Australian Government
Department of Industry, Science and Resources
National Measurement Institute

Proficiency Test Final Report AQA 23-04 Pesticides in Soil

July 2023

ACKNOWLEDGMENTS

This study was conducted by the National Measurement Institute (NMI). Support funding was provided by the Australian Government Department of Industry, Science and Resources.

I would like to thank the management and staff of the participating laboratories for supporting the study. It is only through widespread participation that we can provide an effective service to laboratories.

The assistance of the following NMI staff members in the planning, conduct and reporting of the study is acknowledged.

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SUMMARY

AQA 23-04 Pesticides in Soil commenced in February 2023. Nineteen laboratories enrolled to participate, and seventeen participants submitted results.

Two soil samples were prepared using topsoil bought from a Sydney supplier. The soil was spiked with known amounts of various pesticides (2,4-D, p,p'-DDE, p,p'-DDT and dieldrin for Sample S1, and bifenthrin, diazinon, dicamba and simazine for Sample S2).

Of a possible 153 results, a total of 114 numeric results (75%) were submitted. Three results were submitted as a 'less than' value (<*x*) or Not Reported (NR), and 36 results were submitted as Not Tested (NT).

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

Traceability: 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 the ability of participants to correctly identify environmentally significant pesticides in soil.*

Laboratories **2**, **7**, **8** and **12** reported results for all scored analytes.

Two participants did not report numeric results for spiked analytes that they tested for (total of two results). Eleven participants reported analytes that were not spiked into the test samples (total of 12 results).

- *Compare the performances of participants and assess their accuracy in the measurement of pesticides in soil.*

Of 109 *z*-scores, 90 (83%) returned $|z| \leq 2.0$, indicating a satisfactory performance.

Of 100 E_n-scores, 77 (77%) returned $|E_n| \leq 1.0$, 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.*

Participants used a wide variety of methods, and no correlation with results was evident.

- *Develop the practical application of traceability and measurement uncertainty, and provide participants with information that will be useful in assessing their uncertainty estimates.*

Of 114 numeric results, 105 (92%) were reported with an associated estimate of expanded uncertainty. The magnitude of these expanded uncertainties ranged from 6.9% to 61% of the reported value.

One participant reported an associated estimate standard uncertainty for three of their results.

- *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 NMI Proficiency Testing Program

The National Measurement Institute (NMI) 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 inter-laboratory comparison'.[1] NMI PT studies target chemical testing in areas of high public significance such as trade, environment, law enforcement and food safety. NMI offers studies in:

- pesticide residues in fruit, vegetables and herbs, soil and water;
- petroleum hydrocarbons in soil and water;
- inorganic analytes in soil, water, filters, food and pharmaceuticals;
- controlled drug assay, drugs in wipes and clandestine laboratory;
- per- and polyfluoroalkyl substances in water, soil, biota and food; and
- allergens in food.

1.2 Study Aims

The aims of the study were to:

- assess the ability of participants to correctly identify environmentally significant pesticides in soil;
- compare the performances of participants and assess their accuracy in the measurement of pesticides in soil;
- evaluate participants' methods for the measurement of pesticides in soil;
- develop the practical application of traceability and measurement uncertainty, and provide participants with information that will be useful in assessing their uncertainty estimates; 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 NMI PT studies is described in the NMI Study Protocol for Proficiency Testing.[2] The statistical methods used are described in the NMI 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]

NMI is accredited by the National Association of Testing Authorities, Australia (NATA) to ISO/IEC 17043 as a provider of PT schemes.[1] This study is within the scope of NMI's accreditation.

2 STUDY INFORMATION

2.1 Selection of Pesticides

A list of possible analytes spiked into Samples S1 and S2 is presented in Table 1. This list was also provided to participants.

Table 1 List of Possible Analytes

2,4-D	alpha-Endosulfan	Malathion
Aldrin	beta-Endosulfan	MCPA
Atrazine	Endosulfan sulfate	Metsulfuron-methyl
Bifenthrin	Ethion	p,p'-DDD
cis-Chlordane	Fenitrothion	p,p'-DDE
trans-Chlordane	Fenthion	p,p'-DDT
Total Chlordane	Fenvalerate	Total DDT
Chlorpyrifos	Fipronil	Parathion
Cyfluthrin	Glyphosate	Parathion-methyl
Cypermethrin	Heptachlor	Permethrin
Diazinon	Heptachlor epoxide	Simazine
Dicamba	Hexachlorobenzene	Tebuconazole
Dieldrin	Imidacloprid	Triclopyr
Diuron	Lindane	Trifluralin

The actual spiked pesticides for Samples S1 and S2 are presented in Table 2. 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 Environmental Protection (Assessment of Site Contamination) Measure Schedule B1 *Guideline on Investigation Levels for Soil and Groundwater*.[5]

Table 2 Spiked Values of Test Samples

Sample	Analyte	Spiked Value (mg/kg)	Uncertainty (mg/kg)[a]
S1	2,4-D	2.80	0.14
	p,p'-DDE[b]	1.20	0.06
	p,p'-DDT[b]	0.601	0.030
	Dieldrin	0.0798	0.0040
S2	Bifenthrin	0.198	0.010
	Diazinon	2.10	0.11
	Dicamba	1.10	0.06
	Simazine	1.50	0.07

[a] The uncertainty is an expanded uncertainty at approximately 95% confidence using a coverage factor of 2. It has been estimated with consideration to contributions from the gravimetric and volumetric operations involved in spiking the samples, and the purity of the pesticide reference standards. Stability was not considered in the uncertainty budget and so the expanded uncertainty relates to the mass fraction of analyte at the time of spiking.

[b] Total DDT has also been assessed in this PT study.

2.2 Study Timetable

The timetable of the study was:

Invitations sent	27/02/2023
Samples sent	4/04/2023
Results due	12/05/2023
Interim Report	22/05/2023
Preliminary Report	24/05/2023

2.3 Participation and Laboratory Code

Nineteen laboratories enrolled to participate in this study, and all participants were assigned a confidential laboratory code number for this study. Seventeen participants submitted results.

2.4 Sample Preparation

Two soil samples were prepared by spiking soil purchased from a Sydney supplier with various pesticides to obtain the mass fractions listed in Table 2. Further information on the preparation of the samples is given in Appendix 1.

2.5 Homogeneity and Stability of Test Materials

No homogeneity or stability testing was conducted for this PT study's samples. The samples were prepared, packaged, stored and dispatched using a process that has been demonstrated to produce homogeneous and stable samples in previous NMI Pesticides in Soil PT studies.

Participants' results also gave no reason to question the transport stability or homogeneity of the samples (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 68% to 104% of the spiked value, which is similar to ratios observed in previous NMI Pesticides in Soil PT studies (for example, as presented in PT Report AQA 16-04 Pesticides in Soil).[6] An assigned value was set if there was a reasonable consensus of participants' results.

2.6 Sample Storage, Dispatch and Receipt

The test samples were refrigerated at 4 °C prior to dispatch. Participants were sent 50 g spiked soil for each of Samples S1 and S2. The samples were packed in a polystyrene foam box with cooler bricks and sent by courier on 4 April 2023.

The following items were 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.

2.7 Instructions to Participants

Participants were instructed as follows:

- Quantitatively analyse the samples using your routine test method.
- 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.

- 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 (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 with NT as the result.
- Report the basis of your uncertainty estimates 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.
- Please return the completed results sheet by email (proficiency@measurement.gov.au).
- Return the completed results sheet by 1 May 2023. Late results may not be included in the study report.

The results due date was extended to 12 May 2023 due to courier delivery delays to some participants.

2.8 Interim Report and Preliminary Report

An Interim Report was emailed to all participants on 22 May 2023.

A Preliminary Report was emailed to all participants on 24 May 2023. This report included a summary of the results reported by laboratories, assigned values, performance coefficient of variations, *z*-scores and E_n-scores for each analyte in this study. The following data has been changed from the Preliminary Report in the present Final Report:

- Laboratory **13**: E_n-scores have not been determined for Sample S1 2,4-D and Sample S2 Bifenthrin results reported by this participant. This participant commented that the uncertainties for these analytes had been reported as standard uncertainties with no coverage factor provided (see Section 6.4 for further details).

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.

3.2 Basis of Participants' Measurement Uncertainty Estimates

Participants were requested to provide information about their basis of measurement uncertainty (MU). Responses received are presented in Table 3. Some responses may be modified so that the participant cannot be identified.

Table 3 Basis of Uncertainty Estimate

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
1	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis Instrument calibration	CRM Instrument calibration Recoveries of SS Standard purity	Eurachem/CITAC Guide
2	Standard uncertainty based on historical data	Duplicate analysis Instrument calibration	CRM Instrument calibration Standard purity	Eurachem/CITAC Guide
3	Bottom Up (ISO/GUM, fish bone/cause and effect diagram)			ISO/GUM
4	Top Down - precision and estimates of the method and laboratory bias	Duplicate analysis	CRM Instrument calibration	
6	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis Instrument calibration	Instrument calibration Recoveries of SS	Eurachem/CITAC Guide
7	Bottom Up (ISO/GUM, fish bone/cause and effect diagram)	Duplicate analysis	Recoveries of SS	Eurachem/CITAC Guide
8	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis Instrument calibration		Eurachem/CITAC Guide
9				
10	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS	Recoveries of SS	ISO/GUM
11	Bottom Up (ISO/GUM, fish bone/cause and effect diagram)	Control samples Instrument calibration	Instrument calibration Recoveries of SS Standard purity	ISO/GUM
12	Professional judgment	Duplicate analysis	Laboratory bias from PT studies Recoveries of SS	Nordtest Report TR537

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
13	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS	Recoveries of SS	NATA GAG Estimating and Reporting Measurement Uncertainty of Chemical Test Results
14	Bottom Up (ISO/GUM, fish bone/cause and effect diagram)		Instrument calibration	Eurachem/CITAC Guide
15	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS		ISO/GUM
16	Top Down - precision and estimates of the method and laboratory bias	Duplicate analysis Instrument calibration	CRM Recoveries of SS	ISO/GUM
17	Standard deviation of replicate analyses multiplied by 2 or 3	Standard deviation from PT studies only		Nordtest Report TR537
		Control samples Duplicate analysis	CRM Laboratory bias from PT studies Recoveries of SS	
19		Duplicate analysis Instrument calibration		ISO/GUM

* CRM = Certified Reference Material; RM = 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 4. Some comments may be modified so that the participant cannot be identified.

Table 4 Participants' Comments

Lab. Code	Sample	Participant's Comments	Study Coordinator's Response
6	S2	LOR of 2,4-D raised due to interference from the co-existing components in the soil.	
13	S1	Uncertainty for 2,4-D was stated as standard uncertainty, rather than expanded uncertainty which incorporates bias.	For this PT study, participants were instructed to report the expanded uncertainty associated with their results. As the E_n-score is calculated from the expanded uncertainty, we have not determined the E_n-scores of the analytes listed here.
	S2	Uncertainty for Bifenthrin and Dicamba were stated as standard uncertainty, rather than expanded uncertainty which incorporates bias. Diazinon was detected at 1.4 mg/kg. This value is not normally reported due to low recovery.	

4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

4.1 Results Summary

Participant results are listed in Tables 5 to 13 with the summary statistics: robust average, median, mean, number of numeric results (N), maximum (Max), minimum (Min), robust standard deviation (robust SD) and robust coefficient of variation (robust CV). Bar charts of results and performance scores are presented in Figures 2 to 10, with an example chart with interpretation guide shown in Figure 1.

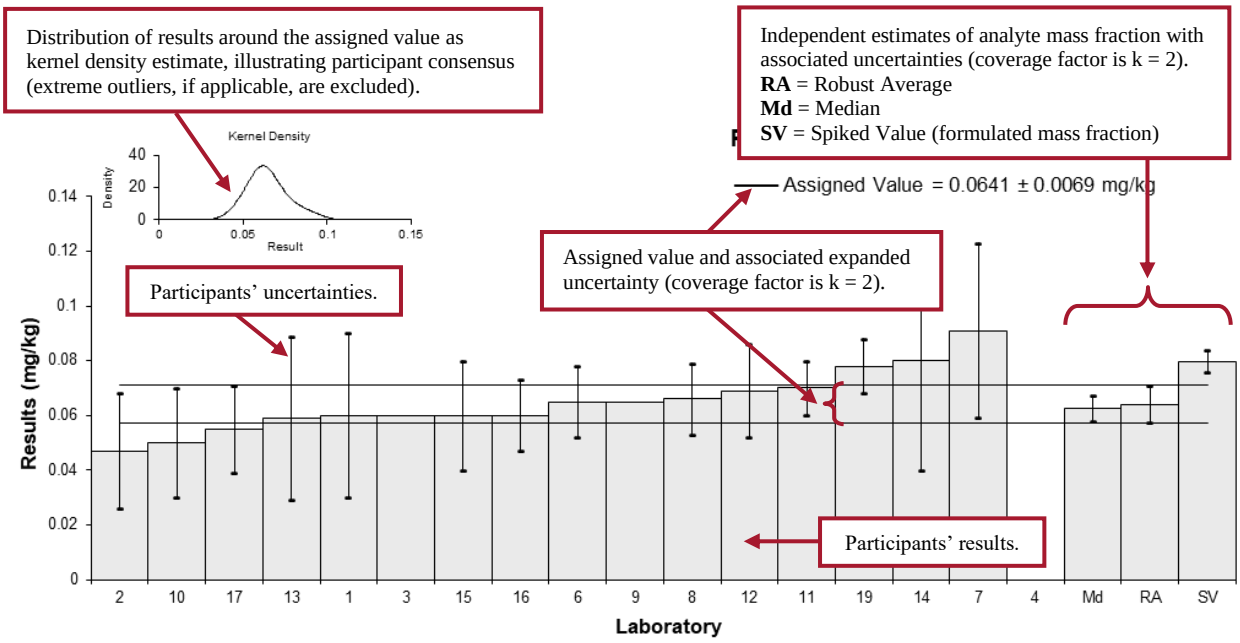


Figure 1 Guide to Presentation of Results

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.[3,4] Extreme outliers, if applicable, were obvious blunders, e.g. results with incorrect units, or for a different analyte or sample, and such results were removed before the calculation of all summary statistics.[3]

4.3 Assigned Value

The assigned value is defined as the 'value attributed to a particular property of a proficiency test item'.[1] In this PT study, the property is the mass fraction of the analytes in the samples. Assigned values were the robust averages of participants' results and the expanded uncertainties were estimated from the associated robust SDs (Appendix 3).

4.4 Robust Average and Robust Between-Laboratory Coefficient of Variation

The robust averages and associated expanded MUs, and robust CVs (a measure of the variability of participants' results) were calculated as described in ISO 13528.[7]

4.5 Performance Coefficient of Variation

The performance coefficient of variation (PCV) is a fixed 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 fixed and realistic value for the PCV, a participant's performance does not depend on other participants' performance and can be compared from study to study.

4.6 Target Standard Deviation for Proficiency Assessment

The target standard deviation for proficiency assessment (σ) 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 target standard deviation from Equation 1

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).

For the absolute value of a *z*-score:

- $|z| \leq 2.0$ is satisfactory;
- $2.0 < |z| < 3.0$ is questionable; and
- $|z| \geq 3.0$ is unsatisfactory.

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 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:

- $|E_n| \leq 1.0$ is satisfactory; and
- $|E_n| > 1.0$ is unsatisfactory.

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 5

Sample Details

Sample No.	S1
Matrix	Soil
Analyte	2,4-D
Unit	mg/kg

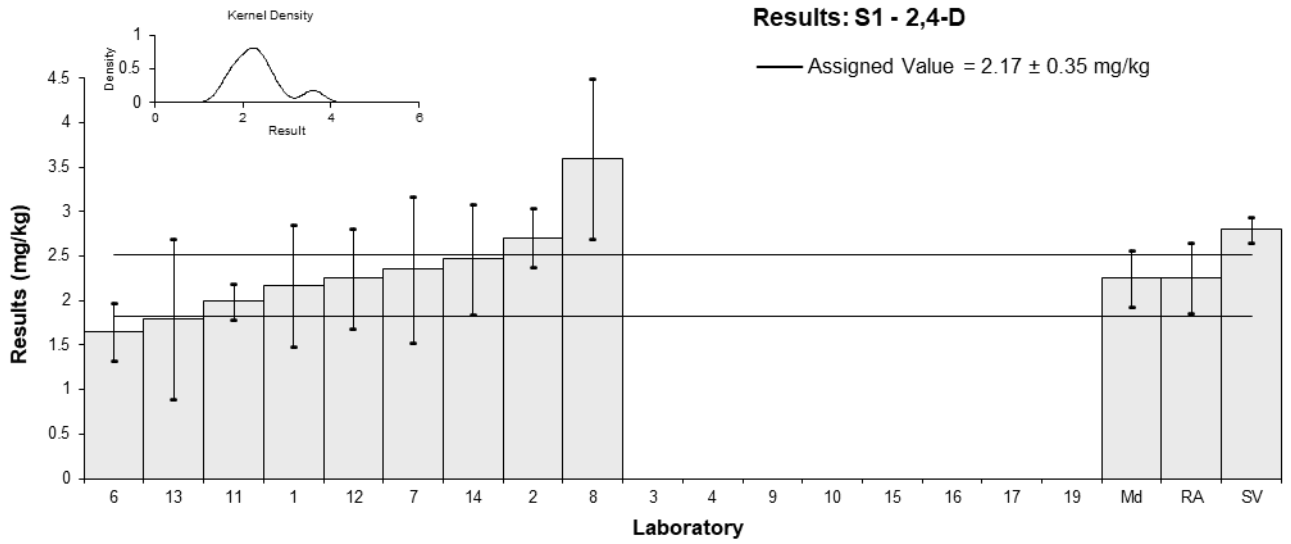
Participant Results

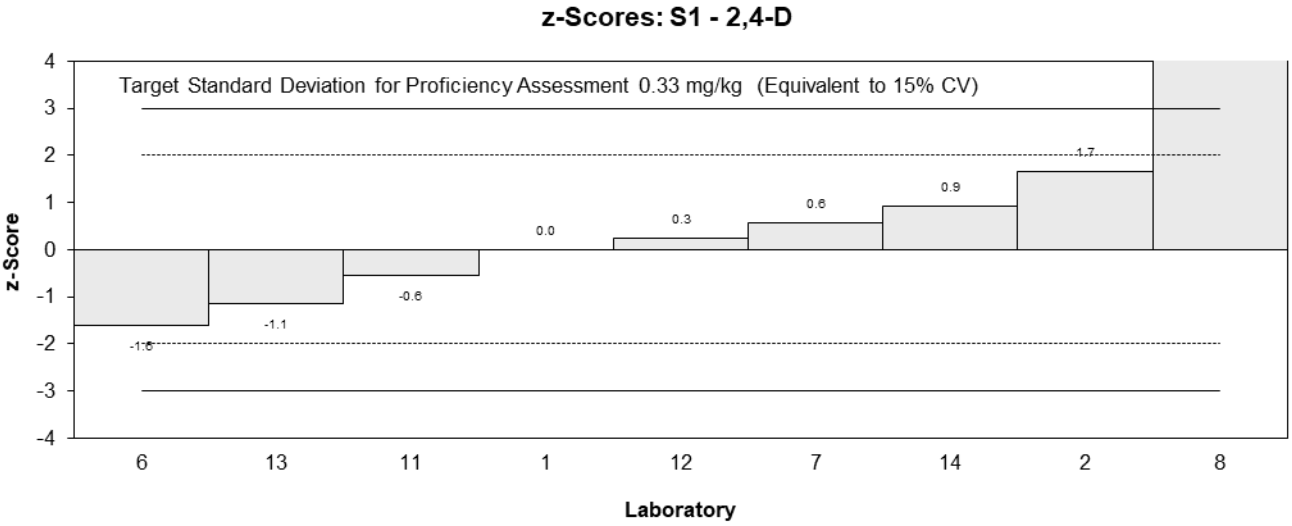
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	2.17	0.69	85	0.00	0.00
2	2.71	0.33	NR	1.66	1.12
3	NT	NT	NT		
4	NT	NT	NT		
6	1.65	0.33	NR	-1.60	-1.08
7	2.352	0.823	91	0.56	0.20
8*	3.6	0.9	103	4.39	1.48
9	NT	NT	NT		
10	NT	NT	NT		
11	1.99	0.20	92	-0.55	-0.45
12	2.25	0.5625	114	0.25	0.12
13[#]	1.8	0.90	54	-1.14	
14	2.47	0.62	NR	0.92	0.42
15	NT	NT	NT		
16	NT	NT	NT		
17	NT	NT	NT		
19	NT	NT	NR		

* Outlier, see Section 4.2; [#] Uncertainty reported as standard uncertainty – E_n-score not determined, see Section 6.4

Statistics

Assigned Value	2.17	0.35
Spike Value	2.80	0.14
Robust Average	2.26	0.40
Median	2.25	0.32
Mean	2.33	
N	9	
Max	3.6	
Min	1.65	
Robust SD	0.48	
Robust CV	21%	


Kernel Density
Density
Result
Results: S1 - 2,4-D
Assigned Value = 2.17 ± 0.35 mg/kg
Results (mg/kg)
Laboratory
6 13 11 1 12 7 14 2 8 3 4 9 10 15 16 17 19 Md RA SV


z-Scores: S1 - 2,4-D
Target Standard Deviation for Proficiency Assessment 0.33 mg/kg (Equivalent to 15% CV)
z-Score
-1.6
-1.1
-0.6
0.0
0.3
0.6
0.9
1.7
Laboratory
6 13 11 1 12 7 14 2 8

