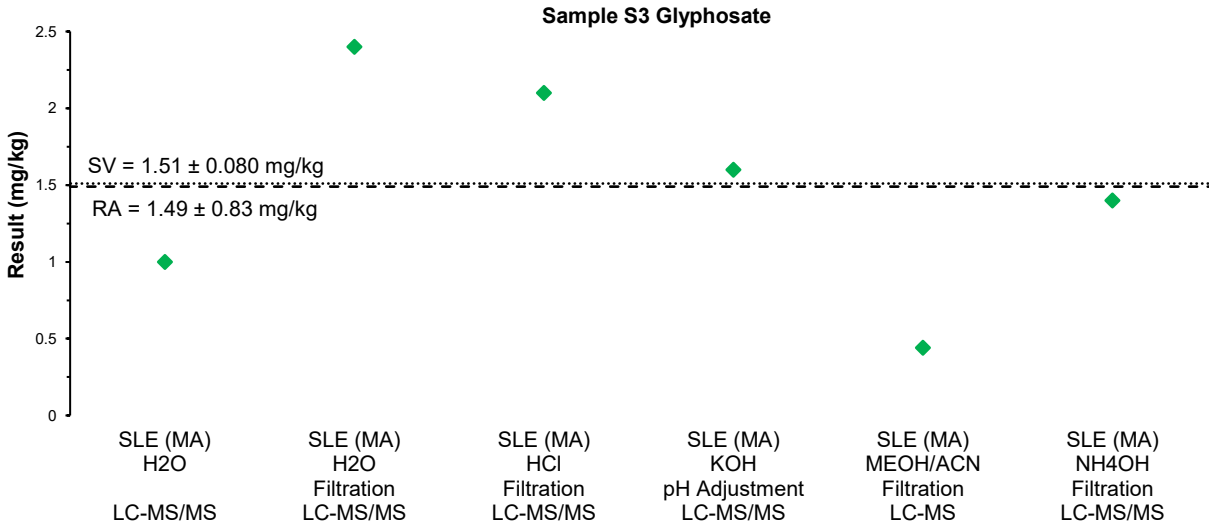


Figure 54 Sample S3 Glyphosate Results vs Methodology

2,4-D results in Sample S2 were in excellent agreement across participating laboratories. However, the reported concentrations corresponded to only approximately 30% of the spiked value (Figure 56). This low recovery has not been observed in previous NMIA PT studies for this analyte. No issues were identified with either the standard solution or the spiking procedure used during sample preparation. An assigned value was not set this analyte. Participants are advised to exercise caution when comparing their reported results with the nominal spike concentration.

MCPA in Sample S2 did not present a significant analytical challenge. All reported results were in excellent agreement with each other and with the spiked value (Figure 57).

Fipronil No assigned value could be set for fipronil in Sample S2. Only 6 participants reported numeric results; the results reported by Laboratories 1, 5, 6, 17, and 24 were in excellent agreement with each other and the spiked value (Figure 55).

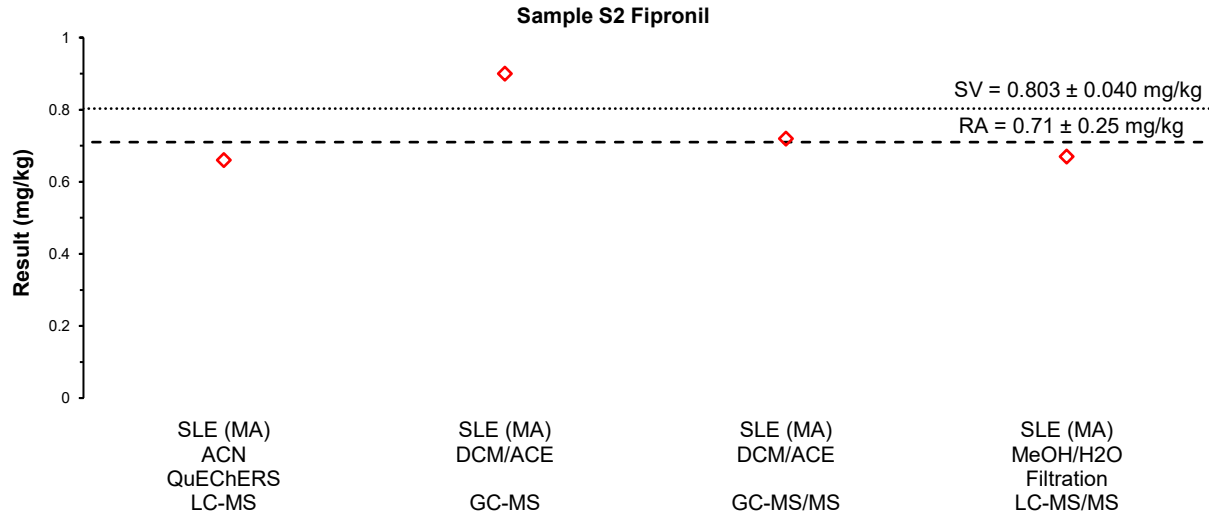


Figure 55 Sample S2 Fipronil Results vs Methodology

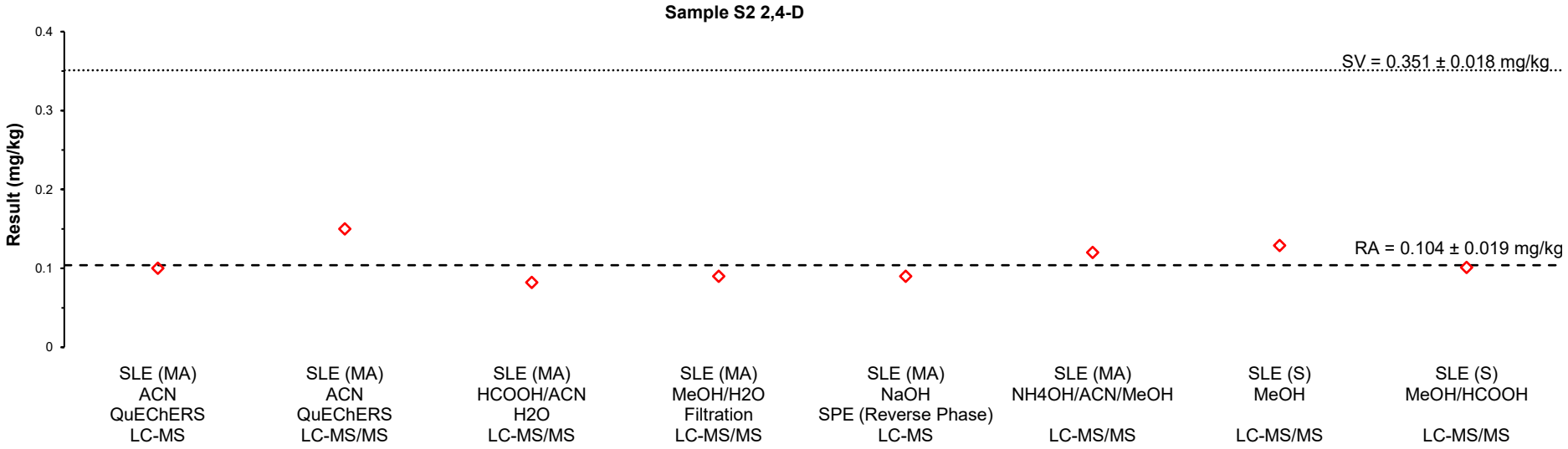


Figure 56 Sample S2 2,4-D Results vs Methodology

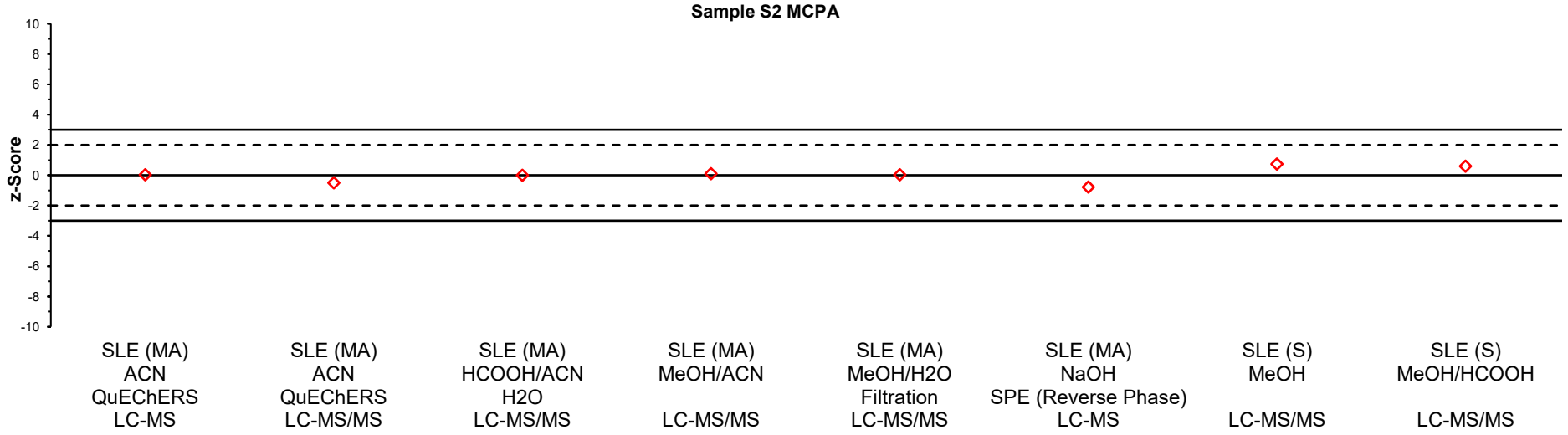


Figure 57 Sample S2 MCPA *z*-Scores vs Methodology

Bifenthrin was an incurred analyte present in Sample S3. In past NMIA PT studies, bifenthrin in soil has not been a challenging analyte for participants, however, in this study, participants' results for bifenthrin in compost were varied (Figure 58). Participants may need to review and, if necessary, reassess the analytical method used for the determination of this analyte in compost.

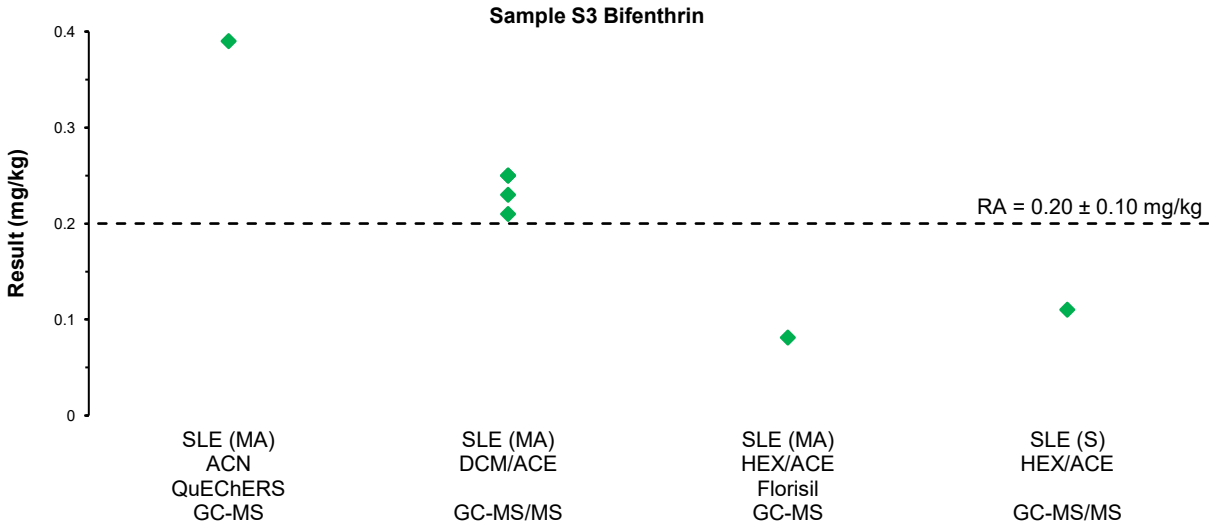


Figure 58 Sample S3 Bifenthrin Results vs Methodology

Fenvalerate No assigned value could be set for Sample S1 fenvalerate as too few results were reported. Results were in reasonable agreement with each other regardless of methodology (Figure 59). Of 14 participants testing for this analyte, 4 reported false negatives; these laboratories may need to reassess their LORs.

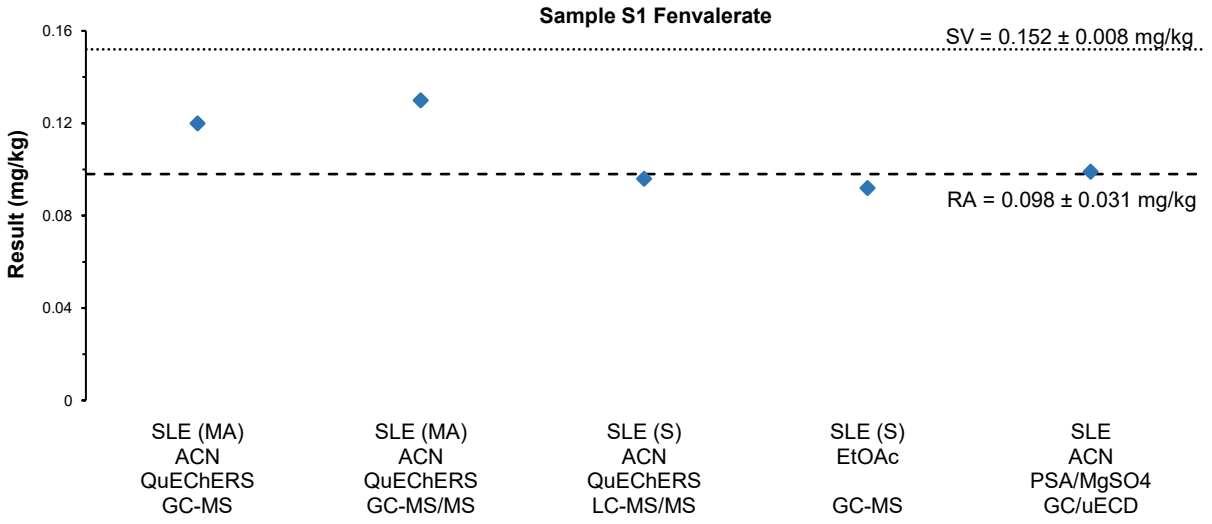


Figure 59 Sample S1 Fenvalerate Results vs Methodology

Metsulfuron-methyl results in Sample S2 were variable and no assigned value could be set (Figure 60). The consensus of participants' results was greater than the spiked value. Incurred metsulfuron-methyl in the soil sample may explain the discrepancy.

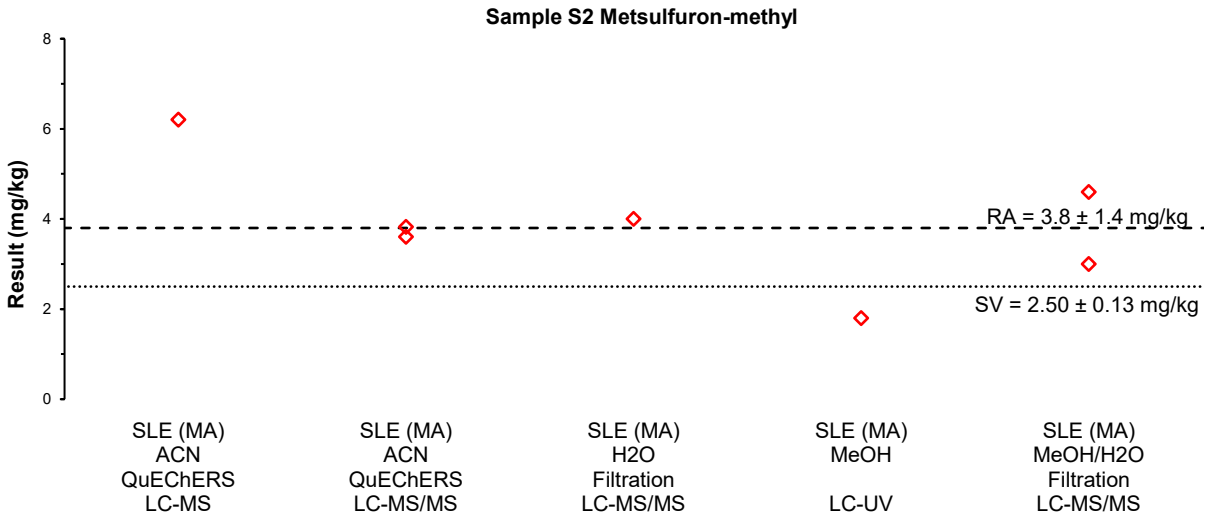


Figure 60 Sample S2 Metsulfuron-methyl Results vs Methodology

Bromacil and **Trifluralin** Participants reported using a variety of analytical methods for the determination of bromacil and trifluralin. Despite the differences in methodology, laboratories produced compatible results for both analytes (Figure 61 and Figure 62).

