Australian Government
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National Measurement Institute

Proficiency Test Final Report AQA 21-06 Pesticides in Fruit & Vegetables

September 2021

ACKNOWLEDGMENTS

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

Jenny Xu

Geoff Morschel

Beth Tully

Raluca Iavetz

Manager, Chemical Reference Values

105 Delhi Rd, North Ryde, NSW 2113, Australia

Phone: +61 2 9449 0178

Email: raluca.iavetz@measurement.gov.au

Accredited for compliance with ISO/IEC 17043

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SUMMARY

AQA 21-06 Pesticides in Fruit & Vegetables commenced in May 2021. Twenty-one laboratories registered to participate, and all participants submitted results.

Four sets of test samples were prepared at the Sydney NMI laboratory. Samples were prepared by adding pesticide standard solutions to pureed tomatoes (Sample S1), bok choy (Sample S2), apples (Sample S3) and oranges (Sample S4).

Of a possible 336 results, 217 numeric results (65%) were submitted. Of the remaining results, 23 results were a 'less than' value (<*x*) or Not Reported (NR), and 96 results were 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 pesticides in fruit and vegetables.*

Laboratories **3**, **5**, **16**, **20** and **21** reported numeric results for all 15 scored analytes.

Eight participants did not report results for analytes that they tested for and were present in the test samples (total of 23 results).

Three participants reported analytes that were not spiked into the samples (total of six results).

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

Of 199 results for which z-scores were calculated, 154 (77%) returned $|z| \leq 2.0$, indicating a satisfactory performance.

Of 199 results for which E_n-scores were calculated, 149 (75%) returned $|E_n| \leq 1.0$, indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

No participant reported results and returned satisfactory z-scores and E_n-scores for all 15 scored analytes. All results reported by Laboratories **7** and **14** (14) returned satisfactory z-scores and E_n-scores.

- *Assess the ability of participants to assess compliance of pesticides in fruit and vegetables against regulatory standards.*

Of 184 results assessed, 132 (72%) gave the correct compliance status (inclusive of uncertainty), while 31 (17%) gave conditionally correct compliance statuses.

No participant reported results for and returned the correct compliance status for all 13 analytes assessed. All assessed results reported by Laboratory **14** (12) returned the correct compliance status.

- *Evaluate the participants' methods for the measurement of pesticides in fruit and vegetables*

Participants used a variety of methods, and no significant trends with any particular sample preparation method or instrumental technique was evident.

- *Develop the practical application of traceability and measurement uncertainty*

Of 217 numerical results, 178 (82%) were reported with an associated expanded measurement uncertainty. The magnitude of the reported uncertainties was within the range 0.044% to 150000% relative.

Laboratories **2**, **6**, **7**, **9**, **10**, **11** and **15** did not provide uncertainties for at least one reported result.

- *Compare the performance of participants with past performance.*

While the number of spiked analytes per study has been increasing over the last several studies, the proportion of numeric results reported by participants has remained fairly steady over this period. The proportion of satisfactory z-score and E_n-scores has been increasing overall across the last several studies.

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

The test samples from this study are homogeneous and are well characterised. Surplus of these samples are available for purchase from NMI and can be used for quality control and 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 interlaboratory 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 PT studies in:

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

1.2 Study Aims

The aims of the study were to:

- assess the ability of participants to correctly identify pesticides in fruit and vegetables;
- compare the performances of participants and assess their accuracy in the measurement of pesticides in fruit and vegetables;
- assess the ability of participants to assess compliance of pesticides in fruit and vegetables against regulatory standards;
- evaluate participants' methods for the measurement of pesticides in fruit and vegetables;
- develop the practical application of traceability and measurement uncertainty;
- compare the performance of participants with past performance; 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 proficiency tests 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,[1] and The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories.[4]

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

2 STUDY INFORMATION

2.1 Selection of Pesticides and Matrices

A list of possible analytes for the samples in this study is presented in Table 1.

Table 1 List of Possible Analytes

Abamectin	Cypermethrin	Fenthion sulfone	Metrafenone
Acetamiprid	Cyprodinil	Fenthion sulfoxide	Mevinphos
Azinphos-methyl	2,4-D	Fenvalerate	Monocrotophos
Azoxystrobin	p,p'-DDT	Fludioxonil	Omethoate
Bifenazate	Deltamethrin	Glyphosate	Parathion
Bifenthrin	Diazinon	Imazalil	Parathion methyl
Buprofezin	Dicofol	Imidacloprid	Penconazole
Captan	Dieldrin	Indoxacarb	Permethrin
Carbaryl	Dimethoate	Iprodione	Pirimicarb
Carbendazim	Dithiocarbamates	Linuron	Procymidone
Chlorfenvinphos	alpha-Endosulfan	Maldison	Profenofos
Chlorothalonil	beta-Endosulfan	Metalaxyl	Propargite
Chlorpyrifos	Endosulfan sulfate	Methamidophos	Pyraclostrobin
Clothianidin	Fenamiphos	Methidathion	Spinosad
Cyfluthrin	Fenitrothion	Methomyl	Thiabendazole
Cyhalothrin	Fenthion	Methomyl oxime	Triadimefon

The spiked values for the samples and corresponding Australian maximum residue limits (MRLs) are presented in Table 2. For matrix and analyte selection, consideration was given to:

- a variety of pesticides, including some amenable to gas and liquid chromatography;
- a variety of matrices, and the availability of matrix material with incurred analytes;
- feedback from participants;
- current Australian agricultural practice; and
- Australian MRLs in the Australia New Zealand Food Standards (ANZFS) Code.[5]

Table 2 Spiked Values of Test Samples

Sample	Analyte	Spiked Value (mg/kg)	Uncertainty (mg/kg)[a]	MRL (mg/kg)[b]
S1 (Tomato)	Cyhalothrin	0.0458	0.0023	0.02[c]
	Dimethoate	0.0548	0.0027	0.02[d]
	Endosulfan sulfate	0.865	0.043	-
	Omethoate	2.28	0.11	1
S2 (Bok Choy)	Cyfluthrin	0.902	0.045	0.5[c]
	Glyphosate	0.250	0.013	*0.1[e]
	Indoxacarb	3.01	0.15	5[f]
	Pyraclostrobin	1.20	0.06	T3

Sample	Analyte	Spiked Value (mg/kg)	Uncertainty (mg/kg)[a]	MRL (mg/kg)[b]
S3 (Apple)	Acetamiprid	0.172	0.009	0.2
	Carbendazim	0.496	0.025	0.2[g]
	Pyraclostrobin	0.0909	0.0045	1
	Triadimefon	2.01	0.10	1[h]
S4 (Orange)	Acetamiprid	1.90	0.09	1
	Azoxystrobin	6.05	0.30	10
	Cyfluthrin	0.220	0.011	0.2[c]
	Imidacloprid	2.79	0.14	2[i]

[a] Estimated expanded uncertainty at 95% confidence interval using a coverage factor of 2.

[b] '*' indicates that the MRL is set at the limit of determination; 'T' indicates that the MRL is a temporary maximum residue limit. In some cases, MRLs are for the sum of a number of different permitted residues.

[c] Sum of isomers.

[d] Sum of dimethoate and omethoate

[e] Sum of glyphosate, N-acetyl-glyphosate and aminomethylphosphonic acid (AMPA) metabolite

[f] Sum of indoxacarb and its R-isomer

[g] Sum of carbendazim and 2-aminobenzimidazole

[h] Sum of triadimefon and triadimenol

[i] Sum of imidacloprid and metabolites containing the 6-chloropyridinylmethylene moiety

2.2 Study Timetable

The timetable of the study was:

Invitation issued 18 May 2021

Samples dispatched 21 June 2021

Results due 2 August 2021

Interim report issued 4 August 2021

2.3 Participation and Laboratory Code

Twenty-one laboratories registered to participate, and all participants were assigned a confidential laboratory code number for this study. All participants submitted results.

2.4 Sample Preparation

Four test samples were prepared by adding pesticide standard solutions to pureed tomatoes (Sample S1), bok choy (Sample S2), apples (Sample S3) and oranges (Sample S4). Additional sample preparation details are provided in Appendix 1.

2.5 Homogeneity of Samples and Stability of Analytes

The samples were prepared and packaged using a process that has been demonstrated to produce homogeneous samples for previous NMI Pesticides in Fruit & Vegetables PT studies. No homogeneity testing was conducted for this study, and the results returned by participants gave no reason to question the homogeneity of these samples (Appendix 2).

No stability testing was conducted for this study. Reports in the Joint FAO/WHO Meeting on Pesticide Residues (JMPR) database,[6] together with previous use of these analytes in NMI PT studies, gave some assurance that the analytes selected were stable in frozen fresh produce. To assess possible instability, the results returned by participants were compared to the spiked concentration. Robust averages of the results were 76% to 107% of the spiked values, which

were similar to values observed in previous studies, and gave no reason to question their stability. Transportation stability was also considered by comparing results and the number of days samples spent in transit, and there no evidence of analyte degradation (Appendix 2).

2.6 Samples Storage and Dispatch

Prior to dispatch, the samples were stored in a freezer at approximately -20 °C. Participants were sent 100 g portions of both spiked and unspiked Samples S1, S2, S3 and S4. The samples were packaged into insulated polystyrene foam boxes with cooler bricks and dispatched by courier on 21 June 2021.

The following items were also sent to participants:

- a letter which included a description of the test samples and instructions for participants; and
- a form for participants to return to confirm receipt and condition of the test 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.
- The unspiked material need not be analysed, it is provided for participants to use if they wish.
- Participants need not test for all analytes listed.
- Please thaw and thoroughly mix the PT samples before analysis.
- For each analyte in each sample report a single result on as received basis in units of mg/kg expressed as if reporting to a client (i.e. corrected for recovery or not, according to your standard procedure). This figure will be used in all statistical analysis in the study report.
- For each analyte in each sample report the associated expanded measurement uncertainty (e.g. 0.50 ± 0.02 mg/kg), if determined.
- Report any listed pesticide not tested as NT.
- Do not correct results for any pesticide found in the unspiked sample.
- No limit of reporting has been set for this study. Report results as you would to a client, applying the limit of reporting of the method used for analysis.
- Give details of your methodology and basis of uncertainty estimate as requested by the results sheet emailed to you.
- If determined, report your percentage recovery. This will be presented in the report for information only.
- Return the completed results sheet by 19 July 2021 by email to proficiency@measurement.gov.au.

The results due date was later extended to 2 August 2021 due to delays with sample delivery to some participants.

2.8 Interim Report

An interim report was emailed to all participants on 4 August 2021.

3 PARTICIPANT LABORATORY INFORMATION

3.1 Test Methods Reported by Participants

Participants were requested to provide information about their test methods. Responses are presented in Appendix 3.

3.2 Basis of Participants' Measurement Uncertainty Estimates

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

Table 3 Basis of MU 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	Duplicate analysis	Recoveries of SS	NMI Uncertainty Course
2				
3	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - SS Duplicate analysis	Recoveries of SS	NATA Technical Note 33
4	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis Instrument calibration	Instrument calibration Recoveries of SS Standard purity	Analytical Quality Control and Method Validation Procedures for Pesticide Residues Analysis
5	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis	Recoveries of SS	Eurachem/CITAC Guide
6		Duplicate analysis	Recoveries of SS	
7	Standard deviation of replicate analyses multiplied by 2 or 3	Duplicate analysis	Instrument calibration Recoveries of SS	Eurachem/CITAC Guide
8	Top Down - precision and estimates of the method and laboratory bias	Duplicate analysis Instrument calibration	Recoveries of SS	SANTE 12682/2019
9	Top Down - precision and estimates of the method and laboratory bias	Control samples Duplicate analysis Instrument calibration	Recoveries of SS	SANTE 12682/2019
10	Top Down - precision and estimates of the method and laboratory bias	Control samples - RM	Standard purity	Eurachem/CITAC Guide
11	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS	Recoveries of SS	Eurachem/CITAC Guide
12	Top Down - precision and estimates of the method and laboratory bias	Control samples	CRM Recoveries of SS Standard purity	ISO/GUM

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
13	Bottom Up (ISO/GUM, fish bone/cause and effect diagram)	Duplicate analysis	Recoveries of SS	ISO/GUM
14	Horwitz formula	Control samples - SS Duplicate analysis Instrument calibration	Recoveries of SS Standard purity	NMI Uncertainty Course
15	Bottom Up (ISO/GUM, fish bone/cause and effect diagram)	Control samples - SS	Recoveries of SS Standard purity	Eurachem/CITAC Guide
16	Top Down - precision and estimates of the method and laboratory bias	Instrument calibration	CRM Recoveries of SS Standard purity	ISO/GUM
17	Top Down - precision and estimates of the method and laboratory bias	Duplicate analysis	Instrument calibration Recoveries of SS Standard purity	SANTE 12682/2019
18	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS	Recoveries of SS	SANTE 12682/2019
19	Top Down - precision and estimates of the method and laboratory bias	Duplicate analysis Instrument calibration	Instrument calibration Recoveries of SS	Eurachem/CITAC Guide
20	Top Down - reproducibility (standard deviation) from PT studies used directly	Control samples - SS	Laboratory bias from PT studies	SANTE 12682/2019
21	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS		NATA Technical Note 33

* CRM = Certified Reference Material; RM = Reference Material; SS = Spiked Samples

3.3 Participants' Comments

Participants were invited to make any comments on the samples, this study, or possible future studies. Such feedback may be useful in improving future studies. Participants' comments, and the study coordinator's response (if applicable) are presented in Table 4. Some responses were modified so that the participant cannot be identified.

Table 4 Participants' Comments

Lab. Code	Sample	Participant's Comments	Study Coordinator's Response
7	S2	S2 untreated found incurred residue of Terbacil @ 0.033 mg/kg.	
8	S2	The containers of the spiked and unspiked samples were broken. We failed to double check the bottles upon receipt.	Please check samples on receipt, and report any problems as soon as possible, so that NMI can replace the sample if deemed necessary. Sample receipt notification from your laboratory had marked the received samples as fit for analysis.

Lab. Code	Sample	Participant's Comments	Study Coordinator's Response
9	S1	Endosulfan ether 0.08mg/kg; Dimethoate is reported as dimethoate only; not FSANZ Schedule 20 definition for Dimethoate (expressed as the sum of dimethoate and omethoate)	Laboratories from different jurisdictions may have different reporting requirements and therefore participants should report their results for the named analyte to ensure consistency of results. Participants can also add comments if they have further information. We will clarify this in future studies. With regards to the results form, we do in-house checking prior to sending it to participants. While we have not encountered any issues ourselves, we will continue to monitor if other participants also report having issues with the results form.
	S3	The report contains the result for Triademefon as triadimefon only. Schedule 20 lists triademefon, as "Sum of triadimefon and triadimenol, expressed as triadimefon"	
	All	It is not clear how you want some results reported. Schedule 20 has residue definitions which include some degradants. In future, it would be useful to have this clarified. We have assumed that this is focused on analytical results, the data should be expressed in a different way. Uncertainty: PS: This form has caused numerous crashes. Not sure why but I'm giving up adding data.	
10	S1	Endosulfan Sulfate and Cyhalothrin are not under laboratory scope	
	S2, S4	Cyfluthrin is not under laboratory scope	
	S3	ND for all tested items	
13	All	Not all analytes are ISO 17025 accredited	
16	All	Our laboratory performs trace level analysis of fruit and vegetables at a sub ppm range (LOR 0.01mg/kg); most residues encountered in this study were in the ppm range. This results in needing to dilute samples which is uncommon for us, resulting in an extra step and source of bias.	Analytes were spiked at a variety of levels to cater for the needs of different participant laboratories. In this study, analytes were spiked at the sub-ppm to ppm range (ranging from 0.0458 mg/kg to 6.05 mg/kg)
17	All	For future studies, we wish to include the following pesticides in the spiked samples, such as cyhalothrin, permethrin, cyfluthrin, cypermethrin, fenvalerate, deltamethrin, isazophos, dimethoate, diazinon or mevinphos.	We will take these analyte suggestions into consideration when planning future PT studies. In this study, samples were spiked with cyhalothrin, cyfluthrin and dimethoate.
18	S1, S2, S4	The concentration of residue reported is an average of three determinations made on the same sample. The unspiked sample was also analysed and found to have no residues at or above the Limit of Quantitation (LOQ) at 0.01 mg/kg. The reported uncertainty of result is an expanded uncertainty calculated using a coverage factor of 2 which gives a level of confidence of approximately 95%.	
	S3	No pesticide residues were detected at and above the LOQ on three determinations made on the same sample. The unspiked sample was also analysed and found to have no residues at or above the Limit of Quantitation (LOQ) at 0.01 mg/kg.	
	All	This PT is important for the reliability and assessment of our laboratory's results, and also for compliance to ISO/IEC 17025 accreditation. We would like to suggest PT studies for pesticide residues in other sample matrices such as rice, banana, pineapple, mango and water.	NMI currently runs a Pesticides in Water PT study annually. The other matrix suggestions will be taken into consideration when planning future PT studies.

Lab. Code	Sample	Participant's Comments	Study Coordinator's Response
		Uncertainty: The reported uncertainty of result is an expanded uncertainty calculated using a coverage factor of 2 which gives a level of confidence of approximately 95%.	
19	All	Coordinate shipment accordingly. Ensure conditions are met	Samples are packaged with cooler bricks in insulated boxes and are dispatched with couriers. While we follow-up with the couriers if there are any delivery issues, we unfortunately have no involvement over the time required for sample delivery and/or customs clearance (for international participants).

4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

4.1 Results Summary

Participant results are listed in Tables 5 to 20 with 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 17. An example chart with interpretation guide is shown in Figure 1.

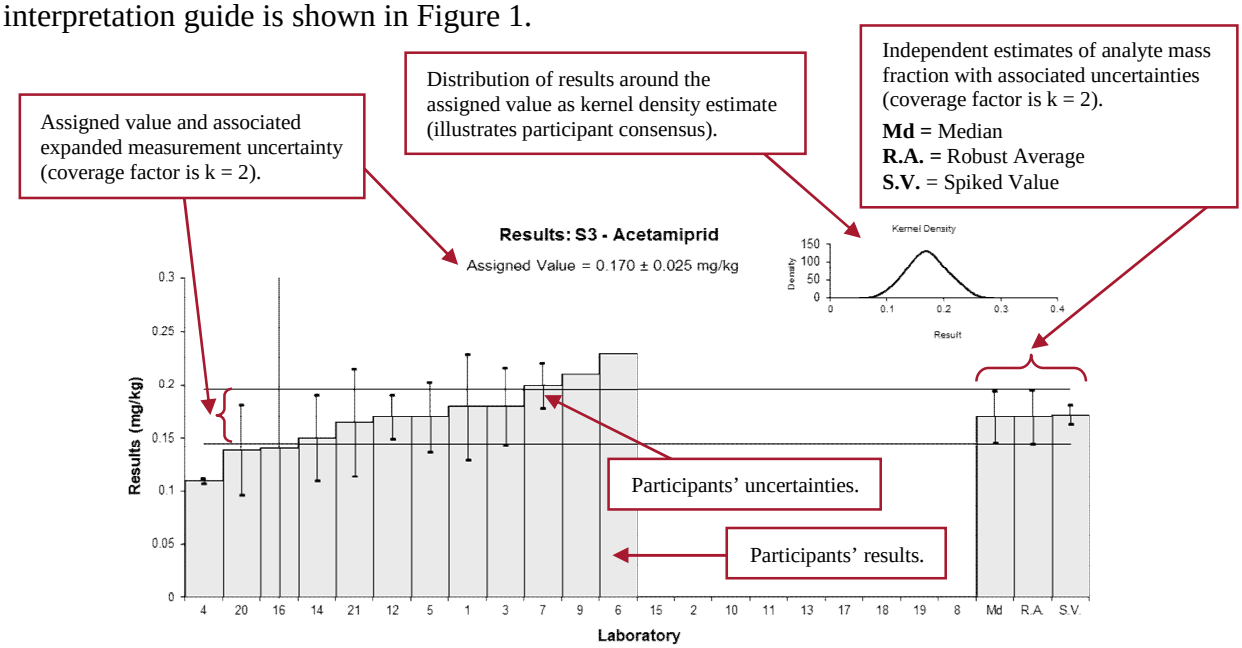


Figure 1 Guide to Presentation of Results

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 (gross errors), and such results were removed for the calculation of all summary statistics.[3,4]

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 in this study were the robust averages of participants' results and the expanded uncertainties were estimated from the associated robust SDs (Appendix 4).