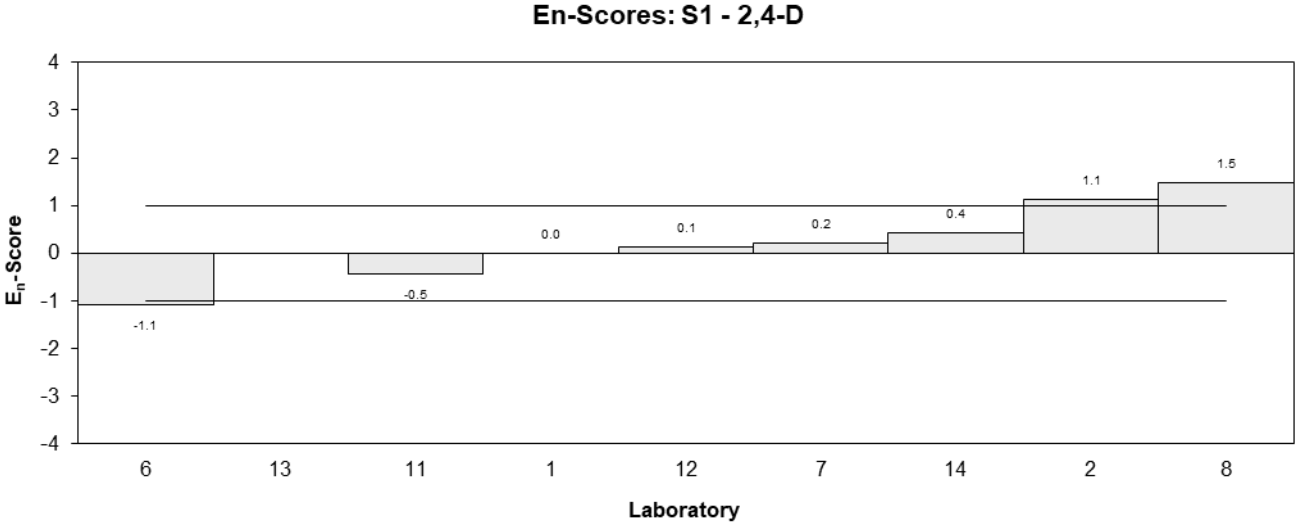

En-Scores: S1 - 2,4-D
En-Score
-1.1
-0.5
0.0
0.1
0.2
0.4
1.1
1.5
Laboratory
6 13 11 1 12 7 14 2 8

Figure 2

Table 6

Sample Details

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

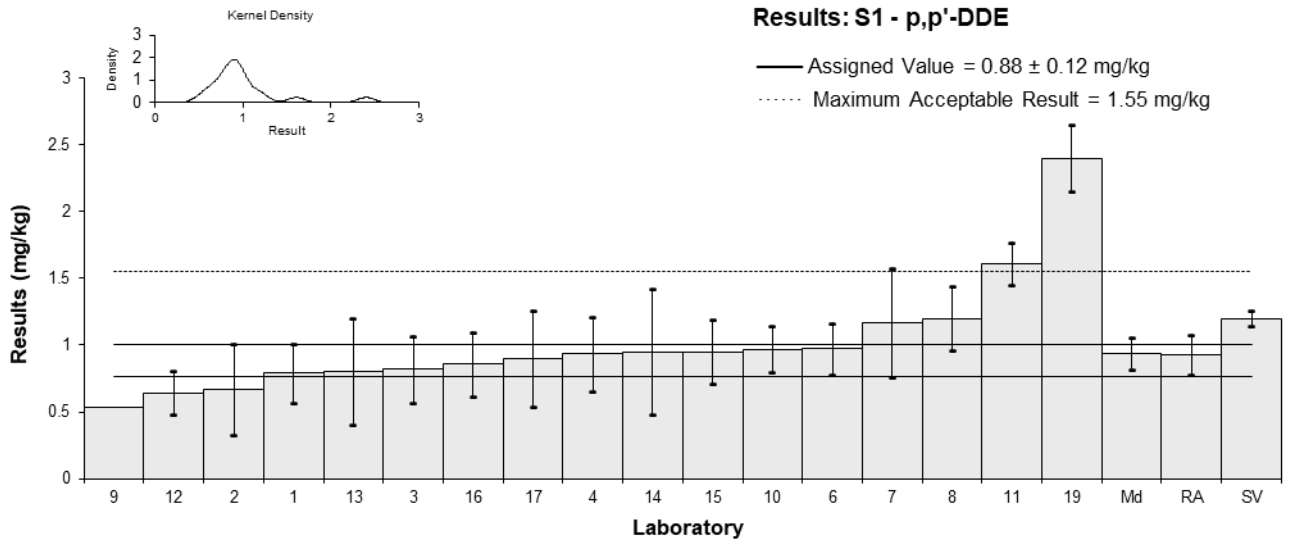
Participant Results

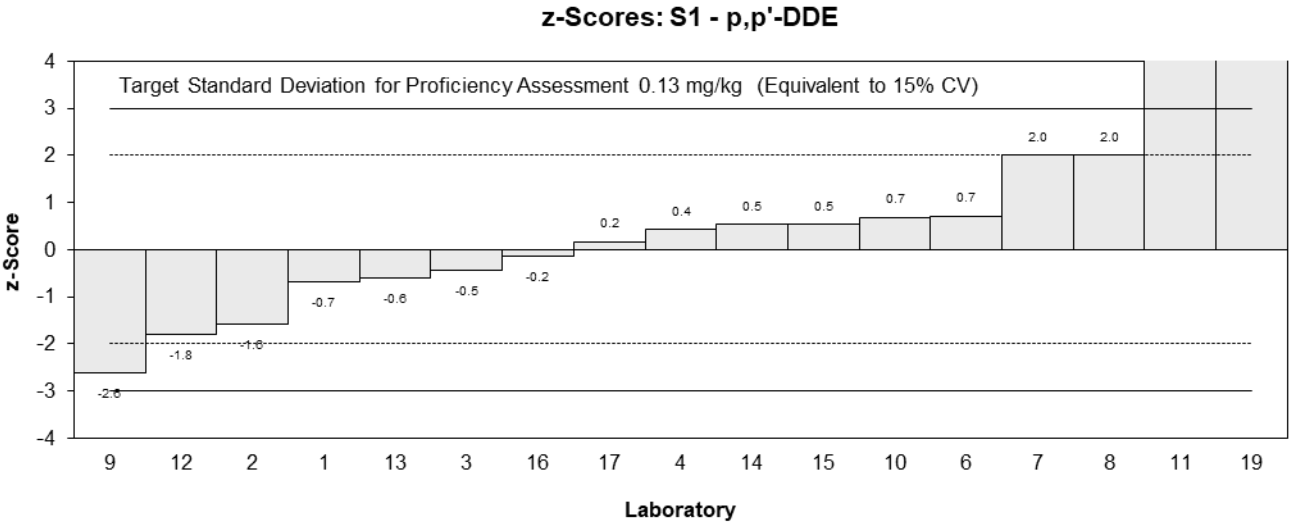
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	0.79	0.22	90	-0.68	-0.36
2	0.67	0.34	NR	-1.59	-0.58
3	0.82	0.25	NR	-0.45	-0.22
4	0.935	0.2805	NR	0.42	0.18
6	0.973	0.1946	NR	0.70	0.41
7	1.167	0.408	78	2.00▼	
8	1.2	0.24	107	2.00▼	
9	0.534	NR	NR	-2.62	-2.88
10	0.97	0.17	NR	0.68	0.43
11*	1.61	0.16	96	5.53	3.65
12	0.643	0.161	80	-1.80	-1.18
13	0.80	0.40	72	-0.61	-0.19
14	0.95	0.47	NR	0.53	0.14
15	0.95	0.24	NR	0.53	0.26
16	0.86	0.24	NR	-0.15	-0.07
17	0.901	0.36	NR	0.16	0.06
19*	2.4	0.25	NR	11.52	5.48

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

Statistics

Assigned Value	0.88	0.12
Spike Value	1.20	0.06
Robust Average	0.93	0.15
Max Acceptable Result	1.55	
Median	0.94	0.12
Mean	1.01	
N	17	
Max	2.4	
Min	0.534	
Robust SD	0.24	
Robust CV	26%	


Kernel Density
Density
Result
Results: S1 - p,p'-DDE
Assigned Value = 0.88 ± 0.12 mg/kg
Maximum Acceptable Result = 1.55 mg/kg
Results (mg/kg)
9 12 2 1 13 3 16 17 4 14 15 10 6 7 8 11 19 Md RA SV
Laboratory


z-Scores: S1 - p,p'-DDE
Target Standard Deviation for Proficiency Assessment 0.13 mg/kg (Equivalent to 15% CV)
z-Score
-2.6 -1.8 -1.6 -0.7 -0.6 -0.5 -0.2 0.2 0.4 0.5 0.5 0.7 0.7 2.0 2.0
9 12 2 1 13 3 16 17 4 14 15 10 6 7 8 11 19
Laboratory

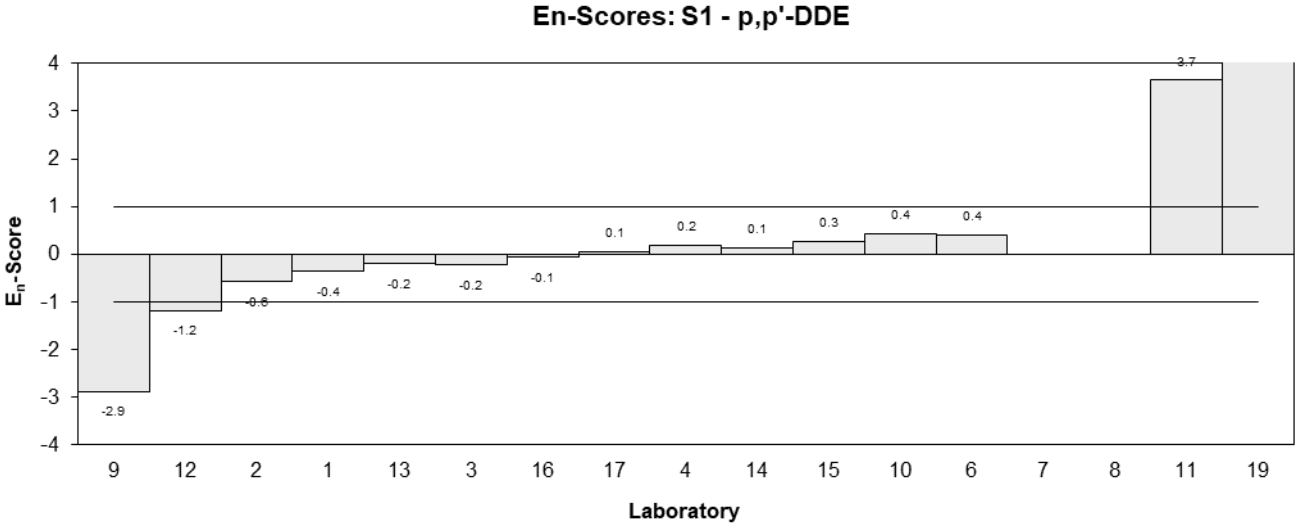

En-Scores: S1 - p,p'-DDE
En-Score
-2.9 -1.2 -0.6 -0.4 -0.2 -0.2 -0.1 0.1 0.2 0.1 0.3 0.4 0.4 3.7
9 12 2 1 13 3 16 17 4 14 15 10 6 7 8 11 19
Laboratory

Figure 3

Table 7

Sample Details

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

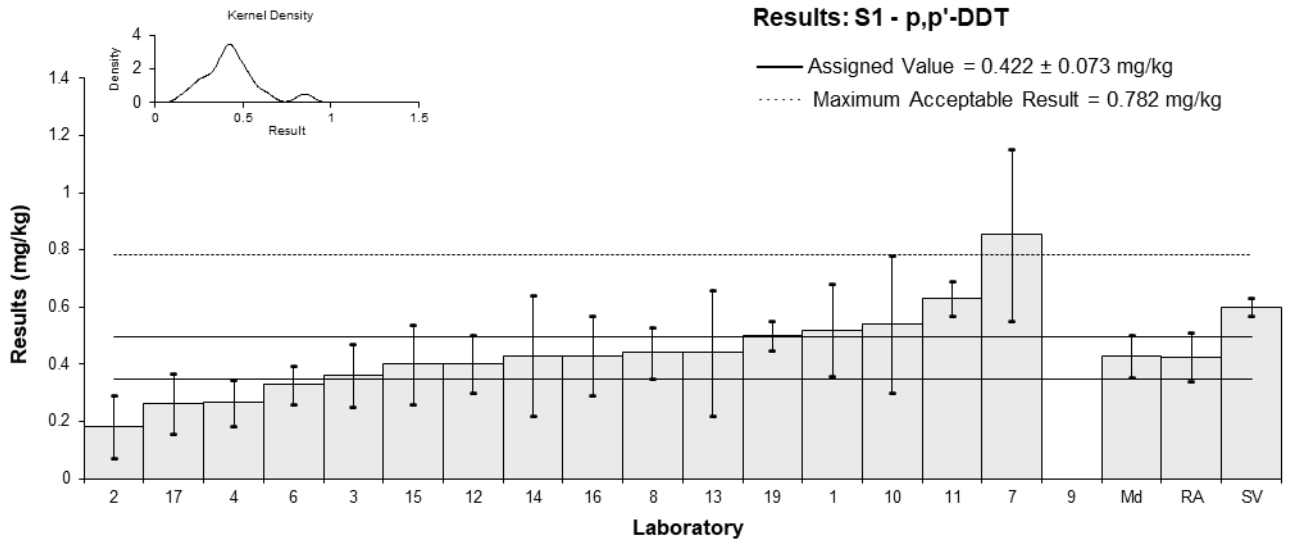
Participant Results

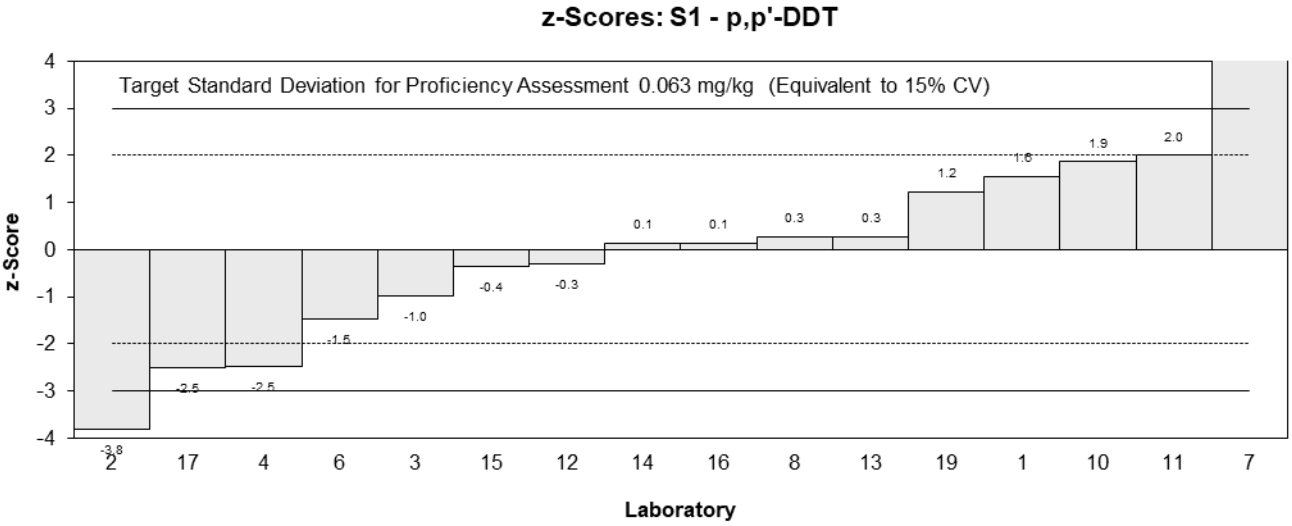
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	0.52	0.16	86	1.55	0.56
2*	0.18	0.11	NR	-3.82	-1.83
3	0.36	0.11	NR	-0.98	-0.47
4	0.265	0.0795	NR	-2.48	-1.45
6	0.328	0.0656	NR	-1.48	-0.96
7*	0.853	0.299	81	6.81	1.40
8	0.44	0.09	94	0.28	0.16
9	NT	NT	NT		
10	0.54	0.24	NR	1.86	0.47
11	0.63	0.06	109	2.00▼	
12	0.403	0.101	122	-0.30	-0.15
13	0.44	0.22	71	0.28	0.08
14	0.43	0.21	NR	0.13	0.04
15	0.4	0.14	NR	-0.35	-0.14
16	0.43	0.14	NR	0.13	0.05
17	0.264	0.106	NR	-2.50	-1.23
19	0.50	0.05	NR	1.23	0.88

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

Statistics

Assigned Value	0.422	0.073
Spike Value	0.601	0.030
Robust Average	0.425	0.086
Max Acceptable Result	0.782	
Median	0.430	0.074
Mean	0.436	
N	16	
Max	0.853	
Min	0.18	
Robust SD	0.14	
Robust CV	32%	


Kernel Density
Density
Result
Results: S1 - p,p'-DDT
Assigned Value = 0.422 ± 0.073 mg/kg
Maximum Acceptable Result = 0.782 mg/kg
Results (mg/kg)
Laboratory
2 17 4 6 3 15 12 14 16 8 13 19 1 10 11 7 9 Md RA SV


z-Scores: S1 - p,p'-DDT
Target Standard Deviation for Proficiency Assessment 0.063 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
2 17 4 6 3 15 12 14 16 8 13 19 1 10 11 7

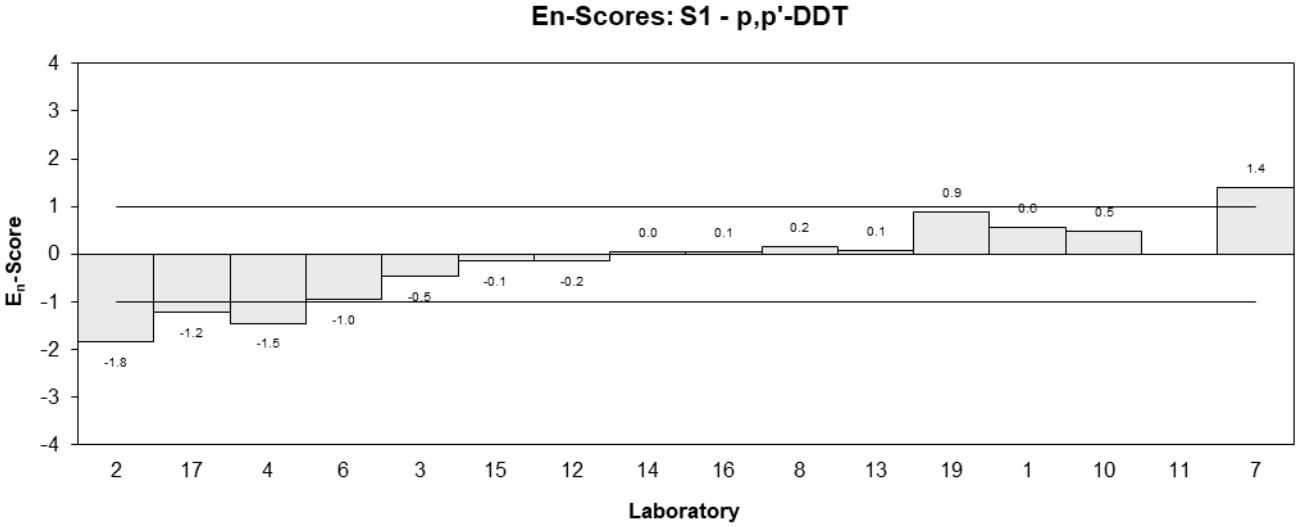

En-Scores: S1 - p,p'-DDT
En-Score
Laboratory
2 17 4 6 3 15 12 14 16 8 13 19 1 10 11 7