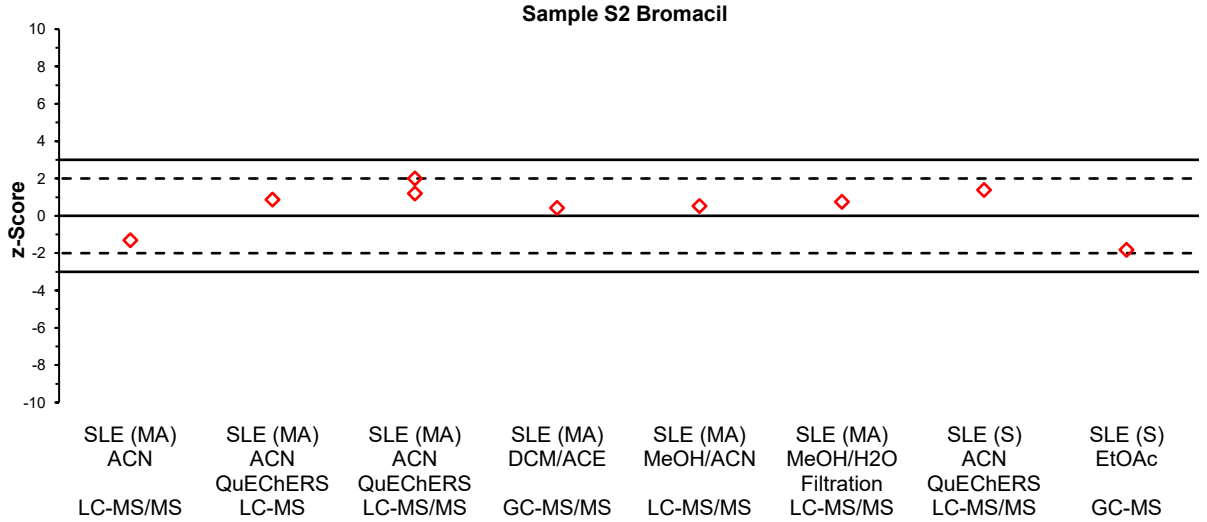


Figure 61 Sample S2 Bromacil *z*-Scores vs Methodology

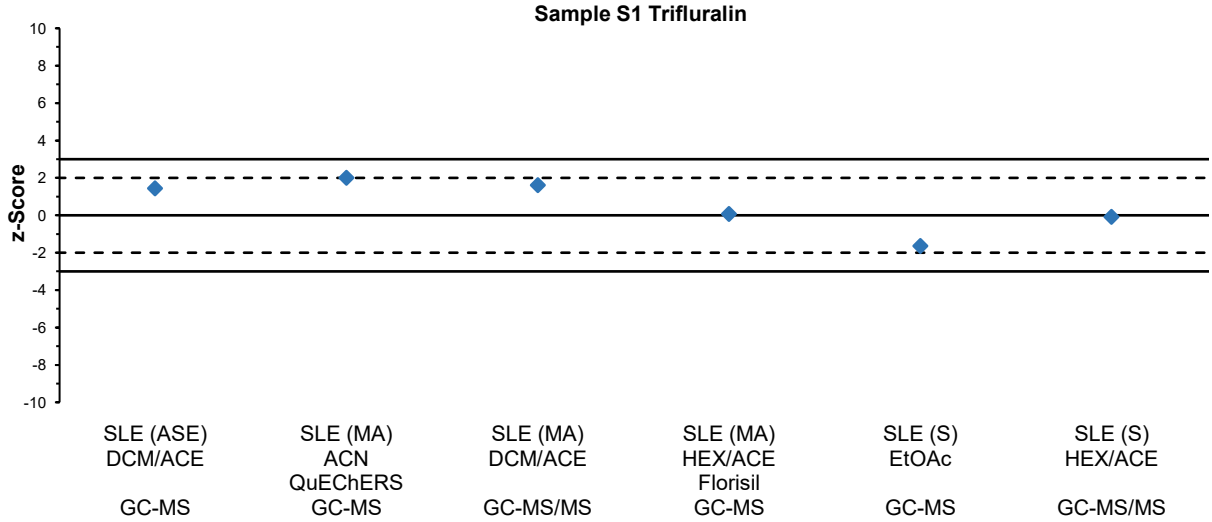


Figure 62 Sample S1 Trifluralin Results vs Methodology

Simazine Overall, participants' results for simazine were in good agreement with each other (between-laboratory CV = 17%). However, of 3 results using QuEChERS clean-up, 2 returned not acceptable *z*-scores (Figure 63).

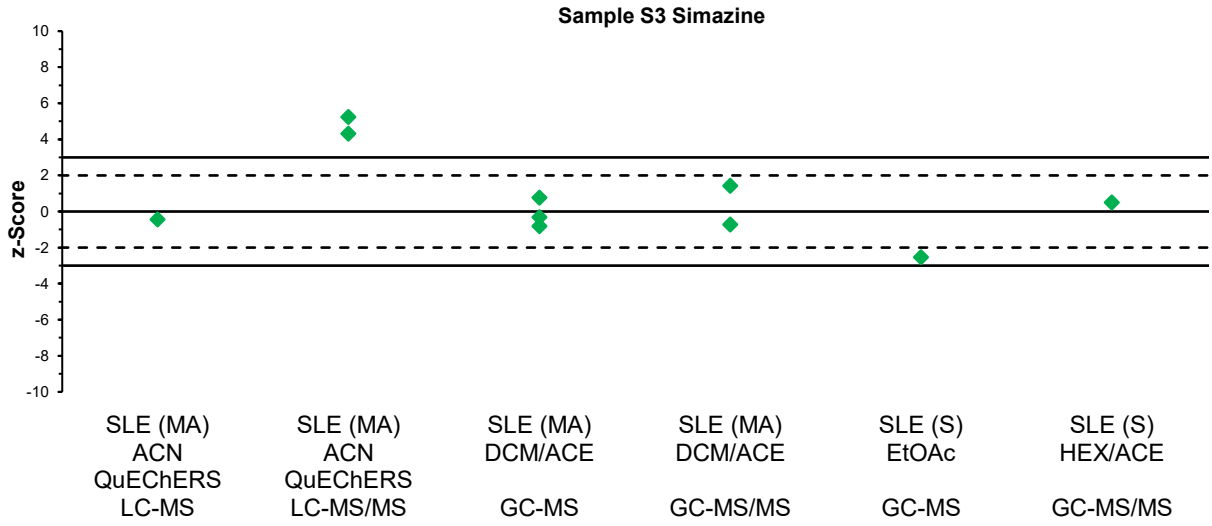


Figure 63 Sample S3 Simazine Results vs Methodology

Carbendazim was one of the analytes with the highest number of false negative reported results – of 11 participants testing for this analyte, 4 reported false negatives. The low level might have challenged participants analytical techniques, and the reported numeric results returned by participants were not in agreement with each other (Figure 64). Laboratory 5 used water as their extraction solvent, and their reported result was very close to the spiked value. Other extraction solvents resulted in poor recovery of this analyte. Laboratories should review their method for this analyte, and also update their LOR.

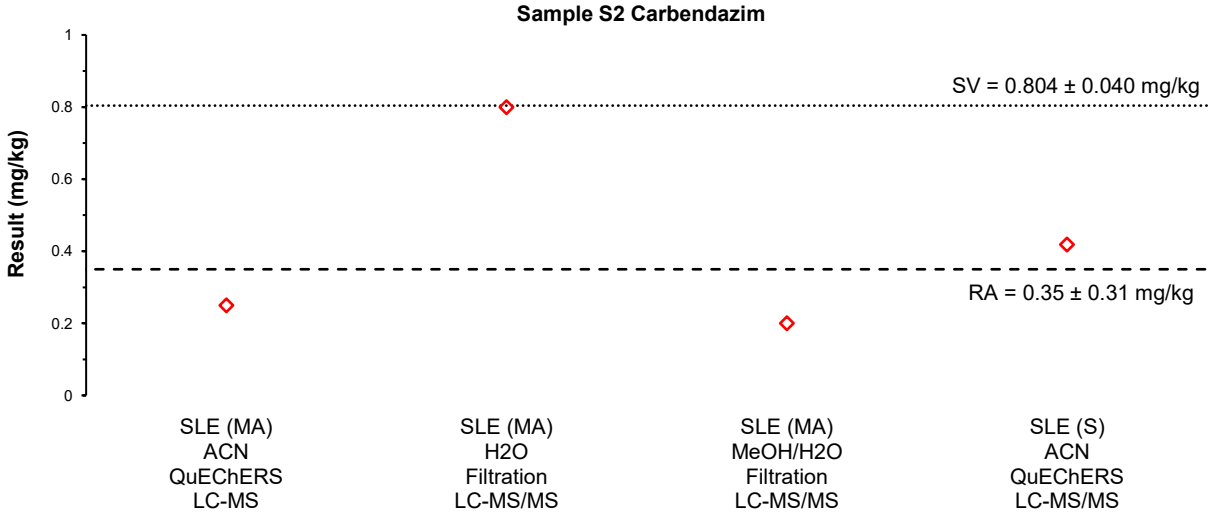


Figure 64 Sample S2 Carbendazim Results vs Methodology

Imidacloprid Laboratories are having difficulties with measuring this analyte at low level, with 4 false negative results in Sample S2. Only 6 labs reported numeric results for this analyte in Sample S2. Laboratories 5, 6, 11, and 22 were in good agreement with each other and the spiked value (Figure 65).

There was lower recovery of the spiked value in Sample S3 as compared to Sample S2 (Figure 66), which may reflect lower extraction efficiency from the compost matrix as compared to soil.

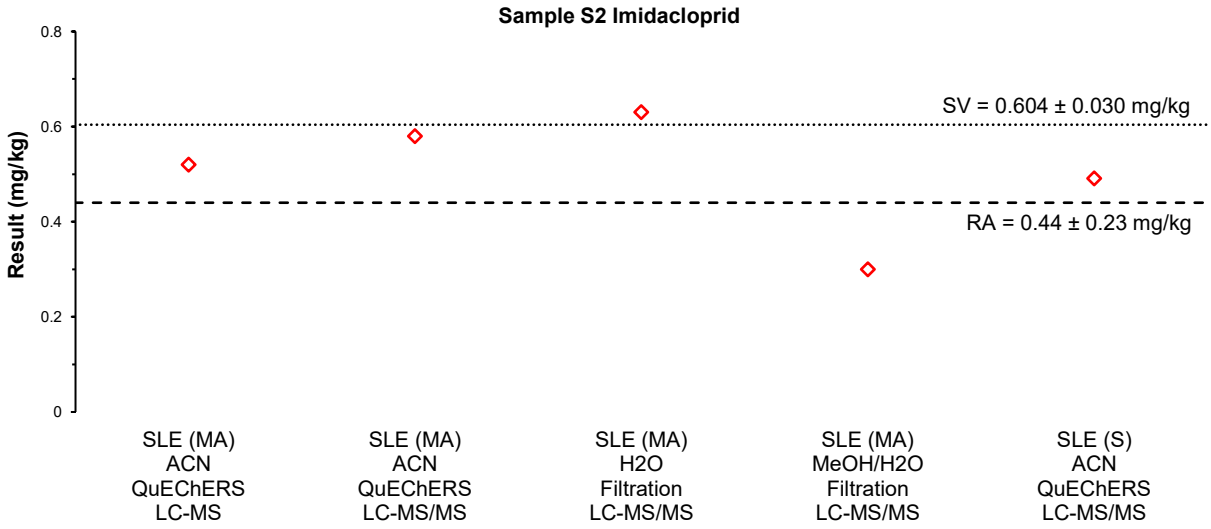


Figure 65 Sample S2 Imidacloprid Results vs Methodology

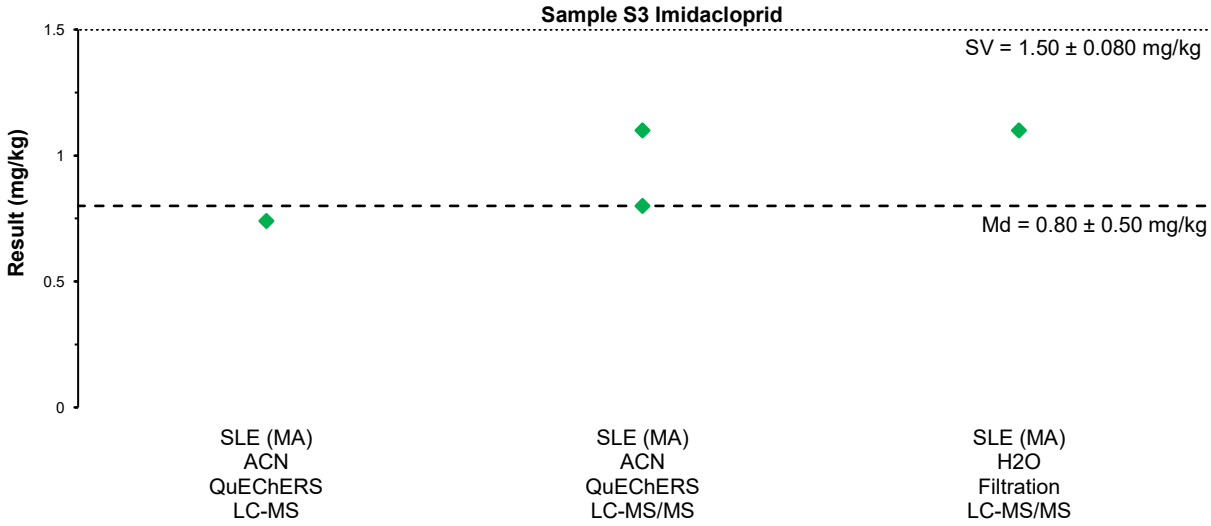


Figure 66 Sample S3 Imidacloprid Results vs Methodology

6.9 Certified Reference Materials

Participants were requested to indicate whether certified standards or matrix reference materials had been used as part of the quality assurance for their analysis. Fourteen participants reported using certified standards, 2 participants reported using matrix reference materials, and 3 participants reported using both.

The following were listed:

- Accustandard
- ERA (e.g. CRM727)
- Dr Ehrenstorfer
- o2si
- PM Separations
- Sigma Aldrich (e.g. SQC009, CRM107, CRM48392)
- ISO 17034 standards

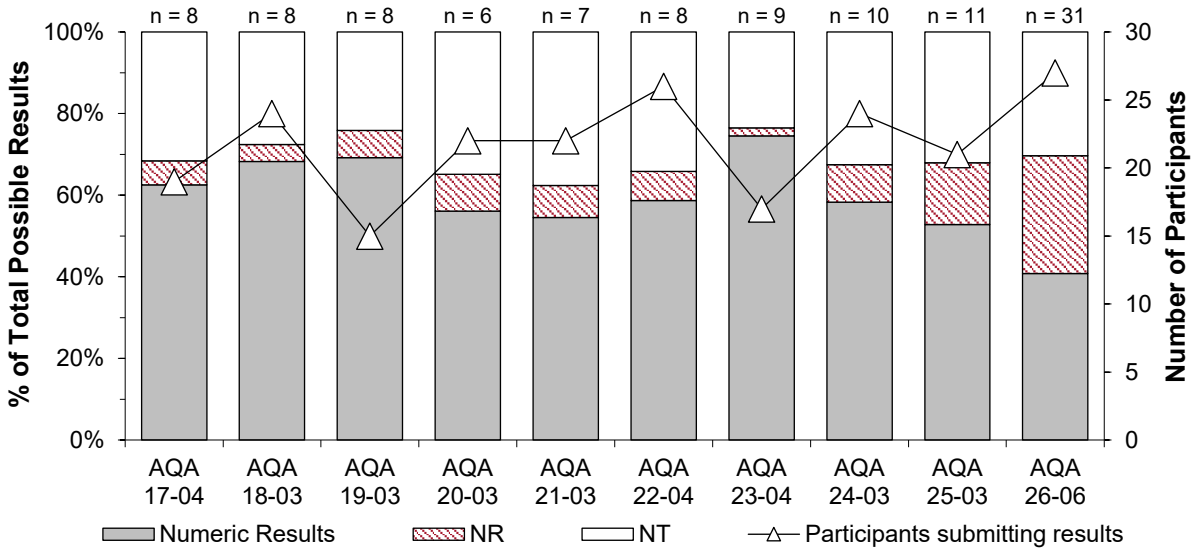
These materials may or may not meet the internationally recognised definition of a CRM:

> *'**reference material**, accompanied by documentation issued by an authoritative body and providing one or more specified property values with associated uncertainties and traceabilities, using valid procedures'*[12]

6.10 Comparison with Previous Pesticides in Soil and Compost PT Studies

This was the first NMIA PT study to assess pesticides in compost. The results for Sample S3 have been included in the following discussion.