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 using the procedure described in ISO 13528:2015.[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 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

The target standard deviation (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

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 measurement 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 measurement uncertainty of the participant's result

U_X is the expanded measurement 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	Tomato
Analyte	Cyhalothrin
Units	mg/kg

Participant Results

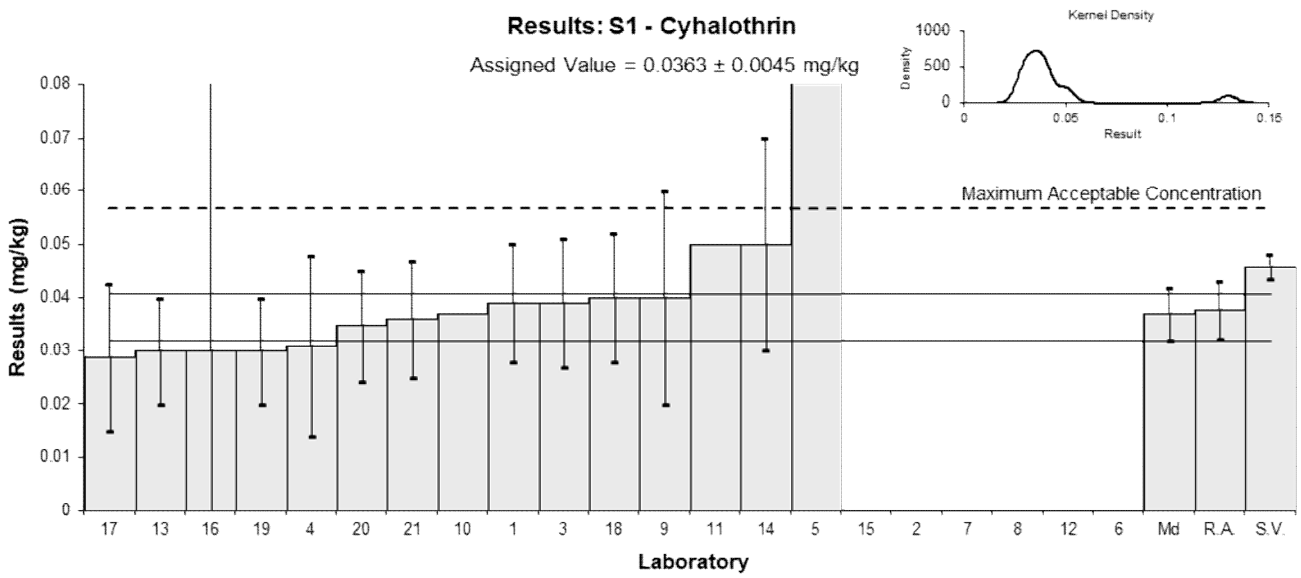
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	0.039	0.011	75	0.50	0.23
2	NR	NR	NR		
3	0.039	0.012	78.18	0.50	0.21
4	0.031	0.017	108	-0.97	-0.30
5	0.13	0.014	103.5	17.21	6.37
6	NT	NT	NT		
7	NR	NR	NR		
8	NR	NR	101		
9	0.04	0.02	91	0.68	0.18
10	0.037	NR	101.72	0.13	0.16
11**	0.05	NR	116	2.00	1.00
12	NT	NT	NT		
13	0.03	0.01	NR	-1.16	-0.57
14**	0.050	0.02	92	2.00	0.67
15	NR	NR	NR		
16	0.03	45	97	-1.16	0.00
17	0.0287	0.0138	123	-1.40	-0.52
18	0.040	0.012	94.42	0.68	0.29
19	0.03	0.01	NR	-1.16	-0.57
20	0.0347	0.0104	83	-0.29	-0.14
21	0.036	0.011	106	-0.06	-0.03

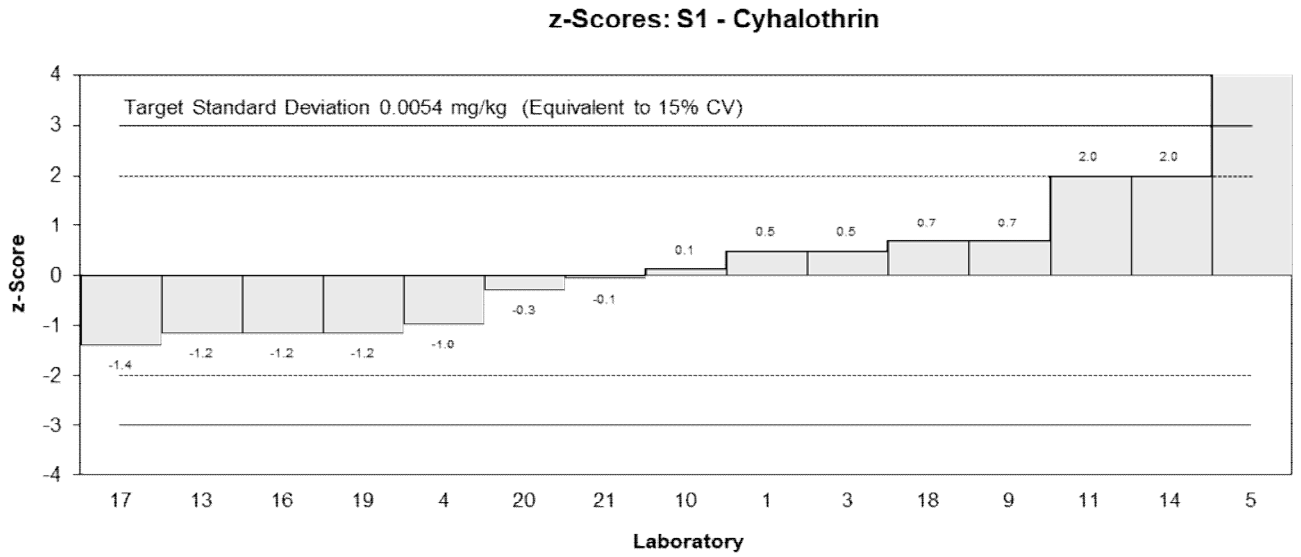
Statistics

Assigned Value*	0.0363	0.0045
Spike	0.0458	0.0023
Max. Acceptable Concentration**	0.0567	
Robust Average	0.0377	0.0055
Median	0.0370	0.0049
Mean	0.0430	
N	15	
Max.	0.13	
Min.	0.0287	
Robust SD	0.0086	
Robust CV	23%	

* Robust average excluding Laboratory 5.

** z-Score adjusted (see Section 6.3).


Results: S1 - Cyhalothrin
Assigned Value = 0.0363 ± 0.0045 mg/kg
Kernel Density
Density
Result
Maximum Acceptable Concentration
Results (mg/kg)
Laboratory
17 13 16 19 4 20 21 10 1 3 18 9 11 14 5 15 2 7 8 12 6 Md R.A. S.V.


z-Scores: S1 - Cyhalothrin
Target Standard Deviation 0.0054 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
17 13 16 19 4 20 21 10 1 3 18 9 11 14 5

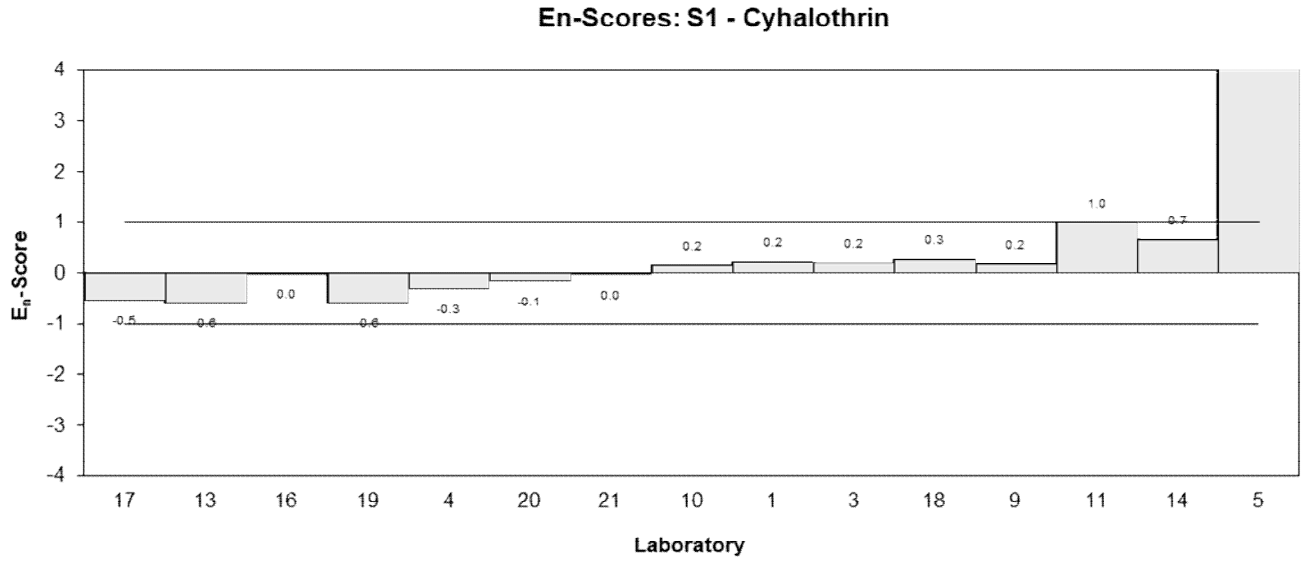

En-Scores: S1 - Cyhalothrin
En-Score
Laboratory
17 13 16 19 4 20 21 10 1 3 18 9 11 14 5

Figure 2

Table 6

Sample Details

Sample No.	S1
Matrix	Tomato
Analyte	Dimethoate
Units	mg/kg

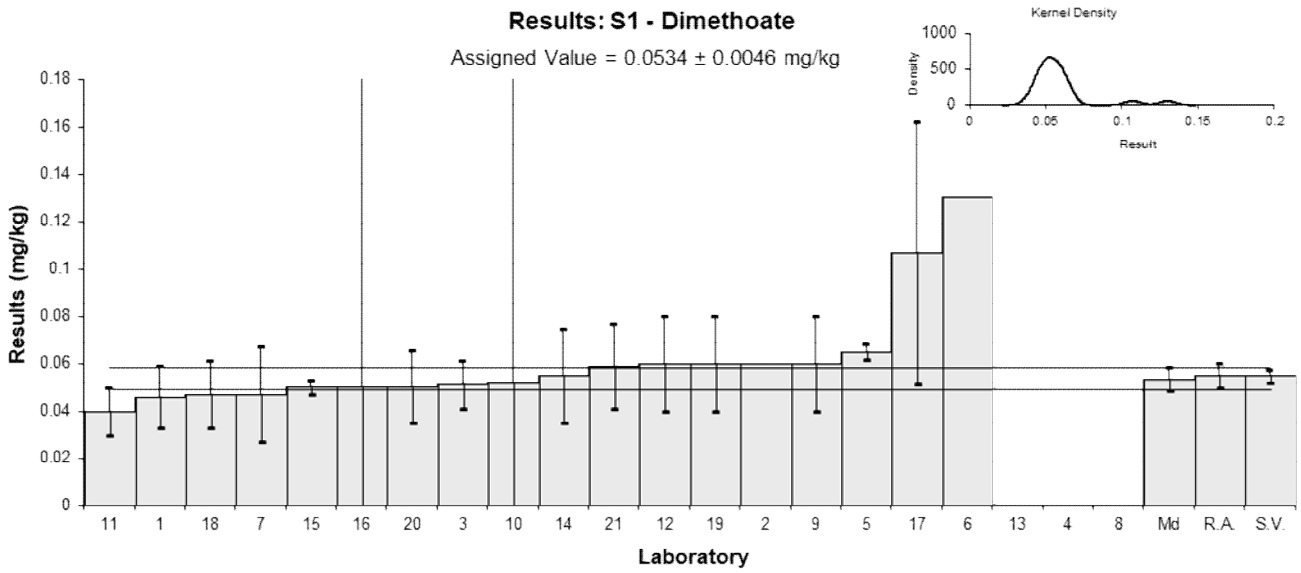
Participant Results

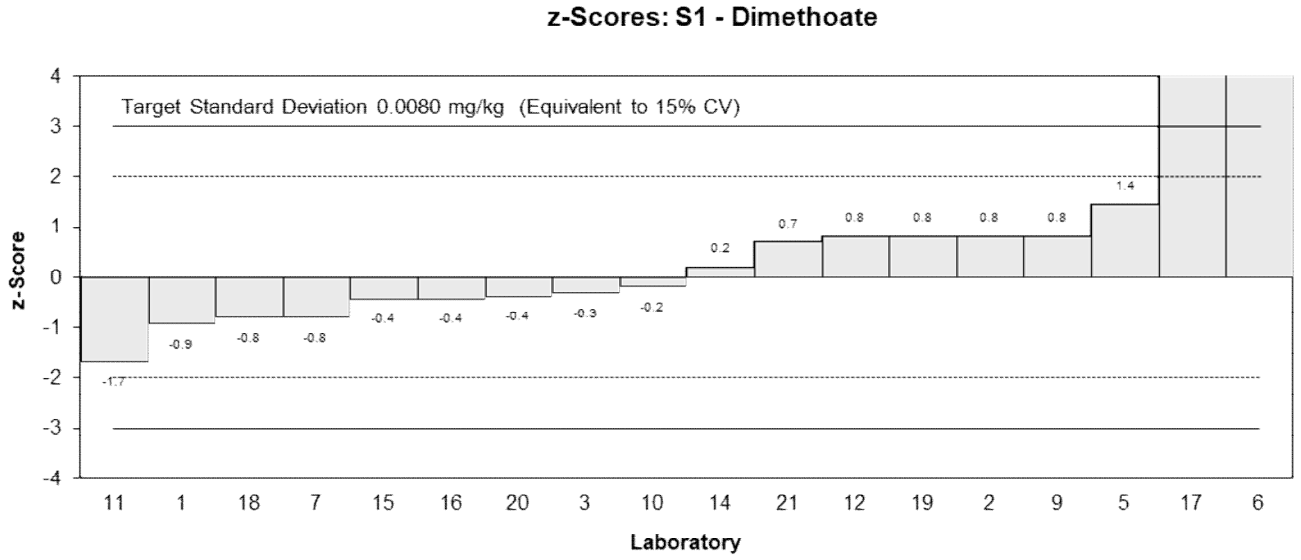
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	0.046	0.013	103	-0.92	-0.54
2	0.06	NR	NR	0.82	1.43
3	0.051	0.010	96.04	-0.30	-0.22
4	NR	NR	NR		
5	0.065	0.003	84.5	1.45	2.11
6	0.13	NR	NR	9.56	16.65
7	0.047	0.02	87.9	-0.80	-0.31
8	NT	NT	NT		
9	0.06	0.02	94	0.82	0.32
10	0.052	0.149	100.48	-0.17	-0.01
11	0.04	0.01	79	-1.67	-1.22
12	0.06	0.02	101	0.82	0.32
13	<0.01	NR	NR		
14	0.055	0.02	102	0.20	0.08
15	0.05	0.003	120	-0.42	-0.62
16	0.05	44	103	-0.42	0.00
17	0.107	0.0556	73	6.69	0.96
18	0.047	0.014	95.61	-0.80	-0.43
19	0.06	0.02	NR	0.82	0.32
20	0.0504	0.015	88	-0.37	-0.19
21	0.059	0.018	113	0.70	0.30

Statistics

Assigned Value*	0.0534	0.0046
Spike	0.0548	0.0027
Robust Average	0.0551	0.0053
Median	0.0535	0.0048
Mean	0.0605	
N	18	
Max.	0.13	
Min.	0.04	
Robust SD	0.0090	
Robust CV	16%	

* Robust average excluding Laboratories 6 and 17.


Results: S1 - Dimethoate
Assigned Value = 0.0534 ± 0.0046 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
11 1 18 7 15 16 20 3 10 14 21 12 19 2 9 5 17 6 13 4 8 Md R.A. S.V.


z-Scores: S1 - Dimethoate
Target Standard Deviation 0.0080 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
11 1 18 7 15 16 20 3 10 14 21 12 19 2 9 5 17 6

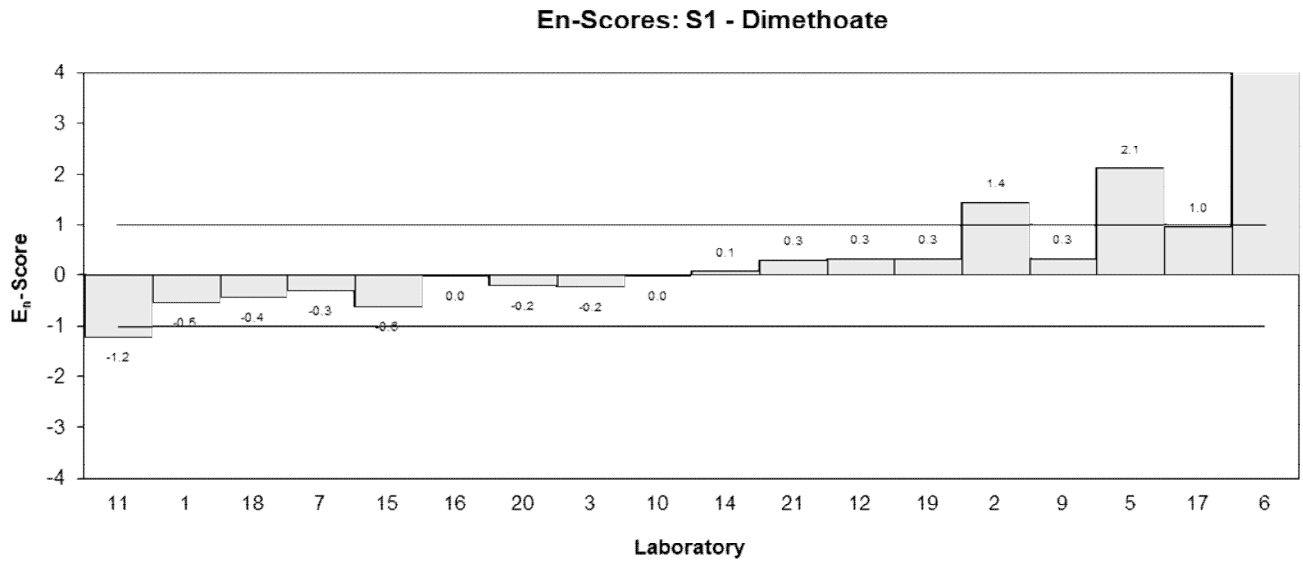

En-Scores: S1 - Dimethoate
En-Score
Laboratory
11 1 18 7 15 16 20 3 10 14 21 12 19 2 9 5 17 6