Figure 4

Table 8

Sample Details

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

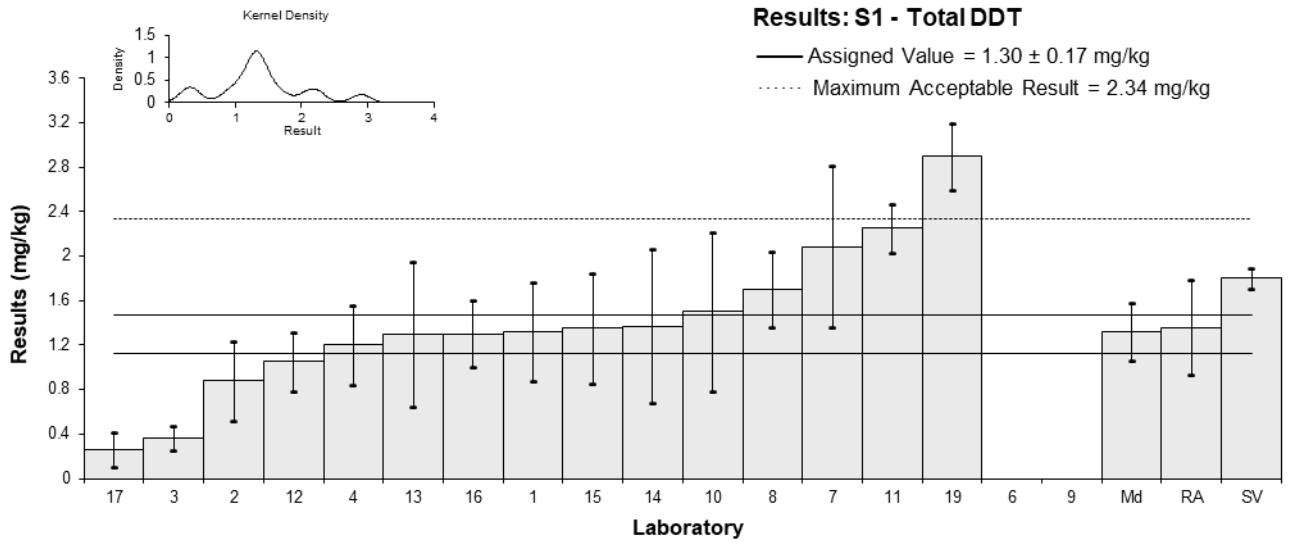
Participant Results

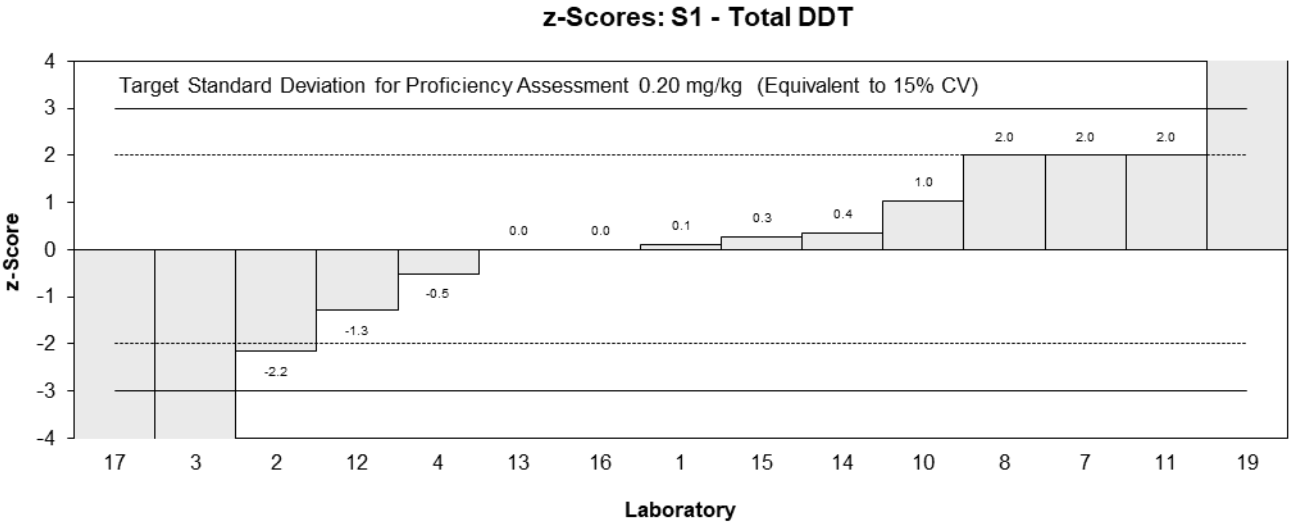
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	1.32	0.44	NR	0.10	0.04
2	0.88	0.36	NR	-2.15	-1.05
3*	0.36	0.11	NR	-4.82	-4.64
4	1.2	0.36	NR	-0.51	-0.25
6	NT	NT	NT		
7*	2.087	0.731	NR	2.00▼	
8	1.7	0.34	94	2.00▼	
9	NT	NT	NT		
10	1.5	0.71	NR	1.03	0.27
11*	2.25	0.22	109	2.00▼	
12	1.05	0.26	NR	-1.28	-0.80
13	1.3	0.65	NR	0.00	0.00
14	1.37	0.69	NR	0.36	0.10
15	1.35	0.5	NR	0.26	0.09
16	1.3	0.30	NR	0.00	0.00
17*	0.264	0.156	NR	-5.31	-4.49
19*	2.9	0.30	NR	8.21	4.64

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

Statistics

Assigned Value	1.30	0.17
Spike Value	1.80	0.09
Robust Average	1.36	0.43
Max Acceptable Result	2.34	
Median	1.32	0.26
Mean	1.39	
N	15	
Max	2.9	
Min	0.264	
Robust SD	0.66	
Robust CV	49%	


Kernel Density
Density
Result
Results: S1 - Total DDT
Assigned Value = 1.30 ± 0.17 mg/kg
Maximum Acceptable Result = 2.34 mg/kg
Results (mg/kg)
Laboratory
17 3 2 12 4 13 16 1 15 14 10 8 7 11 19 6 9 Md RA SV


z-Scores: S1 - Total DDT
Target Standard Deviation for Proficiency Assessment 0.20 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
17 3 2 12 4 13 16 1 15 14 10 8 7 11 19
-2.2 -1.3 -0.5 0.0 0.0 0.1 0.3 0.4 1.0 2.0 2.0 2.0

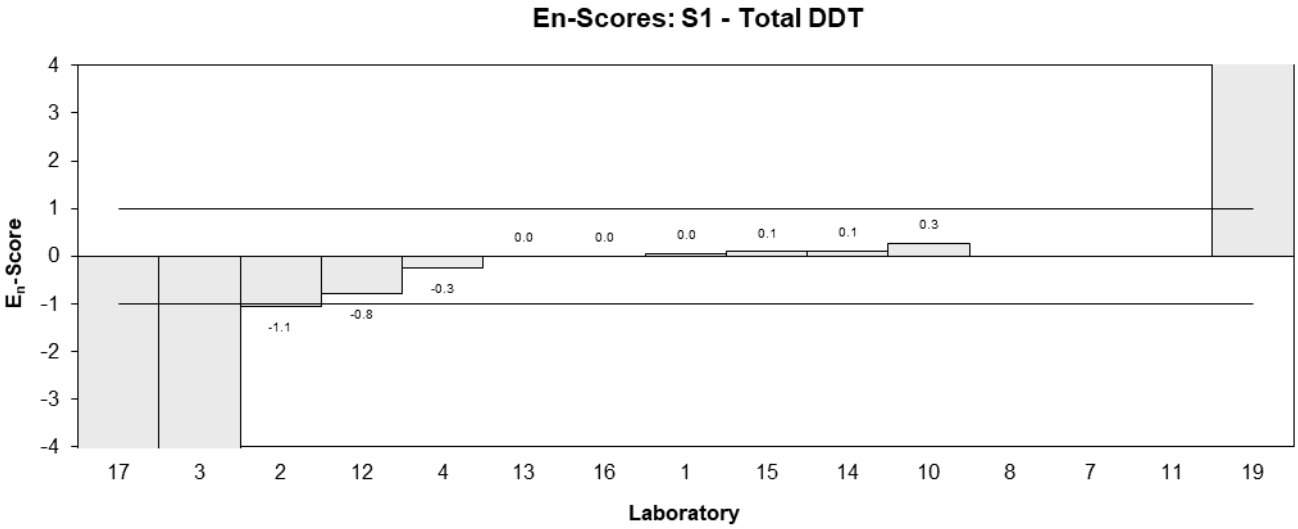

En-Scores: S1 - Total DDT
En-Score
Laboratory
17 3 2 12 4 13 16 1 15 14 10 8 7 11 19
-1.1 -0.8 -0.3 0.0 0.0 0.0 0.1 0.1 0.3

Figure 5

Table 9

Sample Details

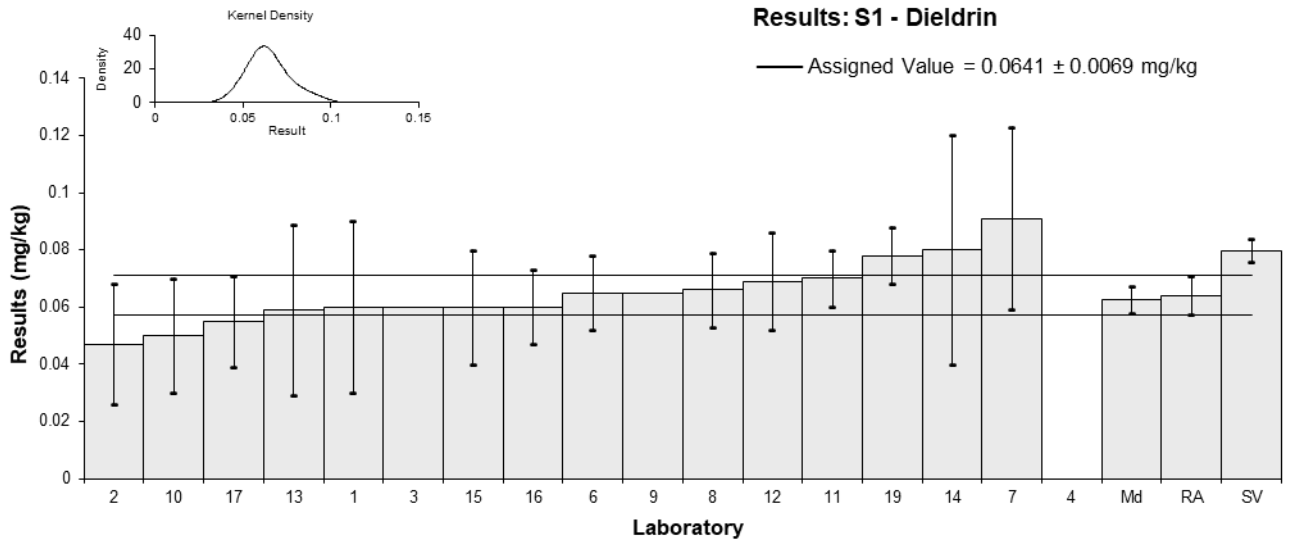
Sample No.	S1
Matrix	Soil
Analyte	Dieldrin
Unit	mg/kg

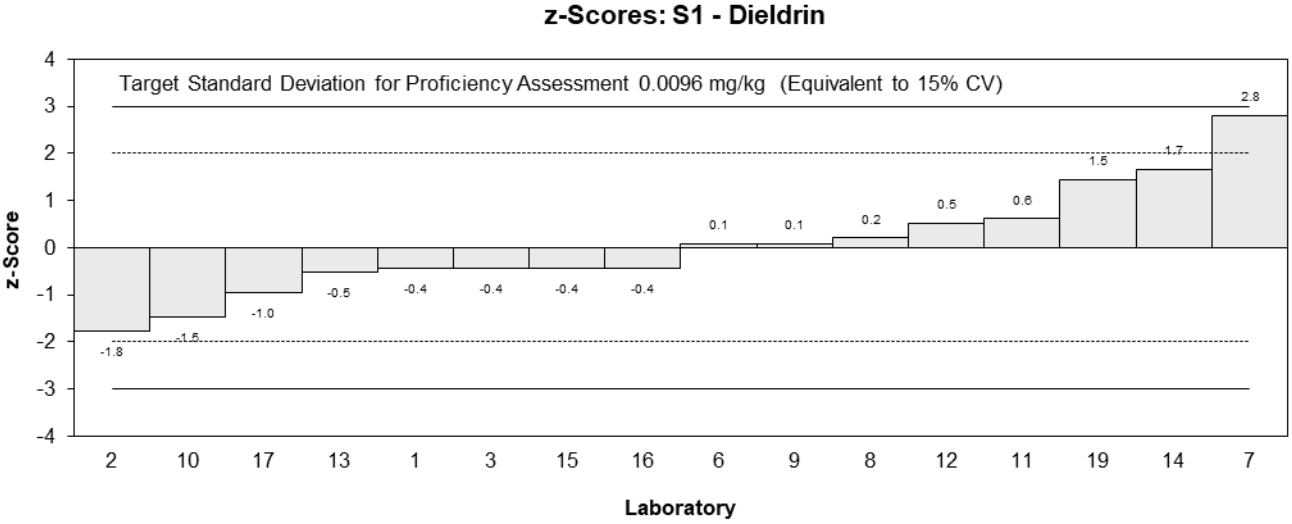
Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E_n
1	0.06	0.03	75	-0.43	-0.13
2	0.047	0.021	NR	-1.78	-0.77
3	0.06	NR	NR	-0.43	-0.59
4	<0.05	NR	NR		
6	0.065	0.013	NR	0.09	0.06
7	0.091	0.032	104	2.80	0.82
8	0.066	0.013	107	0.20	0.13
9	0.065	NR	NR	0.09	0.13
10	0.05	0.02	NR	-1.47	-0.67
11	0.07	0.01	94	0.61	0.49
12	0.069	0.017	113	0.51	0.27
13	0.059	0.030	78	-0.53	-0.17
14	0.08	0.04	NR	1.65	0.39
15	0.06	0.02	NR	-0.43	-0.19
16	0.06	0.013	NR	-0.43	-0.28
17	0.055	0.016	NR	-0.95	-0.52
19	0.078	0.01	NR	1.45	1.14

Statistics

Assigned Value	0.0641	0.0069
Spike Value	0.0798	0.0040
Robust Average	0.0641	0.0069
Median	0.0625	0.0046
Mean	0.0647	
N	16	
Max	0.091	
Min	0.047	
Robust SD	0.011	
Robust CV	17%	


Kernel Density
Density
Result
Results: S1 - Dieldrin
Assigned Value = 0.0641 ± 0.0069 mg/kg
Results (mg/kg)
Laboratory
2 10 17 13 1 3 15 16 6 9 8 12 11 19 14 7 4 Md RA SV


z-Scores: S1 - Dieldrin
Target Standard Deviation for Proficiency Assessment 0.0096 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
2 10 17 13 1 3 15 16 6 9 8 12 11 19 14 7

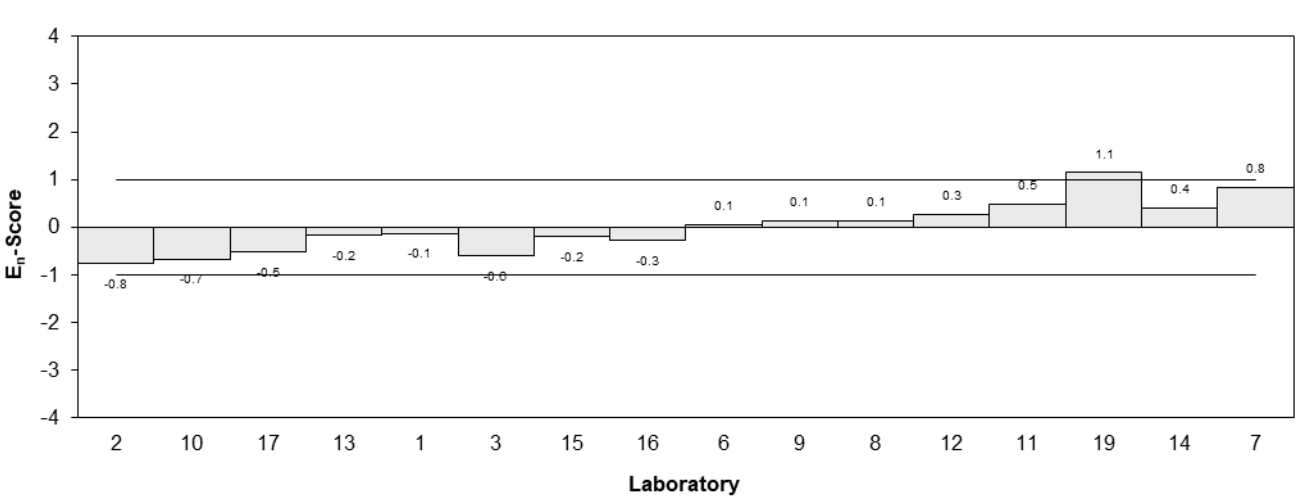

En-Scores: S1 - Dieldrin
En-Score
Laboratory
2 10 17 13 1 3 15 16 6 9 8 12 11 19 14 7

Figure 6

Table 10

Sample Details

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

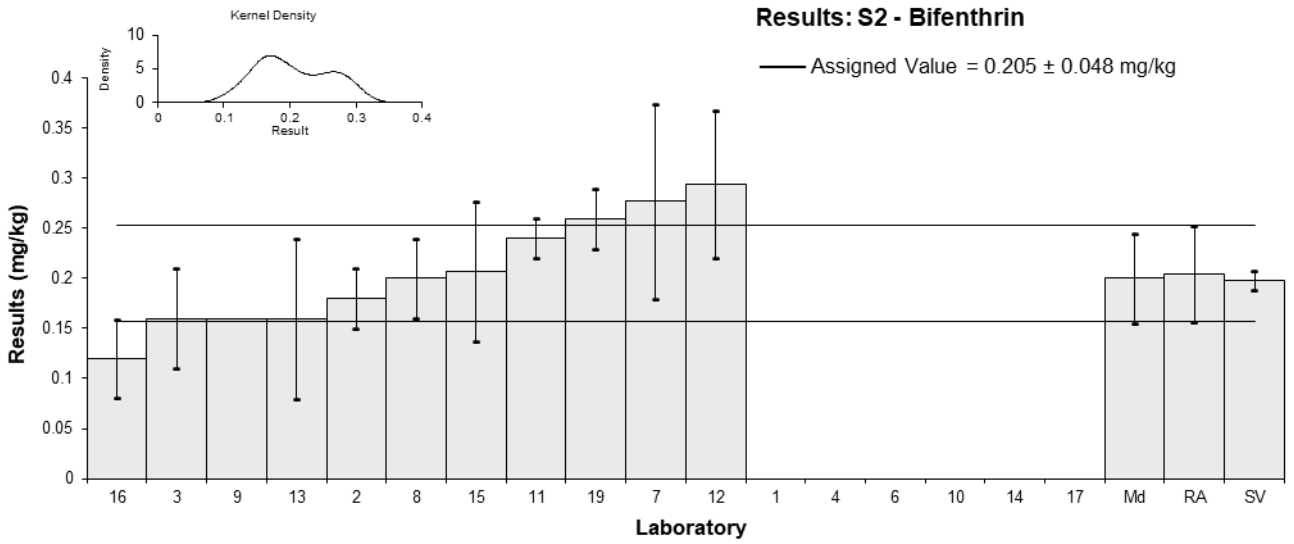
Participant Results

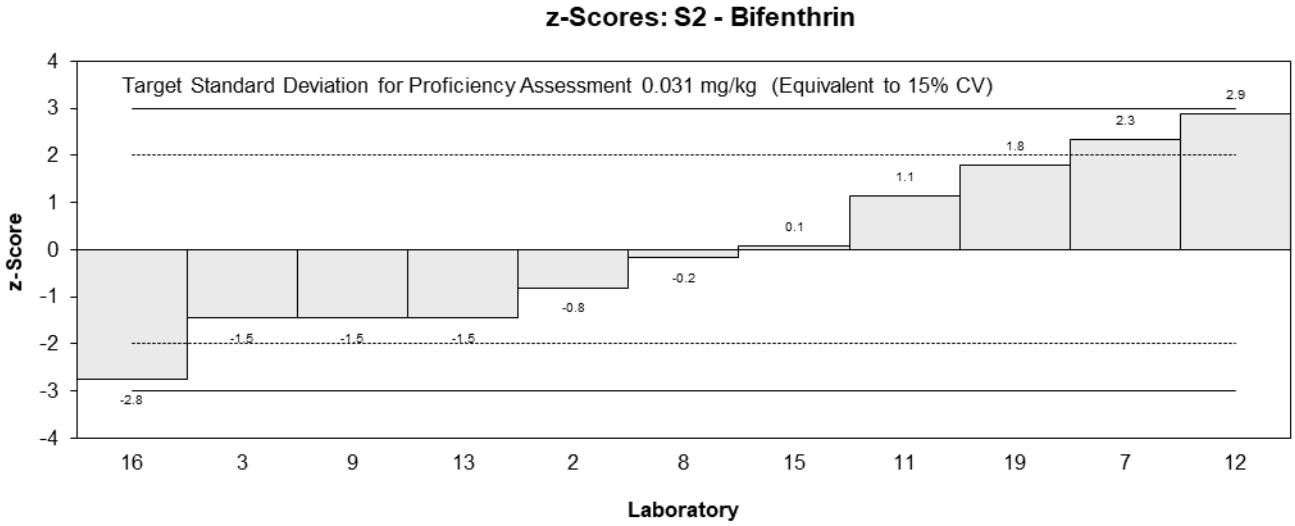
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	NT	NT	NT		
2	0.18	0.03	NR	-0.81	-0.44
3	0.16	0.05	NR	-1.46	-0.65
4	NT	NT	NT		
6	<0.5	NR	NR		
7	0.277	0.097	79	2.34	0.67
8	0.20	0.04	NR	-0.16	-0.08
9	0.16	NR	NR	-1.46	-0.94
10	NT	NT	NT		
11	0.24	0.02	99	1.14	0.67
12	0.294	0.074	82	2.89	1.01
13[#]	0.16	0.08	49	-1.46	
14	NT	NT	NT		
15	0.207	0.07	NR	0.07	0.02
16	0.12	0.039	NR	-2.76	-1.37
17	NT	NT	NT		
19	0.26	0.03	NR	1.79	0.97

[#] Uncertainty reported as standard uncertainty – E_n-score not determined, see Section 6.4

Statistics

Assigned Value	0.205	0.048
Spike Value	0.198	0.010
Robust Average	0.205	0.048
Median	0.200	0.045
Mean	0.205	
N	11	
Max	0.294	
Min	0.12	
Robust SD	0.063	
Robust CV	31%	


Kernel Density
Density
Result
Results: S2 - Bifenthrin
Assigned Value = 0.205 ± 0.048 mg/kg
Results (mg/kg)
Laboratory
16 3 9 13 2 8 15 11 19 7 12 1 4 6 10 14 17 Md RA SV


z-Scores: S2 - Bifenthrin
Target Standard Deviation for Proficiency Assessment 0.031 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
16 3 9 13 2 8 15 11 19 7 12
-2.8
-1.5
-1.5
-1.5
-0.8
-0.2
0.1
1.1
1.8
2.3
2.9