A summary of participation and reported results rates in NMIA pesticides in soil and compost PT studies over the last 10 studies (2017 – 2026) is presented in Figure 67. There was a decrease in the proportion of pesticides being tested for in this study, which is likely due to the low analyte concentrations for several analytes in Samples S1 and S2.



n = number of spiked analytes

Figure 67 Summary of Participation and Reported Results in NMIA Pesticides in Soil and Compost PT Studies

A summary of the acceptable performance (presented as a percentage of the total number of scores for each study) obtained by participants in NMIA pesticides in soil and compost PT studies over the last 10 studies (2017 – 2026) is presented in Figure 68. Over this period, the average proportion of acceptable *z*-scores and E_n-scores was 88% and 84% respectively.

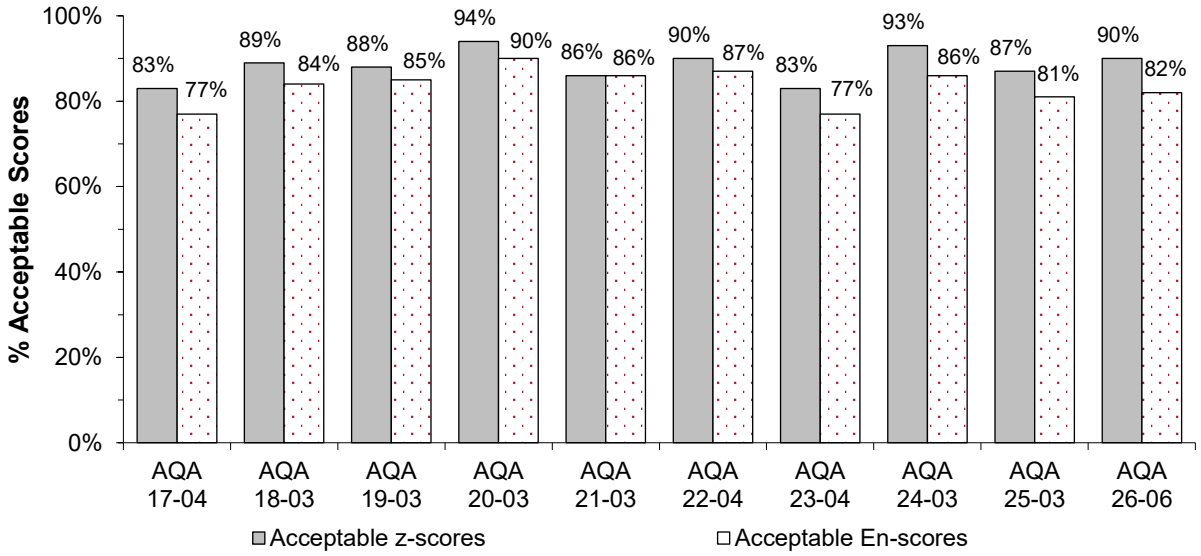


Figure 68 Acceptable *z*-Scores and E_n-scores in NMIA Pesticides in Soil and Compost PT Studies

Individual performance history reports are emailed to participants at the end of each study; the consideration of *z*-scores over time provides much more useful information than a single score. Over time, laboratories should expect at least 95% of their scores to lie within the range $|z| \leq 2.0$. Scores in the range $2.0 < |z| < 3.0$ can occasionally occur, however these should be interpreted in conjunction with the other scores obtained by that laboratory. For example, a trend of *z*-scores on one side of the zero line is an indication of method or laboratory bias.

As discussed in Section 6.2, it is a requirement of ISO/IEC 17025 that laboratories report their uncertainties. Figure 69 presents a summary of the relative uncertainties as reported by participants over the last 10 studies (2017 – 2026). Over this period, most numeric results were reported with uncertainties, and most of these relative uncertainties were between 15% and 50%. However, around 25% of relative uncertainties were outside this range, and these may have been unrealistically small or too large to be fit-for-purpose.

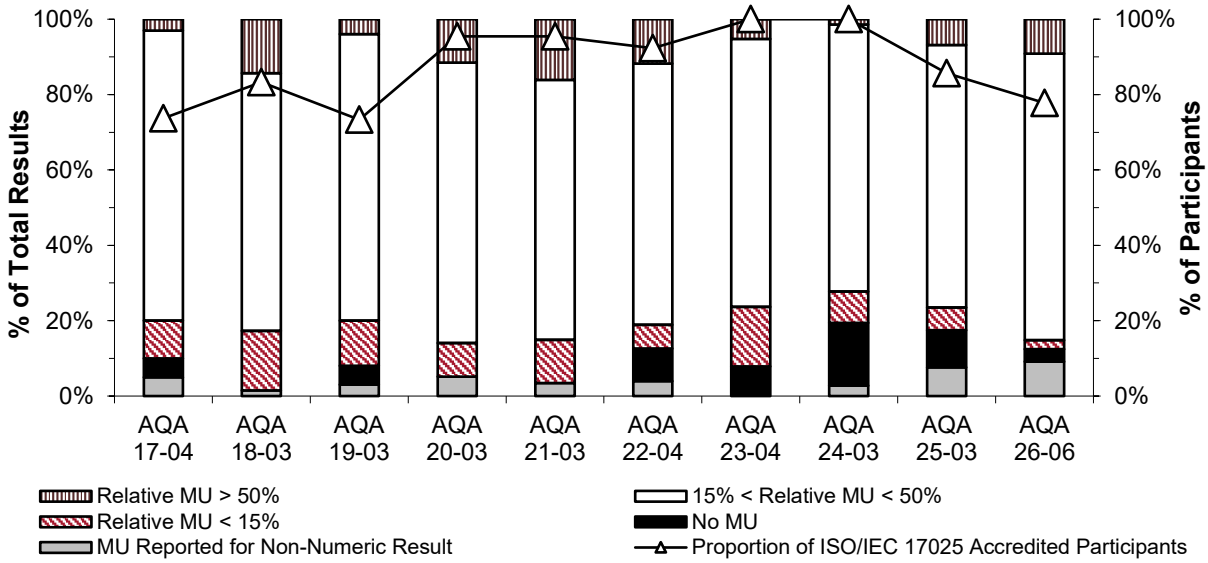


Figure 69 Summary of Participants' Relative Uncertainties for NMIA Pesticides in Soil and Compost PT Studies

7 REFERENCES

Please note that for all undated references, the latest edition of the referenced document (including any amendments) applies.

[1] ISO/IEC 17043, *Conformity assessment – General requirements for the competence of proficiency testing providers*.

[2] Commonwealth of Australia, Department of Industry, Science and Resources, NMIA, 2026, *Study Protocol for Proficiency Testing,* viewed July 2026, <https://www.industry.gov.au/sites/default/files/2020-10/cpt_study_protocol.pdf>.

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APPENDIX 1 SAMPLE PREPARATION

Samples S1 and S2 were prepared from soil collected from a farm in Victoria. The material was dried, sieved, and fortified with the analytes of interest prior to dividing it into equal portions of 50 g each.

Sample S3 was prepared using compost material purchased from a hardware and garden supply store. The material was dried, sieved, and further fortified for the analytes of interest prior to dividing it into equal portions of 45 g each.

All samples were stored at 4°C prior to dispatch.

APPENDIX 2 ASSESSMENT OF HOMOGENEITY AND STABILITY

A2.1 Homogeneity

No homogeneity testing was conducted for Samples S1 and S2 as the samples were prepared using a process previously demonstrated to produce sufficiently homogeneous samples. The results of this study also gave no reason to question the samples' homogeneity.

Sample S3 was prepared in a new matrix (compost), and homogeneity testing was conducted for this sample. The testing was performed using routine methods from the NMIA Analytical Services Branch. Four different methods were used depending on the analyte, as described below.

Total Chlordane, Dieldrin, Endosulfan Sulfate, and Lindane: Sample (10 g) was weighed out, and anhydrous sodium sulfate (5 g) was added to absorb moisture. Acetone/Hexane (2:3, 50 mL) was added to the sample, along with surrogate. The sample was extracted on the rotating shaker for 2 hours, or sonicated for 10 minutes while maintaining the temperature of the sample to be under 30°C by adding ice to the sonication bath. A portion of the sample extract was taken through an alumina clean-up and collected. The portion of cleaned extract then went through a sulfur clean-up followed by a sulfuric acid clean-up. The resulting extract was transferred to a GC vial where it was analysed by a GC-dual ECD system.

Bifenthrin, Diazinon, Ethion, and Simazine: Sample (10 g) was weighed out, and anhydrous sodium sulfate (5 g) was added to absorb moisture. Acetone/Hexane (2:3, 50 mL) was added to the sample, along with surrogate. The sample was extracted on the rotating shaker for 2 hours, or sonicated for 10 minutes while maintaining the temperature of the sample jar to be under 30°C by adding ice to the sonication bath. A portion of the sample extract was solvent exchanged to acetonitrile and a QuECHeRs clean-up was performed. The cleaned sample extract was then solvent exchanged to hexane and a portion was transferred to GC vials. ISTD was added to the GC vials before it was analysed by a GC-MS/MS system.

Acephate and Imidacloprid: Sample (2 g) was weighed out, followed by an addition of 20% methanol in water (10 mL). The samples were tumbled for one hour and centrifuged for 10 minutes at 3500 rpm. The liquid sample extract was filtered using a 0.20 µm syringe filter and transferred to a plastic LC vial with ISTD added. The sample was analysed by a LC-MS/MS system.

Glyphosate: Sample (1 g) was weighed out, followed by an addition of 0.1 M KOH solution (10 mL) and ISTD, and then was tumbled for 30 minutes. The tube was then centrifuged for 10 minutes at 3500 rpm, and the liquid portion of the sample extract was collected. The pH of the sample extract was adjusted to 7, and the sample extract (5 mL) was transferred to a new tube. 5% borate buffer (250 µL) was added to the neutralised extract followed by FMOC derivatization solution (750 µL). The mixture was vortexed and placed in an oven at 40°C overnight. To quench the reaction, 2% phosphoric acid (150 µL) was added to the mixture. The final mixture was filtered and transferred to plastic LC vials. The sample was then analysed by a LC-MS/MS system.

Homogeneity testing for this sample was based on that described by Thompson and Fearn,[13] which is also the procedure as described in the International Harmonised Protocol for Proficiency Testing.[4] A minimum of 6 bottles from Sample S3 were selected at random. Duplicate test portions were taken from each bottle, and the mass fraction of all targeted analytes were measured. Measurements were made under repeatability conditions in random order. The data for the full homogeneity testing of Sample S3 can be found below in Tables 44 to 54.

Table 44 Sample S3 Acephate Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
3	1.02	1.11
19	1.09	1.10
24	1.08	1.03
27	1.04	1.09
30	1.11	1.10
44	1.12	1.11
49	1.06	1.05
Mean	1.08	
CV	3%	

Thompson and Fearn Homogeneity Tests

Test	Value	Critical	Result
Cochran	0.59	0.73	**Pass**
s_{an}/σ	0.14	0.50	**Pass**
s^2_{sam}	0.00012	0.010	**Pass**

Table 45 Sample S3 Bifenthrin Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
3	0.115	0.112
19	0.104	0.105
24	0.101	0.110
27	0.110	0.111
30	0.108	0.101
44	0.100	0.109
49	0.105	0.105
Mean	0.107	
CV	4.3%	

Thompson and Fearn Homogeneity Tests

Test	Value	Critical	Result
Cochran	0.39	0.73	**Pass**
s_{an}/σ	0.18	0.50	**Pass**
s^2_{sam}	0.0000060	0.00011	**Pass**

Table 46 Sample S3 Total Chlordane Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
3	1.73	1.79
19	1.63	1.77
27	1.76	1.70
30	1.66	1.62
44	1.67	1.75
49	1.66	1.70
Mean	1.70	
CV	3.3%	

Thompson and Fearn Homogeneity Tests

Test	Value	Critical	Result
Cochran	0.55	0.78	**Pass**
s_{an}/σ	0.16	0.50	**Pass**
s^2_{sam}	0.00016	0.028	**Pass**

Table 47 Sample S3 Diazinon Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
3	0.639	0.626
19	0.567	0.630
24	0.586	0.613
27	0.664	0.622
30	0.603	0.594
49	0.630	0.649
Mean	0.619	
CV	4.5%	

Thompson and Fearn Homogeneity Tests

Test	Value	Critical	Result
Cochran	0.56	0.78	**Pass**
s_{an}/σ	0.20	0.50	**Pass**
s^2_{sam}	0.00018	0.0041	**Pass**

Table 48 Sample S3 Dieldrin Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
3	0.42	0.42
19	0.39	0.42
27	0.43	0.42
30	0.40	0.40
44	0.40	0.42
49	0.40	0.41
Mean	0.41	
CV	2.8%	

Thompson and Fearn Homogeneity Tests

Test	Value	Critical	Result
Cochran	0.64	0.78	**Pass**
s_{an}/σ	0.13	0.50	**Pass**
s^2_{sam}	0.000016	0.0015	**Pass**

Table 49 Sample S3 Endosulfan Sulfate Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
3	0.61	0.63
19	0.64	0.68
27	0.64	0.62
30	0.61	0.64
44	0.62	0.61
49	0.60	0.62
Mean	0.63	
CV	3.2%	

Thompson and Fearn Homogeneity Tests

Test	Value	Critical	Result
Cochran	0.49	0.78	**Pass**
s_{an}/σ	0.13	0.50	**Pass**
s^2_{sam}	0.00013	0.0036	**Pass**

Table 50 Sample S3 Ethion Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
3	0.147	0.146
19	0.131	0.136
24	0.129	0.140
27	0.142	0.142
30	0.136	0.129
44	0.127	0.142
49	0.135	0.134
Mean	0.137	
CV	4.7%	

Thompson and Fearn Homogeneity Tests

Test	Value	Critical	Result
Cochran	0.53	0.73	**Pass**
s_{an}/σ	0.20	0.50	**Pass**
s^2_{sam}	0.000012	0.00019	**Pass**

Table 51 Sample S3 Glyphosate Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
3	1.81	2.03
19	1.96	2.15
24	1.82	1.97
27	1.88	1.91
30	2.24	2.00
44	1.86	1.94
49	2.07	2.04
Mean	1.98	
CV	6.2%	

Thompson and Fearn Homogeneity Tests

Test	Value	Critical	Result
Cochran	0.32	0.73	**Pass**
s_{an}/σ	0.28	0.50	**Pass**
s^2_{sam}	0.0030	0.047	**Pass**

Table 52 Sample S3 Imidacloprid Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
3	0.50	0.49
19	0.46	0.46
24	0.45	0.52
27	0.49	0.45
30	0.47	0.45
44	0.46	0.49
49	0.49	0.48
Mean	0.48	
CV	4.5%	

Thompson and Fearn Homogeneity Tests

Test	Value	Critical	Result
Cochran	0.58	0.73	**Pass**
s_{an}/σ	0.26	0.50	**Pass**
s^2_{sam}	0.00001	0.0026	**Pass**

Table 53 Sample S3 Lindane Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
3	0.64	0.64
19	0.58	0.64
27	0.66	0.63
30	0.60	0.60
44	0.60	0.63
49	0.64	0.61
Mean	0.62	
CV	3.9%	

Thompson and Fearn Homogeneity Tests

Test	Value	Critical	Result
Cochran	0.51	0.78	**Pass**
s_{an}/σ	0.18	0.50	**Pass**
s^2_{sam}	0.000073	0.0039	**Pass**

Table 54 Sample S3 Simazine Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
3	0.669	0.688
19	0.684	0.701
24	0.625	0.688
27	0.759	0.719
30	0.674	0.628
44	0.588	0.733
49	0.654	0.707
Mean	0.680	
CV	6.7%	

Thompson and Fearn Homogeneity Tests

Test	Value	Critical	Result
Cochran	0.65	0.73	**Pass**
s_{an}/σ	0.35	0.50	**Pass**
s^2_{sam}	0.00001	0.0068	**Pass**

A2.2 Stability

No stability testing was conducted for Samples S1 and S2 as the samples were prepared, stored and dispatched using a process previously demonstrated to produce sufficiently stable samples for similar analytes and matrices over a similar time frame. After preparation and before dispatch, all samples were stored in a refrigerator at approximately 4°C. For dispatch, samples were packaged into insulated polystyrene foam boxes with cooler bricks.