Figure 3

Table 7

Sample Details

Sample No.	S1
Matrix	Tomato
Analyte	Endosulfan sulfate
Units	mg/kg

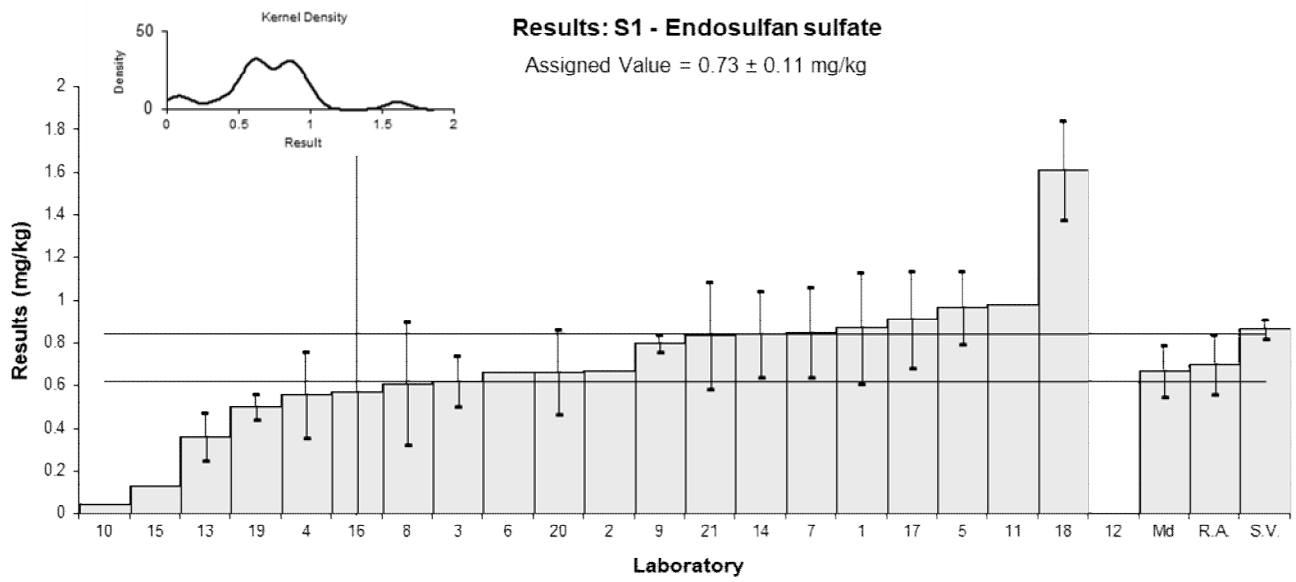
Participant Results

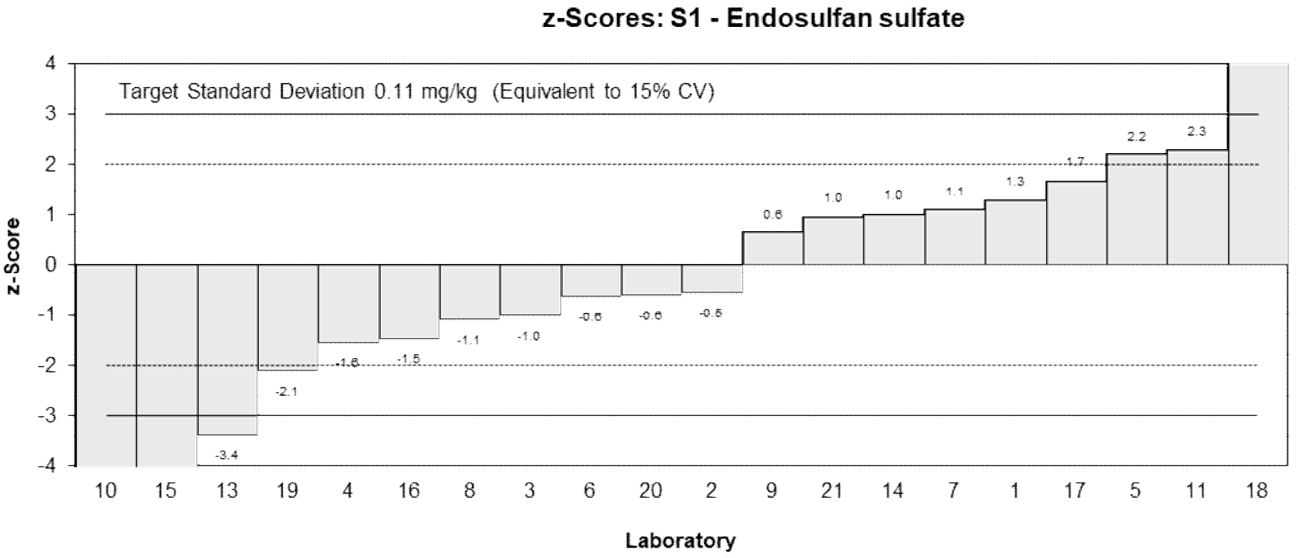
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	0.87	0.26	90	1.28	0.50
2	0.67	NR	NR	-0.55	-0.55
3	0.62	0.12	116.40	-1.00	-0.68
4	0.56	0.20	95	-1.55	-0.74
5	0.97	0.171	109.6	2.19	1.18
6	0.66	NR	NR	-0.64	-0.64
7	0.85	0.21	89.2	1.10	0.51
8	0.61	0.29	97	-1.10	-0.39
9	0.8	0.04	88	0.64	0.60
10	0.043	NR	126.54	-6.27	-6.25
11	0.98	NR	93	2.28	2.27
12	NT	NT	NT		
13	0.36	0.11	NR	-3.38	-2.38
14	0.84	0.2	98	1.00	0.48
15	0.13	NR	53	-5.48	-5.45
16	0.57	49	96	-1.46	0.00
17	0.912	0.228	110	1.66	0.72
18	1.61	0.23	88.76	8.04	3.45
19	0.5	0.06	NR	-2.10	-1.84
20	0.663	0.199	64	-0.61	-0.29
21	0.835	0.25	104	0.96	0.38

Statistics

Assigned Value*	0.73	0.11
Spike	0.865	0.043
Robust Average	0.70	0.14
Median	0.67	0.12
Mean	0.70	
N	20	
Max.	1.61	
Min.	0.043	
Robust SD	0.25	
Robust CV	36%	

* Robust average excluding Laboratories 10, 15 and 18.


Kernel Density
Density
Result
Results: S1 - Endosulfan sulfate
Assigned Value = 0.73 ± 0.11 mg/kg
Results (mg/kg)
10 15 13 19 4 16 8 3 6 20 2 9 21 14 7 1 17 5 11 18 12 Md R.A. S.V.
Laboratory


z-Scores: S1 - Endosulfan sulfate
Target Standard Deviation 0.11 mg/kg (Equivalent to 15% CV)
z-Score
10 15 13 19 4 16 8 3 6 20 2 9 21 14 7 1 17 5 11 18
Laboratory

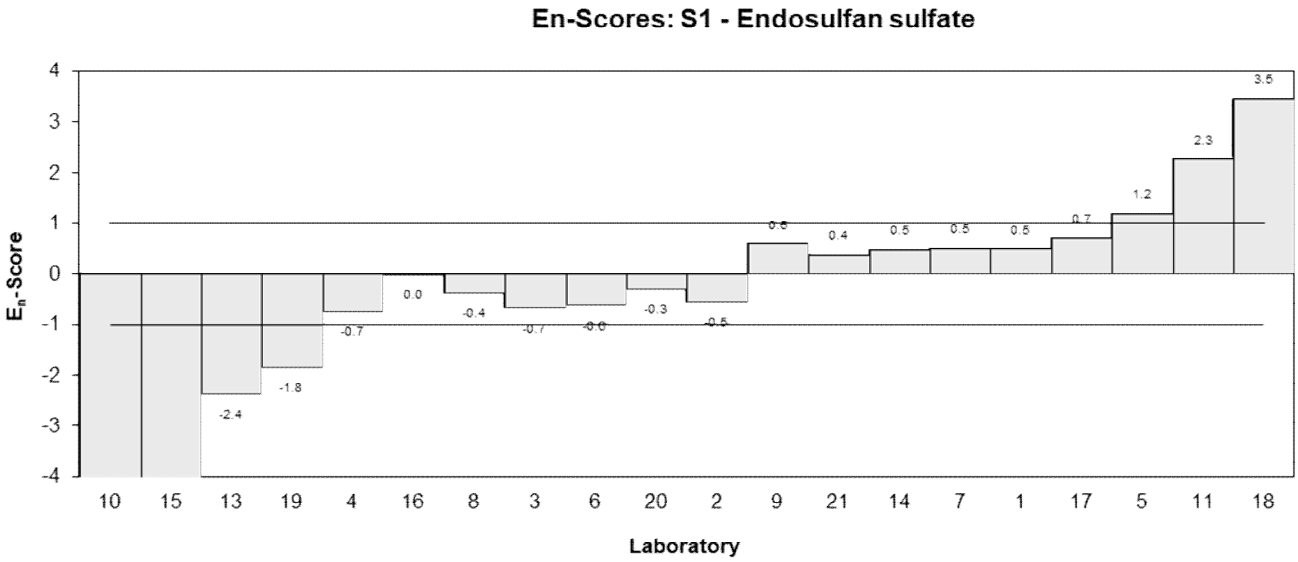

En-Scores: S1 - Endosulfan sulfate
En-Score
10 15 13 19 4 16 8 3 6 20 2 9 21 14 7 1 17 5 11 18
Laboratory

Figure 4

Table 8

Sample Details

Sample No.	S1
Matrix	Tomato
Analyte	Omethoate
Units	mg/kg

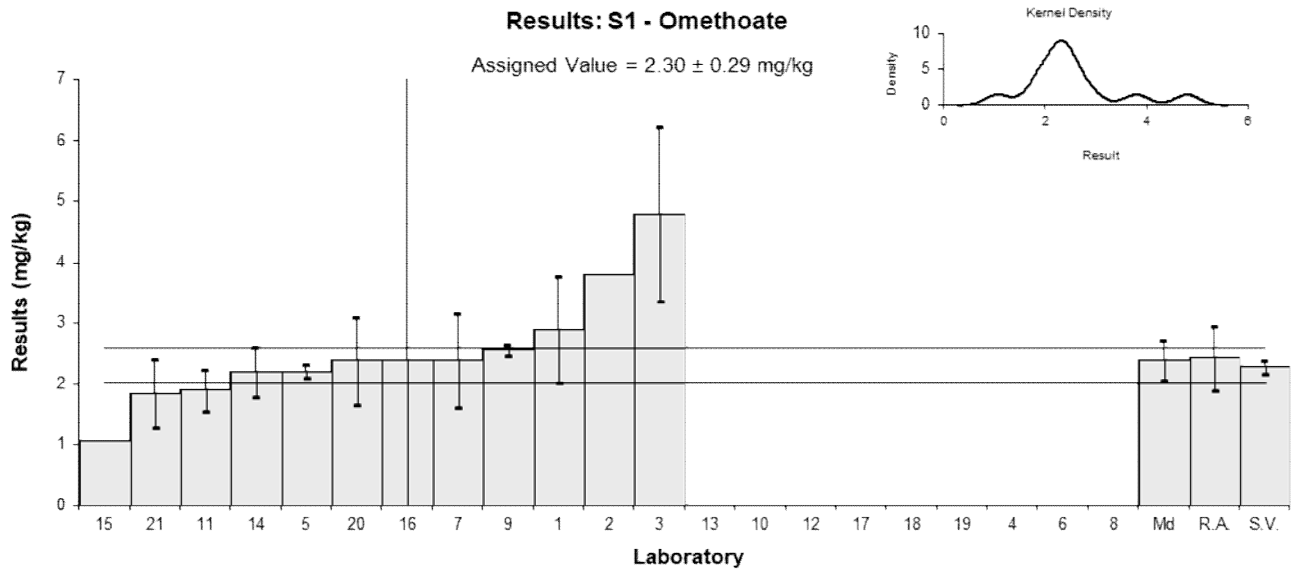
Participant Results

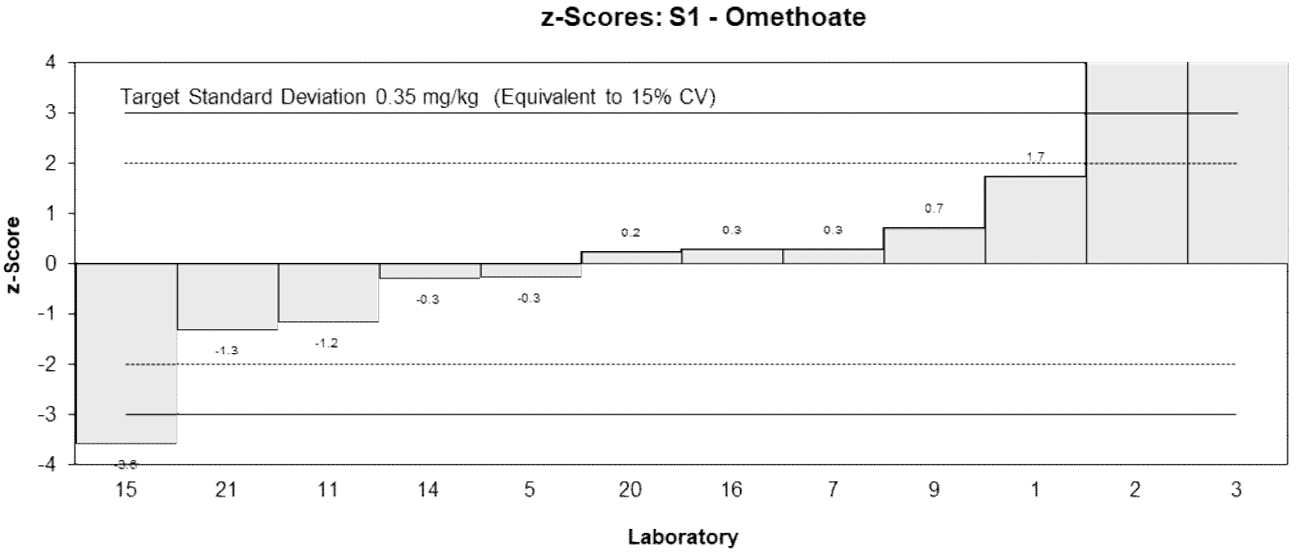
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	2.9	0.87	96	1.74	0.65
2	3.80	NR	NR	4.35	5.17
3	4.8	1.44	97.65	7.25	1.70
4	NT	NT	NT		
5	2.21	0.11	82.9	-0.26	-0.29
6	NT	NT	NT		
7	2.4	0.77	78.9	0.29	0.12
8	NT	NT	NT		
9	2.55	0.08	86	0.72	0.83
10	NR	NR	NR		
11	1.9	0.34	77	-1.16	-0.90
12	NT	NT	NT		
13	<0.5	NR	NR		
14	2.2	0.4	95	-0.29	-0.20
15	1.06	NR	82	-3.59	-4.28
16	2.4	26	89	0.29	0.00
17	NT	NT	NT		
18	NT	NT	NT		
19	NT	NT	NT		
20	2.384	0.715	81	0.24	0.11
21	1.843	0.55	99	-1.32	-0.73

Statistics

Assigned Value*	2.30	0.29
Spike	2.28	0.11
Robust Average	2.43	0.53
Median	2.39	0.32
Mean	2.54	
N	12	
Max.	4.8	
Min.	1.06	
Robust SD	0.73	
Robust CV	30%	

* Robust average excluding Laboratories 2, 3 and 15.


Results: S1 - Omethoate
Assigned Value = 2.30 ± 0.29 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
15 21 11 14 5 20 16 7 9 1 2 3 13 10 12 17 18 19 4 6 8 Md R.A. S.V.
Laboratory


z-Scores: S1 - Omethoate
Target Standard Deviation 0.35 mg/kg (Equivalent to 15% CV)
z-Score
-3.6 -1.3 -1.2 -0.3 -0.3 0.2 0.3 0.3 0.7 1.7
15 21 11 14 5 20 16 7 9 1 2 3
Laboratory

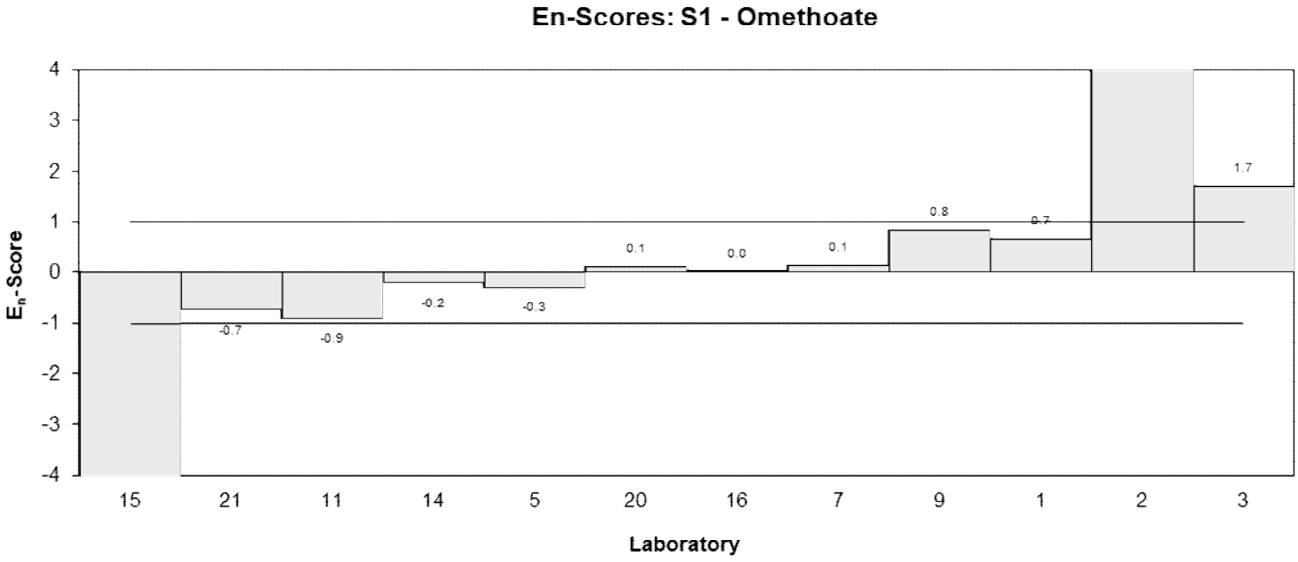

En-Scores: S1 - Omethoate
En-Score
-0.7 -0.9 -0.2 -0.3 0.1 0.0 0.1 0.8 0.7 1.7
15 21 11 14 5 20 16 7 9 1 2 3
Laboratory

Figure 5

Table 9

Sample Details

Sample No.	S2
Matrix	Bok Choy
Analyte	Cyfluthrin
Units	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	Recovery
1	1.0	0.3	84
2	1.17	NR	NR
3	0.66	0.20	78.75
4	0.85	0.38	110
5	0.92	0.145	80
6	NT	NT	NT
7	0.73	0.52	87.8
8	0.46	0.21	83
9	5.23	NR	NR
10	0.880	NR	127.71
11	1.41	NR	98
12	NT	NT	NT
13	NT	NT	NT
14	0.91	0.2	98
15	0.16	NR	68
16	0.66	40	106
17	0.141	0.0566	86
18	3.35	1.09	89.76
19	0.30	0.10	NR
20	0.598	0.179	71
21	0.316	0.095	128

Statistics

Assigned Value	Not Set	
Spike	0.902	0.045
Robust Average	0.79	0.29
Median	0.79	0.20
Mean	1.1	
N	18	
Max.	5.23	
Min.	0.141	
Robust SD	0.49	
Robust CV	62%	