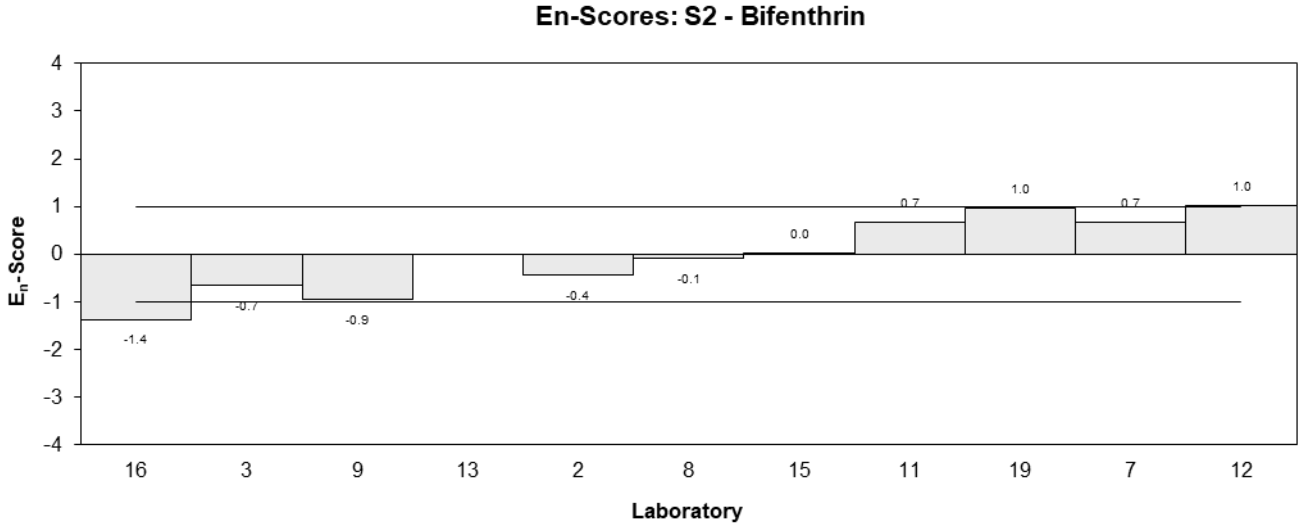

En-Scores: S2 - Bifenthrin
En-Score
Laboratory
16 3 9 13 2 8 15 11 19 7 12
-1.4
-0.7
-0.9
-0.4
-0.1
0.0
0.7
1.0
0.7
1.0

Figure 7

Table 11

Sample Details

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

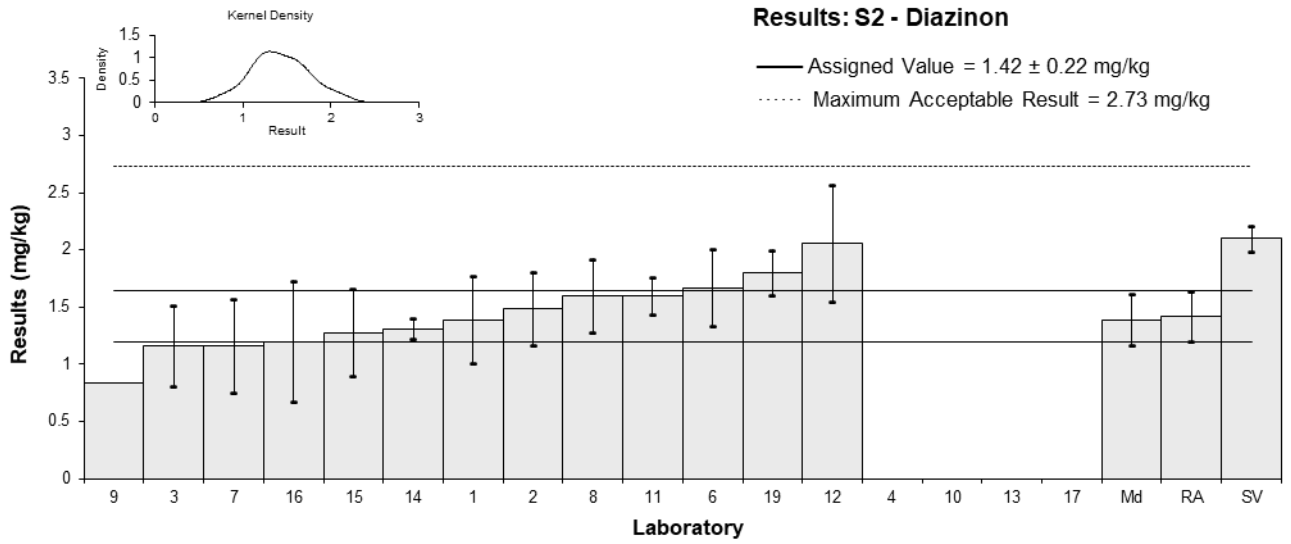
Participant Results

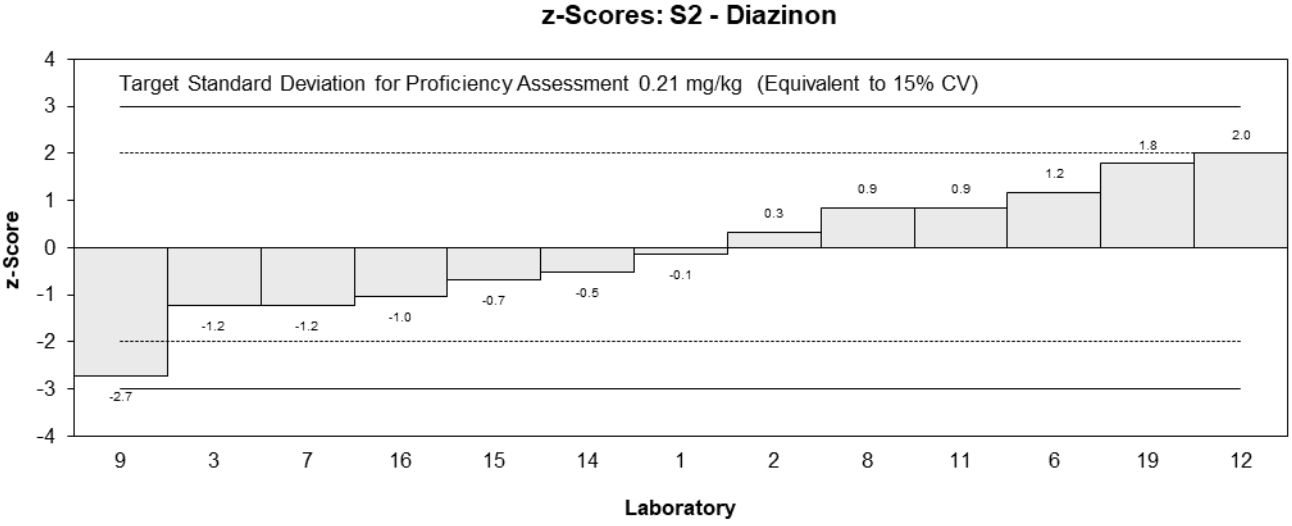
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	1.39	0.38	95	-0.14	-0.07
2	1.49	0.32	NR	0.33	0.18
3	1.16	0.35	NR	-1.22	-0.63
4	NT	NT	NT		
6	1.67	0.334	NR	1.17	0.63
7	1.161	0.406	77	-1.22	-0.56
8	1.6	0.32	NR	0.85	0.46
9	0.8387	NR	NR	-2.73	-2.64
10	NT	NT	NT		
11	1.60	0.16	96	0.85	0.66
12	2.058	0.515	111	2.00▼	
13	NT	NT	NT		
14	1.31	0.09	NR	-0.52	-0.46
15	1.275	0.38	NR	-0.68	-0.33
16	1.2	0.53	NR	-1.03	-0.38
17	<0.05	NR	NR		
19	1.8	0.2	NR	1.78	1.28

▼ Adjusted Score, see Section 6.3

Statistics

Assigned Value	1.42	0.22
Spike Value	2.10	0.11
Robust Average	1.42	0.22
Max Acceptable Result	2.73	
Median	1.39	0.22
Mean	1.43	
N	13	
Max	2.058	
Min	0.8387	
Robust SD	0.32	
Robust CV	22%	


Kernel Density
Density
Result
Results: S2 - Diazinon
Assigned Value = 1.42 ± 0.22 mg/kg
Maximum Acceptable Result = 2.73 mg/kg
Results (mg/kg)
Laboratory
9 3 7 16 15 14 1 2 8 11 6 19 12 4 10 13 17 Md RA SV


z-Scores: S2 - Diazinon
Target Standard Deviation for Proficiency Assessment 0.21 mg/kg (Equivalent to 15% CV)
z-Score
-2.7 -1.2 -1.2 -1.0 -0.7 -0.5 -0.1 0.3 0.9 0.9 1.2 1.8 2.0
Laboratory
9 3 7 16 15 14 1 2 8 11 6 19 12

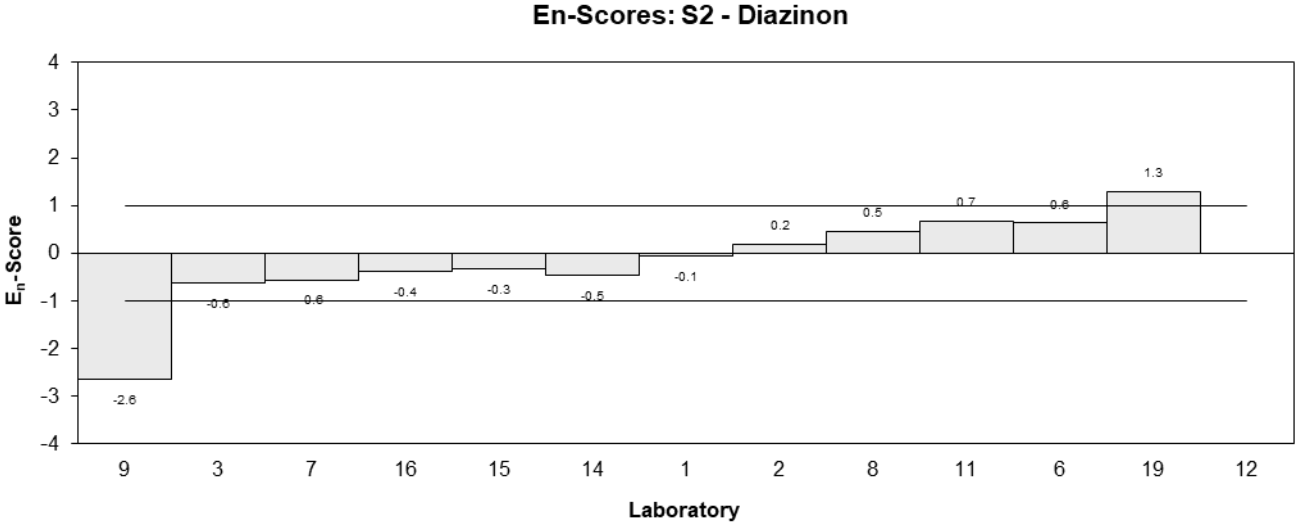

En-Scores: S2 - Diazinon
En-Score
-2.6 -0.6 -0.6 -0.4 -0.3 -0.5 -0.1 0.2 0.5 0.7 0.6 1.3
Laboratory
9 3 7 16 15 14 1 2 8 11 6 19 12

Figure 8

Table 12

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	0.81	0.23	58
2	0.97	0.14	NR
3	NT	NT	NT
4	NT	NT	NT
6	NT	NT	NT
7	NT	NT	NT
8	0.78	0.20	NR
9	NT	NT	NT
10	NT	NT	NT
11	NT	NT	NT
12	NT	NT	NT
13[#]	0.55	0.42	38
14	1.03	0.27	NR
15	NT	NT	NT
16	NT	NT	NT
17	NT	NT	NT
19	NT	NT	NR

[#] Uncertainty reported as standard uncertainty, see Section 6.4

Statistics

Assigned Value	Not Set	
Spike Value	1.10	0.06
Robust Average	NA (N<6)	
Median	0.81	0.27
Mean	0.83	
N	5	
Max	1.03	
Min	0.55	
Robust SD	NA (N<6)	
Robust CV	NA (N<6)	

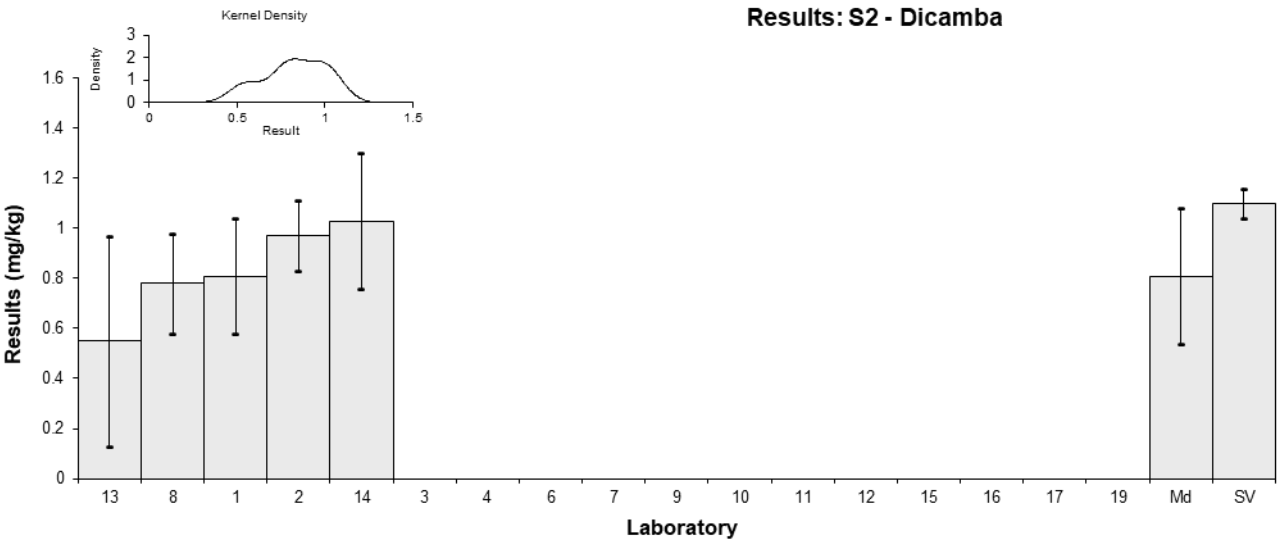

Results: S2 - Dicamba
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
13
8
1
2
14
3
4
6
7
9
10
11
12
15
16
17
19
Md
SV

Figure 9

Table 13

Sample Details

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

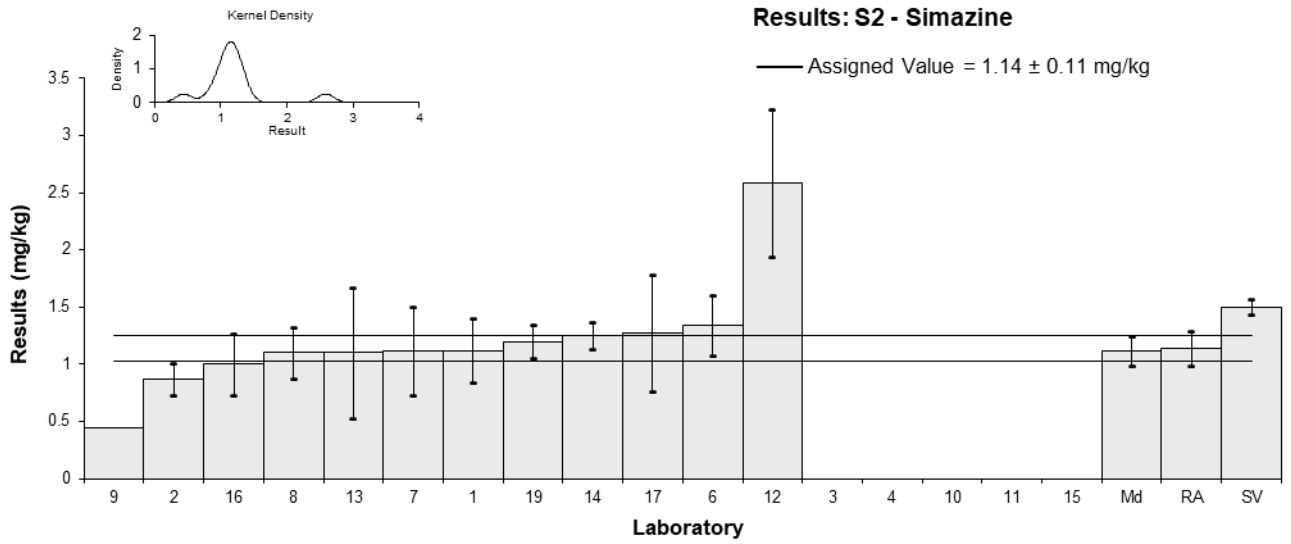
Participant Results

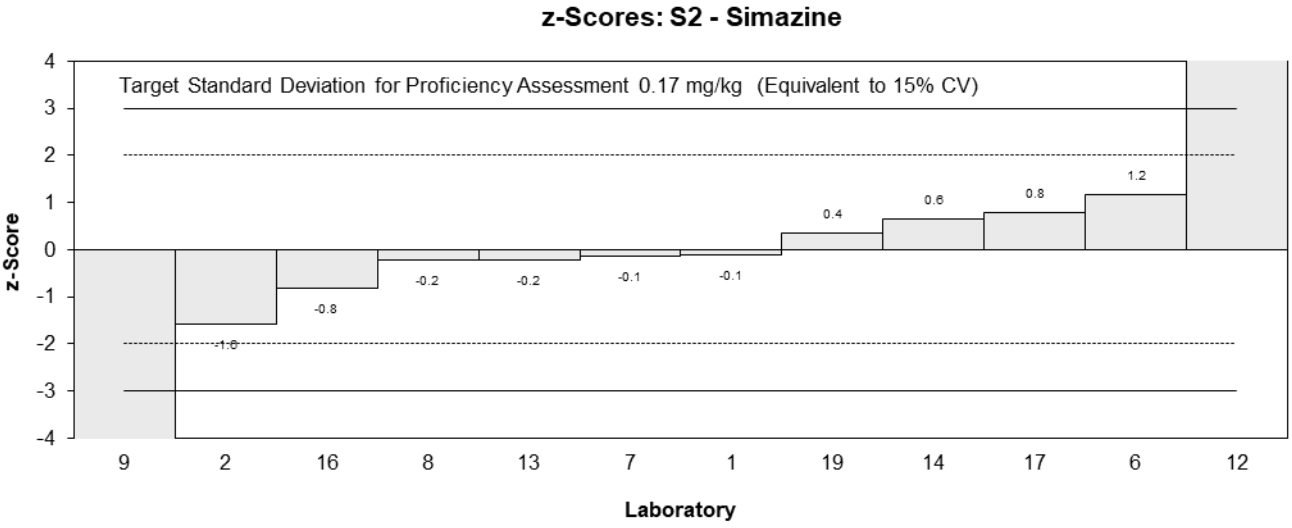
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	1.12	0.28	87	-0.12	-0.07
2	0.87	0.14	NR	-1.58	-1.52
3	NT	NT	NT		
4	NT	NT	NT		
6	1.34	0.268	NR	1.17	0.69
7	1.116	0.391	97	-0.14	-0.06
8	1.1	0.22	NR	-0.23	-0.16
9*	0.441	NR	NR	-4.09	-6.35
10	NT	NT	NT		
11	NT	NT	NT		
12*	2.587	0.647	102	8.46	2.20
13	1.1	0.57	70	-0.23	-0.07
14	1.25	0.12	NR	0.64	0.68
15	NT	NT	NT		
16	1.0	0.27	NR	-0.82	-0.48
17	1.2747	0.50988	NR	0.79	0.26
19	1.2	0.15	NR	0.35	0.32

* Outlier, see Section 4.2

Statistics

Assigned Value	1.14	0.11
Spike Value	1.50	0.07
Robust Average	1.14	0.15
Median	1.12	0.13
Mean	1.20	
N	12	
Max	2.587	
Min	0.441	
Robust SD	0.20	
Robust CV	18%	


Kernel Density
Density
Result
Results: S2 - Simazine
Assigned Value = 1.14 ± 0.11 mg/kg
Results (mg/kg)
Laboratory
9 2 16 8 13 7 1 19 14 17 6 12 3 4 10 11 15 Md RA SV


z-Scores: S2 - Simazine
Target Standard Deviation for Proficiency Assessment 0.17 mg/kg (Equivalent to 15% CV)
z-Score
-1.6 -0.8 -0.2 -0.2 -0.1 -0.1 0.4 0.6 0.8 1.2
Laboratory
9 2 16 8 13 7 1 19 14 17 6 12

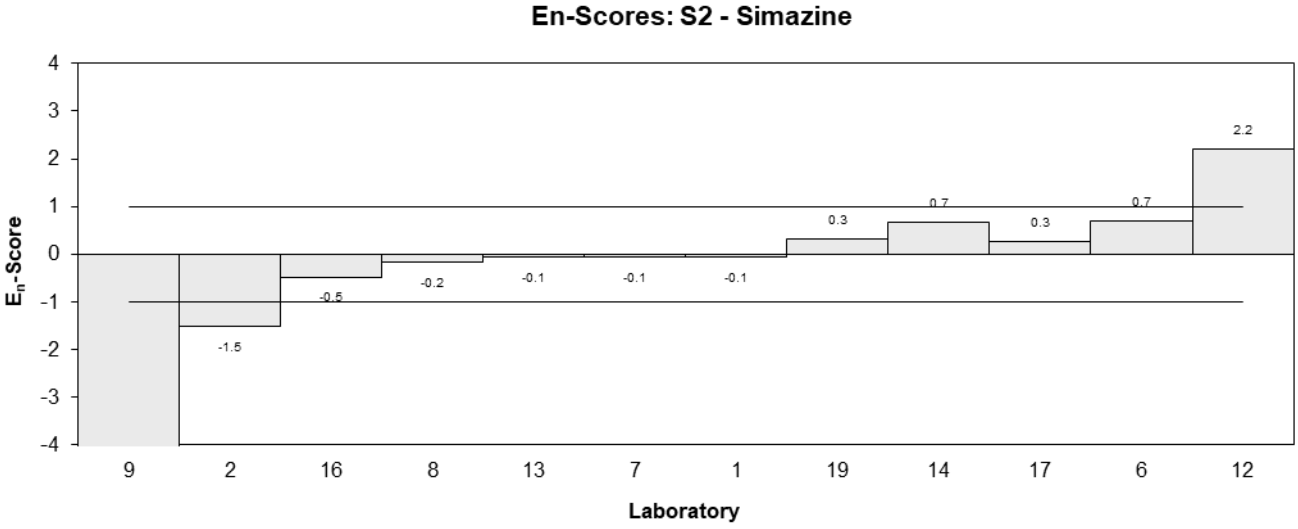

En-Scores: S2 - Simazine
En-Score
-1.5 -0.5 -0.2 -0.1 -0.1 -0.1 0.3 0.7 0.3 0.7 2.2
Laboratory
9 2 16 8 13 7 1 19 14 17 6 12

Figure 10

6 DISCUSSION OF RESULTS

6.1 Assigned Value

The robust averages of participants' results were used as the assigned values for all scored analytes. The robust averages and associated expanded uncertainties were calculated using the procedure described in ISO 13528.[7] Results less than 50% and greater than 150% of the robust average were removed before calculation of the assigned value.[3,4] The calculation of the expanded uncertainty for robust averages is presented in Appendix 3, using Sample S2 diazinon as an example.