Stability testing was conducted for Sample S3. Two units of Sample S3 were packed in the same way as the samples sent to participants and were stored at ambient conditions for the same amount of time as for the longest participant sample delivery time; for this study, this was 3 days. The samples were then stored at 4°C and analysed after the study results due date. These were then compared against samples analysed prior to sample dispatch. The stability study results therefore cover both transport stability as well as analytes stability over the whole study conduct.

The analyses for stability were conducted for: acephate, bifenthrin, total chlordane, diazinon, dieldrin, endosulfan sulfate, ethion, imidacloprid, and lindane. The testing was performed by NMIA Analytical Services Branch as described above.

Figures 70 to 78 present the initial and stability testing results for each analyte. Results were in agreement with each other within their respective uncertainties.

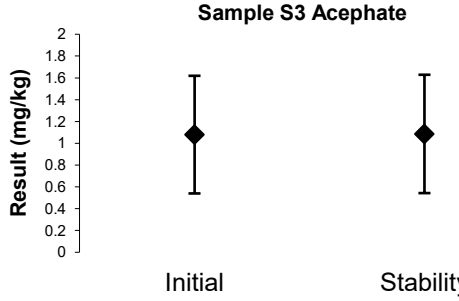


Figure 70 S3 Acephate Stability Results



Figure 71 S3 Bifenthrin Stability Results

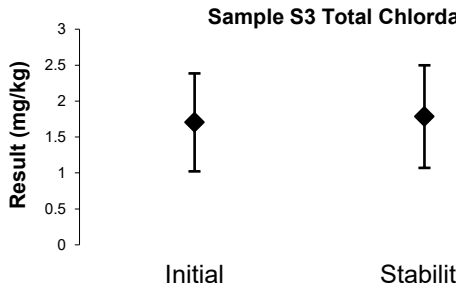


Figure 72 S3 Total Chlordane Stability Results

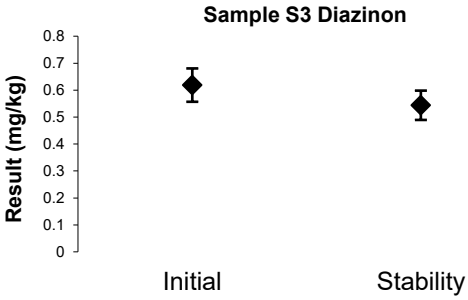


Figure 73 S3 Diazinon Stability Results

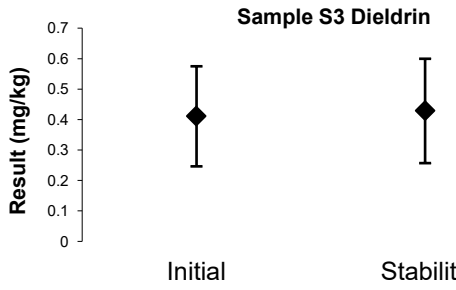


Figure 74 S3 Dieldrin Stability Results

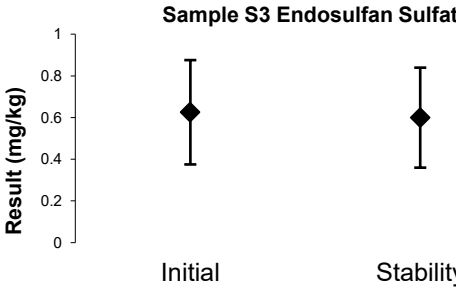


Figure 75 S3 Endosulfan Sulfate Stability Results



Figure 76 S3 Ethion Stability Results

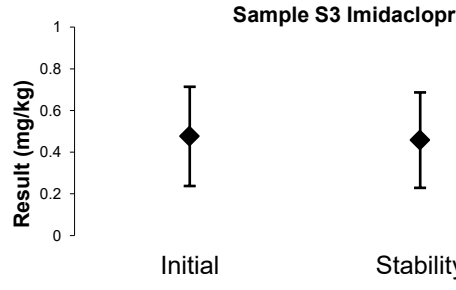


Figure 77 S3 Imidacloprid Stability Results

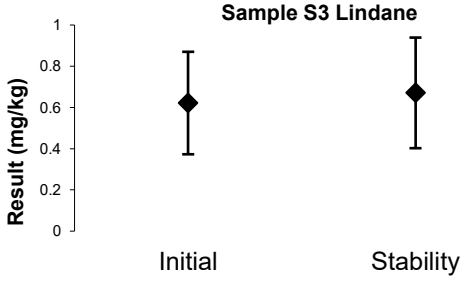


Figure 78 S3 Lindane Stability Results

The results of this study also gave no reason to question the samples' transportation stability. Comparisons of results to days spent in transit for scored analytes are presented in Figures 79 to 92 (solid blue lines correspond to the assigned value ± U for each analyte; results have not been included here if they were excluded from all statistical calculations in Section 5). No significant trend was observed.

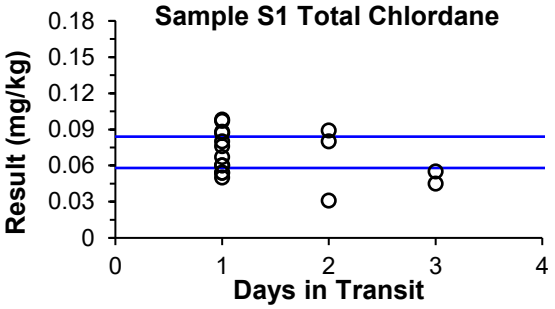


Figure 79 S1 Total Chlordane Results vs Transit Days

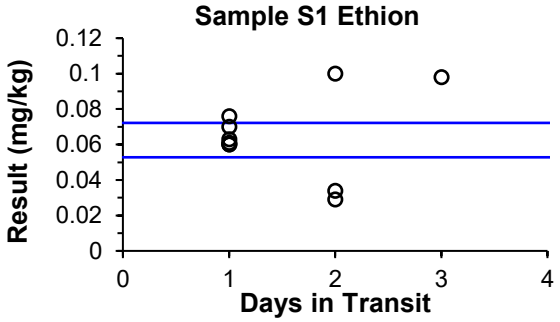


Figure 80 S1 Ethion Results vs Transit Days

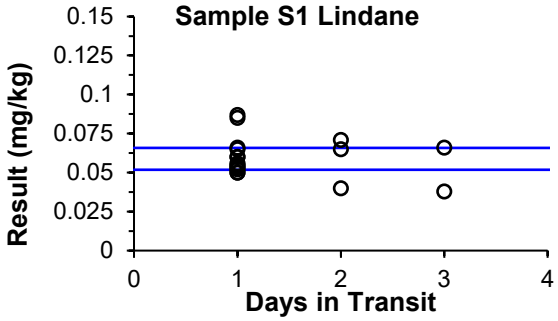


Figure 81 S1 Lindane Results vs Transit Days

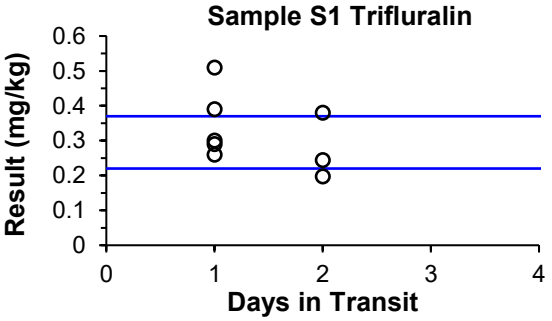


Figure 82 S1 Trifluralin Results vs Transit Days

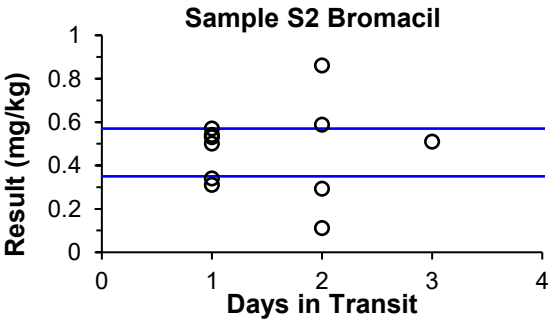


Figure 83 S2 Bromacil Results vs Transit Days

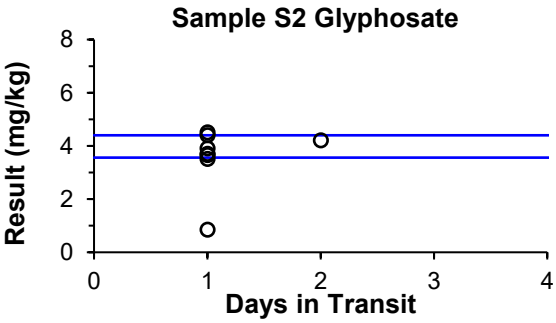


Figure 84 S2 Glyphosate Results vs Transit Days

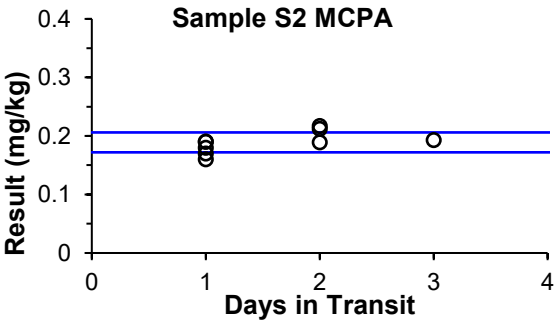


Figure 85 S2 MCPA Results vs Transit Days

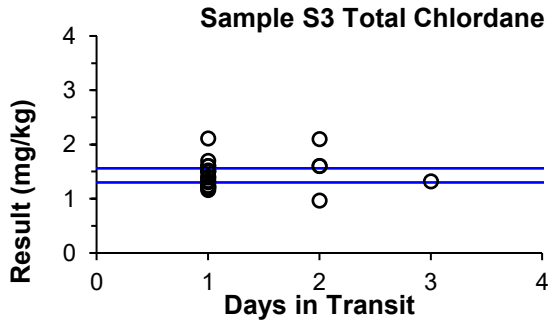


Figure 86 S3 Total Chlordane Results vs Transit Days

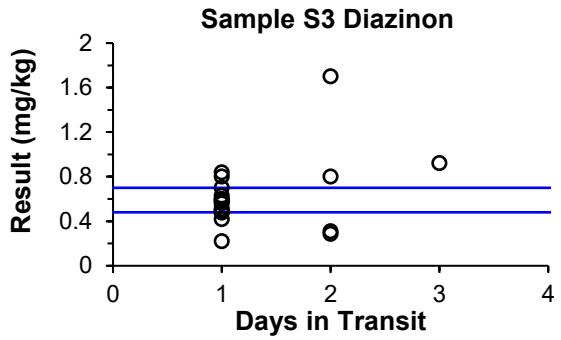


Figure 87 S3 Diazinon Results vs Transit Days

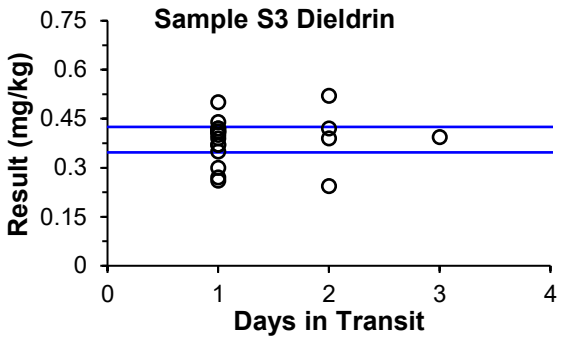
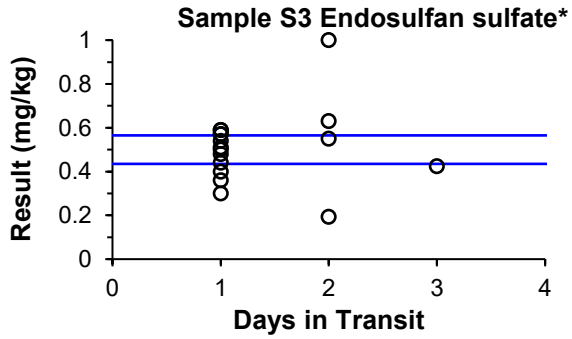


Figure 88 S3 Dieldrin Results vs Transit Days



*Results larger than 1.0 mg/kg have been set as 1.0 mg/kg

Figure 89 S3 Endosulfan Sulfate Results vs Transit Days

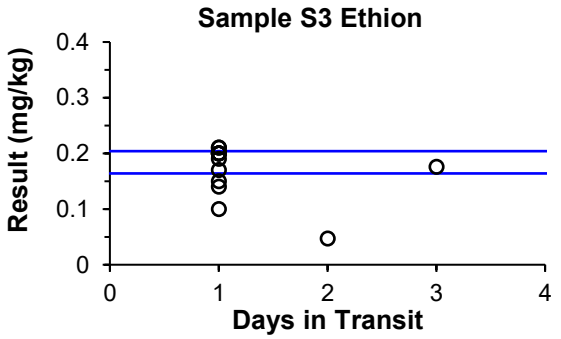


Figure 90 S3 Ethion Results vs Transit Days

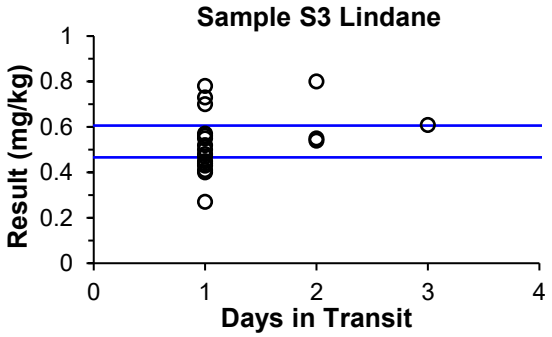


Figure 91 S3 Lindane Results vs Transit Days

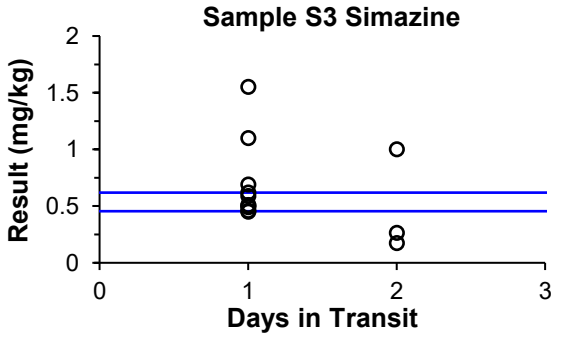


Figure 92 S3 Simazine Results vs Transit Days

APPENDIX 3 ROBUST AVERAGE AND ASSOCIATED UNCERTAINTY, z-SCORE AND E_n-SCORE CALCULATIONS

A3.1 Robust Average and Associated Uncertainty

Robust averages were calculated using the procedure described in ISO 13528.[7] The associated uncertainties were evaluated as according to Equation 4.

$$u_{rob\ av} = \frac{1.25 \times S_{rob\ av}}{\sqrt{p}} \qquad \textit{Equation 4}$$

where:

$u_{rob\ av}$ is the standard uncertainty of the robust average

$S_{rob\ av}$ is the standard deviation of the robust average

p is the number of results

The expanded uncertainty ($U_{rob\ av}$) is the standard uncertainty multiplied by a coverage factor of 2 at approximately 95% confidence level.

A worked example for Sample S1 lindane is set out below in Table 55.

Table 55 Uncertainty of the Robust Average for Sample S1 Lindane

No. results (p)	18
Robust Average	0.0588 mg/kg
$S_{rob\ av}$	0.0120 mg/kg
$u_{rob\ av}$	0.0035 mg/kg
k	2
$U_{rob\ av}$	0.0070 mg/kg

Therefore, the robust average for Sample S1 lindane is 0.0588 ± 0.0070 mg/kg.

A3.2 z-Score and E_n-Score Calculations

For each participant's result, a z-score and E_n-score are calculated according to Equations 2 and 3 respectively (Section 4).

A worked example for the result reported by Laboratory 20 for Sample S1 lindane is set out below in Table 56.