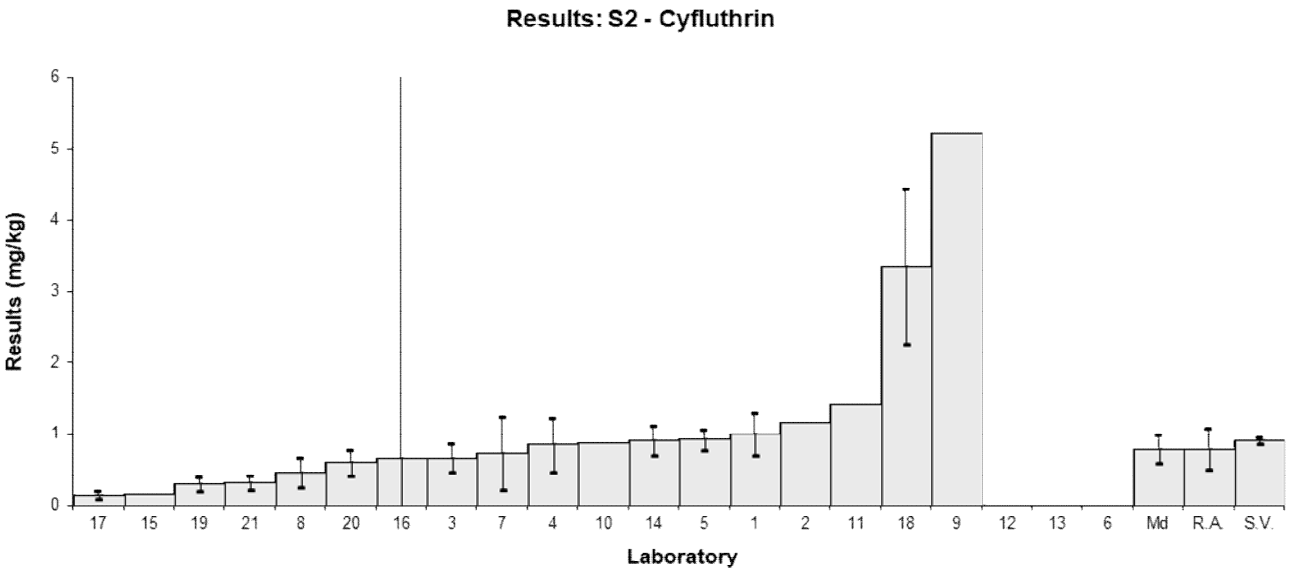

Results: S2 - Cyfluthrin
Results (mg/kg)
0
1
2
3
4
5
6
17
15
19
21
8
20
16
3
7
4
10
14
5
1
2
11
18
9
12
13
6
Md
R.A.
S.V.
Laboratory

Figure 6

Table 10

Sample Details

Sample No.	S2
Matrix	Bok Choy
Analyte	Glyphosate
Units	mg/kg

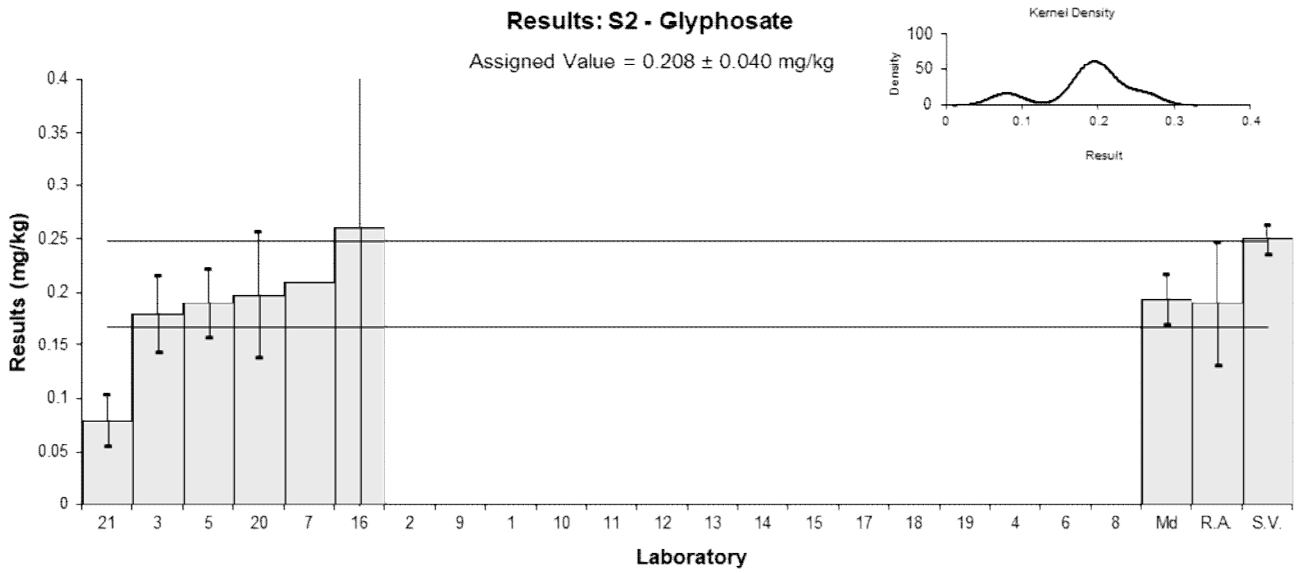
Participant Results

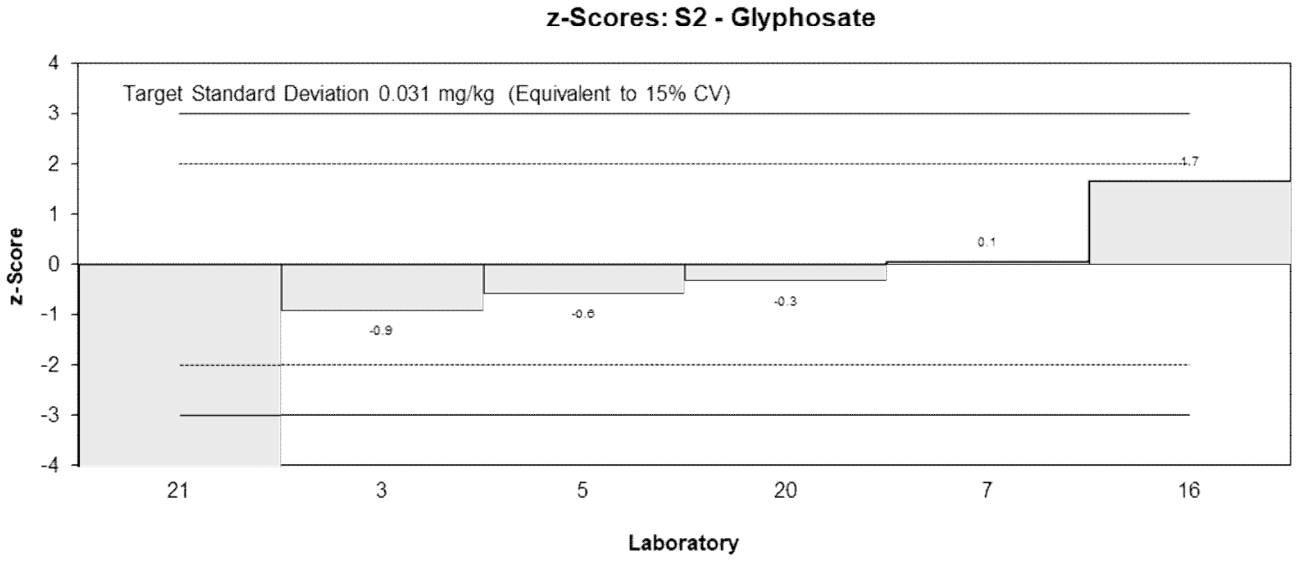
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	NT	NT	NT		
2	NR	NR	NR		
3	0.18	0.036	90	-0.90	-0.52
4	NT	NT	NT		
5	0.19	0.032	84.1	-0.58	-0.35
6	NT	NT	NT		
7	0.21	NR	42	0.06	0.05
8	NT	NT	NT		
9	NR	NR	NR		
10	NT	NT	NT		
11	NT	NT	NT		
12	NT	NT	NT		
13	NT	NT	NT		
14	NT	NT	NT		
15	NT	NT	NT		
16	0.26	17	106	1.67	0.00
17	NT	NT	NT		
18	NT	NT	NT		
19	NT	NT	NT		
20	0.198	0.059	85	-0.32	-0.14
21	0.08	0.024	63	-4.10	-2.74

Statistics

Assigned Value*	0.208	0.040
Spike	0.250	0.013
Robust Average	0.190	0.058
Median	0.194	0.023
Mean	0.186	
N	6	
Max.	0.26	
Min.	0.08	
Robust SD	0.057	
Robust CV	30%	

* Robust average excluding Laboratory 21.


Results: S2 - Glyphosate
Assigned Value = 0.208 ± 0.040 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
21 3 5 20 7 16 2 9 1 10 11 12 13 14 15 17 18 19 4 6 8 Md R.A. S.V.


z-Scores: S2 - Glyphosate
Target Standard Deviation 0.031 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
21 3 5 20 7 16
1.7
0.1
-0.3
-0.6
-0.9

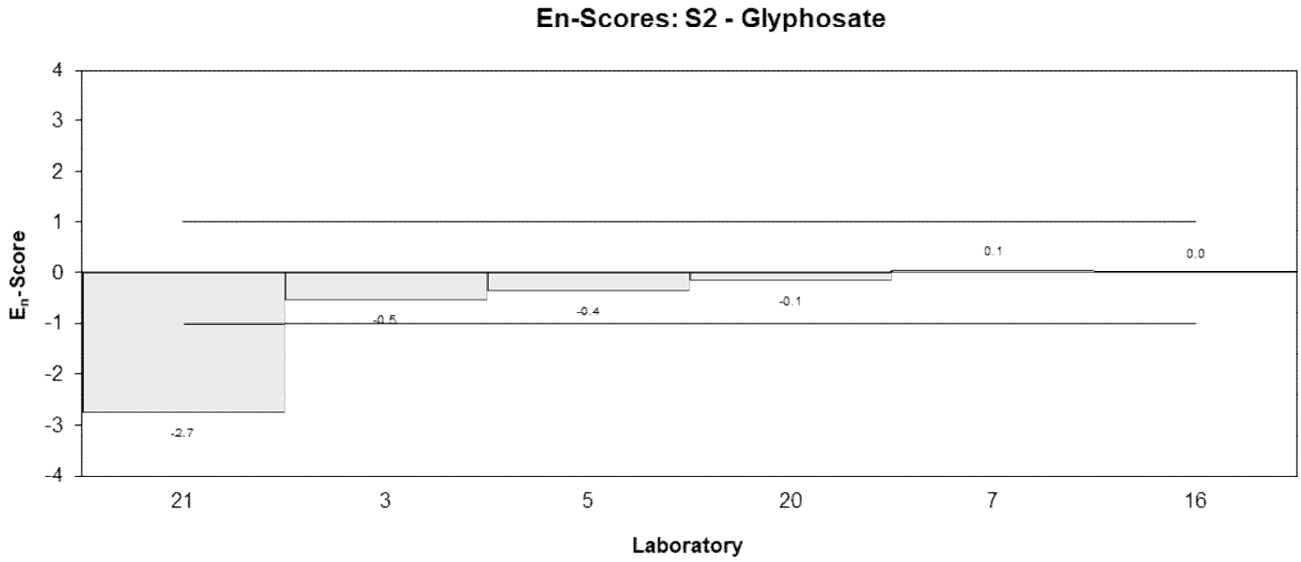

En-Scores: S2 - Glyphosate
En-Score
Laboratory
21 3 5 20 7 16
0.1
0.0
-0.1
-0.4
-0.5
-2.7

Figure 7

Table 11

Sample Details

Sample No.	S2
Matrix	Bok Choy
Analyte	Indoxacarb
Units	mg/kg

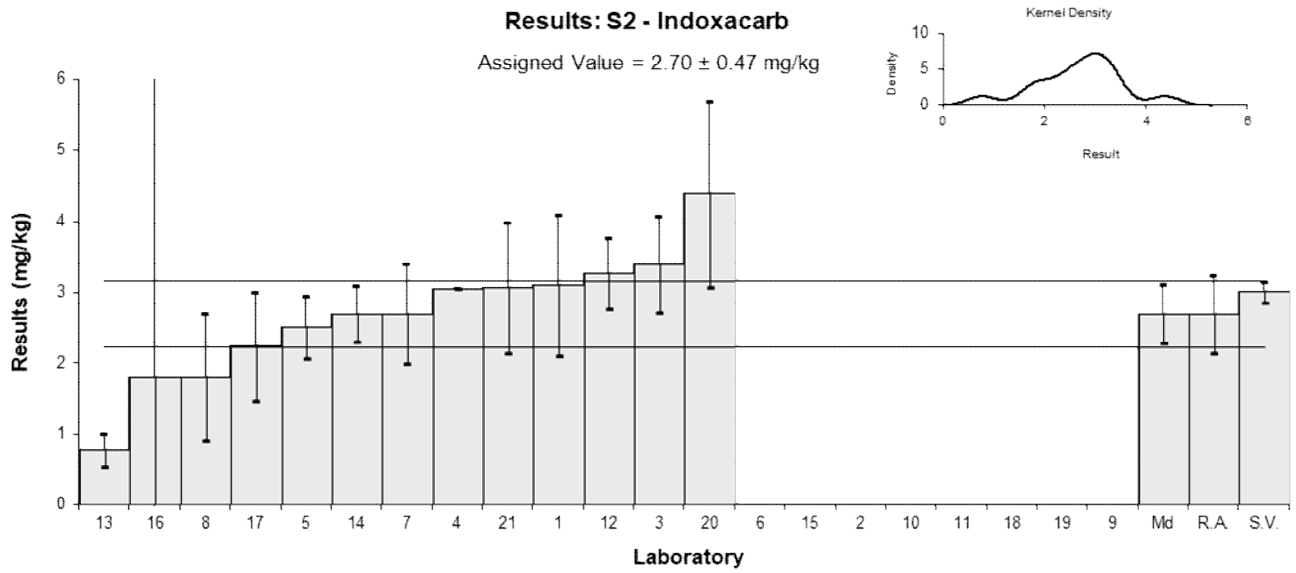
Participant Results

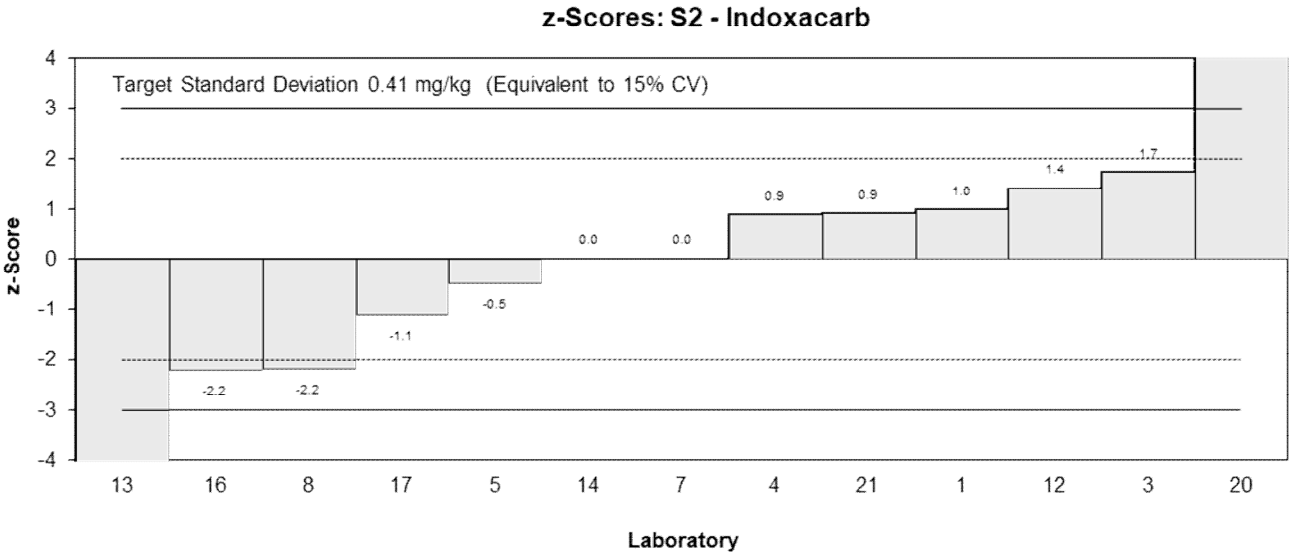
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	3.1	1.0	115	0.99	0.36
2	NR	NR	NR		
3	3.4	0.68	97.55	1.73	0.85
4	3.06	0.002	150	0.89	0.77
5	2.51	0.442	70.9	-0.47	-0.29
6	NT	NT	NT		
7	2.7	0.71	81.3	0.00	0.00
8	1.81	0.9	119	-2.20	-0.88
9	NT	NT	NT		
10	NT	NT	NT		
11	NT	NT	NT		
12	3.27	0.49	99	1.41	0.84
13	0.77	0.23	NR	-4.77	-3.69
14	2.7	0.4	105	0.00	0.00
15	NR	NR	NR		
16	1.8	34	97	-2.22	-0.03
17	2.244	0.7631	129	-1.13	-0.51
18	NT	NT	NT		
19	NT	NT	NT		
20	4.388	1.316	80	4.17	1.21
21	3.069	0.92	94	0.91	0.36

Statistics

Assigned Value*	2.70	0.47
Spike	3.01	0.15
Robust Average	2.70	0.55
Median	2.70	0.41
Mean	2.68	
N	13	
Max.	4.388	
Min.	0.77	
Robust SD	0.80	
Robust CV	30%	

* Robust average excluding Laboratories 13 and 20.


Results: S2 - Indoxacarb
Assigned Value = 2.70 ± 0.47 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
13 16 8 17 5 14 7 4 21 1 12 3 20 6 15 2 10 11 18 19 9 Md R.A. S.V.
Laboratory


z-Scores: S2 - Indoxacarb
Target Standard Deviation 0.41 mg/kg (Equivalent to 15% CV)
z-Score
13 16 8 17 5 14 7 4 21 1 12 3 20
Laboratory