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

No assigned value was set for Sample S2 dicamba as too few numeric results were reported for this analyte.

A comparison of the assigned values (or robust average if no assigned value was set) and the spiked values is presented in Table 14. The assigned values were within the range of 68% to 104% of the spiked values. Similar ratios have been observed in previous NMI Pesticides in Soil PT studies,[6] and an assigned value was set if there was a reasonable consensus of results.

Table 14 Comparison of Assigned Value (Robust Average) and Spiked Value

Sample	Analyte	Assigned Value *(Robust Average)* (mg/kg)	Spiked Value (mg/kg)	Assigned Value *(Robust Average)* / Spiked Value (%)
S1	2,4-D	2.17	2.80	78
	p,p'-DDE	0.88	1.20	73
	p,p'-DDT	0.422	0.601	70
	Total DDT	1.30	1.80	72
	Dieldrin	0.0641	0.0798	80
S2	Bifenthrin	0.205	0.198	104
	Diazinon	1.42	2.10	68
	Dicamba	*(0.83)*	1.10	*(75)*
	Simazine	1.14	1.50	76

The best estimate of the 'true' mass fraction of the pesticides in soil is most likely the spiked value. However, a proportion of the spiked pesticide is strongly bound to the soil and so is not readily extracted and measured. What laboratories measure may best be described as 'extractable pesticide', and the result may be influenced by the efficiency of the extraction process used. Therefore, for this study, the assigned value is the best estimate of the amount of 'extractable pesticide'.

6.2 Measurement Uncertainty Reported by Participants

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

Of 114 numeric results, 105 (92%) were reported with an associated expanded MU, and a further three results (3%) were reported with an associated standard MU. Participants used a

wide variety of procedures to estimate their uncertainties (Table 3). One participant reported using the NATA GAG Estimating and Reporting MU as their guide; NATA no longer publishes these documents.[11]

Laboratory **13** reported three of their uncertainties as a standard uncertainty rather than an expanded uncertainty as requested for this study. This participant did not report a coverage factor.

Laboratory **3** did not report an uncertainty for one of their numeric results; this participant reported being accredited to ISO/IEC 17025. Laboratory **9** did not report uncertainties for all their numeric results; this participant also reported being accredited to ISO/IEC 17025.

The magnitude of the reported expanded uncertainties was within the range 6.9% to 61% of the reported value. In general, an expanded uncertainty of less than 15% is likely to be unrealistically small for the routine measurement of a pesticide residue, while over 50% is likely to be too large and not fit-for-purpose. In this study, 18 expanded uncertainties were less than 15% relative, while six were greater than 50% relative.

Uncertainties associated with results returning a satisfactory *z*-score but an unsatisfactory E_n-score may have been underestimated.

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 2.25 ± 0.5625 mg/kg, it is better to report this as 2.25 ± 0.56 mg/kg.[10]

6.3 *z*-Score

Target SDs equivalent to 15% PCV were used to calculate *z*-scores. CVs predicted by the Thomspon-Horwitz equation,[8] between-laboratory CVs and target SDs (as PCVs) obtained in this study for analytes in this study are presented for comparison in Table 15.

Table 15 Comparison of Thompson-Horwitz CVs, Between-Laboratory CVs and Target SDs

Sample	Analyte	Assigned Value *(Robust Average)* (mg/kg)	Thompson-Horwitz CV[a] (%)	Between-Laboratory CV[b] (%)	Target SD (as PCV) (%)
S1	2,4-D	2.17	14	18	15
	p,p'-DDE	0.88	16	21	15
	p,p'-DDT	0.422	18	26	15
	Total DDT	1.30	15	17	15
	Dieldrin	0.0641	22	17	15
S2	Bifenthrin	0.205	20	31	15
	Diazinon	1.42	15	22	15
	Dicamba	*(0.83)*	16	26	Not Set
	Simazine	1.14	16	12	15

[a] Calculated from the assigned value (robust average).

[b] Robust between-laboratory CV (outliers removed where applicable).

To account for possible low bias in consensus values due to participants using inefficient extraction or analytical techniques, a total of seven *z*-scores were adjusted across the

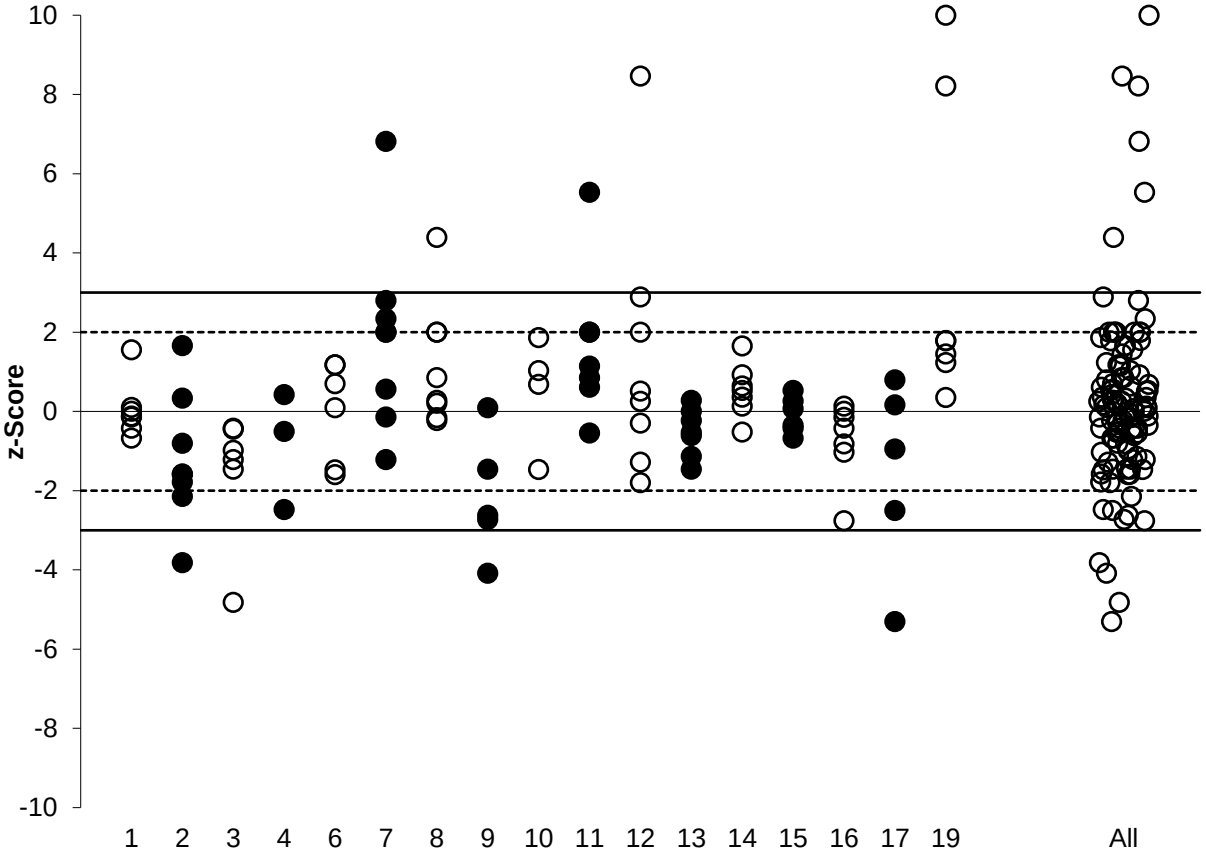
following analytes: Sample S1 p,p'-DDE, p,p'-DDT and total DDT, and Sample S2 diazinon. A maximum acceptable result was set as the spiked value plus two target SDs of the spiked value. Results lower than the maximum acceptable result but with a *z*-score greater than 2.0 had their *z*-score adjusted 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 109 results for which *z*-scores were calculated, 90 (83%) returned a satisfactory *z*-score of $|z| \leq 2.0$, indicating a satisfactory performance.

Laboratories **2**, **7**, **8** and **12** reported results for all eight analytes for which *z*-scores were calculated. No participant returned satisfactory *z*-scores for all eight scored analytes.

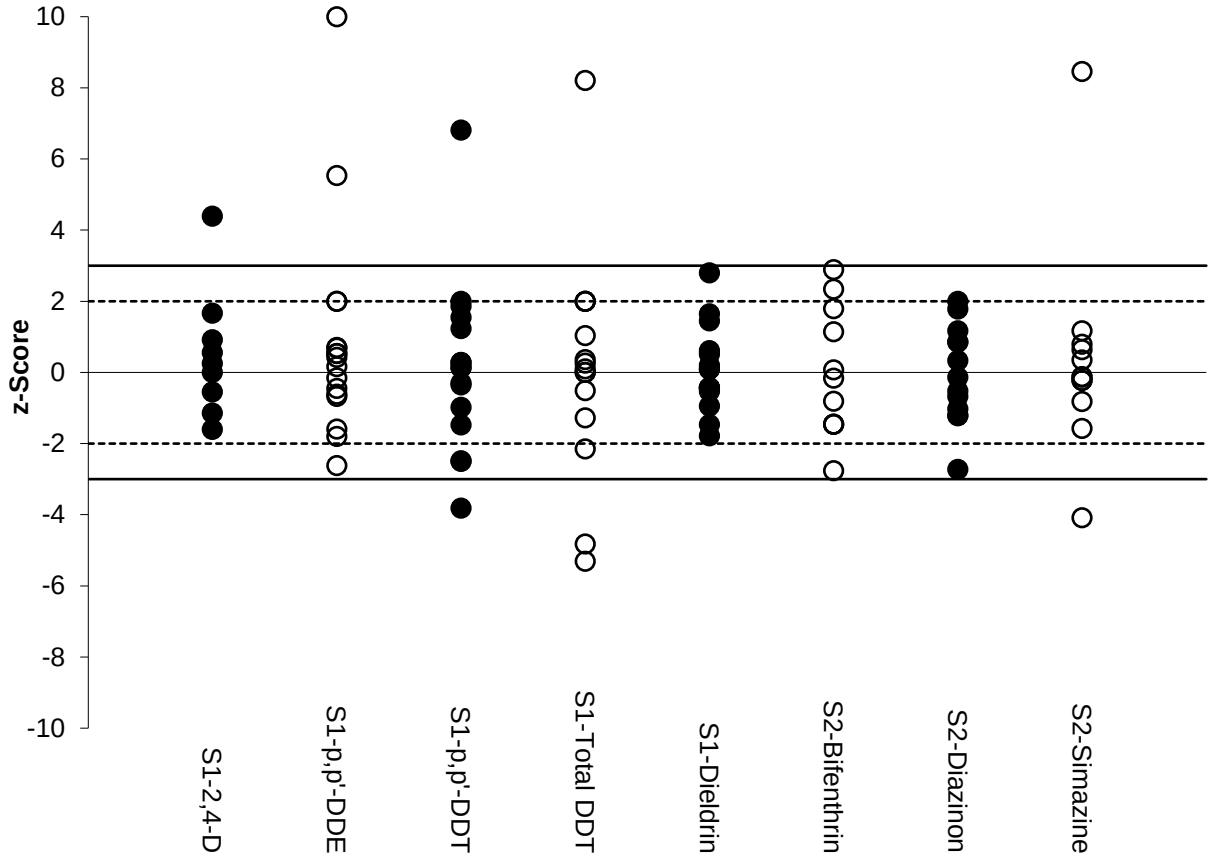
A number of participants received satisfactory *z*-scores for all analytes they reported results for: Laboratories **1** (7), **13** (7), **14** (7), **6** (6), **15** (6) and **10** (4).

The dispersal of participants' *z*-scores is presented graphically by laboratory in Figure 11 and by analyte in Figure 12.



z-Scores greater than 10.0 have been plotted at 10.0.

Figure 11 *z*-Score Dispersal by Laboratory



z-Scores greater than 10.0 have been plotted at 10.0.

Figure 12 *z*-Score Dispersal by Analyte

6.4 E_n-Score

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 *z*-Score, no E_n-score has been calculated.

Laboratory **13** reported standard uncertainties for Sample S1 2,4-D, and Sample S2 bifenthrin and dicamba, and did not report a coverage factor. For this PT study, participants were instructed to report the expanded uncertainty associated with their results. As the E_n-score is calculated using the expanded uncertainty of a result (Equation 3, Section 4.8), the E_n-scores for these results (where applicable) have not been reported.

Of 100 results for which E_n-scores were calculated, 77 (77%) were satisfactory with $|E_n| \leq 1.0$, indicating agreement of the participant's result with the assigned value within their respective uncertainties.

No participant returned satisfactory E_n-scores for all eight scored analytes.

A number of participants received satisfactory E_n-scores for all analytes they reported results for: Laboratories **1** (7), **14** (7), **15** (6) and **10** (4).

The dispersal of participants' E_n-scores is presented graphically by laboratory in Figure 13.

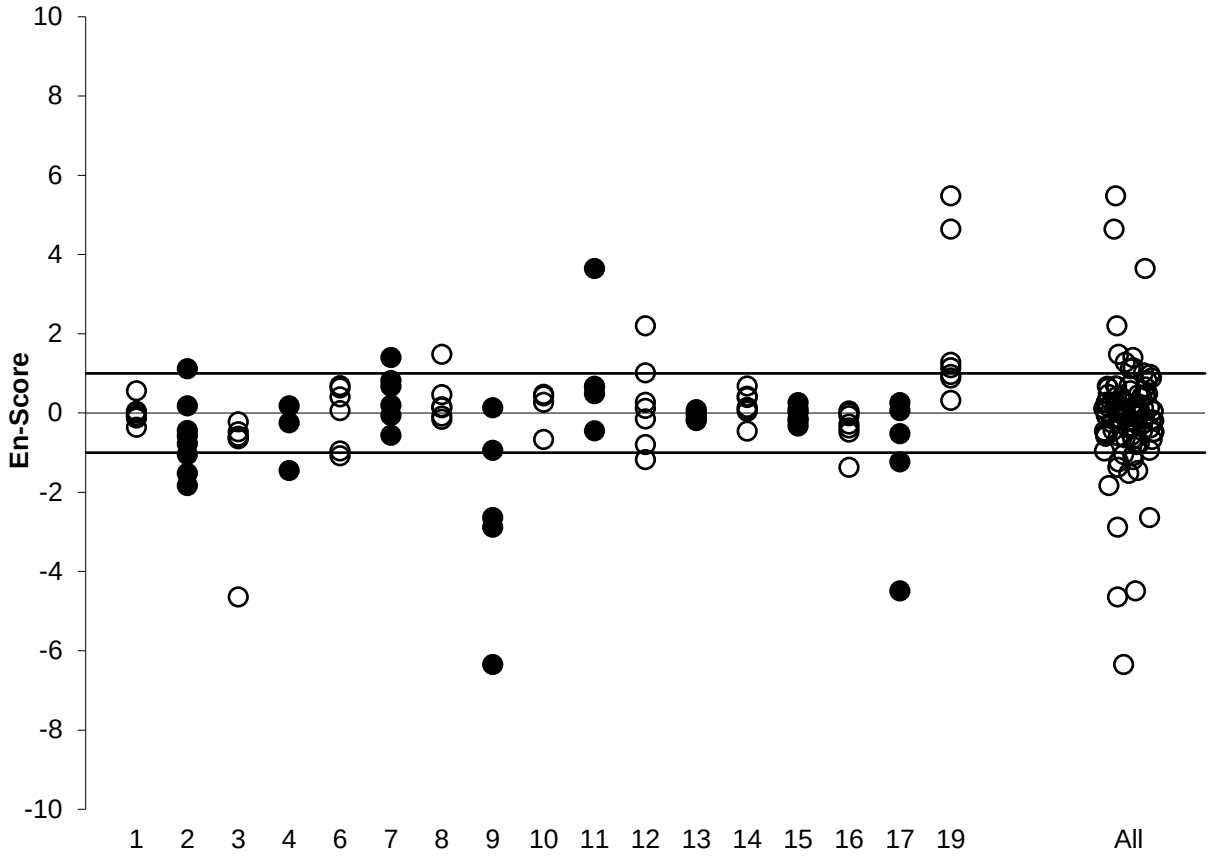


Figure 13 E_n-Score Dispersal by Laboratory

6.5 Range of Pesticides Analysed by Participants

Participants were provided with a list of potential analytes that could have been spiked into the test samples (Table 1). Of these analytes, eight different ones were spiked into the samples for this study (participants were also assessed on total DDT for this study). Participants were not required to test for all potential analytes, and were requested to report 'NT' (for 'Not Tested') for pesticides they did not analyse the samples for.

A summary of the participants' testing of the spiked pesticides is presented in Table 16.

Laboratories **2** and **8** reported testing for all spiked pesticides in this study, as well as reporting for Total DDT. All participants tested for at least one of the spiked pesticides, with the proportion of pesticides analysed by each participant ranging from 44% to 100%.

The proportion of participants analysing each pesticide in this study ranged from 29% (dicamba) to 100% (p,p'-DDE and dieldrin).

Table 16 Summary of Pesticides Analysed by Participants

Lab. Code	Bifenthrin	2,4-D	p,p'-DDE	p,p'-DDT	Total DDT	Diazinon	Dicamba	Dieldrin	Simazine	Proportion of Analytes (%)
1	NT	✓	✓	✓	✓	✓	✓	✓	✓	89
2	✓	✓	✓	✓	✓	✓	✓	✓	✓	100
3	✓	NT	✓	✓	✓	✓	NT	✓	NT	67
4	NT	NT	✓	✓	✓	NT	NT	✓	NT	44
6	✓	✓	✓	✓	NT	✓	NT	✓	✓	78
7	✓	✓	✓	✓	✓	✓	NT	✓	✓	89
8	✓	✓	✓	✓	✓	✓	✓	✓	✓	100
9	✓	NT	✓	NT	NT	✓	NT	✓	✓	56
10	NT	NT	✓	✓	✓	NT	NT	✓	NT	44
11	✓	✓	✓	✓	✓	✓	NT	✓	NT	78
12	✓	✓	✓	✓	✓	✓	NT	✓	✓	89
13	✓	✓	✓	✓	✓	NT	✓	✓	✓	89
14	NT	✓	✓	✓	✓	✓	✓	✓	✓	89
15	✓	NT	✓	✓	✓	✓	NT	✓	NT	67
16	✓	NT	✓	✓	✓	✓	NT	✓	✓	78
17	NT	S1: NT S2: ✓	✓	✓	✓	✓	NT	✓	✓	72
19	✓	NT	✓	✓	✓	✓	NT	✓	✓	78
Proportion of Participants (%)	71	56	100	94	88	82	29	100	71	76

6.6 False Negatives

Table 17 presents false negative results. 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 robust average and spiked value were significantly higher than the participants' LOR (i.e. the sum of the robust average and expanded uncertainty, and the spiked value and expanded uncertainty, were both greater than the LOR), or if no value was reported.

Table 17 False Negatives

Lab. Code	Sample	Analyte	Assigned Value (mg/kg)	Spiked Value (mg/kg)	Result (mg/kg)
4	S1	Dieldrin	0.0641	0.0798	<0.05
17	S2	Diazinon	1.42	2.10	<0.05

6.7 Reporting of Additional Analytes

Additional analytes as reported by participants are presented in Table 18.

Several participants reported for p,p'-DDD in Sample S1, however this sample was spiked with p,p'-DDT and p,p'-DDE only. Samples were stored at 4 °C and so there was unlikely to have been significant breakdown of the p,p'-DDT to p,p'-DDD. The p,p'-DDD reported by participants in this sample may be the result of the break-down of p,p'-DDT during analysis in, for example, hot GC injector liners.[12] Participants reporting p,p'-DDD at significant levels should revise their method to minimise the breakdown.