Table 56 z-Score and E_n-Score Calculation for Sample S1 Lindane Result Reported by Laboratory 20

Participant Result (mg/kg)	Assigned Value (mg/kg)	Standard Deviation for Proficiency Assessment	z-Score	E_n-Score
0.056 ± 0.02	0.0588 ± 0.0070	20% as PCV, or: 0.20 × 0.0588 = 0.01176 mg/kg	$z = \frac{0.056 - 0.0588}{0.01176} = -0.24$	$E_n = \frac{0.056 - 0.0588}{\sqrt{0.02^2 + 0.0070^2}} = -0.13$

APPENDIX 4 TEST METHODS REPORTED BY PARTICIPANTS

A4.1 Sample S1 Methodology

Table 57 Measurement Methods and Instrument Techniques for Total Chlordane

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
1	5	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	25mL, 1 step	60	Room temp	N/A	N/A	GC-MS/MS	Yes, Mixed internal standard
2	10	Yes, before clean-up	Solid-Liquid (Sonication)	Acetone/ Hexane 2:3	50mL, 1 step	2x20 minutes				GC-ECD	Yes, DB-DDE, DF-DDE, TCMX, TPP
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	GC-MS/MS	Phenthrene-d10
5	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: ACETONE	20mL 1 step	120	n/a	n/a	none	GC-MS	Deuterated PAH
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	GC-MS	yes, chlorpyrifos D10
7	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	20 mL, 1 Step	60	NA	NA	NA	GC-MS	Yes, various
8	5	No	Solid-Liquid	Acetonitrile	10 mL, 1 step	240	40	10	PSA,MgSO 4	GC/uECD	No
9	10	No	Solid-Liquid (Mechanical Agitation)	Acetone hexan 1/2	10/20mL	60	25	0	No	GC-MS	no

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
11	2	Yes, before clean-up	Solid-Liquid (Sonication)	Hex:Ace	20mL, 1 step	120	NA	NA	NA	GC-MS/MS	Yes, PBB7 & PBB18
12	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20 mL, 1 step	60	Room Temperature	N/A	N/A	GC-MS	Yes, d12-Chrysene
13	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1:1 DCM/ Acetone	20mL	60 min	n/a	n/a	n/a	GC-MS	EPA 8270 Semivolatile Internal Standard Mix
14	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	60				GC-MS/MS	Yes, 1,4-Dichlorobenzene-D4 and Bromophos-ethyl
15	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone: Methanol	20 mL, 1 step	120	N/A	N/A	None	GC-MS	Yes, 8270 Internal Standard Mix
17	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone (1:1)	30 mL, 1 step	120	N/A	N/A	N/A	GC-MS/MS	Yes: Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12
18	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ ACETONE	30mL, 1 step	60				GC-MS	Yes- Semivolatile internal Standard
19	10	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromonitrobenzene

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
20	5	YES	Solid-Liquid (Mechanical Agitation)	ACETONE/ DCM	10 mL, 1 step	60			NO	GC-MS/MS	Yes, Bromonitrobenzene and Bromophos-ethyl
21	2.5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1:1 DCM: Acetone	5 mL, 1 step	30	NA	NA	No cleanup	GC-MS/MS	Yes, Phenanthrene-d10
22	20	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetone, Hexane	50 mL, 3 steps	60	40	30	Florisil clean-up	GC-MS	Yes, Naphtalene-D8
23	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10 mL, 1 step	10	No concentration	-	QuEChERS (dSPE)	GC-MS/MS	Yes, acenaphthene-d10 and chrysene-d12
24	10	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromonitrobenzene
25	10	Yes, before clean-up	Solid-Liquid (Sonication)	DCM: Acetone	20ml, 1 step	30	N/A	N/A	Silica	GC-ECD	Yes, 2,4,6-Tribromobiphenyl
26	2	Yes, before clean-up	Solid-Liquid (ASE)	DCM: acetone 1:1		10	100	5	None	GC-MS	Yes,phenanthrene-d10
27	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	120	N/A	N/A	None	GC-MS/MS	Yes, 8270 Internal Standard Mix

Table 58 Measurement Methods and Instrument Techniques for Chlorpyrifos

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
1	5	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	25mL, 1 step	60	Room temp	N/A	N/A	GC-MS/MS	Yes, Mixed internal standard
2	10	Yes, before clean-up	Solid-Liquid (Sonication)	Acetone/ Hexane 2:3	50mL, 1 step	2x20 minutes				GC-MS/MS	Yes, DB-DDE, DF-DDE, TCMX, TPP
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	GC-MS/MS	Phenthrene-d10
5	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: ACETONE	20mL 1 step	120	n/a	n/a	none	GC-MS	Deuterated PAH
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	GC-MS	yes, chlorpyrifos D10
7	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	20 mL, 1 Step	60	NA	NA	NA	GC-MS	Yes, various
8	5	No	Solid-Liquid	Acetonitrile	10 mL, 1 step	240	40	10	PSA,MgSO 4	GC/FPD	No
11	5	Yes, before clean-up	Solid-Liquid (Sonication)	Acetonitrile	15mL, 1 step	120	NA	NA	QuEChERS (dSPE)	LC-MS/MS	NO
12	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20 mL, 1 step	60	Room Temperature	N/A	N/A	GC-MS	Yes, d10-Phenanthrene

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
13	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1:1 DCM/ Acetone	20mL	60 min	n/a	n/a	n/a	GC-MS	EPA 8270 Semivolatile Internal Standard Mix
14	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	60				GC-MS/MS	Yes, 1,4-Dichlorobenzene-D4 and Bromophos-ethyl
15	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone: Methanol	20 mL, 1 step	120	N/A	N/A	None	GC-MS	Yes, 8270 Internal Standard Mix
17	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone (1:1)	30 mL, 1 step	120	N/A	N/A	N/A	GC-MS/MS	Yes: Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12
18	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	20mL, 1 Step	60	25	0	Nil	LC-MS/MS	No
19	10	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes.Bromophos-ethyl
20	5	YES	Solid-Liquid (Mechanical Agitation)	ACETONE/ DCM	10 mL, 1 step	60			NO	GC-MS/MS	Yes, Bromonitrobenzene and Bromophos-ethyl
21	2.5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1:1 DCM: Acetone	5 mL, 1 step	30	NA	NA	No cleanup	GC-MS/MS	

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
22	20	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetone, Hexane	50 mL, 3 steps	60	40	30	Florisil clean-up	GC-MS	Yes, Naphtalene-D8
23	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10 mL, 1 step	10	No concentration	-	QuEChERS (dSPE)	GC-MS/MS	Yes, acenaphthene-d10 and chrysene-d12
24	10	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromophos-ethyl
25	8.5	Yes, no clean-up	Solid-Liquid (Sonication)	Ethyl acetate	20ml, 1 step	60	40	14	Nil	GC-MS	Yes,various ONOP internal standards
26	2	Yes, before clean-up	Solid-Liquid (ASE)	DCM: acetone 1:1		10	100	5	None	GC-MS	Yes,phenanthrene-d10
27	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	120	N/A	N/A	None	GC-MS/MS	Yes, 8270 Internal Standard Mix

Table 59 Measurement Methods and Instrument Techniques for p,p’-DDD

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
1	5	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	25mL, 1 step	60	Room temp	N/A	N/A	GC-MS/MS	Yes, Mixed internal standard

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
2	10	Yes, before clean-up	Solid-Liquid (Sonication)	Acetone/ Hexane 2:3	50mL, 1 step	2x20 minutes				GC-ECD	Yes, DB-DDE, DF-DDE, TCMX, TPP
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	GC-MS/MS	Phenthrene-d10
5	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: ACETONE	20mL 1 step	120	n/a	n/a	none	GC-MS	Deuterated PAH
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	GC-MS	yes, chlorpyrifos D10
7	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	20 mL, 1 Step	60	NA	NA	NA	GC-MS	Yes, various
8	5	No	Solid-Liquid	Acetonitrile	10 mL, 1 step	240	40	10	PSA,MgSO 4	GC/uECD	No
9	10	No	Solid-Liquid (Mechanical Agitation)	Acetone hexan 1/2	10/20mL	60	25	0	No	GC-MS	no
11	2	Yes, before clean-up	Solid-Liquid (Sonication)	Hex:Ace	20mL, 1 step	120	NA	NA	NA	GC-MS/MS	Yes, PBB7 & PBB18
12	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20 mL, 1 step	60	Room Temperature	N/A	N/A	GC-MS	Yes, d12-Chrysene

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
13	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1:1 DCM/ Acetone	20mL	60 min	n/a	n/a	n/a	GC-MS	EPA 8270 Semivolatile Internal Standard Mix
14	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	60				GC-MS/MS	Yes, 1,4-Dichlorobenzene-D4 and Bromophos-ethyl
15	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone: Methanol	20 mL, 1 step	120	N/A	N/A	None	GC-MS	Yes, 8270 Internal Standard Mix
17	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone (1:1)	30 mL, 1 step	120	N/A	N/A	N/A	GC-MS/MS	Yes: Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12
18	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ ACETONE	30mL, 1 step	60				GC-MS	Yes- Semivolatile internal Standard
19	10	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromonitrobenzene
20	5	YES	Solid-Liquid (Mechanical Agitation)	ACETONE/ DCM	10 mL, 1 step	60			NO	GC-MS/MS	Yes, Bromonitrobenzene and Bromophos-ethyl
21	2.5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1:1 DCM: Acetone	5 mL, 1 step	30	NA	NA	No cleanup	GC-MS/MS	Yes, Phenanthrene-d10

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
22	20	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetone, Hexane	50 mL, 3 steps	60	40	30	Florisil clean-up	GC-MS	Yes, Naphtalene-D8
23	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10 mL, 1 step	10	No concentration	-	QuEChERS (dSPE)	GC-MS/MS	Yes, acenaphthene-d10 and chrysene-d12
24	10	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromonitrobenzene
25	10	Yes, before clean-up	Solid-Liquid (Sonication)	DCM: Acetone	20ml, 1 step	30	N/A	N/A	Silica	GC-ECD	Yes, 2,4,6-Tribromobiphenyl
26	2	Yes, before clean-up	Solid-Liquid (ASE)	DCM: acetone 1:1		10	100	5	None	GC-MS	Yes,phenanthrene-d10
27	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	120	N/A	N/A	None	GC-MS/MS	Yes, 8270 Internal Standard Mix

Table 60 Measurement Methods and Instrument Techniques for Dieldrin

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
1	5	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	25mL, 1 step	60	Room temp	N/A	N/A	GC-MS/MS	Yes, Mixed internal standard

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
2	10	Yes, before clean-up	Solid-Liquid (Sonication)	Acetone/ Hexane 2:3	50mL, 1 step	2x20 minutes				GC-ECD	Yes, DB-DDE, DF-DDE, TCMX, TPP
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	GC-MS/MS	Phenthrene-d10
5	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: ACETONE	20mL 1 step	120	n/a	n/a	none	GC-MS	Deuterated PAH
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	GC-MS	yes, chlorpyrifos D10
7	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	20 mL, 1 Step	60	NA	NA	NA	GC-MS	Yes, various
8	5	No	Solid-Liquid	Acetonitrile	10 mL, 1 step	240	40	10	PSA,MgSO 4	GC/uECD	No
9	10	No	Solid-Liquid (Mechanical Agitation)	Acetone hexan 1/2	10/20mL	60	25	0	No	GC-MS	no
11	2	Yes, before clean-up	Solid-Liquid (Sonication)	Hex:Ace	20mL, 1 step	120	NA	NA	NA	GC-MS/MS	Yes, PBB7 & PBB18
12	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20 mL, 1 step	60	Room Temperature	N/A	N/A	GC-MS	Yes, d12-Chrysene

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
13	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1:1 DCM/ Acetone	20mL	60 min	n/a	n/a	n/a	GC-MS	EPA 8270 Semivolatile Internal Standard Mix
14	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	60				GC-MS/MS	Yes, 1,4-Dichlorobenzene-D4 and Bromophos-ethyl
15	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone: Methanol	20 mL, 1 step	120	N/A	N/A	None	GC-MS	Yes, 8270 Internal Standard Mix
17	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone (1:1)	30 mL, 1 step	120	N/A	N/A	N/A	GC-MS/MS	Yes: Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12
18	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ ACETONE	30mL, 1 step	60				GC-MS	Yes- Semivolatile internal Standard
19	10	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromonitrobenzene
20	5	YES	Solid-Liquid (Mechanical Agitation)	ACETONE/ DCM	10 mL, 1 step	60			NO	GC-MS/MS	Yes, Bromonitrobenzene and Bromophos-ethyl
21	2.5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1:1 DCM: Acetone	5 mL, 1 step	30	NA	NA	No cleanup	GC-MS/MS	Yes, Phenanthrene-d10

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
22	20	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetone, Hexane	50 mL, 3 steps	60	40	30	Florisil clean-up	GC-MS	Yes, Naphtalene-D8
23	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10 mL, 1 step	10	No concentration	-	QuEChERS (dSPE)	GC-MS/MS	Yes, acenaphthene-d10 and chrysene-d12
24	10	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromonitrobenzene
25	8.5	Yes, no clean-up	Solid-Liquid (Sonication)	Ethyl acetate	20ml, 1 step	60	40	14	Nil	GC-MS	Yes,various ONOP internal standards
26	2	Yes, before clean-up	Solid-Liquid (ASE)	DCM: acetone 1:1		10	100	5	None	GC-MS	Yes,phenanthrene-d10
27	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	120	N/A	N/A	None	GC-MS/MS	Yes, 8270 Internal Standard Mix

Table 61 Measurement Methods and Instrument Techniques for Endosulfan Sulfate

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
1	5	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	25mL, 1 step	60	Room temp	N/A	N/A	GC-MS/MS	Yes, Mixed internal standard

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
2	10	Yes, before clean-up	Solid-Liquid (Sonication)	Acetone/ Hexane 2:3	50mL, 1 step	2x20 minutes				GC-ECD	Yes, DB-DDE, DF-DDE, TCMX, TPP
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	GC-MS/MS	Phenthrene-d10
5	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: ACETONE	20mL 1 step	120	n/a	n/a	none	GC-MS	Deuterated PAH
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	GC-MS	yes, chlorpyrifos D10
7	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	20 mL, 1 Step	60	NA	NA	NA	GC-MS	Yes, various
8	5	No	Solid-Liquid	Acetonitrile	10 mL, 1 step	240	40	10	PSA,MgSO 4	GC/uECD	No
11	2	Yes, before clean-up	Solid-Liquid (Sonication)	Hex:Ace	20mL, 1 step	120	NA	NA	NA	GC-MS/MS	Yes, PBB7 & PBB18
12	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20 mL, 1 step	60	Room Temperature	N/A	N/A	GC-MS	Yes, d12-Chrysene
13	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1:1 DCM/ Acetone	20mL	60 min	n/a	n/a	n/a	GC-MS	EPA 8270 Semivolatile Internal Standard Mix

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
14	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	60				GC-MS/MS	Yes, 1,4-Dichlorobenzene-D4 and Bromophos-ethyl
15	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone: Methanol	20 mL, 1 step	120	N/A	N/A	None	GC-MS	Yes, 8270 Internal Standard Mix
17	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone (1:1)	30 mL, 1 step	120	N/A	N/A	N/A	GC-MS/MS	Yes: Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12
18	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ ACETONE	30mL, 1 step	60				GC-MS	Yes- Semivolatile internal Standard
19	10	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromonitrobenzene
20	5	YES	Solid-Liquid (Mechanical Agitation)	ACETONE/ DCM	10 mL, 1 step	60			NO	GC-MS/MS	Yes, Bromonitrobenzene and Bromophos-ethyl
21	2.5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1:1 DCM: Acetone	5 mL, 1 step	30	NA	NA	No cleanup	GC-MS/MS	Yes, Phenanthrene-d10
22	20	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetone, Hexane	50 mL, 3 steps	60	40	30	Florisil clean-up	GC-MS	Yes, Naphtalene-D8

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
23	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10 mL, 1 step	10	No concentration	-	QuEChERS (dSPE)	GC-MS/MS	Yes, acenaphthene-d10 and chrysene-d12
24	10	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromonitrobenzene
25	10	Yes, before clean-up	Solid-Liquid (Sonication)	DCM: Acetone	20ml, 1 step	30	N/A	N/A	Silica	GC-ECD	Yes, 2,4,6-Tribromobiphenyl
26	2	Yes, before clean-up	Solid-Liquid (ASE)	DCM: acetone 1:1		10	100	5	None	GC-MS	Yes,phenanthrene-d10
27	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	120	N/A	N/A	None	GC-MS/MS	Yes, 8270 Internal Standard Mix

Table 62 Measurement Methods and Instrument Techniques for Ethion

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
1	5	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	25mL, 1 step	60	Room temp	N/A	N/A	GC-MS/MS	Yes, Mixed internal standard
2	10	Yes, before clean-up	Solid-Liquid (Sonication)	Acetone/ Hexane 2:3	50mL, 1 step	2x20 minutes				GC-MS/MS	Yes, DB-DDE, DF-DDE, TCMX, TPP

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	GC-MS/MS	Phenthrene-d10
5	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: ACETONE	20mL 1 step	120	n/a	n/a	none	GC-MS	Deuterated PAH
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	GC-MS	yes, chlorpyrifos D10
7	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	20 mL, 1 Step	60	NA	NA	NA	GC-MS	Yes, various
8	5	No	Solid-Liquid	Acetonitrile	10 mL, 1 step	240	40	10	PSA,MgSO 4	GC/FPD	No
12	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20 mL, 1 step	60	Room Temperature	N/A	N/A	GC-MS	Yes, d12-Chrysene
13	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1:1 DCM/ Acetone	20mL	60 min	n/a	n/a	n/a	GC-MS	EPA 8270 Semivolatile Internal Standard Mix
14	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	60				GC-MS/MS	Yes, 1,4-Dichlorobenzene-D4 and Bromophos-ethyl
15	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone: Methanol	20 mL, 1 step	120	N/A	N/A	None	GC-MS	Yes, 8270 Internal Standard Mix