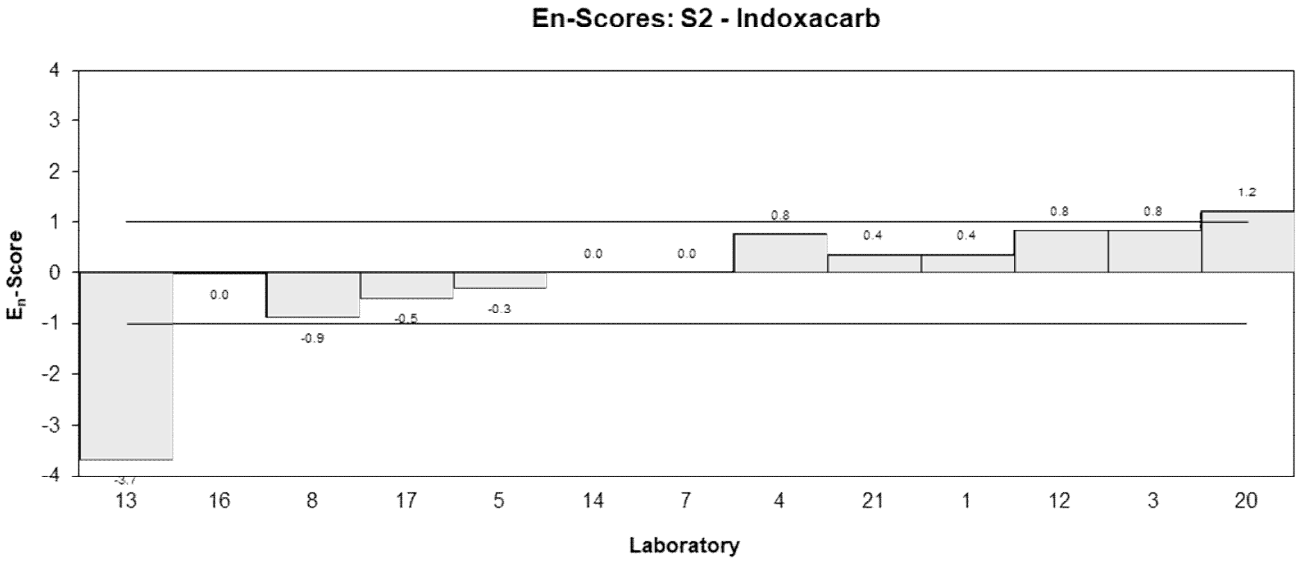

En-Scores: S2 - Indoxacarb
En-Score
13 16 8 17 5 14 7 4 21 1 12 3 20
Laboratory

Figure 8

Table 12

Sample Details

Sample No.	S2
Matrix	Bok Choy
Analyte	Pyraclostrobin
Units	mg/kg

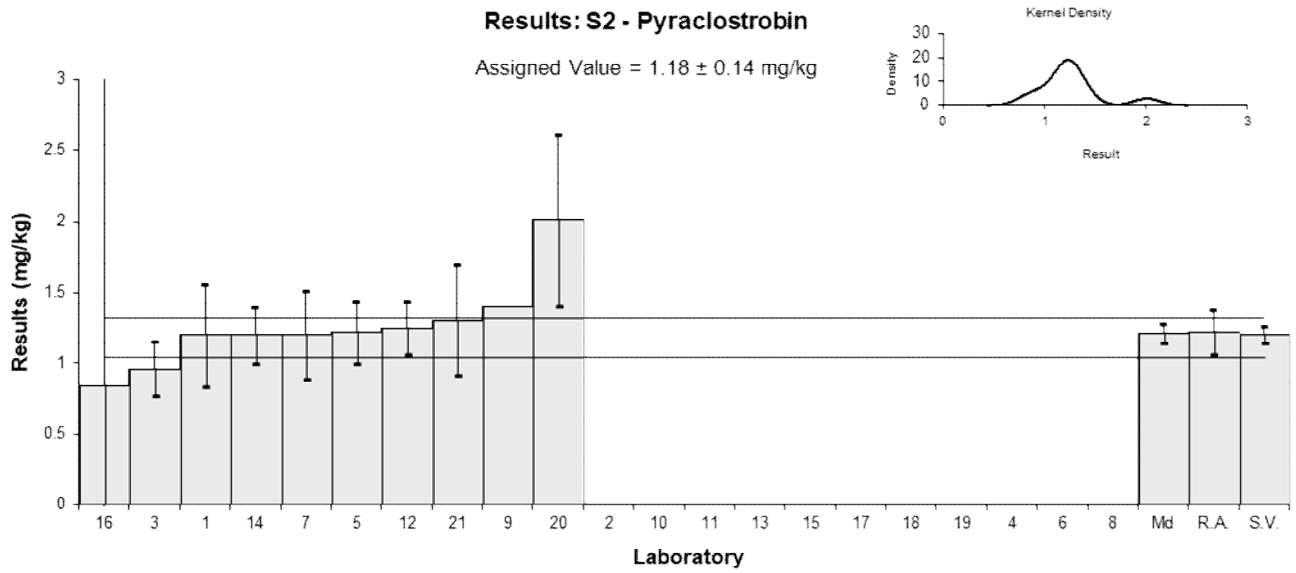
Participant Results

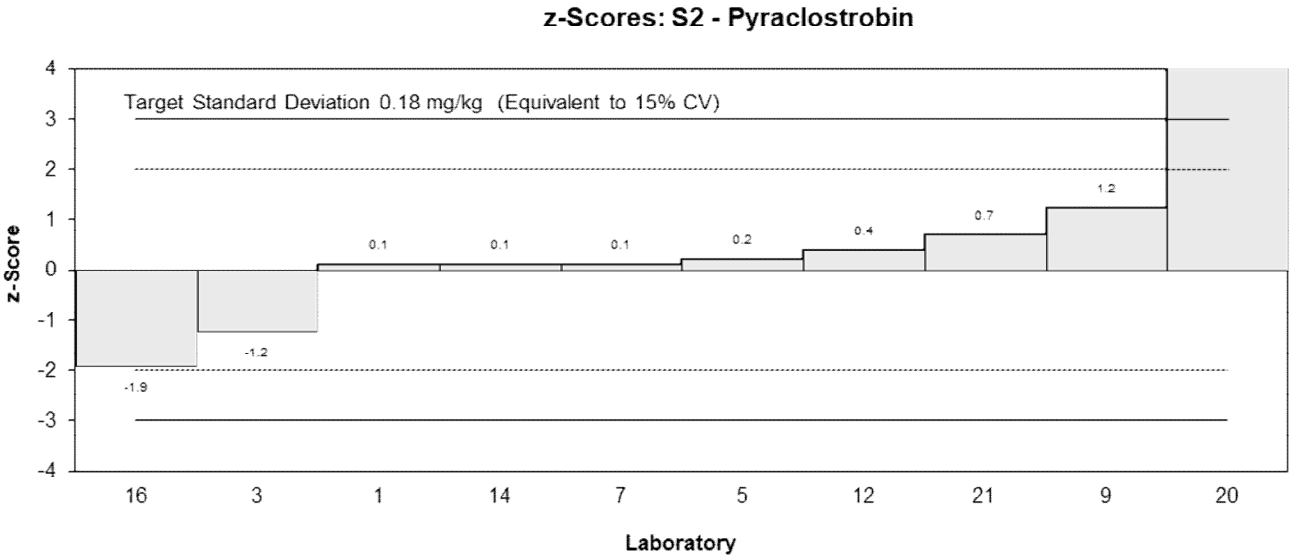
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	1.2	0.36	117	0.11	0.05
2	NR	NR	NR		
3	0.96	0.19	95.57	-1.24	-0.93
4	NT	NT	NT		
5	1.22	0.219	97.9	0.23	0.15
6	NT	NT	NT		
7	1.2	0.31	70.3	0.11	0.06
8	NT	NT	NT		
9	1.4	NR	NR	1.24	1.57
10	NT	NT	NT		
11	NT	NT	NT		
12	1.25	0.19	104	0.40	0.30
13	NT	NT	NT		
14	1.2	0.2	109	0.11	0.08
15	NT	NT	NT		
16	0.84	27	98	-1.92	-0.01
17	NT	NT	NT		
18	NT	NT	NT		
19	NT	NT	NT		
20	2.010	0.603	82	4.69	1.34
21	1.304	0.39	93	0.70	0.30

Statistics

Assigned Value*	1.18	0.14
Spike	1.20	0.06
Robust Average	1.22	0.16
Median	1.21	0.07
Mean	1.26	
N	10	
Max.	2.01	
Min.	0.84	
Robust SD	0.21	
Robust CV	17%	

* Robust average excluding Laboratory 20.


Results: S2 - Pyraclostrobin
Assigned Value = 1.18 ± 0.14 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
16 3 1 14 7 5 12 21 9 20 2 10 11 13 15 17 18 19 4 6 8 Md R.A. S.V.
Laboratory


z-Scores: S2 - Pyraclostrobin
Target Standard Deviation 0.18 mg/kg (Equivalent to 15% CV)
z-Score
-1.9
-1.2
0.1
0.1
0.1
0.2
0.4
0.7
1.2
16 3 1 14 7 5 12 21 9 20
Laboratory

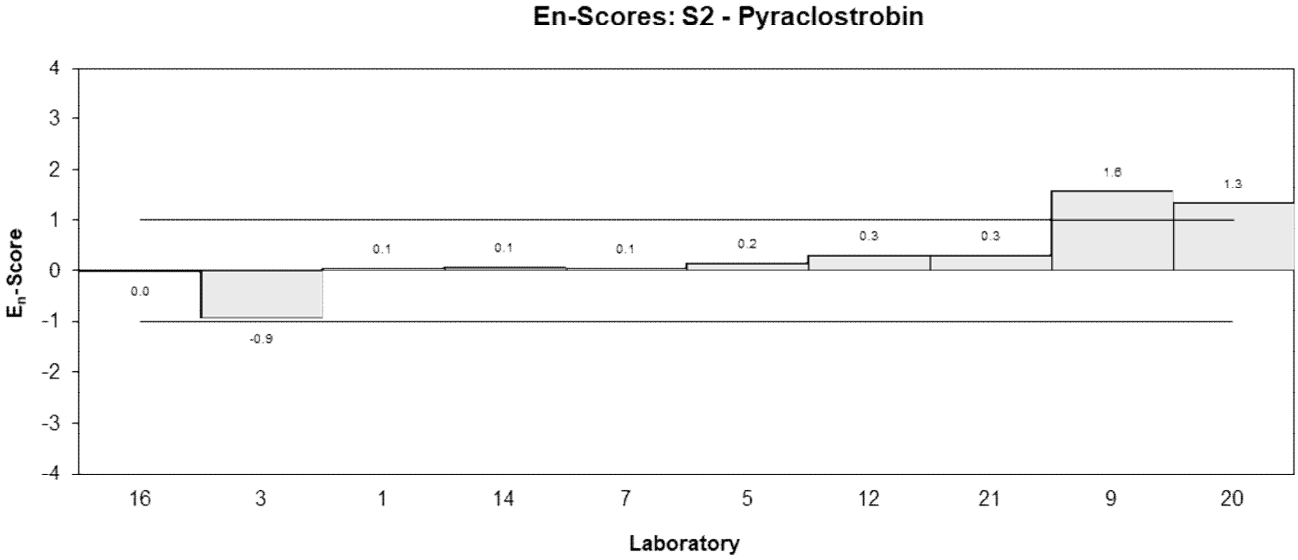

En-Scores: S2 - Pyraclostrobin
En-Score
0.0
-0.9
0.1
0.1
0.1
0.2
0.3
0.3
1.6
1.3
16 3 1 14 7 5 12 21 9 20
Laboratory

Figure 9

Table 13

Sample Details

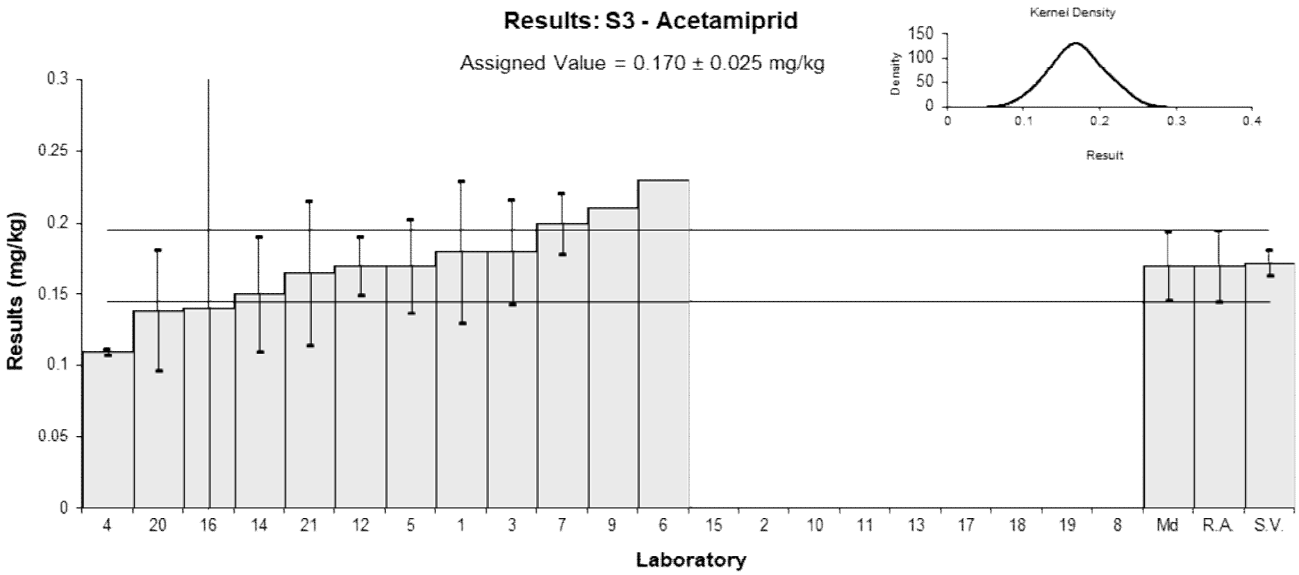
Sample No.	S3
Matrix	Apple
Analyte	Acetamiprid
Units	mg/kg

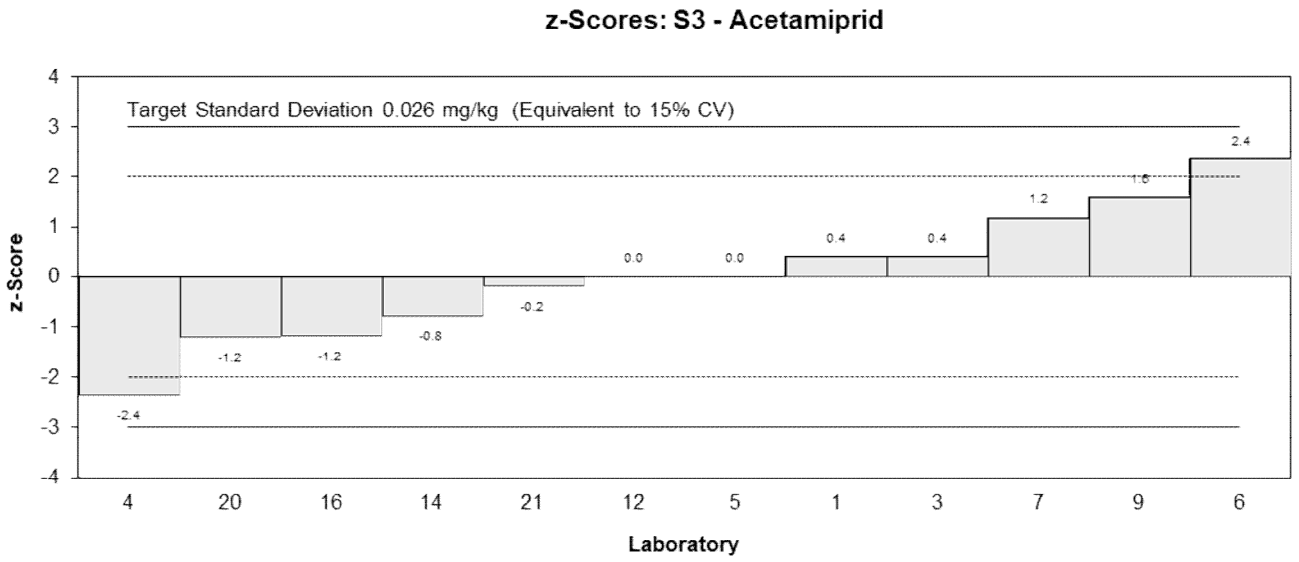
Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	0.18	0.05	102	0.39	0.18
2	NR	NR	NR		
3	0.18	0.036	111.49	0.39	0.23
4	0.11	0.002	84	-2.35	-2.39
5	0.17	0.033	95.1	0.00	0.00
6	0.23	NR	NR	2.35	2.40
7	0.2	0.021	97	1.18	0.92
8	NT	NT	NT		
9	0.21	NR	NR	1.57	1.60
10	NT	NT	NT		
11	NT	NT	NT		
12	0.17	0.02	104	0.00	0.00
13	NT	NT	NT		
14	0.15	0.04	100	-0.78	-0.42
15	NR	NR	NR		
16	0.14	51	87	-1.18	0.00
17	NT	NT	NT		
18	NT	NT	NT		
19	NT	NT	NT		
20	0.139	0.042	70	-1.22	-0.63
21	0.165	0.050	95	-0.20	-0.09

Statistics

Assigned Value	0.170	0.025
Spike	0.172	0.009
Robust Average	0.170	0.025
Median	0.170	0.024
Mean	0.170	
N	12	
Max.	0.23	
Min.	0.11	
Robust SD	0.035	
Robust CV	21%	


Results: S3 - Acetamiprid
Assigned Value = 0.170 ± 0.025 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
4 20 16 14 21 12 5 1 3 7 9 6 15 2 10 11 13 17 18 19 8 Md R.A. S.V.


z-Scores: S3 - Acetamiprid
Target Standard Deviation 0.026 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
4 20 16 14 21 12 5 1 3 7 9 6