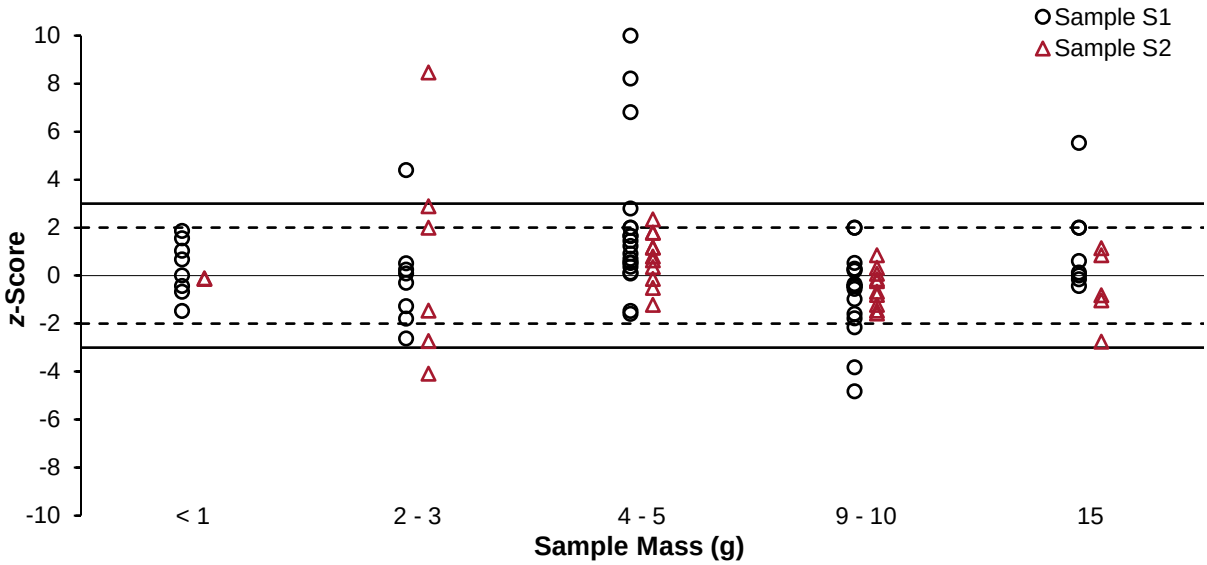
Table 18 Reported Results for Additional Analytes

Lab. Code	Sample	Analyte	Result (mg/kg)	Uncertainty (mg/kg)	Recovery (%)
1	S1	p,p'-DDD	0.01	0.005	85
2	S1	p,p'-DDD	0.026	0.013	NR
4	S1	p,p'-DDD	0.015	0.0045	NR
7	S1	p,p'-DDD	0.068	0.024	76
8	S1	p,p'-DDD	0.022	0.004	NR
9	S1	p,p'-DDD	0.093	NR	NR
11	S1	p,p'-DDD	0.01	0.001	100
	S2	Cypermethrin	0.01	0.001	106
13	S1	p,p'-DDD	0.052	0.035	64
16	S1	p,p'-DDD	0.04	0.012	NR
17	S1	p,p'-DDD	0.057	0.023	NR
19	S1	p,p'-DDD	0.019	0.002	NR

6.8 Participants' Analytical Methods

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

Participants reported using a sample size between 0.1 g and 15 g per analysis. There was no significant trend between the results obtained and the sample mass used for analysis (Figure 14).



z-Scores greater than 10.0 have been plotted at 10.0.

Figure 14 *z*-Score vs Sample Mass Used for Analysis

Participants used a variety of extraction techniques including solid-liquid extraction (SLE), QuEChERS and sonication. Participants also used a range of extraction solvents, such as acetone (ACE), acetonitrile (ACN), dichloromethane (DCM), ethyl acetate (EtOAc), hexane (HEX), methanol (MeOH), toluene (TOL), water, acids and combinations of these solvents. Several participants reported using a clean-up step for their analyses.

Instrumental techniques employed by participants for the analysis of pesticides of interest in this study included gas chromatography (GC) coupled with mass spectrometry (MS), tandem mass spectrometry (MS/MS), electron capture detection (ECD) or flame photometric detection (FPD), liquid chromatography (LC) coupled with MS/MS or diode array detection (DAD), and high performance liquid chromatography (HPLC).

Plots of results reported and methodology used are presented in Figures 15 to 23. If a participant did not report any methodology, this has been recorded as 'NR' (for 'Not Reported'). For scored analytes, participants' results yielding unsatisfactory *z*-scores ($|z| \geq 3.0$) have been circled for reference.

There was a very wide variety of methodologies employed across the analytes in this study, and no significant trend was observed.

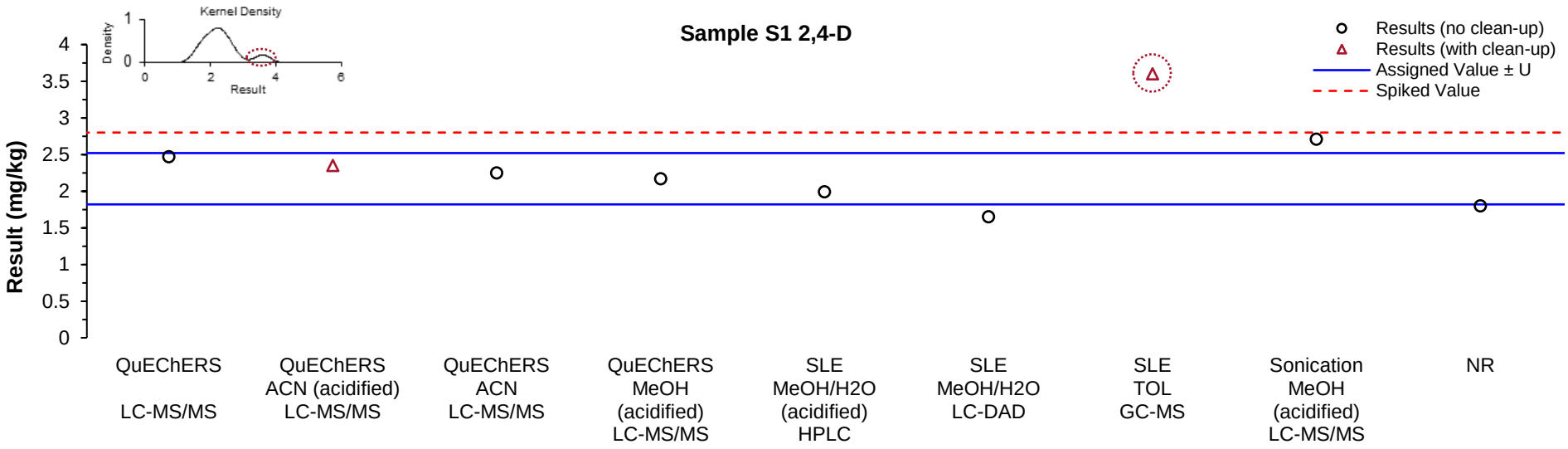


Figure 15 Sample S1 2,4-D Results vs Methodology

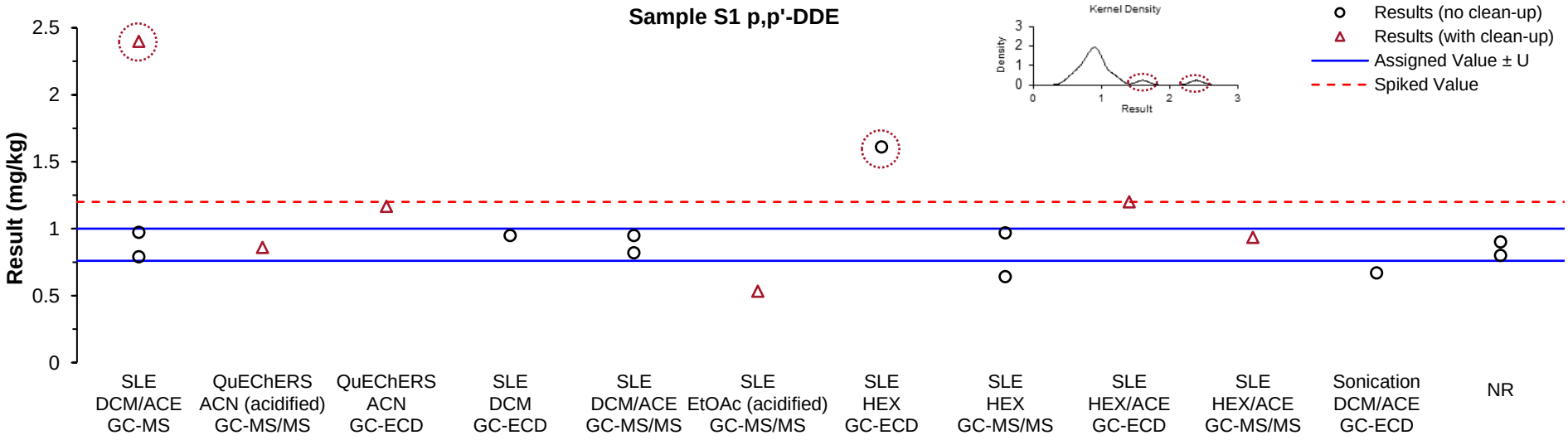


Figure 16 Sample S1 p,p’-DDE Results vs Methodology

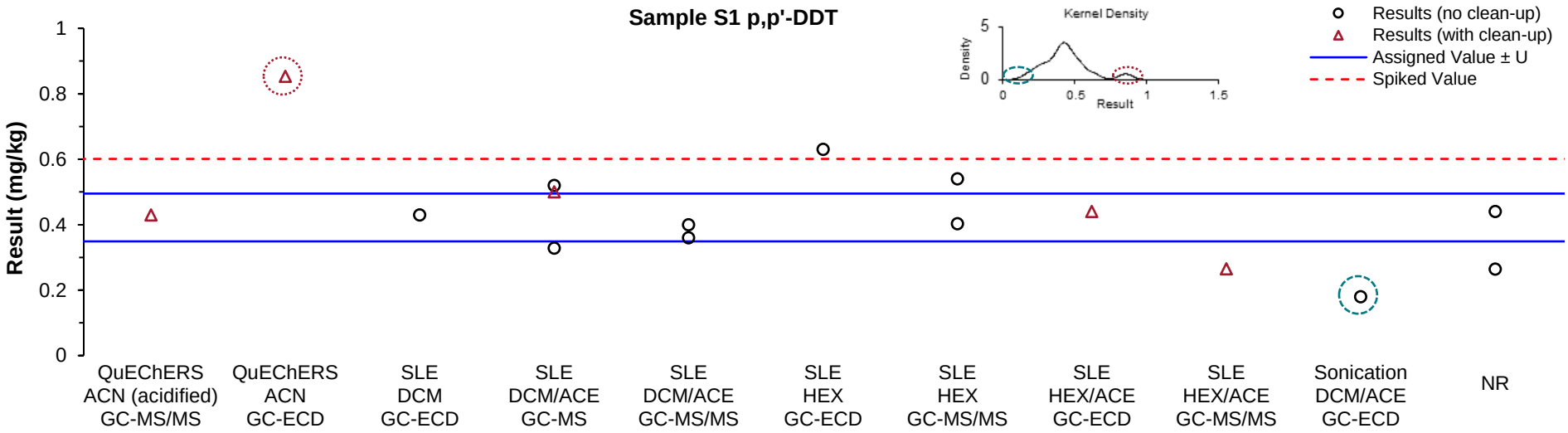


Figure 17 Sample S1 p,p'-DDT Results vs Methodology

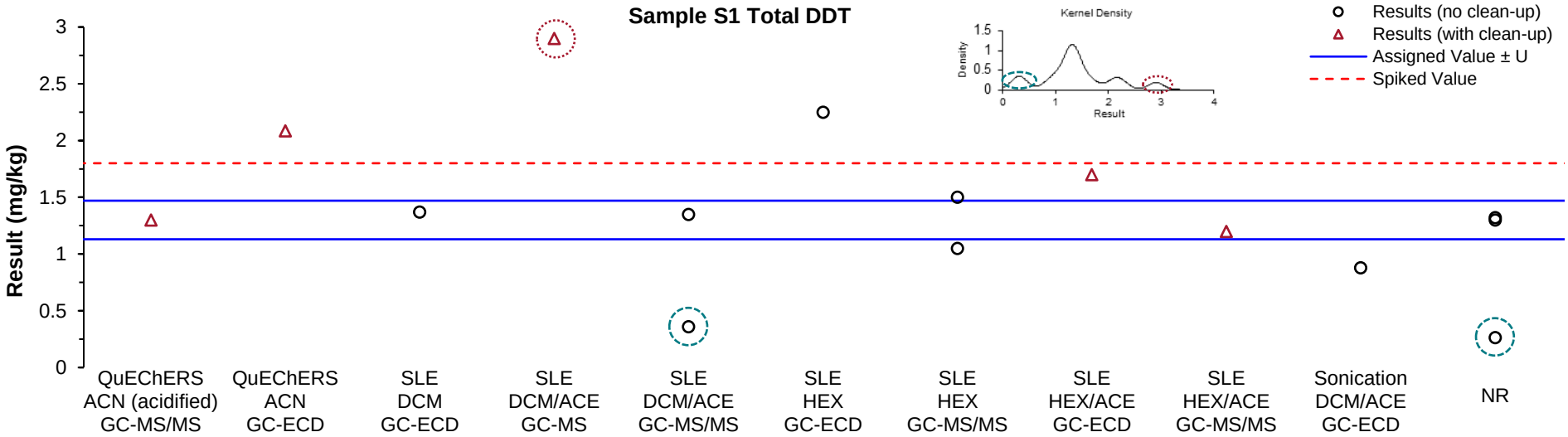


Figure 18 Sample S1 Total DDT Results vs Methodology

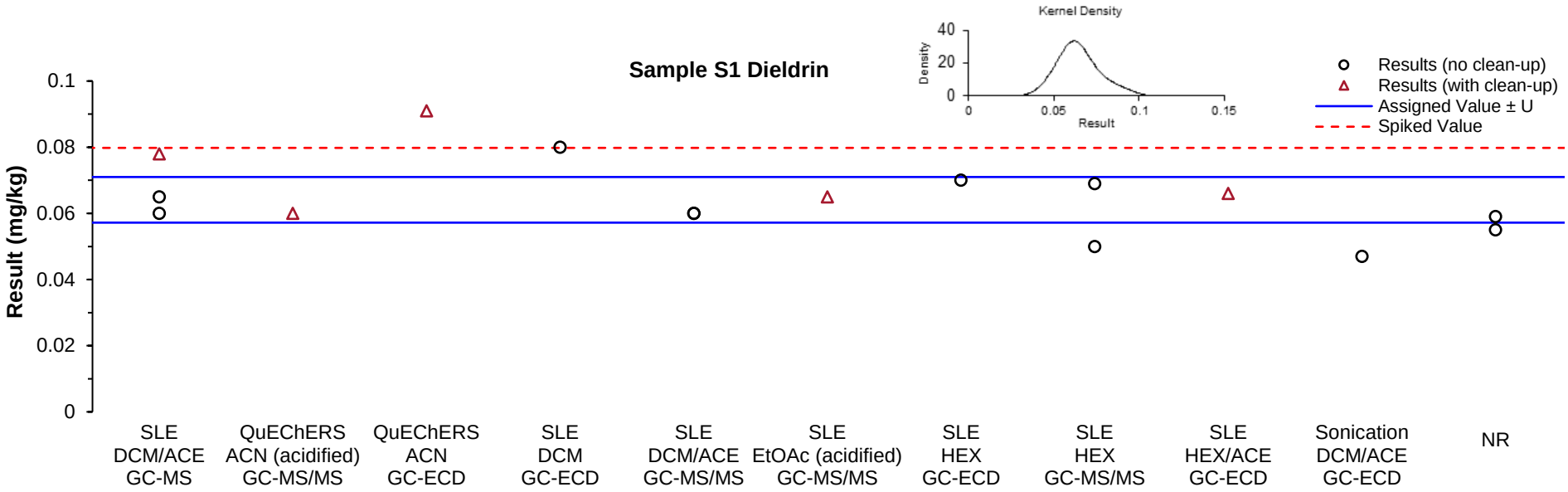


Figure 19 Sample S1 Dieldrin Results vs Methodology

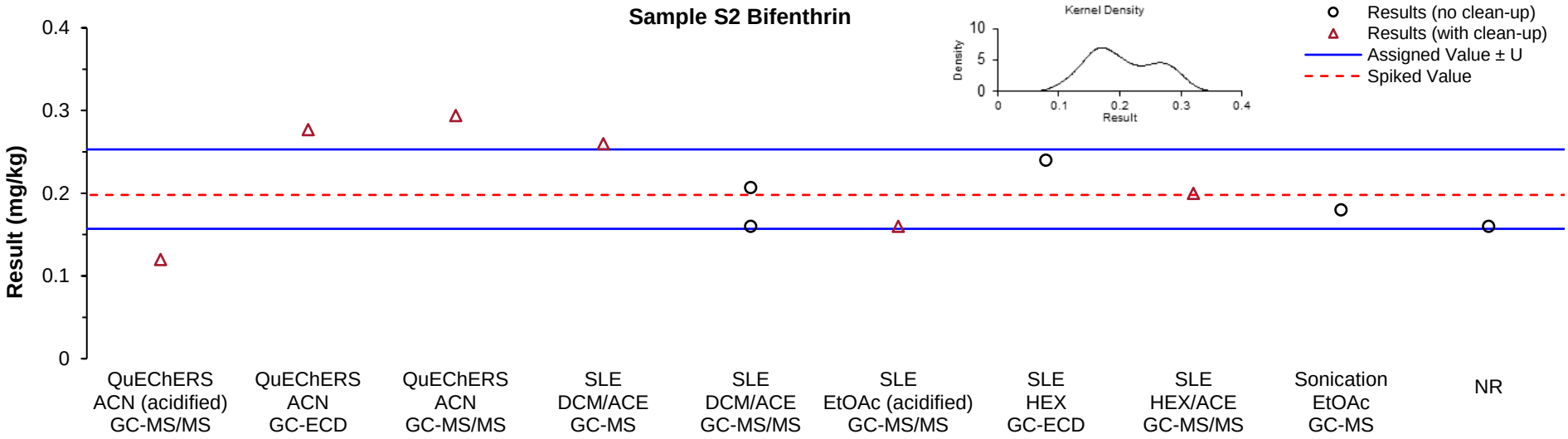


Figure 20 Sample S2 Bifenthrin Results vs Methodology

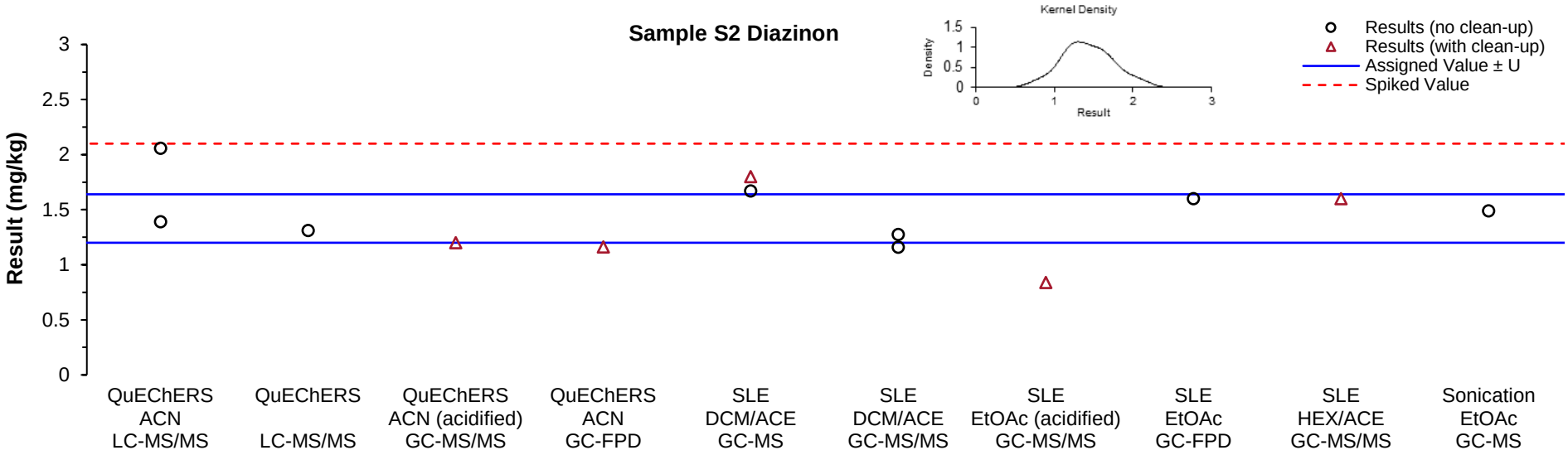


Figure 21 Sample S2 Diazinon Results vs Methodology

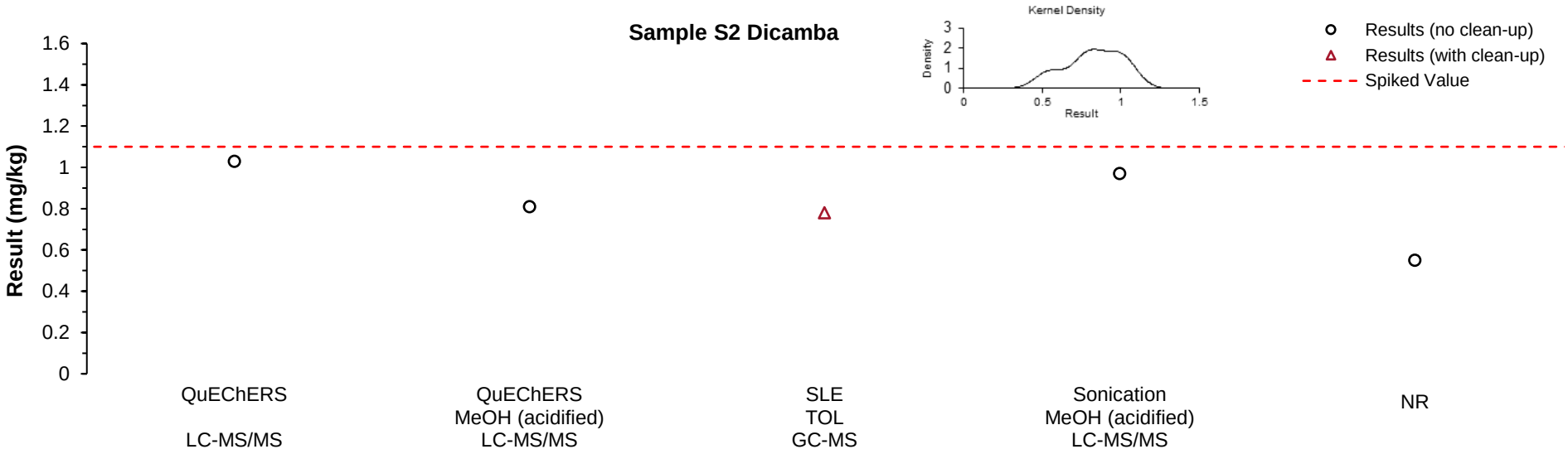


Figure 22 Sample S2 Dicamba Results vs Methodology

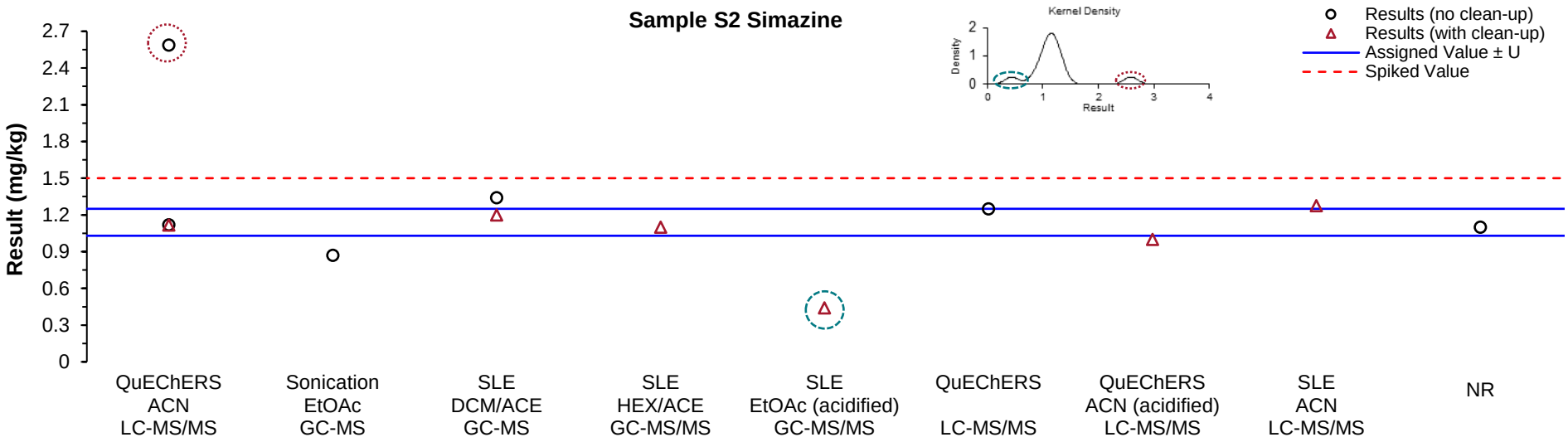


Figure 23 Sample S2 Simazine Results vs Methodology

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**, **7**, **8**, **11**, **12** and **13** reported recoveries for at least one analyte considered in this study, and the recoveries reported were in the range of 38% to 122%. Laboratory **9** reported that they corrected results for recovery.