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
17	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone (1:1)	30 mL, 1 step	120	N/A	N/A	N/A	GC-MS/MS	Yes: Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12
18	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ ACETONE	30mL, 1 step	60				GC-MS	Yes- Semivolatile internal Standard
19	10	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromophos-ethyl
20	5	YES	Solid-Liquid (Mechanical Agitation)	ACETONE/ DCM	10 mL, 1 step	60			NO	GC-MS/MS	Yes, Bromonitrobenzene and Bromophos-ethyl
22	20	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetone, Hexane	50 mL, 3 steps	60	40	30	Florisil clean-up	GC-MS	Yes, Naphtalene-D8
23	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10 mL, 1 step	10	No concentration	-	QuEChERS (dSPE)	GC-MS/MS	Yes, acenaphthene-d10 and chrysene-d12
24	10	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromophos-ethyl
25	8.5	Yes, no clean-up	Solid-Liquid (Sonication)	Ethyl acetate	20ml, 1 step	60	40	14	Nil	GC-MS	Yes,various ONOP internal standards
27	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	120	N/A	N/A	None	GC-MS/MS	Yes, 8270 Internal Standard Mix

Table 63 Measurement Methods and Instrument Techniques for Fenitrothion

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
1	5	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	25mL, 1 step	60	Room temp	N/A	N/A	GC-MS/MS	Yes, Mixed internal standard
2	10	Yes, before clean-up	Solid-Liquid (Sonication)	Acetone/ Hexane 2:3	50mL, 1 step	2x20 minutes				GC-MS/MS	Yes, DB-DDE, DF-DDE, TCMX, TPP
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	GC-MS/MS	Phenthrene-d10
5	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: ACETONE	20mL 1 step	120	n/a	n/a	none	GC-MS	Deuterated PAH
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	GC-MS	yes, chlorpyrifos D10
8	5	No	Solid-Liquid	Acetonitrile	10 mL, 1 step	240	40	10	PSA,MgSO 4	GC/FPD	No
12	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20 mL, 1 step	60	Room Temperature	N/A	N/A	GC-MS	Yes, d10-Phenanthrene
14	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	60				GC-MS/MS	Yes, 1,4-Dichlorobenzene-D4 and Bromophos-ethyl

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
15	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone: Methanol	20 mL, 1 step	120	N/A	N/A	None	GC-MS	Yes, 8270 Internal Standard Mix
17	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone (1:1)	30 mL, 1 step	120	N/A	N/A	N/A	GC-MS/MS	Yes: Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12
19	10	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromophos-ethyl
20	5	YES	Solid-Liquid (Mechanical Agitation)	ACETONE/ DCM	10 mL, 1 step	60			NO	GC-MS/MS	Yes, Bromonitrobenzene and Bromophos-ethyl
21	2.5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1:1 DCM: Acetone	5 mL, 1 step	30	NA	NA	No cleanup	GC-MS/MS	Yes, Phenanthrene-d10
22	20	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetone, Hexane	50 mL, 3 steps	60	40	30	Florisil clean-up	GC-MS	Yes, Naphtalene-D8
23	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10 mL, 1 step	10	No concentration	-	QuEChERS (dSPE)	GC-MS/MS	Yes, acenaphthene-d10 and chrysene-d12
24	10	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromophos-ethyl

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
25	8.5	Yes, no clean-up	Solid-Liquid (Sonication)	Ethyl acetate	20ml, 1 step	60	40	14	Nil	GC-MS	Yes,various ONOP internal standards
27	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	120	N/A	N/A	None	GC-MS/MS	Yes, 8270 Internal Standard Mix

Table 64 Measurement Methods and Instrument Techniques for Fenvalerate

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
1	5	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	25mL, 1 step	60	Room temp	N/A	N/A	GC-MS/MS	Yes, Mixed internal standard
2	10	Yes, before clean-up	Solid-Liquid (Sonication)	Acetone/ Hexane 2:3	50mL, 1 step	2x20 minutes				GC-MS/MS	Yes, DB-DDE, DF-DDE, TCMX, TPP
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	GC-MS/MS	Phenthrene-d10
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	GC-MS	yes, chlorpyrifos D10
8	5	No	Solid-Liquid	Acetonitrile	10 mL, 1 step	240	40	10	PSA,MgSO 4	GC/uECD	No

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
11	5	Yes, before clean-up	Solid-Liquid (Sonication)	Acetonitrile	15mL, 1 step	120	NA	NA	QuEChERS (dSPE)	LC-MS/MS	NO
20	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10 mL, 1 step	60			NO	LC-UV	NO
23	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10 mL, 1 step	10	No concentration	-	QuEChERS (dSPE)	GC-MS/MS	Yes, acenaphthene-d10 and chrysene-d12
25	8.5	Yes, no clean-up	Solid-Liquid (Sonication)	Ethyl acetate	20ml, 1 step	60	40	14	Nil	GC-MS	Yes,various ONOP internal standards

Table 65 Measurement Methods and Instrument Techniques for Lindane

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
1	5	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	25mL, 1 step	60	Room temp	N/A	N/A	GC-MS/MS	Yes, Mixed internal standard
2	10	Yes, before clean-up	Solid-Liquid (Sonication)	Acetone/ Hexane 2:3	50mL, 1 step	2x20 minutes				GC-ECD	Yes, DB-DDE, DF-DDE, TCMX, TPP
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	GC-MS/MS	Phenthrene-d10

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
5	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: ACETONE	20mL 1 step	120	n/a	n/a	none	GC-MS	Deuterated PAH
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	GC-MS	yes, chlorpyrifos D10
7	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	20 mL, 1 Step	60	NA	NA	NA	GC-MS	Yes, various
8	5	No	Solid-Liquid	Acetonitrile	10 mL, 1 step	240	40	10	PSA,MgSO 4	GC/uECD	No
9	10	No	Solid-Liquid (Mechanical Agitation)	Acetone hexan 1/2	10/20mL	60	25	0	No	GC-MS	no
11	2	Yes, before clean-up	Solid-Liquid (Sonication)	Hex:Ace	20mL, 1 step	120	NA	NA	NA	GC-MS/MS	Yes, PBB7 & PBB18
12	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20 mL, 1 step	60	Room Temperature	N/A	N/A	GC-MS	Yes, d10-Phenanthrene
13	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1:1 DCM/ Acetone	20mL	60 min	n/a	n/a	n/a	GC-MS	EPA 8270 Semivolatile Internal Standard Mix
14	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	60				GC-MS/MS	Yes, 1,4-Dichlorobenzene-D4 and Bromophos-ethyl

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
15	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone: Methanol	20 mL, 1 step	120	N/A	N/A	None	GC-MS	Yes, 8270 Internal Standard Mix
17	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone (1:1)	30 mL, 1 step	120	N/A	N/A	N/A	GC-MS/MS	Yes: Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12
18	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ ACETONE	30mL, 1 step	60				GC-MS	Yes- Semivolatile internal Standard
19	10	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromonitrobenzene
20	5	YES	Solid-Liquid (Mechanical Agitation)	ACETONE/ DCM	10 mL, 1 step	60			NO	GC-MS/MS	Yes, Bromonitrobenzene and Bromophos-ethyl
21	2.5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1:1 DCM: Acetone	5 mL, 1 step	30	NA	NA	No cleanup	GC-MS/MS	Yes, Phenanthrene-d10
22	20	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetone, Hexane	50 mL, 3 steps	60	40	30	Florisil clean-up	GC-MS	Yes, Naphtalene-D8
23	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10 mL, 1 step	10	No concentration	-	QuEChERS (dSPE)	GC-MS/MS	Yes, acenaphthene-d10 and chrysene-d12

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
24	10	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromonitrobenzene
25	10	Yes, before clean-up	Solid-Liquid (Sonication)	DCM: Acetone	20ml, 1 step	30	N/A	N/A	Silica	GC-ECD	Yes, 2,4,6-Tribromobiphenyl
26	2	Yes, before clean-up	Solid-Liquid (ASE)	DCM: acetone 1:1		10	100	5	None	GC-MS	Yes,phenanthrene-d10
27	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	120	N/A	N/A	None	GC-MS/MS	Yes, 8270 Internal Standard Mix

Table 66 Measurement Methods and Instrument Techniques for Trifluralin

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
1	5	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	25mL, 1 step	60	Room temp	N/A	N/A	GC-MS/MS	Yes, Mixed internal standard
2	10	Yes, before clean-up	Solid-Liquid (Sonication)	Acetone/ Hexane 2:3	50mL, 1 step	2x20 minutes				GC-MS/MS	Yes, DB-DDE, DF-DDE, TCMX, TPP
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	GC-MS/MS	Phenthrene-d10

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction Technique	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
5	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: ACETONE	20mL 1 step	120	n/a	n/a	none	GC-MS	Deuterated PAH
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	GC-MS	yes, chlorpyrifos D10
15	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone: Methanol	20 mL, 1 step	120	N/A	N/A	None	GC-MS	Yes, 8270 Internal Standard Mix
17	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone (1:1)	30 mL, 1 step	120	N/A	N/A	N/A	GC-MS/MS	Yes: Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12
20	5	YES	Solid-Liquid (Mechanical Agitation)	ACETONE/ DCM	10 mL, 1 step	60			NO	GC-MS/MS	Yes, Bromonitrobenzene and Bromophos-ethyl
22	20	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetone, Hexane	50 mL, 3 steps	60	40	30	Florisil clean-up	GC-MS	Yes, Naphtalene-D8
25	8.5	Yes, no clean-up	Solid-Liquid (Sonication)	Ethyl acetate	20ml, 1 step	60	40	14	Nil	GC-MS	Yes,various ONOP internal standards
26	2	Yes, before clean-up	Solid-Liquid (ASE)	DCM: acetone 1:1		10	100	5	None	GC-MS	Yes,phenanthrene-d10
27	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	120	N/A	N/A	None	GC-MS/MS	Yes, 8270 Internal Standard Mix

Table 67 Sample S1 Measurement Methods and Instrument Techniques Additional Information

Lab. Code	Additional Information
6	Results reported <0.02mg/kg is reported below laboratory LOR.
25	Trans-Chlordane reported. It is noted that the surrogate was added before the extraction solvent was added for the above methods.

A4.2 Sample S2 Methodology

Table 68 Measurement Methods and Instrument Techniques for Acephate

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
1	5	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM:Acetone	25mL, 1 step	60	Room temp	N/A	N/A	GC-MS/MS	Yes, Mixed internal standard
2	1	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	20% MeOH in Water	10mL, 1 step	1 hour			Filtration	LC-MS/MS	Carbofuran d3, Diuron-d6, isoproturon-d6, imidacloprid-d4, pirimicarb-d6,
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	LC-MS/MS	Triphenyl phosphate
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	LC-MS	yes, pirimicarb D6
8	5	No	Solid-Liquid	Acetonitrile	10 mL, 1 step	240	40	10	PSA,MgSO4	GC/FPD	No
11	5	Yes, before clean-up	Solid-Liquid (Sonication)	Acetonitrile	15mL, 1 step	120	NA	NA	QuEChERS (dSPE)	LC-MS/MS	NO
26	2	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	acetonitrile	10 ml,2 steps	5	25	5	QuEChERS (dSPE)	LC-MS/MS	atrazine-d10,diazinon-d10,diuron-d6,dichlorvos-d5

Table 69 Measurement Methods and Instrument Techniques for Bromacil

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
1	5	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	25mL, 1 step	60	Room temp	N/A	N/A	GC-MS/MS	Yes, Mixed internal standard
2	1	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	20% MeOH in Water	10mL, 1 step	1 hour			Filtration	LC-MS/MS	Carbofuran d3, Diuron-d6, isoproturon-d6, imidacloprid-d4, pirimicarb-d6,
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	GC-MS/MS	Phenthrene-d10
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	LC-MS	yes, pirimicarb D6
9	5	no	Solid-Liquid (Mechanical Agitation)	MeOH/ACN	10ml, 1 step	60	20	0		LCMSMS	no
11	5	Yes, before clean-up	Solid-Liquid (Sonication)	Acetonitrile	15mL, 1 step	120	NA	NA	QuEChERS (dSPE)	LC-MS/MS	NO
15	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone: Methanol	20 mL, 1 step	120	N/A	N/A	None	GC-MS	Yes, 8270 Internal Standard Mix
18	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	20mL, 1 Step	60	25	0	Nil	LC-MS/MS	No

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
22	20	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL,1 step	10	40	30	QuEChERS (dSPE)	LC-MS/MS	Yes, Atrazine D5, 2,4,5-T 13C6
25	8.5	Yes, no clean-up	Solid-Liquid (Sonication)	Ethyl acetate	20ml, 1 step	60	40	14	Nil	GC-MS	Yes,various ONOP internal standards
26	2	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	acetonitrile	10 ml,2 steps	5	25	5	QuEChERS (dSPE)	LC-MS/MS	atrazine-d10,diazinon-d10,diuron-d6,dichlorvos-d5
27	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	120	N/A	N/A	None	GC-MS/MS	Yes, 8270 Internal Standard Mix

Table 70 Measurement Methods and Instrument Techniques for Carbendazim

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
1	2	No	Solid-Liquid (Mechanical Agitation)	50:50 Methanol: H2O	10mL, 1 step	120	Room temp	N/A	Filtration	LC-MS/MS	No
2	1	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	20% MeOH in Water	10mL, 1 step	1 hour			Filtration	LC-MS/MS	Carbofuran d3, Diuron-d6, isoproturon-d6, imidacloprid-d4, pirimicarb-d6,

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	LC-MS/MS	Triphenyl phosphate
5	2.5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Aqeous	5mL, 1 step	60	n/a	n/a	Filtration	LC-MS/MS	Isotope labelled
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	LC-MS	yes, pirimicarb D6
11	5	Yes, before clean-up	Solid-Liquid (Sonication)	Acetonitrile	15mL, 1 step	120	NA	NA	QuEChERS (dSPE)	LC-MS/MS	NO

Table 71 Measurement Methods and Instrument Techniques for 2,4-D

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
1	2	No	Solid-Liquid (Mechanical Agitation)	50:50 Methanol: H2O	10mL, 1 step	120	Room temp	N/A	Filtration	LC-MS/MS	No
3	2	No	Solid-Liquid (Mechanical Agitation)	10% NH4OH ACN: MeOH	10mL, 1 step	60	21	N/A	N/A	LC-MS/MS	Yes, 2,4-D 13C6

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
5	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: ACETONE	20mL 1 step	120	n/a	n/a	Derivitizatio n	GC-MS	Isotope labelled
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	LC-MS	yes, pirimicarb D6
11	2	Yes, before clean-up	Solid-Liquid (Sonication)	Methanol	20mL, 1 step	120	NA	NA	NA	LC-MS/MS	Yes, Trichlorophenylaceti c acid-13C6, D2-Clopyralid, Pentachlorophenol-13C6 & Dichorprop-13C6
15	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone: Methanol	20 mL, 1 step	120	N/A	N/A	Derivatisatio n	GC-MS	Yes, 8270 Internal Standard Mix
18	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	0.1M NaOH	20mL, 1 Step	60	25	30	SPE (Reverse Phase)	LC-MS	No
20	5	YES	Solid-Liquid (Mechanical Agitation)	Methanol	10 mL, 1 step	60			NO	LC-UV	NO
22	20	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL,1 step	10	40	30	QuEChERS (dSPE)	LC-MS/MS	Yes, Atrazine D5, 2,4,5-T 13C6
25	5	Yes, no clean-up	Solid-Liquid (Sonication)	MeOH: Formic acid	18ml, 1 step	60	N/A	N/A	Nil	LC-MS/MS	No

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
26	2	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1% formic acid/ACN	10 ml,2 step	5	25	5	Dilute water	LC-MS/MS	2,4-D labelled
27	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Basified Methanol	10mL, 1 step	60	N/A	N/A	None	LC-MS/MS	Yes, 2,4,5-T D4, Mecoprop D3 , Bentazone D6 , 2,4-D-d3 , Dichloroprop D6

Table 72 Measurement Methods and Instrument Techniques for Fipronil

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
1	2	No	Solid-Liquid (Mechanical Agitation)	50:50 Methanol: H2O	10mL, 1 step	120	Room temp	N/A	Filtration	LC-MS/MS	No
2	1	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	20% MeOH in Water	10mL, 1 step	1 hour			Filtration	LC-MS/MS	Carbofuran d3, Diuron-d6, isoproturon-d6, imidacloprid-d4, pirimicarb-d6,
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	GC-MS/MS	Phenthrene-d10