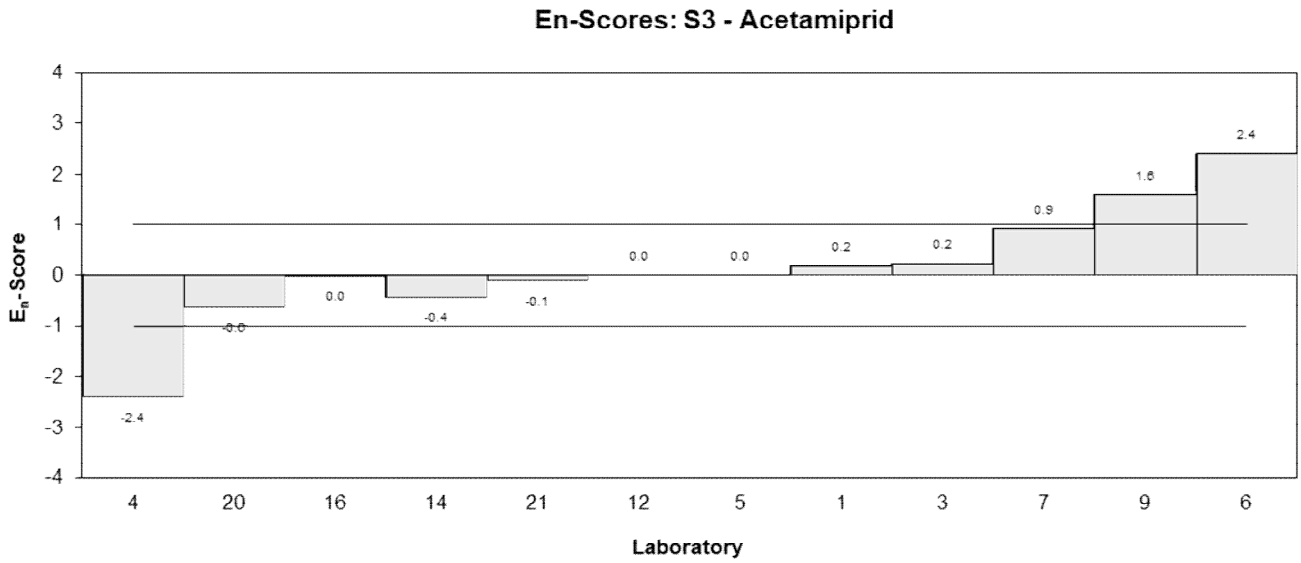

En-Scores: S3 - Acetamiprid
En-Score
Laboratory
4 20 16 14 21 12 5 1 3 7 9 6

Figure 10

Table 14

Sample Details

Sample No.	S3
Matrix	Apple
Analyte	Carbendazim
Units	mg/kg

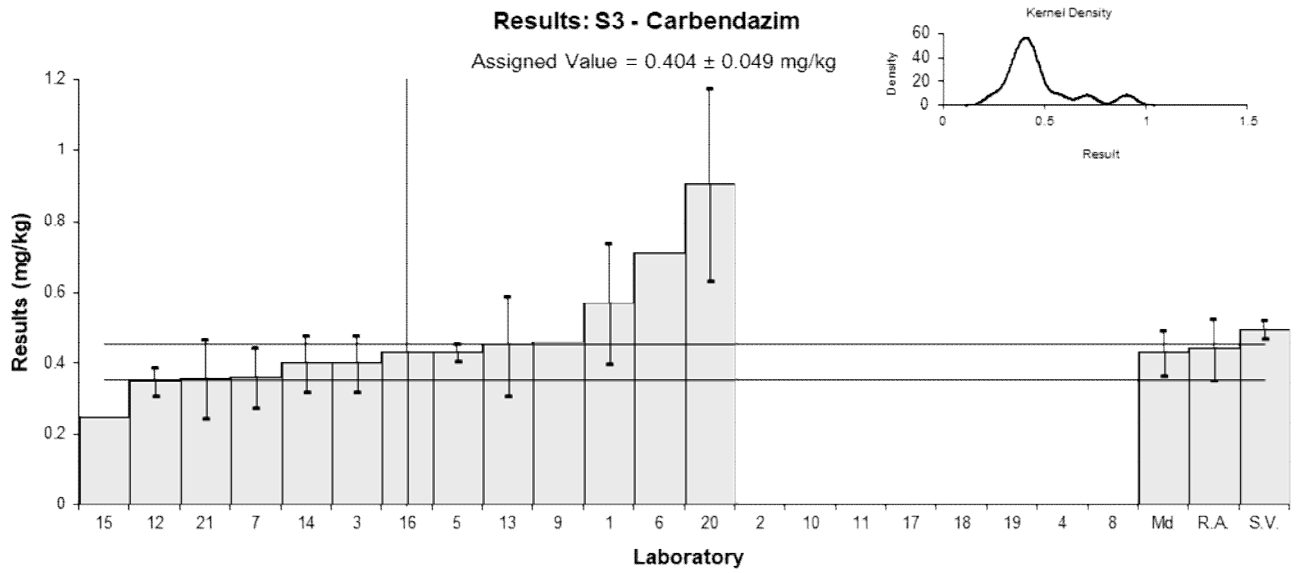
Participant Results

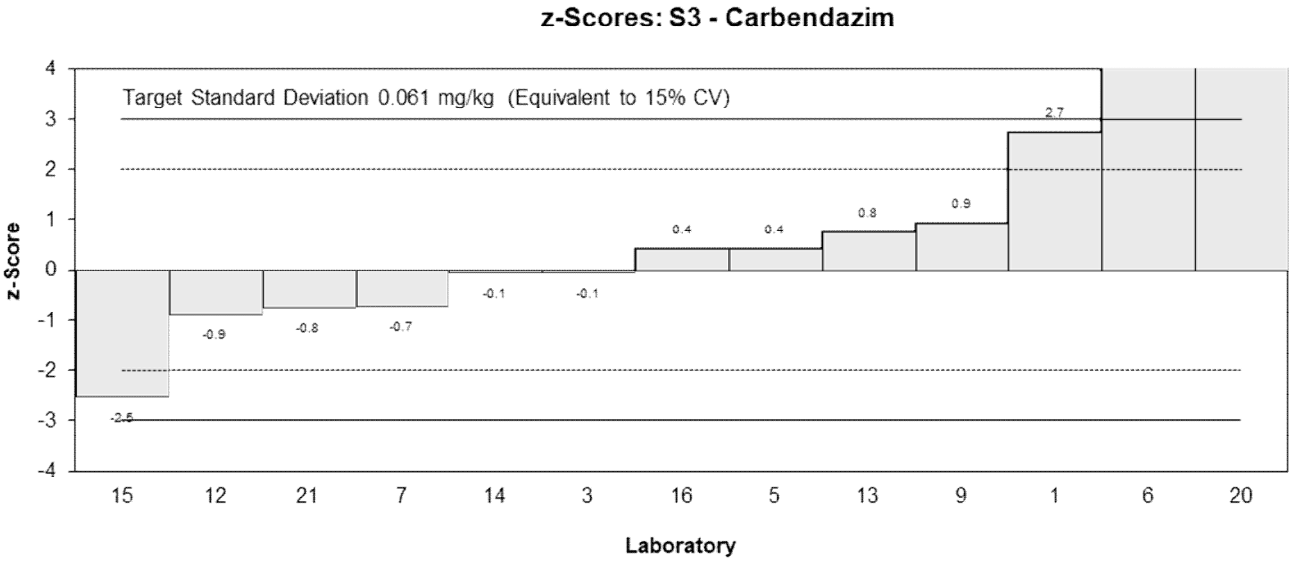
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	0.57	0.17	100	2.74	0.94
2	NR	NR	NR		
3	0.4	0.080	85.52	-0.07	-0.04
4	NT	NT	NT		
5	0.43	0.022	103.6	0.43	0.48
6	0.71	NR	NR	5.05	6.24
7	0.36	0.082	92.1	-0.73	-0.46
8	NT	NT	NT		
9	0.46	NR	NR	0.92	1.14
10	NT	NT	NT		
11	NT	NT	NT		
12	0.35	0.04	92	-0.89	-0.85
13	0.45	0.14	NR	0.76	0.31
14	0.40	0.08	96	-0.07	-0.04
15	0.25	NR	100	-2.54	-3.14
16	0.43	37	88	0.43	0.00
17	NT	NT	NT		
18	NT	NT	NT		
19	NT	NT	NT		
20	0.905	0.271	70	8.27	1.82
21	0.358	0.11	86	-0.76	-0.38

Statistics

Assigned Value*	0.404	0.049
Spike	0.496	0.025
Robust Average	0.440	0.087
Median	0.430	0.063
Mean	0.467	
N	13	
Max.	0.905	
Min.	0.25	
Robust SD	0.13	
Robust CV	29%	

* Robust average excluding Laboratories 6 and 20.


Results: S3 - Carbendazim
Assigned Value = 0.404 ± 0.049 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
15 12 21 7 14 3 16 5 13 9 1 6 20 2 10 11 17 18 19 4 8 Md R.A. S.V.


z-Scores: S3 - Carbendazim
Target Standard Deviation 0.061 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
15 12 21 7 14 3 16 5 13 9 1 6 20

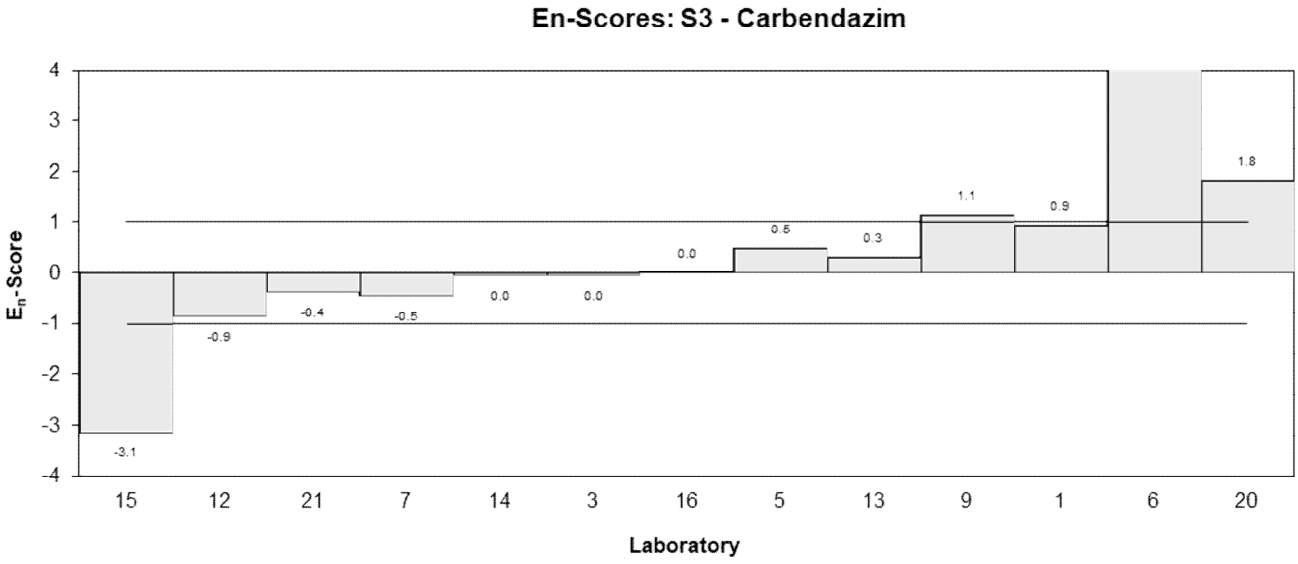

En-Scores: S3 - Carbendazim
En-Score
Laboratory
15 12 21 7 14 3 16 5 13 9 1 6 20

Figure 11

Table 15

Sample Details

Sample No.	S3
Matrix	Apple
Analyte	Pyraclostrobin
Units	mg/kg

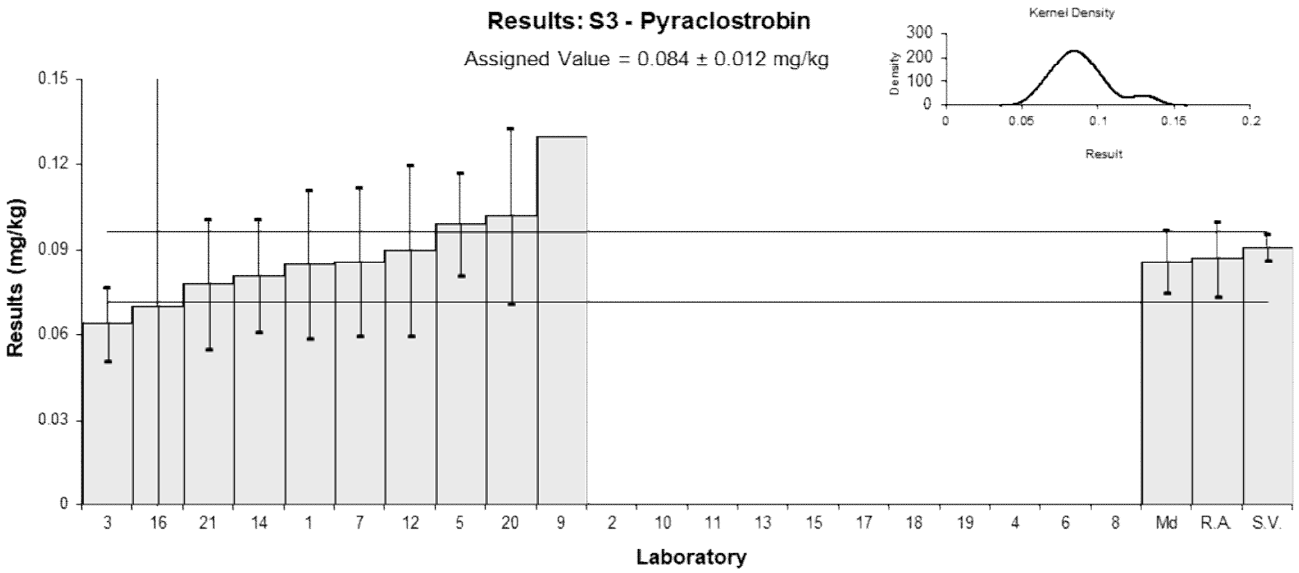
Participant Results

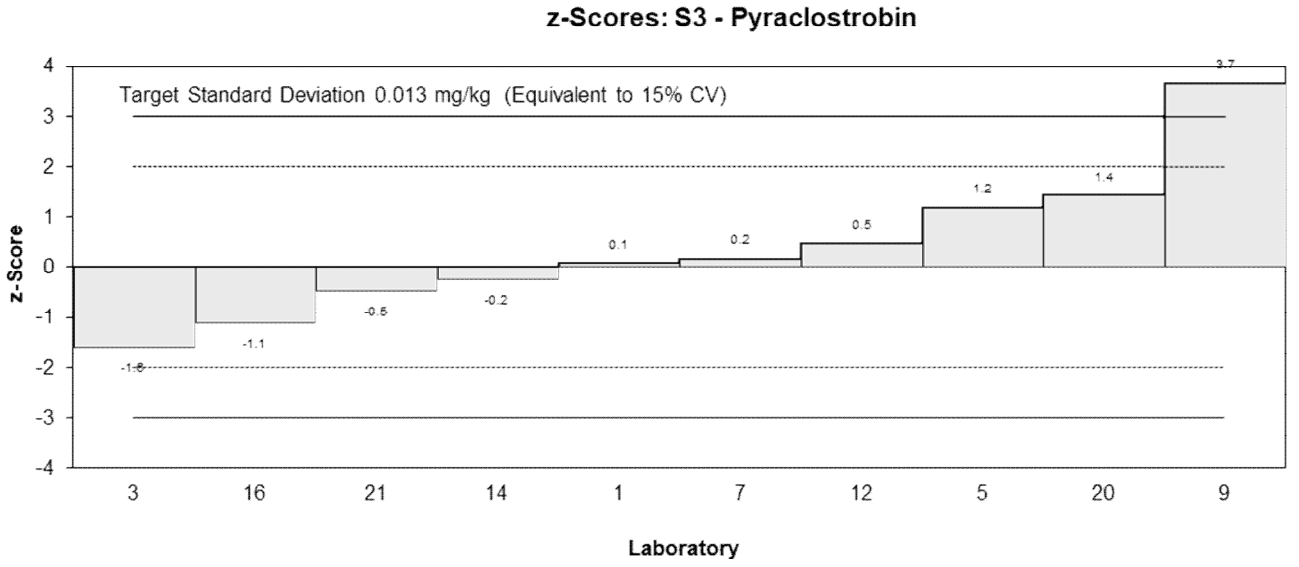
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	0.085	0.026	91	0.08	0.03
2	NR	NR	NR		
3	0.064	0.013	93.24	-1.59	-1.13
4	NT	NT	NT		
5	0.099	0.018	95.4	1.19	0.69
6	NT	NT	NT		
7	0.086	0.026	80.7	0.16	0.07
8	NT	NT	NT		
9	0.13	NR	NR	3.65	3.83
10	NT	NT	NT		
11	NT	NT	NT		
12	0.09	0.03	104	0.48	0.19
13	NT	NT	NT		
14	0.081	0.02	104	-0.24	-0.13
15	NT	NT	NT		
16	0.07	27	98	-1.11	0.00
17	NT	NT	NT		
18	NT	NT	NT		
19	NT	NT	NT		
20	0.102	0.031	93	1.43	0.54
21	0.078	0.023	95	-0.48	-0.23

Statistics

Assigned Value*	0.084	0.012
Spike	0.0909	0.0045
Robust Average	0.087	0.013
Median	0.086	0.011
Mean	0.089	
N	10	
Max.	0.13	
Min.	0.064	
Robust SD	0.017	
Robust CV	19%	

* Robust average excluding Laboratory 9.


Results: S3 - Pyraclostrobin
Assigned Value = 0.084 ± 0.012 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
3 16 21 14 1 7 12 5 20 9 2 10 11 13 15 17 18 19 4 6 8 Md R.A. S.V.


z-Scores: S3 - Pyraclostrobin
Target Standard Deviation 0.013 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
3 16 21 14 1 7 12 5 20 9

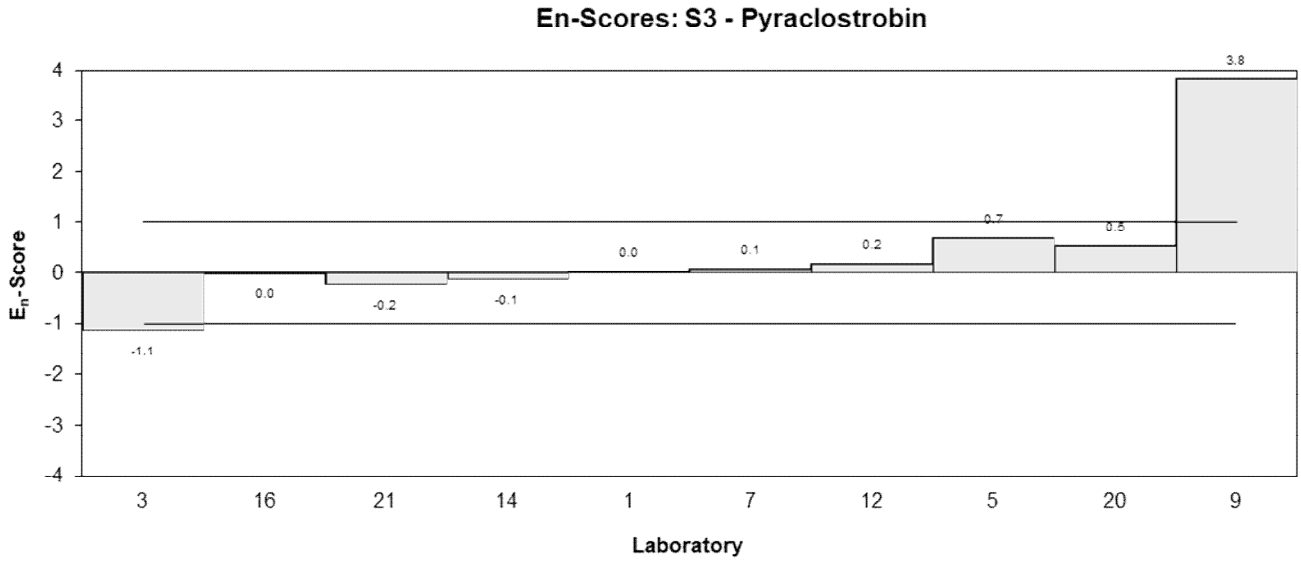

En-Scores: S3 - Pyraclostrobin
En-Score
Laboratory
3 16 21 14 1 7 12 5 20 9

Figure 12

Table 16

Sample Details

Sample No.	S3
Matrix	Apple
Analyte	Triadimefon
Units	mg/kg

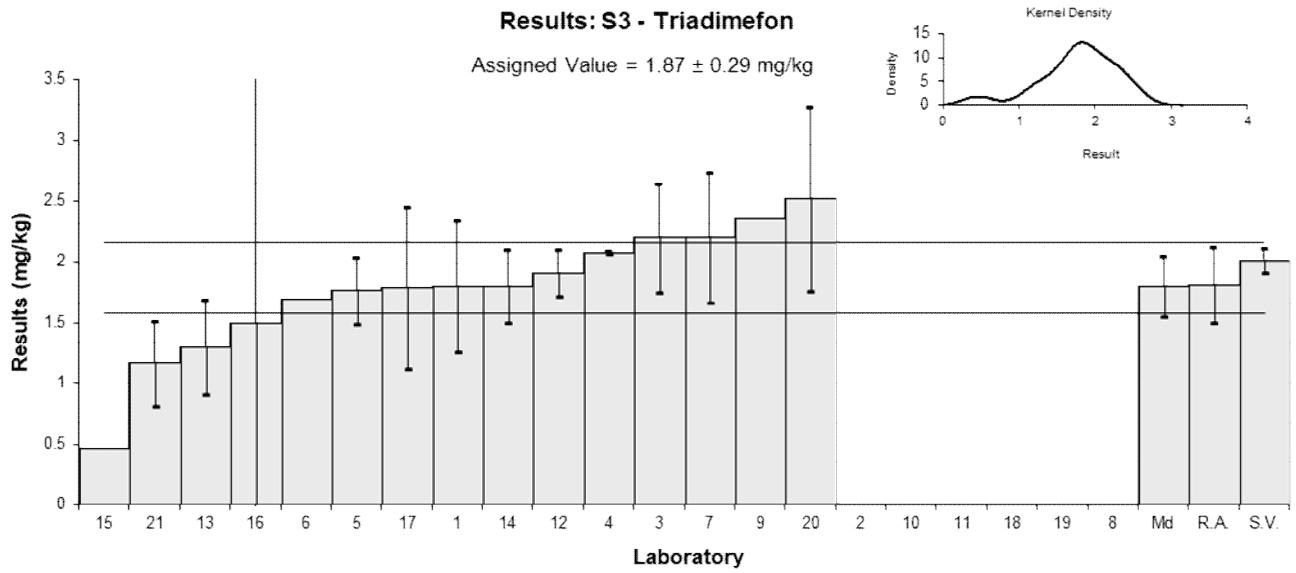
Participant Results

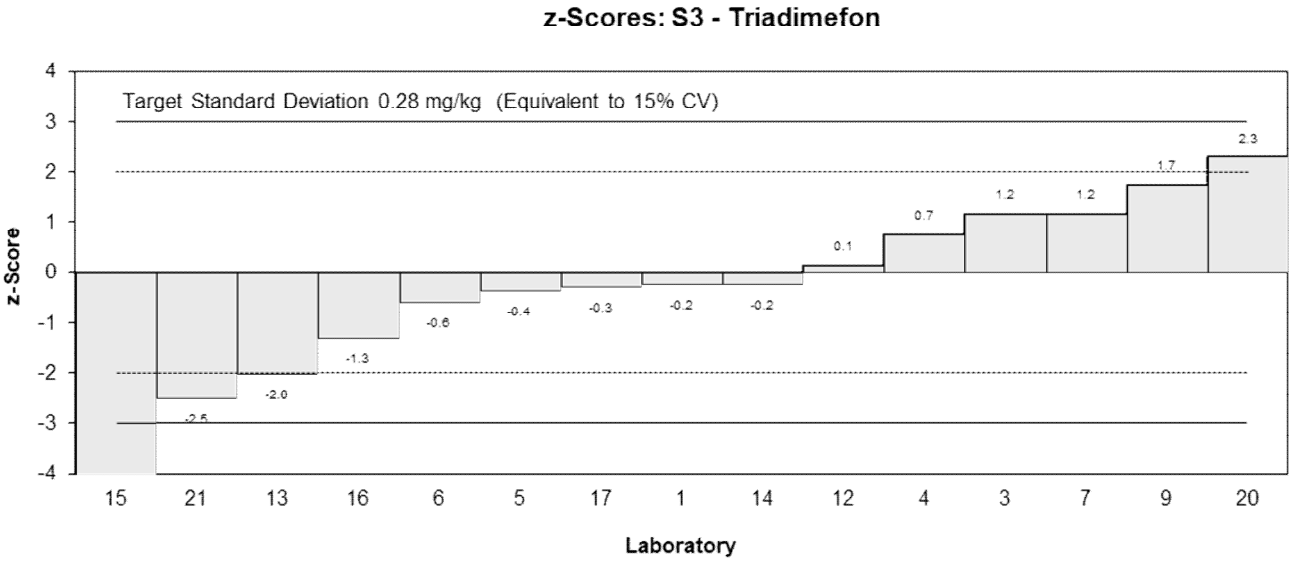
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	1.8	0.54	102	-0.25	-0.11
2	NR	NR	NR		
3	2.2	0.44	134.20	1.18	0.63
4	2.08	0.01	91	0.75	0.72
5	1.77	0.274	84.8	-0.36	-0.25
6	1.7	NR	NR	-0.61	-0.59
7	2.2	0.53	96.4	1.18	0.55
8	NT	NT	NT		
9	2.36	NR	NR	1.75	1.69
10	NT	NT	NT		
11	NT	NT	NT		
12	1.91	0.19	97	0.14	0.12
13	1.3	0.39	NR	-2.03	-1.17
14	1.8	0.3	105	-0.25	-0.17
15	0.46	NR	86	-5.03	-4.86
16	1.5	30	94	-1.32	-0.01
17	1.789	0.6618	105	-0.29	-0.11
18	NT	NT	NT		
19	NT	NT	NT		
20	2.515	0.754	74	2.30	0.80
21	1.165	0.35	84	-2.51	-1.55

Statistics

Assigned Value*	1.87	0.29
Spike	2.01	0.10
Robust Average	1.81	0.31
Median	1.80	0.25
Mean	1.77	
N	15	
Max.	2.515	
Min.	0.46	
Robust SD	0.48	
Robust CV	26%	

* Robust average excluding Laboratory 15.