6.9 Certified Reference Materials (CRM)

Participants were requested to indicate whether certified standards or matrix reference materials had been used as part of the quality assurance for their analysis. Eight participants reported using certified standards, two participants reported using matrix reference materials, and two participants reported using both. The following were listed:

- AccuStandard
- LGC
- Neochema
- Sigma Aldrich (e.g. CRM821, CRM107, SQC009)
- Dr Ehrenstorfer
- o2si
- PM Separations
- Other pesticide 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'*[13]

6.10 Summary of Participants' Results and Performances

Summaries of participants' results and performances for scored analytes in this PT study are presented in Table 19 and Figure 24.

Table 19 Summary of Participants' Results*

Lab. Code	S1 2,4-D	S1 p,p'-DDE	S1 p,p'-DDT	S1 Total DDT	S1 Dieldrin	S2 Bifenthrin	S2 Diazinon	S2 Simazine
AV	2.17	0.88	0.422	1.30	0.0641	0.205	1.42	1.14
SV	2.80	1.20	0.601	1.80	0.0798	0.198	2.10	1.50
1	2.17	0.79	0.52	1.32	0.06	NT	1.39	1.12
2	2.71	0.67	0.18	0.88	0.047	0.18	1.49	0.87
3	NT	0.82	0.36	0.36	0.06	0.16	1.16	NT
4	NT	0.935	0.265	1.2	<0.05	NT	NT	NT
6	1.65	0.973	0.328	NT	0.065	<0.5	1.67	1.34
7	2.352	1.167	0.853	2.087	0.091	0.277	1.161	1.116
8	3.6	1.2	0.44	1.7	0.066	0.20	1.6	1.1
9	NT	0.534	NT	NT	0.065	0.16	0.8387	0.441
10	NT	0.97	0.54	1.5	0.05	NT	NT	NT
11	1.99	1.61	0.63	2.25	0.07	0.24	1.60	NT
12	2.25	0.643	0.403	1.05	0.069	0.294	2.058	2.587
13	1.8	0.80	0.44	1.3	0.059	0.16	NT	1.1
14	2.47	0.95	0.43	1.37	0.08	NT	1.31	1.25
15	NT	0.95	0.4	1.35	0.06	0.207	1.275	NT
16	NT	0.86	0.43	1.3	0.06	0.12	1.2	1.0
17	NT	0.901	0.264	0.264	0.055	NT	<0.05	1.2747
19	NT	2.4	0.50	2.9	0.078	0.26	1.8	1.2

* All results are given in mg/kg. Shaded cells are results which returned a questionable or unsatisfactory *z*-score. AV = Assigned Value; SV = Spiked Value.

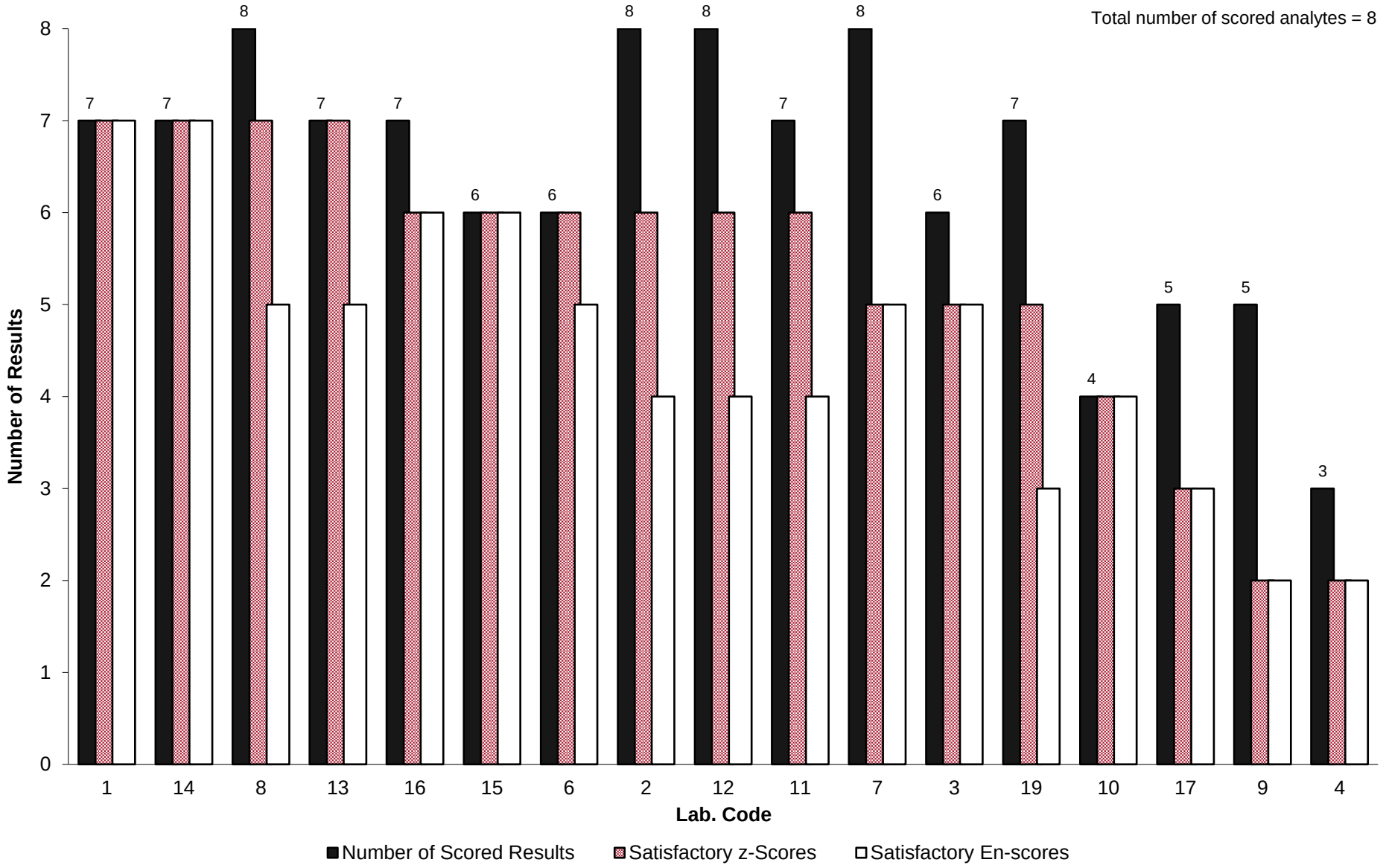


Figure 24 Summary of Participants' Performance

6.11 Comparison with Previous Pesticides in Soil PT Studies

A summary of participation and reported results rates in NMI Pesticides in Soil PT studies over the last 10 studies (2015 – 2023) is presented in Figure 25. The proportion of pesticides being tested for by participants has remained relatively steady over the last few years.

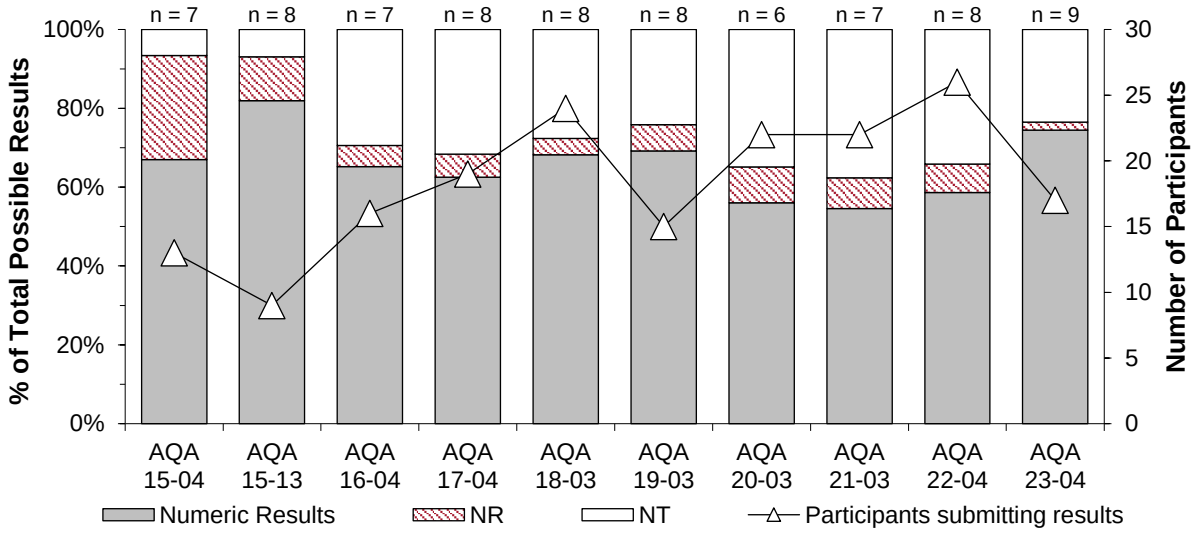


Figure 25 Summary of Participation and Reported Results in Pesticides in Soil PT Studies (n = number of spiked analytes)

A summary of the satisfactory performance (presented as a percentage of the total number of scores for each study) obtained by participants in NMI Pesticides in Soil PT studies over the last 10 studies (2015 – 2023) is presented in Figure 26. To enable direct comparison, the target SD used to calculate *z*-scores has been kept constant at 15% PCV. Over this period, the average proportion of satisfactory *z*-scores and E_n-scores was 84% and 83% respectively.

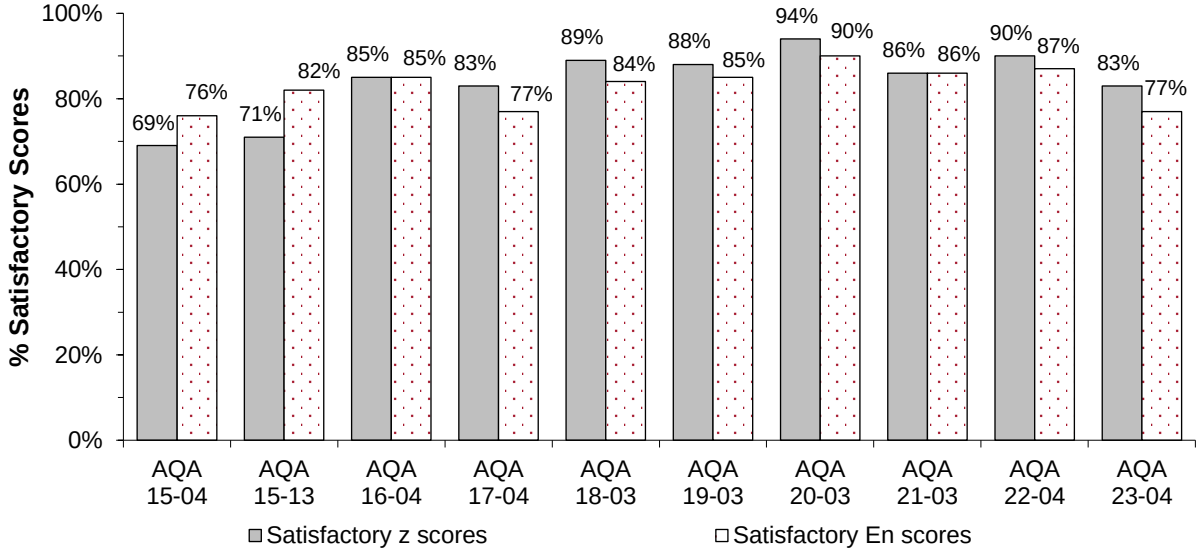


Figure 26 Satisfactory *z*-Scores and E_n-scores in Pesticides in Soil PT Studies

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 27 presents a summary of the relative uncertainties as reported by participants over the last 10 studies (2015–2023). Over this time period, the vast majority of numeric results were reported with uncertainties (96%), with on average 86% of participants in each study reporting that they were accredited to ISO/IEC 17025.

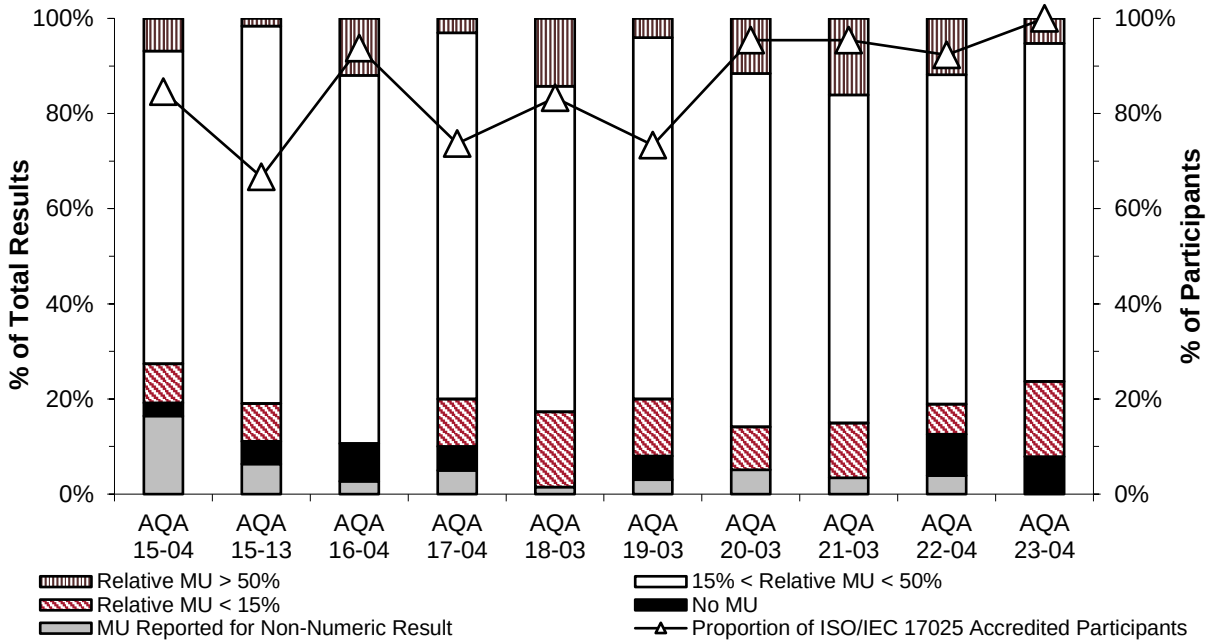


Figure 27 Satisfactory *z*-Scores and E_n-scores in Pesticides in Soil PT Studies

7 REFERENCES

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

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APPENDIX 1 SAMPLE PREPARATION

Forty bottles of each of Sample S1 and Sample S2 were prepared using dried, ground and sieved Australian Native Landscapes Menangle topsoil. The 350 µm to 850 µm fraction was used to prepare the samples.

Sample S1 was prepared by weighing 2201 g of soil into a stainless steel drum, adding acetone to cover the soil, and allowing it to be stirred. The stirred soil suspension was spiked with pesticide standard solutions. The solvent was allowed to evaporate in the fume cupboard. After drying, the soil was divided using a Retsch PT100 sample divider and dispensed into 65 mL glass jars.

Sample S2 was prepared by weighing 2216 g of soil into a stainless steel drum, adding acetone to cover the soil, and allowing it to be stirred. The stirred soil suspension was spiked with pesticide standard solutions. The solvent was allowed to evaporate in the fume cupboard. After drying, the soil was divided using a Retsch PT100 sample divider and dispensed into 65 mL glass jars.

APPENDIX 2 ASSESSMENT OF STABILITY AND HOMOGENEITY

A2.1 Transportation Stability

No stability testing was conducted for this study, though previous use of these pesticides and similar analytes gave some assurance they were stable. Samples were refrigerated at 4 °C after preparation and prior to dispatch. For dispatch, samples were packaged into insulated polystyrene foam boxes with cooler bricks. Comparisons of results to days spent in transit for scored analytes are presented in Figures 28 to 35 (solid lines correspond to the assigned value ± U for each analyte). No significant trend was observed.