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
5	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: ACETONE	20mL 1 step	120	n/a	n/a	none	GC-MS	Deuterated PAH
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	LC-MS	yes, pirimicarb D6
14	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	60				GC-MS/MS	Yes, 1,4-Dichlorobenzene-D4 and Bromophos-ethyl
17	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone (1:1)	30 mL, 1 step	120	N/A	N/A	N/A	GC-MS/MS	Yes: Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12

Table 73 Measurement Methods and Instrument Techniques for Glyphosate

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
1	2.5	No	Solid-Liquid (Mechanical Agitation)	5ml 0.6M KOH, 1 mL of extact + 4mL DI H2O, 0.5 mL of diluted	10mL, 3 steps	180	Room temp	N/A	Filtration	LC-MS	Yes, Glyphosate 2-13C

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
				extract + 0.5mL 0.1M Borate Bufer + 0.5 mL of 10,000 mg/L FMOC-Cl solution							
2	0.5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	0.1M HCl	60mL, 1 step				Filtration	LC-MS/MS	Gly-13C215N and AMPA-13C15ND2
5	1	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Aqeous	4mL, 1 Step	50	n/a	n/a	Filtration	LC-MS/MS	Isotope labelled
6	5	No	Solid-Liquid (Mechanical Agitation)	MeOH and Acetonitrile	20mL, 1step	10	NA	NA	Filtration	LC-MS	Glyphosate 13C15N
14	5	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	Ammonium Hydroxide	10mL, 1 step	30			Filtration	LC-MS/MS	Yes, 1,2-13-C2 15N-Glyphosate and 13C-15N-AMPA
18	2	No	Solid-Liquid (Mechanical Agitation)	Water	20mL, 1 Step	60	25	0	Nil	LC-MS/MS	No
22	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	water	10mL, 1 step	10			Filtration	LC-MS/MS	Yes, 13C 15N AMPA, 13C 15N Glyphosate

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
26	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	6N KOH	10 ml,2 steps	5	25	5	ph to 7, dilute water	LC-MS/MS	13C2,15N-glyphosate

Table 74 Measurement Methods and Instrument Techniques for Imidacloprid

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
1	2	No	Solid-Liquid (Mechanical Agitation)	50:50 Methanol: H2O	10mL, 1 step	120	Room temp	N/A	Filtration	LC-MS/MS	No
2	1	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	20% MeOH in Water	10mL, 1 step	1 hour			Filtration	LC-MS/MS	Carbofuran d3, Diuron-d6, isoproturon-d6, imidacloprid-d4, pirimicarb-d6,
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	LC-MS/MS	Triphenyl phosphate
5	2.5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Aqeous	5mL, 1 step	60	n/a	n/a	Filtration	LC-MS/MS	Isotope labelled
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	LC-MS	yes, pirimicarb D6

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
11	5	Yes, before clean-up	Solid-Liquid (Sonication)	Acetonitrile	15mL, 1 step	120	NA	NA	QuEChERS (dSPE)	LC-MS/MS	NO
22	20	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL,1 step	10	40	30	QuEChERS (dSPE)	LC-MS/MS	Yes, Atrazine D5, 2,4,5-T 13C6
26	2	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	acetonitrile	10 ml,2 steps	5	25	5	QuEChERS (dSPE)	LC-MS/MS	atrazine-d10,diazinon-d10,diuron-d6,dichlorvos-d5

Table 75 Measurement Methods and Instrument Techniques for MCPA

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
1	2	No	Solid-Liquid (Mechanical Agitation)	50:50 Methanol: H2O	10mL, 1 step	120	Room temp	N/A	Filtration	LC-MS/MS	No
5	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: ACETONE	20mL 1 step	120	n/a	n/a	Derivitizatio n	GC-MS	Isotope labelled
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	LC-MS	yes, pirimicarb D6
9	5	no	Solid-Liquid (Mechanical Agitation)	MeOH/ACN	10ml, 1 step	60	20			LCMSMS	no

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
11	2	Yes, before clean-up	Solid-Liquid (Sonication)	Methanol	20mL, 1 step	120	NA	NA	NA	LC-MS/MS	Yes, Trichlorophenylaceti c acid-13C6, D2-Clopyralid, Pentachlorophenol-13C6 & Dichorprop-13C6
15	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone: Methanol	20 mL, 1 step	120	N/A	N/A	Derivatisatio n	GC-MS	Yes, 8270 Internal Standard Mix
18	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	0.1M NaOH	20mL, 1 Step	60	25	30	SPE (Reverse Phase)	LC-MS	No
20	5	YES	Solid-Liquid (Mechanical Agitation)	Methanol	10 mL, 1 step	60			NO	LC-UV	NO
22	20	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL,1 step	10	40	30	QuEChERS (dSPE)	LC-MS/MS	Yes, Atrazine D5, 2,4,5-T 13C6
25	5	Yes, no clean-up	Solid-Liquid (Sonication)	MeOH: Formic acid	18ml, 1 step	60	N/A	N/A	Nil	LC-MS/MS	No
26	2	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1% formic acid/ACN	10 ml,2 step	5	25	5	Dilute water	LC-MS/MS	2,4-D labelled
27	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Basified Methanol	10mL, 1 step	60	N/A	N/A	None	LC-MS/MS	Yes, 2,4,5-T D4, Mecoprop D3 , Bentazone D6 , 2,4-D-d3 , Dichloroprop D6

Table 76 Measurement Methods and Instrument Techniques for Metsulfuron-methyl

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
1	2	No	Solid-Liquid (Mechanical Agitation)	50:50 Methanol: H2O	10mL, 1 step	120	Room temp	N/A	Filtration	LC-MS/MS	No
2	1	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	20% MeOH in Water	10mL, 1 step	1 hour			Filtration	LC-MS/MS	Carbofuran d3, Diuron-d6, isoproturon-d6, imidacloprid-d4, pirimicarb-d6,
5	2.5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Aqeous	5mL, 1 step	60	n/a	n/a	Filtration	LC-MS/MS	Isotope labelled
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	LC-MS	yes, pirimicarb D6
20	5	YES	Solid-Liquid (Mechanical Agitation)	Methanol	10 mL, 1 step	60			NO	LC-UV	NO
22	20	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL,1 step	10	40	30	QuEChERS (dSPE)	LC-MS/MS	Yes, Atrazine D5, 2,4,5-T 13C6
26	2	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	acetonitrile	10 ml,2 steps	5	25	5	QuEChERS (dSPE)	LC-MS/MS	atrazine-d10,diazinon-d10,diuron-d6,dichlorvos-d5

Table 77 Measurement Methods and Instrument Techniques for Omethoate

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
1	5	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	25mL, 1 step	60	Room temp	N/A	N/A	GC-MS/MS	Yes, Mixed internal standard
2	1	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	20% MeOH in Water	10mL, 1 step	1 hour			Filtration	LC-MS/MS	Carbofuran d3, Diuron-d6, isoproturon-d6, imidacloprid-d4, pirimicarb-d6,
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	GC-MS/MS	Phenthrene-d10
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	LC-MS	yes, pirimicarb D6
8	5	No	Solid-Liquid	Acetonitrile	10 mL, 1 step	240	40	10	PSA,MgSO4	GC/FPD	No
11	5	Yes, before clean-up	Solid-Liquid (Sonication)	Acetonitrile	15mL, 1 step	120	NA	NA	QuEChERS (dSPE)	LC-MS/MS	NO
14	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	60				GC-MS/MS	Yes, 1,4-Dichlorobenzene-D4 and Bromophos-ethyl
18	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	20mL, 1 Step	60	25	0	Nil	LC-MS/MS	No

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
19	10	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes.Bromophos-ethyl
20	5	YES	Solid-Liquid (Mechanical Agitation)	ACETONE/ DCM	10 mL, 1 step	60			NO	GC-MS/MS	Yes, Bromonitrobenzene and Bromophos-ethyl
24	10	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes.Bromophos-ethyl
25	8.5	Yes, no clean-up	Solid-Liquid (Sonication)	Ethyl acetate	20ml, 1 step	60	40	14	Nil	GC-MS	Yes,various ONOP internal standards

Table 78 Sample S2 Measurement Methods and Instrument Techniques Additional Information

Lab. Code	Additional Information
25	It is noted that the surrogate was added before the extraction solvent was added for the above methods.

A4.3 Sample S3 Methodology

Table 79 Measurement Methods and Instrument Techniques for Acephate

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
2	1	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	20% MeOH in Water	10mL, 1 step	1 hour			Filtration	LC-MS/MS	Carbofuran d3, Diuron-d6, isoproturon-d6, imidacloprid-d4, pirimicarb-d6,
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	LC-MS/MS	Triphenyl phosphate
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	LC-MS	yes, pirimicarb D6
8	5	No	Solid-Liquid	Acetonitrile	10 mL, 1 step	240	40	10	PSA,MgSO4	GC/FPD	No
25	2.6	Yes, before clean-up	Solid-Liquid (Sonication)	MeOH: Formic acid	18ml, 1 step	60	N/A	N/A	C18 & Alumina	LC-MS/MS	No
26	2	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	acetonitrile	10 ml,2 steps	5	25	5	QuEChERS (dSPE)	LC-MS/MS	atrazine-d10,diazinon-d10,diuron-d6,dichlorvos-d5

Table 80 Measurement Methods and Instrument Techniques for Bifenthrin

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
2	10	Yes, before clean-up	Solid-Liquid (Sonication)	Acetone/ Hexane 2:3	50mL, 1 step	2x20 minutes				GC-MS/MS	Yes, DB-DDE, DF-DDE, TCMX, TPP
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	GC-MS/MS	Phenthrene-d10
5	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: ACETONE	20mL 1 step	120	n/a	n/a	none	GC-MS	Deuterated PAH
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	GC-MS	yes, chlorpyrifos D10
8	5	No	Solid-Liquid	Acetonitrile	10 mL, 1 step	240	40	10	PSA,MgSO4	GC/uECD	No
13	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1:1 DCM/ Acetone	20mL	60 min	n/a	n/a	n/a	GC-MS	EPA 8270 Semivolatile Internal Standard Mix
14	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	60				GC-MS/MS	Yes, 1,4-Dichlorobenzene-D4 and Bromophos-ethyl
15	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone: Methanol	20 mL, 1 step	120	N/A	N/A	None	GC-MS	Yes, 8270 Internal Standard Mix

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
17	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone (1:1)	30 mL, 1 step	120	N/A	N/A	N/A	GC-MS/MS	Yes: Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12
19	5	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromonitrobenzene
20	5	YES	Solid-Liquid (Mechanical Agitation)	ACETONE/ DCM	10 mL, 1 step	60			NO	GC-MS/MS	Yes, Bromonitrobenzene and Bromophos-ethyl
22	20	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetone, Hexane	50 mL, 3 steps	60	40	30	Florisil clean-up	GC-MS	Yes, Naphtalene-D8
24	5	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromonitrobenzene
25	8.5	Yes, no clean-up	Solid-Liquid (Sonication)	Ethyl acetate	20ml, 1 step	60	40	14	Nil	GC-MS	Yes,various ONOP internal standards
27	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	120	N/A	N/A	None	GC-MS/MS	Yes, 8270 Internal Standard Mix

Table 81 Measurement Methods and Instrument Techniques for Total Chlordane

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
2	10	Yes, before clean-up	Solid-Liquid (Sonication)	Acetone/ Hexane 2:3	50mL, 1 step	2x20 minutes				GC-ECD	Yes, DB-DDE, DF-DDE, TCMX, TPP
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	GC-MS/MS	Phenthrene-d10
5	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: ACETONE	20mL 1 step	120	n/a	n/a	none	GC-MS	Deuterated PAH
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	GC-MS	yes, chlorpyrifos D10
7	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	20 mL, 1 Step	60	NA	NA	NA	GC-MS	Yes, various
8	5	No	Solid-Liquid	Acetonitrile	10 mL, 1 step	240	40	10	PSA,MgSO4	GC/uECD	No
12	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20 mL, 1 step	60	Room Temperature	N/A	N/A	GC-MS	Yes, d12-Chrysene
13	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1:1 DCM/ Acetone	20mL	60 min	n/a	n/a	n/a	GC-MS	EPA 8270 Semivolatile Internal Standard Mix

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
14	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	60				GC-MS/MS	Yes, 1,4-Dichlorobenzene-D4 and Bromophos-ethyl
15	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone: Methanol	20 mL, 1 step	120	N/A	N/A	None	GC-MS	Yes, 8270 Internal Standard Mix
17	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone (1:1)	30 mL, 1 step	120	N/A	N/A	N/A	GC-MS/MS	Yes: Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12
18	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ ACETONE	30mL, 1 step	60				GC-MS	Yes- Semivolatile internal Standard
19	5	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromonitrobenzene
20	5	YES	Solid-Liquid (Mechanical Agitation)	ACETONE/ DCM	10 mL, 1 step	60			NO	GC-MS/MS	Yes, Bromonitrobenzene and Bromophos-ethyl
22	20	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetone, Hexane	50 mL, 3 steps	60	40	30	Florisil clean-up	GC-MS	Yes, Naphtalene-D8
24	5	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromonitrobenzene

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
25	10	Yes, before clean-up	Solid-Liquid (Sonication)	DCM: Acetone	20ml, 1 step	30	N/A	N/A	Silica	GC-ECD	Yes, 2,4,6-Tribromobiphenyl
26	2	Yes, before clean-up	Solid-Liquid (ASE)	DCM: acetone 1:1		10	100	5	None	GC-MS	Yes,phenanthrene-d10
27	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	120	N/A	N/A	None	GC-MS/MS	Yes, 8270 Internal Standard Mix

Table 82 Measurement Methods and Instrument Techniques for Diazinon

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
2	10	Yes, before clean-up	Solid-Liquid (Sonication)	Acetone/ Hexane 2:3	50mL, 1 step	2x20 minutes				GC-ECD	Yes, DB-DDE, DF-DDE, TCMX, TPP
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	GC-MS/MS	Phenthrene-d10
5	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: ACETONE	20mL 1 step	120	n/a	n/a	none	GC-MS	Deuterated PAH
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	LC-MS	yes, pirimicarb D6

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
7	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	20 mL, 1 Step	60	NA	NA	NA	GC-MS	Yes, various
8	5	No	Solid-Liquid	Acetonitrile	10 mL, 1 step	240	40	10	PSA,MgSO4	GC/FPD	No
12	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20 mL, 1 step	60	Room Temperature	N/A	N/A	GC-MS	Yes, d10-Phenanthrene
13	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1:1 DCM/ Acetone	20mL	60 min	n/a	n/a	n/a	GC-MS	EPA 8270 Semivolatile Internal Standard Mix
14	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	60				GC-MS/MS	Yes, 1,4-Dichlorobenzene-D4 and Bromophos-ethyl
15	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone: Methanol	20 mL, 1 step	120	N/A	N/A	None	GC-MS	Yes, 8270 Internal Standard Mix
17	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone (1:1)	30 mL, 1 step	120	N/A	N/A	N/A	GC-MS/MS	Yes: Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12
18	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ ACETONE	30mL, 1 step	60				GC-MS	Yes- Semivolatile internal Standard

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
20	5	YES	Solid-Liquid (Mechanical Agitation)	ACETONE/ DCM	10 mL, 1 step	60			NO	GC-MS/MS	Yes, Bromonitrobenzene and Bromophos-ethyl
25	8.5	Yes, no clean-up	Solid-Liquid (Sonication)	Ethyl acetate	20ml, 1 step	60	40	14	Nil	GC-MS	Yes,various ONOP internal standards
26	2	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	acetonitrile	10 ml,2 steps	5	25	5	QuEChERS (dSPE)	LC-MS/MS	atrazine-d10,diazinon-d10,diuron-d6,dichlorvos-d5
27	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	120	N/A	N/A	None	GC-MS/MS	Yes, 8270 Internal Standard Mix

Table 83 Measurement Methods and Instrument Techniques for Dieldrin

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
2	10	Yes, before clean-up	Solid-Liquid (Sonication)	Acetone/ Hexane 2:3	50mL, 1 step	2x20 minutes				GC-ECD	Yes, DB-DDE, DF-DDE, TCMX, TPP
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	GC-MS/MS	Phenthrene-d10