Results: S3 - Triadimefon
Assigned Value = 1.87 ± 0.29 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
15 21 13 16 6 5 17 1 14 12 4 3 7 9 20 2 10 11 18 19 8 Md R.A. S.V.


z-Scores: S3 - Triadimefon
Target Standard Deviation 0.28 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
15 21 13 16 6 5 17 1 14 12 4 3 7 9 20
-2.5 -2.0 -1.3 -0.6 -0.4 -0.3 -0.2 -0.2 0.1 0.7 1.2 1.2 1.7 2.3

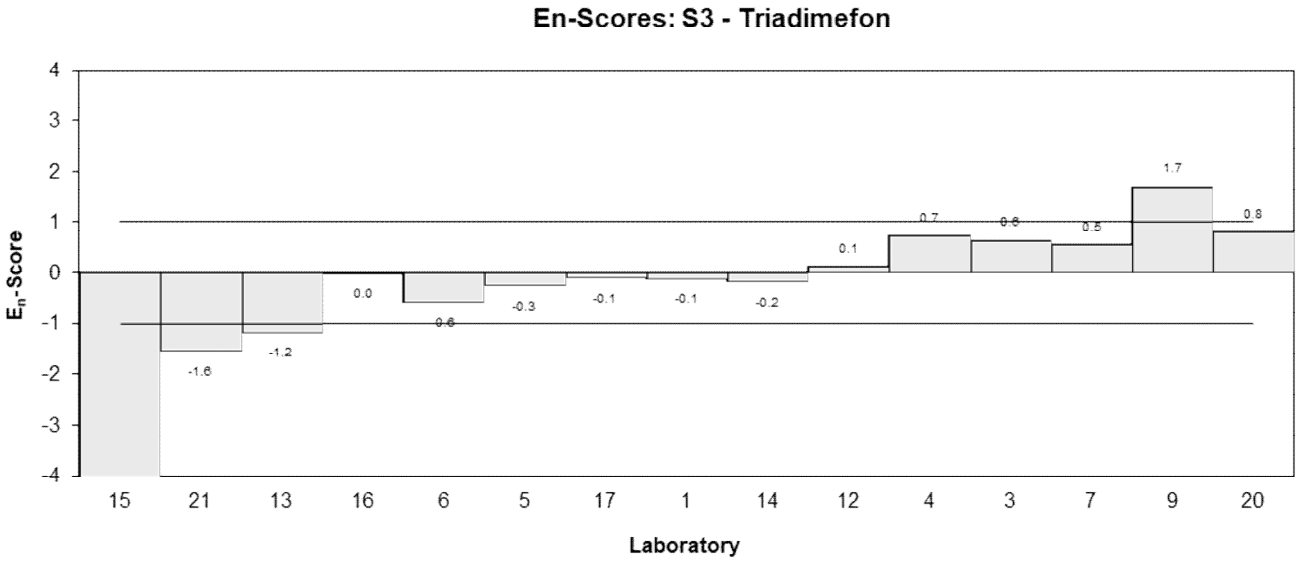

En-Scores: S3 - Triadimefon
En-Score
Laboratory
15 21 13 16 6 5 17 1 14 12 4 3 7 9 20
-1.6 -1.2 0.0 -0.6 -0.3 -0.1 -0.1 -0.2 0.1 0.7 0.6 0.5 1.7 0.8

Figure 13

Table 17

Sample Details

Sample No.	S4
Matrix	Orange
Analyte	Acetamiprid
Units	mg/kg

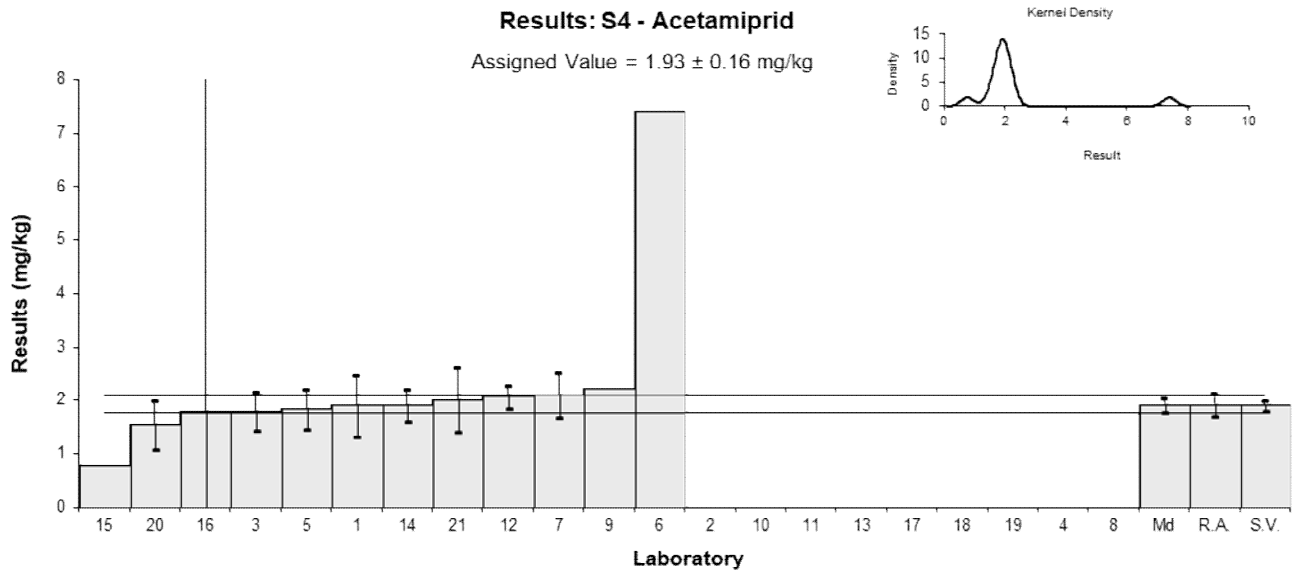
Participant Results

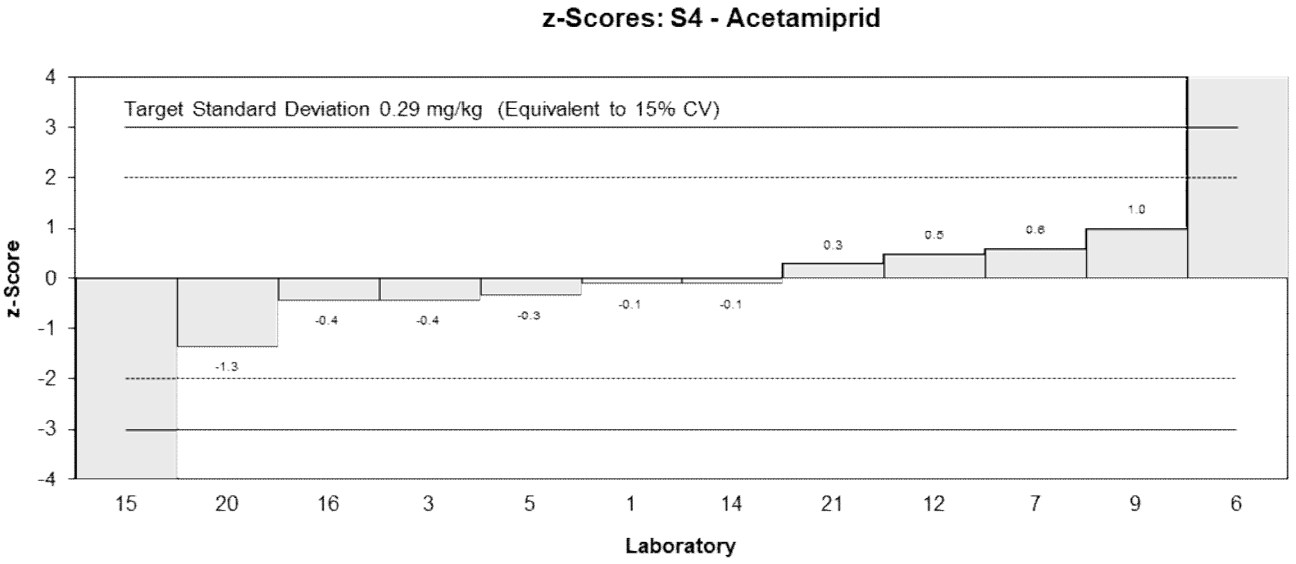
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	1.9	0.57	117	-0.10	-0.05
2	NR	NR	NR		
3	1.8	0.36	106.71	-0.45	-0.33
4	NT	NT	NT		
5	1.83	0.368	90.8	-0.35	-0.25
6	7.4	NR	NR	18.89	34.19
7	2.1	0.42	81.2	0.59	0.38
8	NT	NT	NT		
9	2.22	NR	NR	1.00	1.81
10	NT	NT	NT		
11	NT	NT	NT		
12	2.07	0.21	104	0.48	0.53
13	NT	NT	NT		
14	1.9	0.3	98	-0.10	-0.09
15	0.77	NR	142	-4.01	-7.25
16	1.8	51	87	-0.45	0.00
17	NT	NT	NT		
18	NT	NT	NT		
19	NT	NT	NT		
20	1.540	0.462	61	-1.35	-0.80
21	2.014	0.60	77	0.29	0.14

Statistics

Assigned Value*	1.93	0.16
Spike	1.90	0.09
Robust Average	1.92	0.21
Median	1.90	0.13
Mean	2.28	
N	12	
Max.	7.4	
Min.	0.77	
Robust SD	0.29	
Robust CV	15%	

* Robust average excluding Laboratories 6 and 15.


Results: S4 - Acetamiprid
Assigned Value = 1.93 ± 0.16 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
15 20 16 3 5 1 14 21 12 7 9 6 2 10 11 13 17 18 19 4 8 Md R.A. S.V.


z-Scores: S4 - Acetamiprid
Target Standard Deviation 0.29 mg/kg (Equivalent to 15% CV)
z-Score
-1.3 -0.4 -0.4 -0.3 -0.1 -0.1 0.3 0.5 0.6 1.0
Laboratory
15 20 16 3 5 1 14 21 12 7 9 6

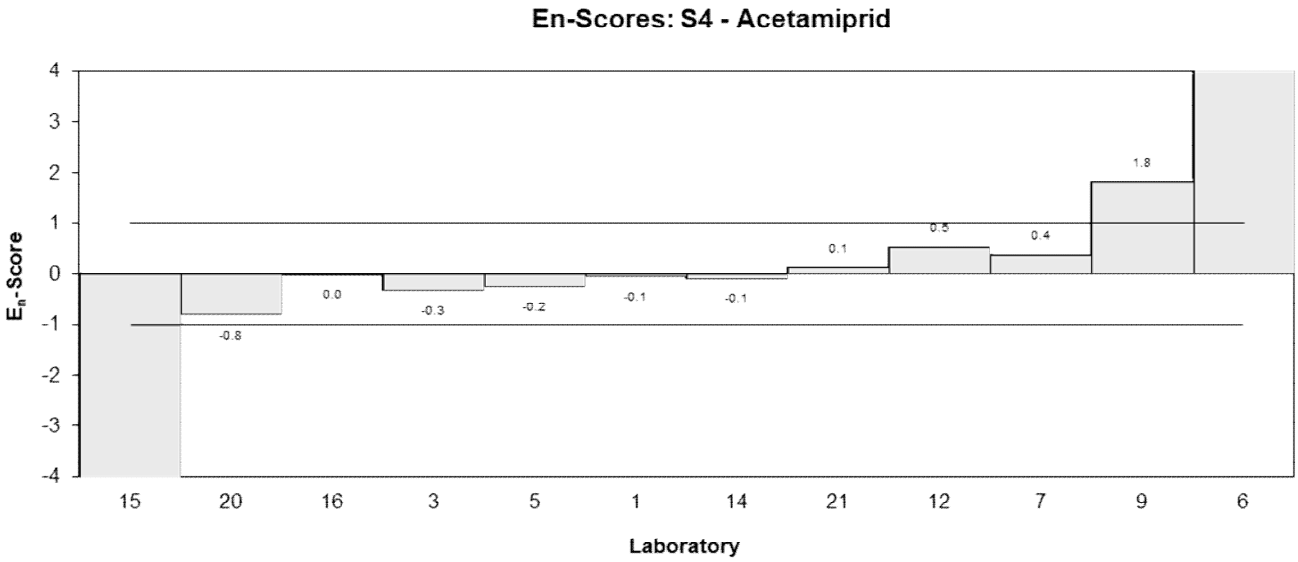

En-Scores: S4 - Acetamiprid
En-Score
-0.8 0.0 -0.3 -0.2 -0.1 -0.1 0.1 0.5 0.4 1.8
Laboratory
15 20 16 3 5 1 14 21 12 7 9 6

Figure 14

Table 18

Sample Details

Sample No.	S4
Matrix	Orange
Analyte	Azoxystrobin
Units	mg/kg

Participant Results

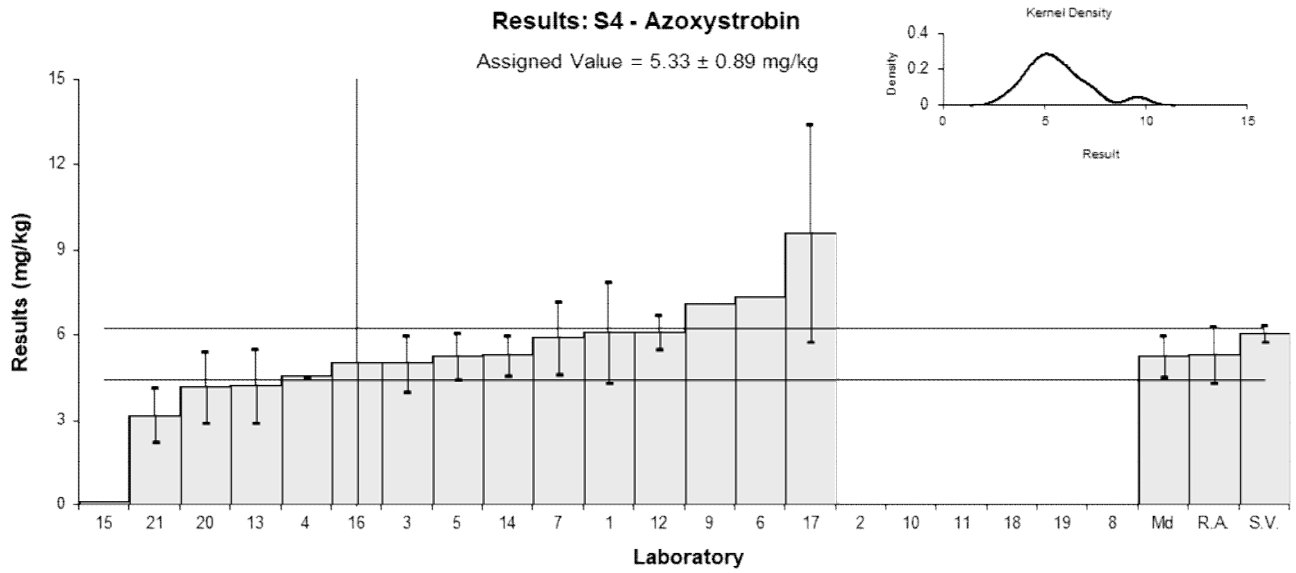
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	6.1	1.8	105	0.96	0.38
2	NR	NR	NR		
3	5.0	1.0	83.48	-0.41	-0.25
4	4.55	0.002	158	-0.98	-0.88
5	5.26	0.81	102.6	-0.09	-0.06
6	7.35	NR	NR	2.53	2.27
7	5.9	1.29	88.6	0.71	0.36
8	NT	NT	NT		
9	7.08	NR	NR	2.19	1.97
10	NT	NT	NT		
11	NT	NT	NT		
12	6.12	0.61	101	0.99	0.73
13	4.2	1.3	NR	-1.41	-0.72
14	5.3	0.7	98	-0.04	-0.03
15	0.08	NR	95	-6.57	-5.90
16	5.0	22	96	-0.41	-0.01
17	9.595	3.838	128	5.33	1.08
18	NT	NT	NT		
19	NT	NT	NT		
20	4.165	1.249	62	-1.46	-0.76
21	3.181	0.95	104	-2.69	-1.65

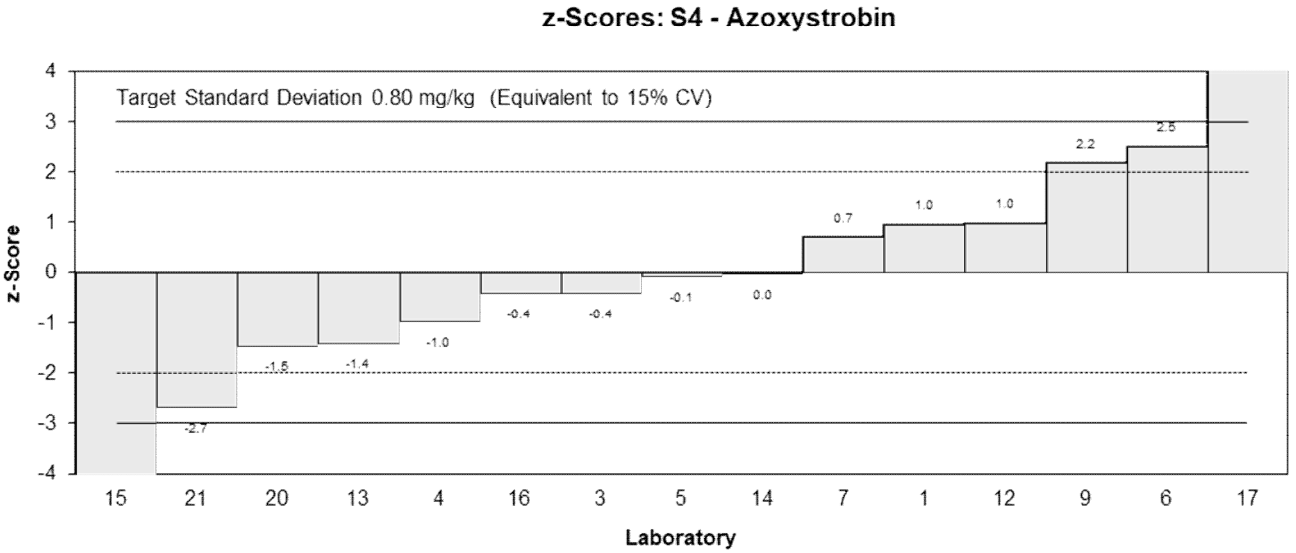
Statistics*

Assigned Value**	5.33	0.89
Spike	6.05	0.30
Robust Average	5.50	0.97
Median	5.28	0.71
Mean	5.63	
N	14	
Max.	9.595	
Min.	3.181	
Robust SD	1.4	
Robust CV	26%	

* Laboratory 15 excluded from all statistical calculations as an extreme outlier.