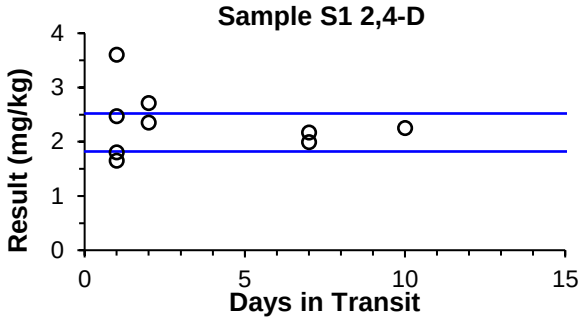

Figure 28 S1 2,4-D Results vs Transit Days

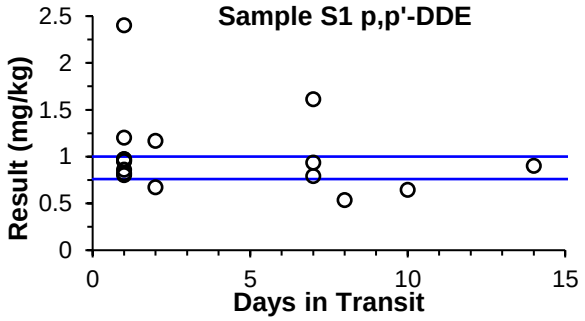

Figure 29 S1 p,p’-DDE Results vs Transit Days

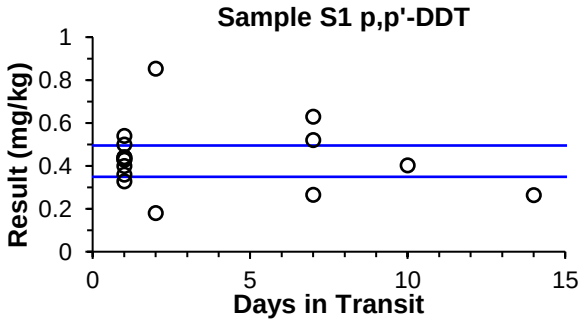

Figure 30 S1 p,p’-DDT Results vs Transit Days

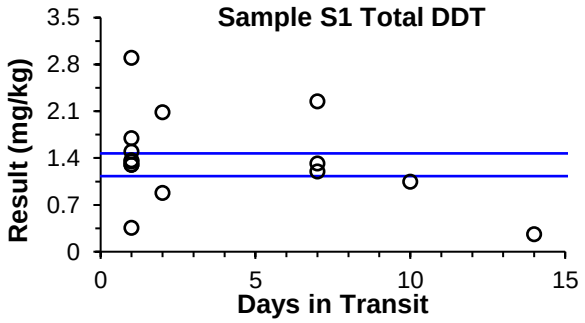

Figure 31 S1 Total DDT Results vs Transit Days

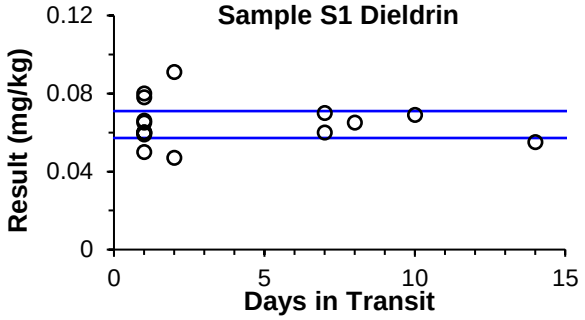

Figure 32 S1 Dieldrin Results vs Transit Days

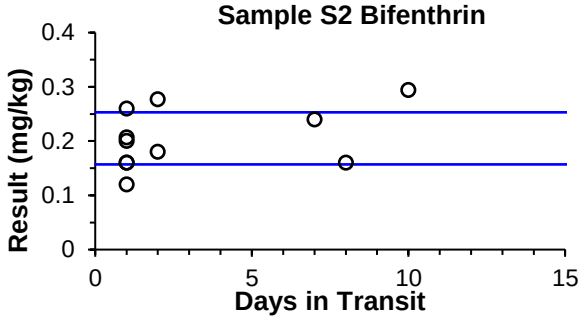

Figure 33 S2 Bifenthrin Results vs Transit Days

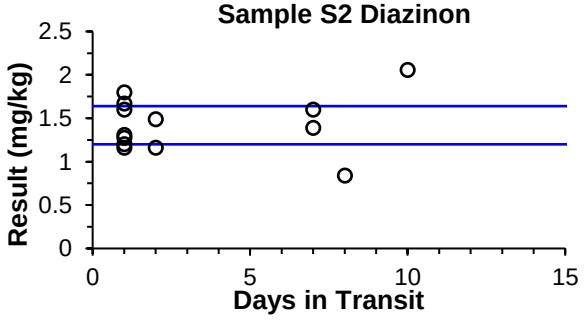

Figure 34 S2 Diazinon Results vs Transit Days

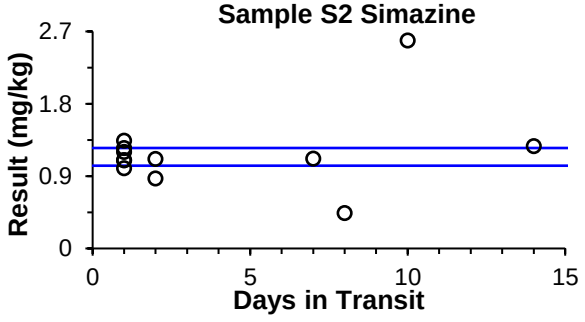

Figure 35 S2 Simazine Results vs Transit Days

A2.2 Homogeneity

No homogeneity testing was completed for this study as the samples were prepared using a process previously demonstrated to produce homogeneous samples. The results of this study also gave no reason to question the samples' homogeneity. Comparisons of results to bottle number for scored analytes are presented in Figures 36 to 43 (solid lines correspond to the assigned value ± U for each analyte). No significant fill order trend was observed.

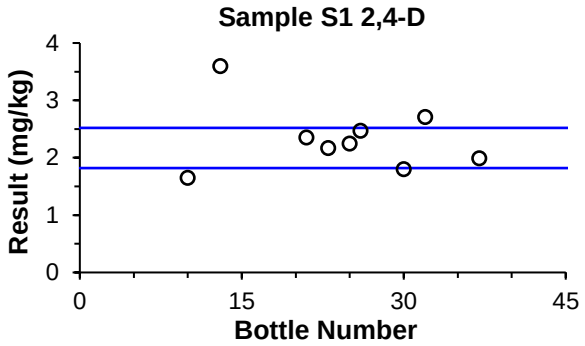

Figure 36 S1 2,4-D Results vs Bottle Number

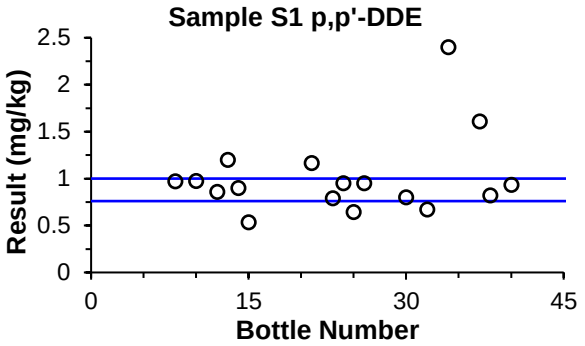

Figure 37 S1 p,p'-DDE Results vs Bottle Number

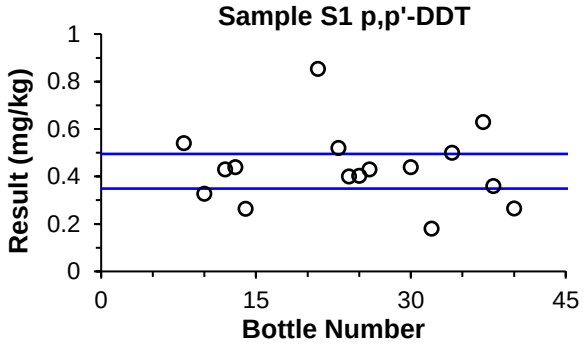

Figure 38 S1 p,p'-DDT Results vs Bottle Number

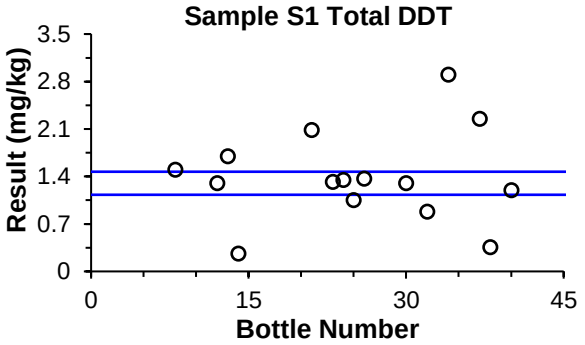

Figure 39 S1 Total DDT Results vs Bottle Number

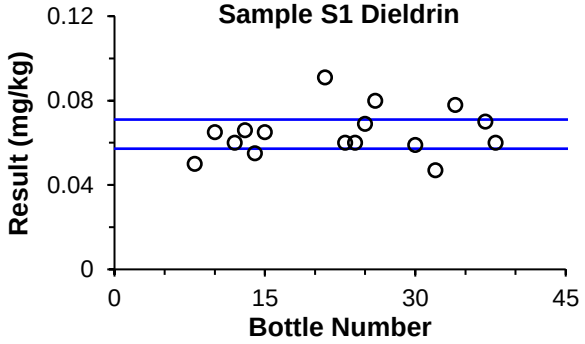

Figure 40 S1 Dieldrin Results vs Bottle Number

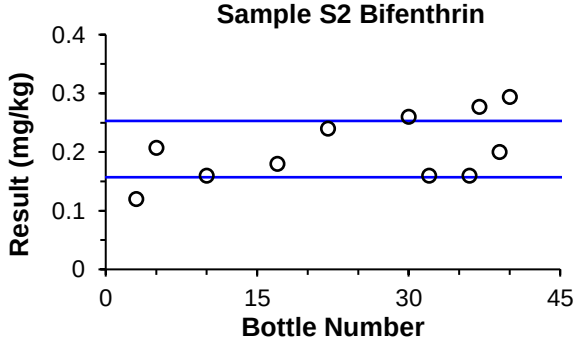

Figure 41 S2 Bifenthrin Results vs Bottle Number

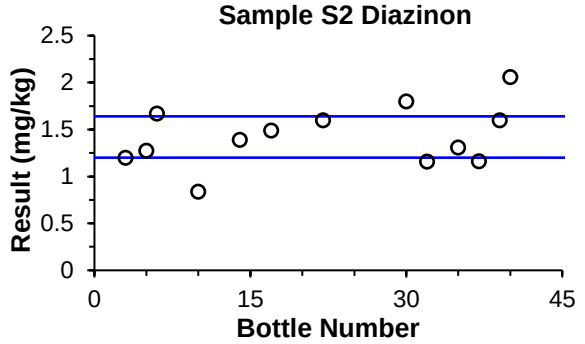

Figure 42 S2 Diazinon Results vs Bottle Number

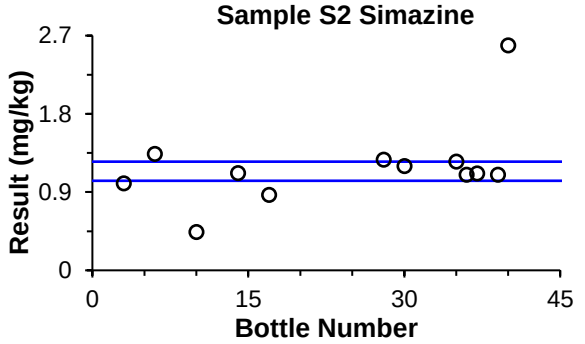

Figure 43 S2 Simazine Results vs Bottle Number

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 estimated 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 S2 diazinon is set out below in Table 20.

Table 20 Uncertainty of the Robust Average for Sample S2 Diazinon

No. results (p)	13
Robust Average	1.42 mg/kg
$S_{rob\ av}$	0.32 mg/kg
$u_{rob\ av}$	0.11 mg/kg
k	2
$U_{rob\ av}$	0.22 mg/kg

Therefore, the robust average for Sample S2 Diazinon is 1.42 ± 0.22 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 is set out below in Table 21.

Table 21 *z*-Score and E_n-Score Calculation for Sample S1 p,p'-DDE Result Reported by Laboratory 1

Participant Result (mg/kg)	Assigned Value (mg/kg)	Target SD	*z*-Score	E_n-Score
0.79 ± 0.22	0.88 ± 0.12	15% as PCV, or: 0.15 × 0.88 = 0.132 mg/kg	$z\text{-Score} = \frac{0.79-0.88}{0.132}$ = -0.68	$E_n\text{-Score} = \frac{0.79-0.88}{\sqrt{0.22^2+0.12^2}}$ = -0.36

APPENDIX 4 TEST METHODS REPORTED BY PARTICIPANTS

Participants were requested to provide information about their test methods. Responses are presented in Tables 22 to 30. Some responses may be modified so that the participant cannot be identified.

Table 22 Methodology – Bifenthrin

Lab. Code	Sample Mass (g)	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument
1	NT				
2	9	Sonication	Ethyl acetate	Nil	GC-MS
3	10	Solid-Liquid	DCM:Ace	N/A	GC-MS/MS
4	NT				
6	4	Solid-Liquid	DCM/ACETONE	Nil	GC-MS
7	5	QuEChERS	Acetonitrile	dSPE	GC-ECD
8	10	Solid-Liquid	Hexane/Acetone	Filtration	GC-MS/MS
9	3	Solid-Liquid	acidified ethyl acetate	PSA	GC-MS/MS
10	NT				
11	15	Solid-Liquid	Hexane	None	GC-ECD
12	2	QuEChERS	ACN	aqua+Cyclohexane shaking	GC-MS/MS
13					
14	NT				
15	10	Solid-Liquid	DCM:Acetone		GC-MS/MS
16	15	QuEChERS	ACN 0.1% Acetic Acid	DSPE	GC-MS/MS
17	NT				
19	4	Solid-Liquid	DCM/Acetone	Centrifuge	GC-MS

Table 23 Methodology – 2,4-D

Lab. Code	Sample Mass (g)	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument
1	0.1 to 0.5	QuEChERS	1%formic/Meoh	None	LC-MS/MS
2	5	Sonication	MeOH:Formic acid 98:2	Nil	LC-MS/MS
3	NT				
4	NT				
6	5	Solid-Liquid	METHANOL/WATER	Nil	LC-DAD
7	5	QuEChERS	5% FA in Acetonitrile	dSPE	LC-MS/MS
8	2	Solid-Liquid	Toluene	Resin column	GC-MS
9	NT				
10	NT				
11	10	Solid-Liquid	Methanol:Water:Acetic acid (80:20:2.5 v/v/v)	None	HPLC
12	2	QuEChERS	ACN	none	LC-MS/MS

Lab. Code	Sample Mass (g)	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument
13					
14	5	QuEChERS			LC-MS/MS
15	NT				
16	NT				
17	5	Solid-Liquid	Acetonitrile	centrifugation	LC-MS/MS
19	NT				

Table 24 Methodology – Diazinon

Lab. Code	Sample Mass (g)	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument
1	0.1 to 0.5	QuEChERS	AcN	None	LC-MS/MS
2	9	Sonication	Ethyl acetate	Nil	GC-MS
3	10	Solid-Liquid	DCM:Ace	N/A	GC-MS/MS
4	NT				
6	4	Solid-Liquid	DCM/ACETONE	Nil	GC-MS
7	5	QuEChERS	Acetonitrile	dSPE	GC-FPD
8	10	Solid-Liquid	Hexane/Acetone	Filtration	GC-MS/MS
9	3	Solid-Liquid	acidified ethyl acetate	PSA	GC-MS/MS
10	NT				
11	15	Solid-Liquid	Ethyl acetate	None	GC-FPD
12	2	QuEChERS	ACN	none	LC-MS/MS
13	NT				
14	5	QuEChERS			LC-MS/MS
15	10	Solid-Liquid	DCM:Acetone		GC-MS/MS
16	15	QuEChERS	ACN 0.1% Acetic Acid	DSPE	GC-MS/MS
17					
19	4	Solid-Liquid	DCM/Acetone	Centrifuge	GC-MS

Table 25 Methodology – Dicamba

Lab. Code	Sample Mass (g)	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument
1	0.1 to 0.5	QuEChERS	1%formic/Meoh	None	LC-MS/MS
2	5	Sonication	MeOH:Formic acid 98:2	Nil	LC-MS/MS
3	NT				
4	NT				
6	NT				
7	NT				
8	2	Solid-Liquid	Toluene	Resin column	GC-MS

Lab. Code	Sample Mass (g)	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument
9	NT				
10	NT				
11	NT				
12	NT				
13					
14	5	QuEChERS			LC-MS/MS
15	NT				
16	NT				
17	NT				
19	NT				

Table 26 Methodology – p,p'-DDE

Lab. Code	Sample Mass (g)	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument
1	0.1 to 1	Solid-Liquid	DCM/acetone	None	GC-MS
2	10	Sonication	DCM:Acetone 1:1	Nil	GC-ECD
3	10	Solid-Liquid	DCM:Ace	N/A	GC-MS/MS
4		Solid-Liquid	Hexane/Acetone	Florisil	GC-MS/MS
6	4	Solid-Liquid	DCM/ACETONE	Nil	GC-MS
7	5	QuEChERS	Acetonitrile	dSPE	GC-ECD
8	10	Solid-Liquid	Hexane/Acetone	Alumina	GC-ECD
9	3	Solid-Liquid	acidified ethyl acetate	PSA	GC-MS/MS
10	0.5	Solid-Liquid	Hexane	None	GC-MS/MS
11	15	Solid-Liquid	Hexane	None	GC-ECD
12	2.5	Solid-Liquid	Hexane	none	GC-MS/MS
13					
14	5	Solid-Liquid	DCM		GC-ECD
15	10	Solid-Liquid	DCM:Acetone		GC-MS/MS
16	15	QuEChERS	ACN 0.1% Acetic Acid	DSPE	GC-MS/MS
17					
19	4	Solid-Liquid	DCM/Acetone	Centrifuge	GC-MS

Table 27 Methodology – p,p'-DDT

Lab. Code	Sample Mass (g)	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument
1	0.1 to 1	Solid-Liquid	DCM/acetone	None	GC-MS
2	10	Sonication	DCM:Acetone 1:1	Nil	GC-ECD
3	10	Solid-Liquid	DCM:Ace	N/A	GC-MS/MS

Lab. Code	Sample Mass (g)	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument
4		Solid-Liquid	Hexane/Acetone	Florisil	GC-MS/MS
6	4	Solid-Liquid	DCM/ACETONE	Nil	GC-MS
7	5	QuEChERS	Acetonitrile	dSPE	GC-ECD
8	10	Solid-Liquid	Hexane/Acetone	Alumina	GC-ECD
9	NT				
10	0.5	Solid-Liquid	Hexane	None	GC-MS/MS
11	15	Solid-Liquid	Hexane	None	GC-ECD
12	2.5	Solid-Liquid	Hexane	none	GC-MS/MS
13					
14	5	Solid-Liquid	DCM		GC-ECD
15	10	Solid-Liquid	DCM:Acetone		GC-MS/MS
16	15	QuEChERS	ACN 0.1% Acetic Acid	DSPE	GC-MS/MS
17					
19	4	Solid-Liquid	DCM/Acetone	Centrifuge	GC-MS

Table 28 Methodology – Total DDT

Lab. Code	Sample Mass (g)	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument
1					
2	10	Sonication	DCM:Acetone 1:1	Nil	GC-ECD
3	10	Solid-Liquid	DCM:Ace	N/A	GC-MS/MS
4		Solid-Liquid	Hexane/Acetone	Florisil	GC-MS/MS
6	NT				
7	5	QuEChERS	Acetonitrile	dSPE	GC-ECD
8	10	Solid-Liquid	Hexane/Acetone	Alumina	GC-ECD
9	NT				
10	0.5	Solid-Liquid	Hexane	None	GC-MS/MS
11	15	Solid-Liquid	Hexane	None	GC-ECD
12	2.5	Solid-Liquid	Hexane	none	GC-MS/MS
13					
14	5	Solid-Liquid	DCM		GC-ECD
15	10	Solid-Liquid	DCM:Acetone		GC-MS/MS
16	15	QuEChERS	ACN 0.1% Acetic Acid	DSPE	GC-MS/MS
17					
19	4	Solid-Liquid	DCM/Acetone	Centrifuge	GC-MS

Table 29 Methodology – Dieldrin

Lab. Code	Sample Mass (g)	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument
1	0.1 to 1	Solid-Liquid	DCM/acetone	None	GC-MS
2	10	Sonication	DCM:Acetone 1:1	Nil	GC-ECD
3	10	Solid-Liquid	DCM:Ace	N/A	GC-MS/MS
4					
6	4	Solid-Liquid	DCM/ACETONE	Nil	GC-MS
7	5	QuEChERS	Acetonitrile	dSPE	GC-ECD
8	10	Solid-Liquid	Hexane/Acetone	Alumina	GC-ECD
9	3	Solid-Liquid	acidified ethyl acetate	PSA	GC-MS/MS
10	0.5	Solid-Liquid	Hexane	None	GC-MS/MS
11	15	Solid-Liquid	Hexane	None	GC-ECD
12	2.5	Solid-Liquid	Hexane	none	GC-MS/MS
13					
14	5	Solid-Liquid	DCM		GC-ECD
15	10	Solid-Liquid	DCM:Acetone		GC-MS/MS
16	15	QuEChERS	ACN 0.1% Acetic Acid	DSPE	GC-MS/MS
17					
19	4	Solid-Liquid	DCM/Acetone	Centrifuge	GC-MS

Table 30 Methodology – Simazine

Lab. Code	Sample Mass (g)	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument
1	0.1 to 0.5	QuEChERS	AcN	None	LC-MS/MS
2	9	Sonication	Ethyl acetate	Nil	GC-MS
3	NT				
4	NT				
6	4	Solid-Liquid	DCM/ACETONE	Nil	GC-MS
7	5	QuEChERS	Acetonitrile	dSPE	LC-MS/MS
8	10	Solid-Liquid	Hexane/Acetone	Filtration	GC-MS/MS
9	3	Solid-Liquid	acidified ethyl acetate	PSA	GC-MS/MS
10	NT				
11	NT				
12	2	QuEChERS	ACN	none	LC-MS/MS
13					
14	5	QuEChERS			LC-MS/MS
15	NT				
16	15	QuEChERS	ACN 0.1% Acetic Acid	DSPE	LC-MS/MS
17	5	Solid-Liquid	Acetonitrile	centrifugation	LC-MS/MS
19	4	Solid-Liquid	DCM/Acetone	Centrifuge	GC-MS

APPENDIX 5 ACRONYMS AND ABBREVIATIONS

2,4-D	2,4-Dichlorophenoxyacetic acid
ACE	Acetone
ACN	Acetonitrile
AV	Assigned Value
CITAC	Cooperation on International Traceability in Analytical Chemistry
CRM	Certified Reference Material
CV	Coefficient of Variation
DAD	Diode Array Detection
DCM	Dichloromethane
dSPE	Dispersive Solid Phase Extraction
ECD	Electron Capture Detection
EtOAc	Ethyl Acetate
FPD	Flame Photometric Detection
GAG	General Accreditation Guidance (NATA)
GC	Gas Chromatography
GUM	Guide to the expression of Uncertainty in Measurement
HEX	Hexane
HPLC	High Performance Liquid Chromatography
IEC	International Electrotechnical Commission
ISO	International Organization for Standardization
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
NMI	National Measurement Institute, Australia
NR	Not Reported

NT	Not Tested
p,p’-DDD	Dichlorodiphenyldichloroethane
p,p’-DDE	Dichlorodiphenyldichloroethylene
p,p’-DDT	Dichlorodiphenyltrichloroethane
PCV	Performance Coefficient of Variation
PSA	Primary-Secondary Amine
PT	Proficiency Testing
QuEChERS	Quick, Easy, Cheap, Effective, Rugged, and Safe preparation method
RA	Robust Average
Rec	Recovery
RM	Reference Material
SD	Standard Deviation
SI	International System of Units
SLE	Solid-Liquid Extraction
SS	Spiked Samples
SV	Spiked Value
TOL	Toluene
Total DDT	Sum of DDD, DDE and DDT analytes
U	Expanded Uncertainty

END OF REPORT