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
5	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: ACETONE	20mL 1 step	120	n/a	n/a	none	GC-MS	Deuterated PAH
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	GC-MS	yes, chlorpyrifos D10
7	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	20 mL, 1 Step	60	NA	NA	NA	GC-MS	Yes, various
8	5	No	Solid-Liquid	Acetonitrile	10 mL, 1 step	240	40	10	PSA,MgSO4	GC/uECD	No
12	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20 mL, 1 step	60	Room Temperature	N/A	N/A	GC-MS	Yes, d12-Chrysene
13	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1:1 DCM/ Acetone	20mL	60 min	n/a	n/a	n/a	GC-MS	EPA 8270 Semivolatile Internal Standard Mix
14	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	60				GC-MS/MS	Yes, 1,4-Dichlorobenzene-D4 and Bromophos-ethyl
15	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone: Methanol	20 mL, 1 step	120	N/A	N/A	None	GC-MS	Yes, 8270 Internal Standard Mix
17	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone (1:1)	30 mL, 1 step	120	N/A	N/A	N/A	GC-MS/MS	Yes: Naphthalene-d8, Acenaphthene-d10, Phenanthren

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
											e-d10, Chrysene-d12, Perylene-d12
18	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ ACETONE	30mL, 1 step	60				GC-MS	Yes- Semivolatile internal Standard
19	5	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromonitrobenzene
20	5	YES	Solid-Liquid (Mechanical Agitation)	ACETONE/ DCM	10 mL, 1 step	60			NO	GC-MS/MS	Yes, Bromonitrobenzene and Bromophos-ethyl
22	20	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetone, Hexane	50 mL, 3 steps	60	40	30	Florisil clean-up	GC-MS	Yes, Naphtalene-D8
24	5	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromonitrobenzene
25	10	Yes, before clean-up	Solid-Liquid (Sonication)	DCM: Acetone	20ml, 1 step	30	N/A	N/A	Silica	GC-ECD	Yes, 2,4,6-Tribromobiphenyl
26	2	Yes, before clean-up	Solid-Liquid (ASE)	DCM: acetone 1:1		10	100	5	None	GC-MS	Yes,phenanthrene-d10
27	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	120	N/A	N/A	None	GC-MS/MS	Yes, 8270 Internal Standard Mix

Table 84 Measurement Methods and Instrument Techniques for Endosulfan sulfate

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
2	10	Yes, before clean-up	Solid-Liquid (Sonication)	Acetone/ Hexane 2:3	50mL, 1 step	2x20 minutes				GC-ECD	Yes, DB-DDE, DF-DDE, TCMX, TPP
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	GC-MS/MS	Phenthrene-d10
5	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: ACETONE	20mL 1 step	120	n/a	n/a	none	GC-MS	Deuterated PAH
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	GC-MS	yes, chlorpyrifos D10
7	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	20 mL, 1 Step	60	NA	NA	NA	GC-MS	Yes, various
8	5	No	Solid-Liquid	Acetonitrile	10 mL, 1 step	240	40	10	PSA,MgSO4	GC/uECD	No
12	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20 mL, 1 step	60	Room Temperature	N/A	N/A	GC-MS	Yes, d12-Chrysene
13	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1:1 DCM/ Acetone	20mL	60 min	n/a	n/a	n/a	GC-MS	EPA 8270 Semivolatile Internal Standard Mix

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
14	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	60				GC-MS/MS	Yes, 1,4-Dichlorobenzene-D4 and Bromophos-ethyl
15	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone: Methanol	20 mL, 1 step	120	N/A	N/A	None	GC-MS	Yes, 8270 Internal Standard Mix
17	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone (1:1)	30 mL, 1 step	120	N/A	N/A	N/A	GC-MS/MS	Yes: Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12
18	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ ACETONE	30mL, 1 step	60				GC-MS	Yes- Semivolatile internal Standard
19	5	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromonitrobenzene
20	5	YES	Solid-Liquid (Mechanical Agitation)	ACETONE/ DCM	10 mL, 1 step	60			NO	GC-MS/MS	Yes, Bromonitrobenzene and Bromophos-ethyl
22	20	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetone, Hexane	50 mL, 3 steps	60	40	30	Florisil clean-up	GC-MS	Yes, Naphtalene-D8
24	5	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromonitrobenzene

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
25	10	Yes, before clean-up	Solid-Liquid (Sonication)	DCM: Acetone	20ml, 1 step	30	N/A	N/A	Silica	GC-ECD	Yes, 2,4,6-Tribromobiphenyl
26	2	Yes, before clean-up	Solid-Liquid (ASE)	DCM: acetone 1:1		10	100	5	None	GC-MS	Yes,phenanthrene-d10
27	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	120	N/A	N/A	None	GC-MS/MS	Yes, 8270 Internal Standard Mix

Table 85 Measurement Methods and Instrument Techniques for Ethion

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
2	10	Yes, before clean-up	Solid-Liquid (Sonication)	Acetone/ Hexane 2:3	50mL, 1 step	2x20 minutes				GC-MS/MS	Yes, DB-DDE, DF-DDE, TCMX, TPP
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	GC-MS/MS	Phenthrene-d10
5	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: ACETONE	20mL 1 step	120	n/a	n/a	none	GC-MS	Deuterated PAH
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	GC-MS	yes, chlorpyrifos D10

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
7	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	20 mL, 1 Step	60	NA	NA	NA	GC-MS	Yes, various
8	5	No	Solid-Liquid	Acetonitrile	10 mL, 1 step	240	40	10	PSA,MgSO4	GC/FPD	No
12	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20 mL, 1 step	60	Room Temperature	N/A	N/A	GC-MS	Yes, d12-Chrysene
13	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1:1 DCM/ Acetone	20mL	60 min	n/a	n/a	n/a	GC-MS	EPA 8270 Semivolatile Internal Standard Mix
14	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	60				GC-MS/MS	Yes, 1,4-Dichlorobenzene-D4 and Bromophos-ethyl
15	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone: Methanol	20 mL, 1 step	120	N/A	N/A	None	GC-MS	Yes, 8270 Internal Standard Mix
17	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone (1:1)	30 mL, 1 step	120	N/A	N/A	N/A	GC-MS/MS	Yes: Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12
18	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ ACETONE	30mL, 1 step	60				GC-MS	Yes- Semivolatile internal Standard

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
19	5	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromonitrobenzene
20	5	YES	Solid-Liquid (Mechanical Agitation)	ACETONE/ DCM	10 mL, 1 step	60			NO	GC-MS/MS	Yes, Bromonitrobenzene and Bromophos-ethyl
22	20	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetone, Hexane	50 mL, 3 steps	60	40	30	Florisil clean-up	GC-MS	Yes, Naphtalene-D8
24	5	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromonitrobenzene
25	8.5	Yes, no clean-up	Solid-Liquid (Sonication)	Ethyl acetate	20ml, 1 step	60	40	14	Nil	GC-MS	Yes,various ONOP internal standards
27	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	120	N/A	N/A	None	GC-MS/MS	Yes, 8270 Internal Standard Mix

Table 86 Measurement Methods and Instrument Techniques for Glyphosate

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
2	0.5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	0.1M HCl	60mL, 1 step				Filtration	LC-MS/MS	Gly-13C215N and AMPA-13C15ND2
5	1	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Aqeous	4mL, 1 Step	50	n/a	n/a	Filtration	LC-MS/MS	Isotope labelled
6	5	No	Solid-Liquid (Mechanical Agitation)	MeOH and Acetonitrile	20mL, 1step	10	NA	NA	Filtration	LC-MS	Glyphosate 13C15N
14	5	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	Ammonium Hydroxide	10mL, 1 step	30			Filtration	LC-MS/MS	Yes, 1,2-13-C2 15N-Glyphosate and 13C-15N-AMPA
18	2	No	Solid-Liquid (Mechanical Agitation)	Water	20mL, 1 Step	60	25	0	Nil	LC-MS/MS	No
22	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	water	10mL, 1 step	10			Filtration	LC-MS/MS	Yes, 13C 15N AMPA, 13C 15N Glyphosate
26	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	6N KOH	10 ml,2 steps	5	25	5	ph to 7, dilute water	LC-MS/MS	13C2,15N-glyphosate

Table 87 Measurement Methods and Instrument Techniques for Imidacloprid

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
2	1	Yes, after clean-up	Solid-Liquid (Mechanical Agitation)	20% MeOH in Water	10mL, 1 step	1 hour			Filtration	LC-MS/MS	Carbofuran d3, Diuron-d6, isoproturon-d6, imidacloprid-d4, pirimicarb-d6,
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	GC-MS/MS	Phenthrene-d10
5	2.5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Aqeous	5mL, 1 step	60	n/a	n/a	Filtration	LC-MS/MS	Isotope labelled
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	LC-MS	yes, pirimicarb D6
22	20	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL,1 step	10	40	30	QuEChERS (dSPE)	LC-MS/MS	Yes, Atrazine D5, 2,4,5-T 13C6
26	2	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	acetonitrile	10 ml,2 steps	5	25	5	QuEChERS (dSPE)	LC-MS/MS	atrazine-d10,diazinon-d10,diuron-d6,dichlorvos-d5

Table 88 Measurement Methods and Instrument Techniques for Lindane

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
2	10	Yes, before clean-up	Solid-Liquid (Sonication)	Acetone/ Hexane 2:3	50mL, 1 step	2x20 minutes				GC-ECD	Yes, DB-DDE, DF-DDE, TCMX, TPP
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	GC-MS/MS	Phenthrene-d10
5	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: ACETONE	20mL 1 step	120	n/a	n/a	none	GC-MS	Deuterated PAH
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	GC-MS	yes, chlorpyrifos D10
7	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	20 mL, 1 Step	60	NA	NA	NA	GC-MS	Yes, various
8	5	No	Solid-Liquid	Acetonitrile	10 mL, 1 step	240	40	10	PSA,MgSO4	GC/uECD	No
12	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20 mL, 1 step	60	Room Temperature	N/A	N/A	GC-MS	Yes, d10-Phenanthrene
13	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1:1 DCM/ Acetone	20mL	60 min	n/a	n/a	n/a	GC-MS	EPA 8270 Semivolatile Internal Standard Mix

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
14	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	60				GC-MS/MS	Yes, 1,4-Dichlorobenzene-D4 and Bromophos-ethyl
15	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone: Methanol	20 mL, 1 step	120	N/A	N/A	None	GC-MS	Yes, 8270 Internal Standard Mix
17	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone (1:1)	30 mL, 1 step	120	N/A	N/A	N/A	GC-MS/MS	Yes: Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12
18	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ ACETONE	30mL, 1 step	60				GC-MS	Yes- Semivolatile internal Standard
19	5	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromonitrobenzene
20	5	YES	Solid-Liquid (Mechanical Agitation)	ACETONE/ DCM	10 mL, 1 step	60			NO	GC-MS/MS	Yes, Bromonitrobenzene and Bromophos-ethyl
22	20	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetone, Hexane	50 mL, 3 steps	60	40	30	Florisil clean-up	GC-MS	Yes, Naphtalene-D8
24	5	Before adding solvent	Solid-Liquid (Mechanical Agitation)	Acetone/ DCM (1:1)	20ml; 1 step	60				GC/MS/MS	Yes. Bromonitrobenzene

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
25	10	Yes, before clean-up	Solid-Liquid (Sonication)	DCM: Acetone	20ml, 1 step	30	N/A	N/A	Silica	GC-ECD	Yes, 2,4,6-Tribromobiphenyl
26	2	Yes, before clean-up	Solid-Liquid (ASE)	DCM: acetone 1:1		10	100	5	None	GC-MS	Yes,phenanthrene-d10
27	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	120	N/A	N/A	None	GC-MS/MS	Yes, 8270 Internal Standard Mix

Table 89 Measurement Methods and Instrument Techniques for Simazine

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
2	10	Yes, before clean-up	Solid-Liquid (Sonication)	Acetone/ Hexane 2:3	50mL, 1 step	2x20 minutes				GC-MS/MS	Yes, DB-DDE, DF-DDE, TCMX, TPP
4	3	Yes, before clean-up	Vortex for 1 min	Acidified ethyl acetate	15 mL in 2 steps (10 mL + 5 mL)	NA	38		PSA	GC-MS/MS	Phenthrene-d10
5	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: ACETONE	20mL 1 step	120	n/a	n/a	none	GC-MS	Deuterated PAH
6	5	No	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL , 1step	15	NA	NA	QuEChERS (dSPE)	LC-MS	yes, pirimicarb D6

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
7	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	50:50 DCM: Acetone	20 mL, 1 Step	60	NA	NA	NA	GC-MS	Yes, various
13	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	1:1 DCM/ Acetone	20mL	60 min	n/a	n/a	n/a	GC-MS	EPA 8270 Semivolatile Internal Standard Mix
15	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone: Methanol	20 mL, 1 step	120	N/A	N/A	None	GC-MS	Yes, 8270 Internal Standard Mix
17	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM: Acetone (1:1)	30 mL, 1 step	120	N/A	N/A	N/A	GC-MS/MS	Yes: Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12
18	10	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ ACETONE	30mL, 1 step	60				GC-MS	Yes- Semivolatile internal Standard
20	5	YES	Solid-Liquid (Mechanical Agitation)	ACETONE/ DCM	10 mL, 1 step	60			NO	GC-MS/MS	Yes, Bromonitrobenzene and Bromophos-ethyl
22	20	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	Acetonitrile	10mL,1 step	10	40	30	QuEChERS (dSPE)	LC-MS/MS	Yes, Atrazine D5, 2,4,5-T 13C6
25	8.5	Yes, no clean-up	Solid-Liquid (Sonication)	Ethyl acetate	20ml, 1 step	60	40	14	Nil	GC-MS	Yes,various ONOP internal standards

Lab. Code	Sample Mass for Analysis (g)	Was surrogate used? If yes, when was it added?	Extraction	Extraction Solvent	Extraction Volume and Number of steps	Extraction Time (min)	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Measurement Instrument	Were internal standards used? If yes, what internal standard?
26	2	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	acetonitrile	10 ml,2 steps	5	25	5	QuEChERS (dSPE)	LC-MS/MS	atrazine-d10,diazinon-d10,diuron-d6,dichlorvos-d5
27	5	Yes, before clean-up	Solid-Liquid (Mechanical Agitation)	DCM/ Acetone	20mL, 1 step	120	N/A	N/A	None	GC-MS/MS	Yes, 8270 Internal Standard Mix

Table 90 Sample S3 Measurement Methods and Instrument Techniques Additional Information

Lab. Code	Additional Information
6	Results reported <0.02mg/kg is reported below laboratory LOR.
18	In-house method LOR raised due to sample matrix interferences.
25	Cis and trans-Chlordane reported. It is noted that the surrogate was added before the extraction solvent was added for the above methods.
27	Sample was light, LOR raised 2x.

APPENDIX 5 ACRONYMS AND ABBREVIATIONS

ACE	Acetone
ACN	Acetonitrile
AV	Assigned Value
CITAC	Cooperation on International Traceability in Analytical Chemistry
CRM	Certified Reference Material
CV	Coefficient of Variation
2,4-D	2,4-Dichlorophenoxyacetic acid
DCM	Dichloromethane
p,*p*'-DDD	Dichlorodiphenyldichloroethane
p,*p*'-DDE	Dichlorodiphenyldichloroethylene
p,*p*'-DDT	Dichlorodiphenyltrichloroethane
Total DDT	Sum of DDD, DDE and DDT analytes
dSPE	Dispersive Solid Phase Extraction
ECD	Electron Capture Detection
EtOAc	Ethyl Acetate
FPD	Flame Photometric Detection
GC	Gas Chromatography
GUM	Guide to the expression of Uncertainty in Measurement
HEX	Hexane
HV	Homogeneity Value
IEC	International Electrotechnical Commission
ISO	International Organization for Standardization
k	Coverage Factor
LC	Liquid Chromatography
LOR	Limit Of Reporting
Max	Maximum
MCPA	2-Methyl-4-chlorophenoxyacetic acid
Md	Median
MeOH	Methanol
Min	Minimum
MS	Mass Spectrometry
MS/MS	Tandem Mass Spectrometry
MU	Measurement Uncertainty
N	Number of numeric results

NATA	National Association of Testing Authorities, Australia
NMIA	National Measurement Institute Australia
NR	Not Reported
NS	Not Supplied
NT	Not Tested
PCV	Performance Coefficient of Variation
PSA	Primary-Secondary Amine
PT	Proficiency Testing
QuEChERS	Quick, Easy, Cheap, Effective, Rugged, and Safe preparation method
Rec	Recovery
SD	Standard Deviation
SDPA	Standard Deviation for Proficiency Assessment
SI	International System of Units
SLE	Solid-Liquid Extraction
SLE (ASE)	Solid-Liquid Extraction Accelerated Solvent Extraction
SLE (MA)	Solid-Liquid Extraction Mechanical Agitation
SLE (S)	Solid-Liquid Extraction Sonication
SPE	Solid-Phase Extraction
SS	Spiked Samples
SV	Spiked Value
U	Expanded Uncertainty

END OF REPORT