** Robust average excluding Laboratory 17.


Results: S4 - Azoxystrobin
Assigned Value = 5.33 ± 0.89 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
15 21 20 13 4 16 3 5 14 7 1 12 9 6 17 2 10 11 18 19 8 Md R.A. S.V.


z-Scores: S4 - Azoxystrobin
Target Standard Deviation 0.80 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
15 21 20 13 4 16 3 5 14 7 1 12 9 6 17
-2.7 -1.5 -1.4 -1.0 -0.4 -0.4 -0.1 0.0 0.7 1.0 1.0 2.2 2.5

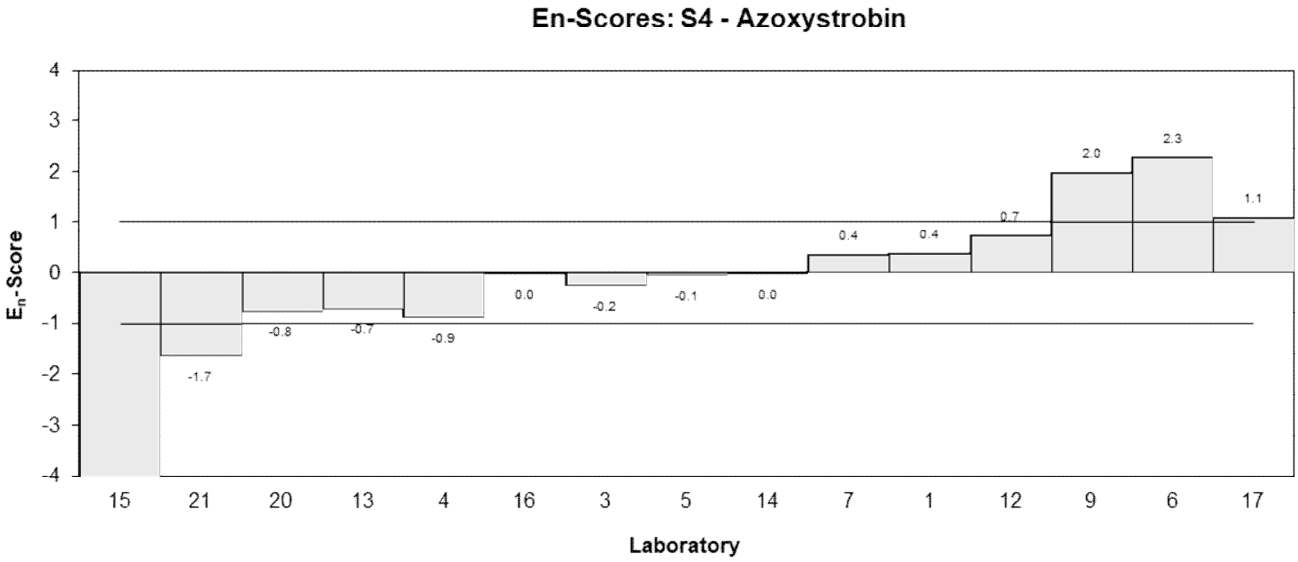

En-Scores: S4 - Azoxystrobin
En-Score
Laboratory
15 21 20 13 4 16 3 5 14 7 1 12 9 6 17
-1.7 -0.8 -0.7 -0.9 0.0 -0.2 -0.1 0.0 0.4 0.4 0.7 2.0 2.3 1.1

Figure 15

Table 19

Sample Details

Sample No.	S4
Matrix	Orange
Analyte	Cyfluthrin
Units	mg/kg

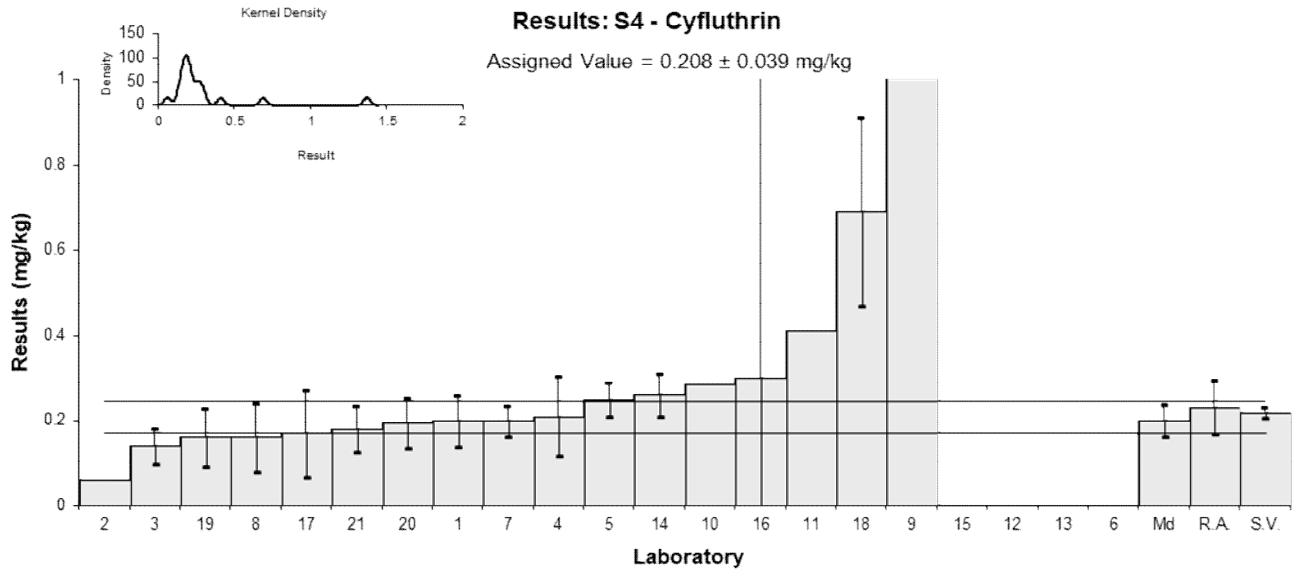
Participant Results

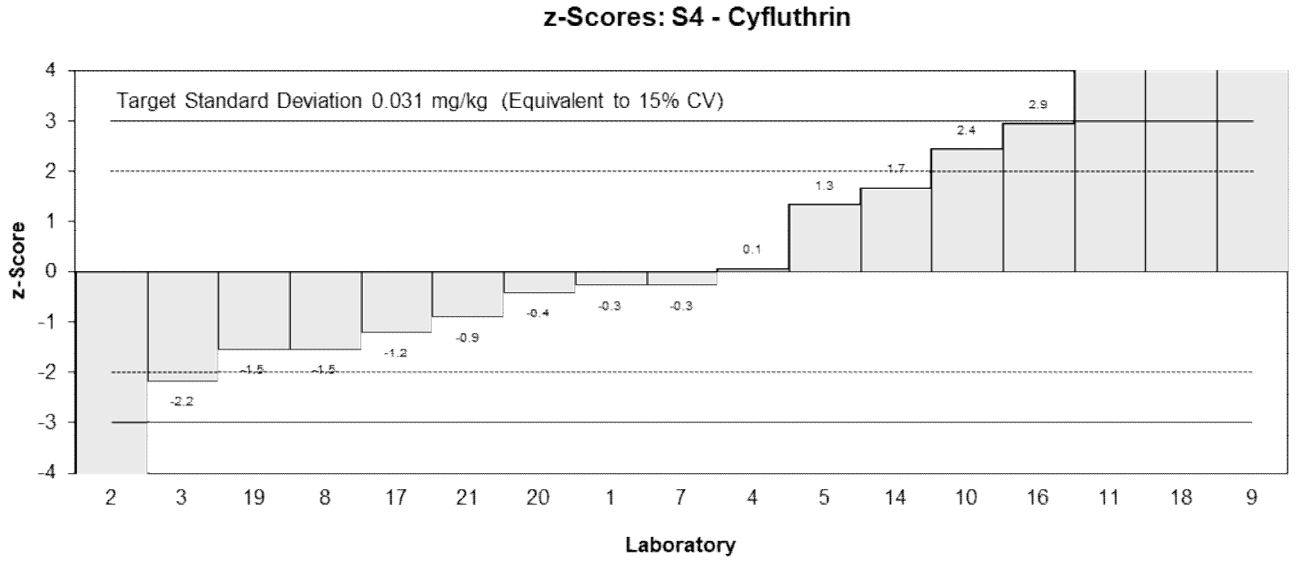
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	0.20	0.06	86	-0.26	-0.11
2	0.06	NR	NR	-4.74	-3.79
3	0.14	0.042	73.25	-2.18	-1.19
4	0.21	0.093	90	0.06	0.02
5	0.25	0.038	84.1	1.35	0.77
6	NT	NT	NT		
7	0.20	0.036	88.6	-0.26	-0.15
8	0.16	0.08	90	-1.54	-0.54
9	1.37	NR	NR	37.24	29.79
10	0.284	NR	127.71	2.44	1.95
11	0.41	NR	98	6.47	5.18
12	NT	NT	NT		
13	NT	NT	NT		
14	0.26	0.05	90	1.67	0.82
15	NR	NR	NR		
16	0.30	40	106	2.95	0.00
17	0.17	0.102	115	-1.22	-0.35
18	0.69	0.22	89.76	15.45	2.16
19	0.16	0.07	NR	-1.54	-0.60
20	0.195	0.058	89	-0.42	-0.19
21	0.18	0.054	130	-0.90	-0.42

Statistics

Assigned Value*	0.208	0.039
Spike	0.220	0.011
Robust Average	0.232	0.063
Median	0.200	0.038
Mean	0.308	
N	17	
Max.	1.37	
Min.	0.06	
Robust SD	0.10	
Robust CV	45%	

* Robust average excluding Laboratories 2, 9, 11 and 18.


Kernel Density
Density
Result
Results: S4 - Cyfluthrin
Assigned Value = 0.208 ± 0.039 mg/kg
Results (mg/kg)
Laboratory
2 3 19 8 17 21 20 1 7 4 5 14 10 16 11 18 9 15 12 13 6 Md R.A. S.V.


z-Scores: S4 - Cyfluthrin
Target Standard Deviation 0.031 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
2 3 19 8 17 21 20 1 7 4 5 14 10 16 11 18 9

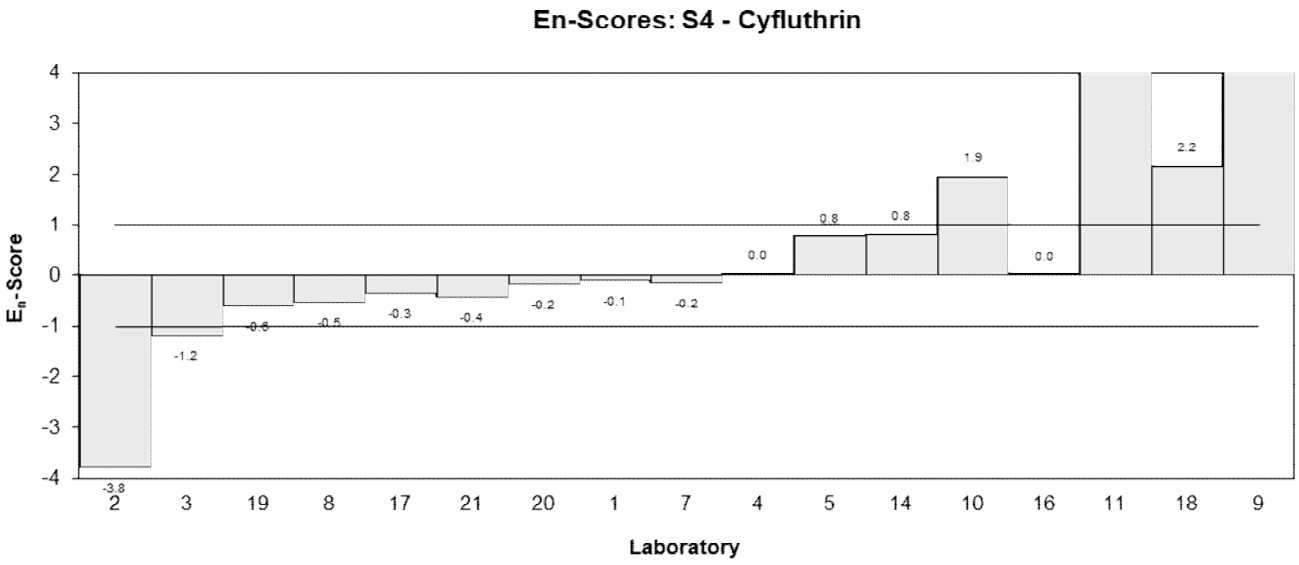

En-Scores: S4 - Cyfluthrin
En-Score
Laboratory
2 3 19 8 17 21 20 1 7 4 5 14 10 16 11 18 9

Figure 16

Table 20

Sample Details

Sample No.	S4
Matrix	Orange
Analyte	Imidacloprid
Units	mg/kg

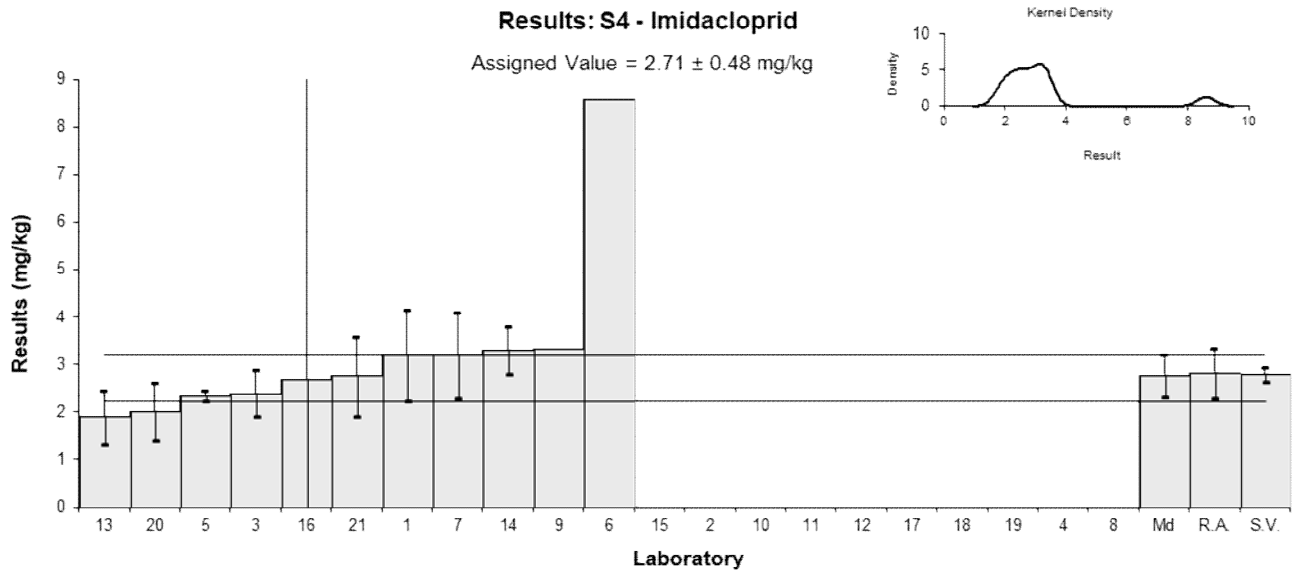
Participant Results

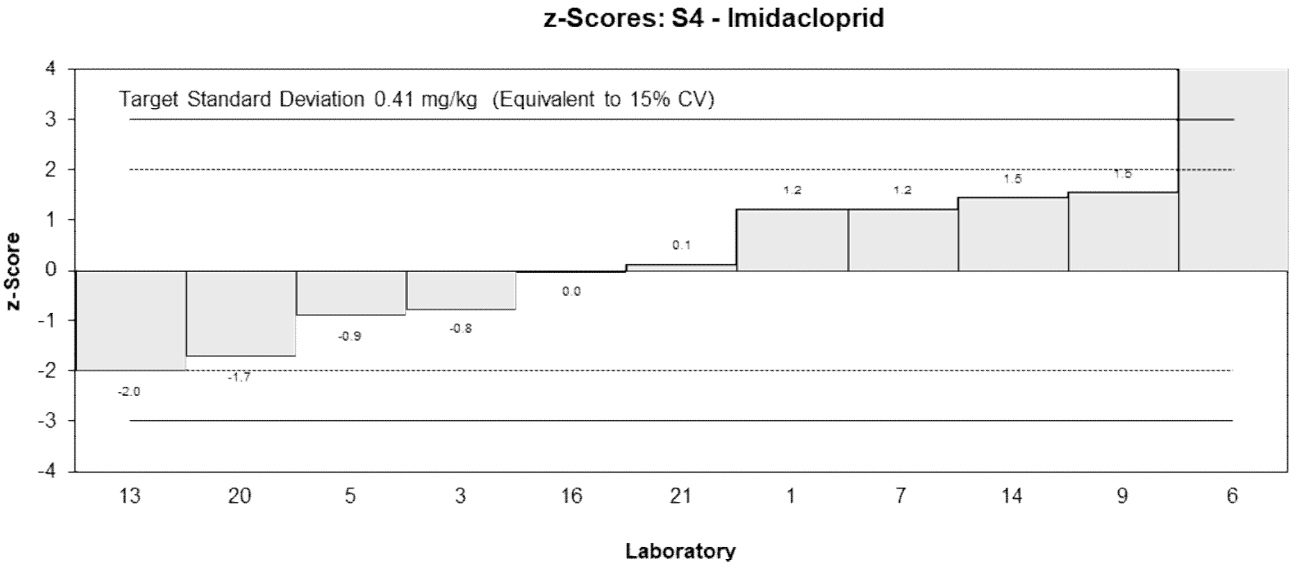
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	3.2	0.96	111	1.21	0.46
2	NR	NR	NR		
3	2.4	0.48	110.94	-0.76	-0.46
4	NT	NT	NT		
5	2.34	0.112	108.9	-0.91	-0.75
6	8.6	NR	NR	14.49	12.27
7	3.2	0.9	91.2	1.21	0.48
8	NT	NT	NT		
9	3.34	NR	NR	1.55	1.31
10	NT	NT	NT		
11	NT	NT	NT		
12	NT	NT	NT		
13	1.9	0.57	NR	-1.99	-1.09
14	3.3	0.5	101	1.45	0.85
15	NR	NR	NR		
16	2.7	55	83	-0.02	0.00
17	NT	NT	NT		
18	NT	NT	NT		
19	NT	NT	NT		
20	2.010	0.603	63	-1.72	-0.91
21	2.758	0.83	79	0.12	0.05

Statistics

Assigned Value*	2.71	0.48
Spike	2.79	0.14
Robust Average	2.82	0.53
Median	2.76	0.44
Mean	3.25	
N	11	
Max.	8.6	
Min.	1.9	
Robust SD	0.70	
Robust CV	25%	

* Robust average excluding Laboratory 6.


Results: S4 - Imidacloprid
Assigned Value = 2.71 ± 0.48 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
13 20 5 3 16 21 1 7 14 9 6 15 2 10 11 12 17 18 19 4 8 Md R.A. S.V.
Laboratory


z-Scores: S4 - Imidacloprid
Target Standard Deviation 0.41 mg/kg (Equivalent to 15% CV)
z-Score
-2.0 -1.7 -0.9 -0.8 0.0 0.1 1.2 1.2 1.5 1.5
13 20 5 3 16 21 1 7 14 9 6
Laboratory

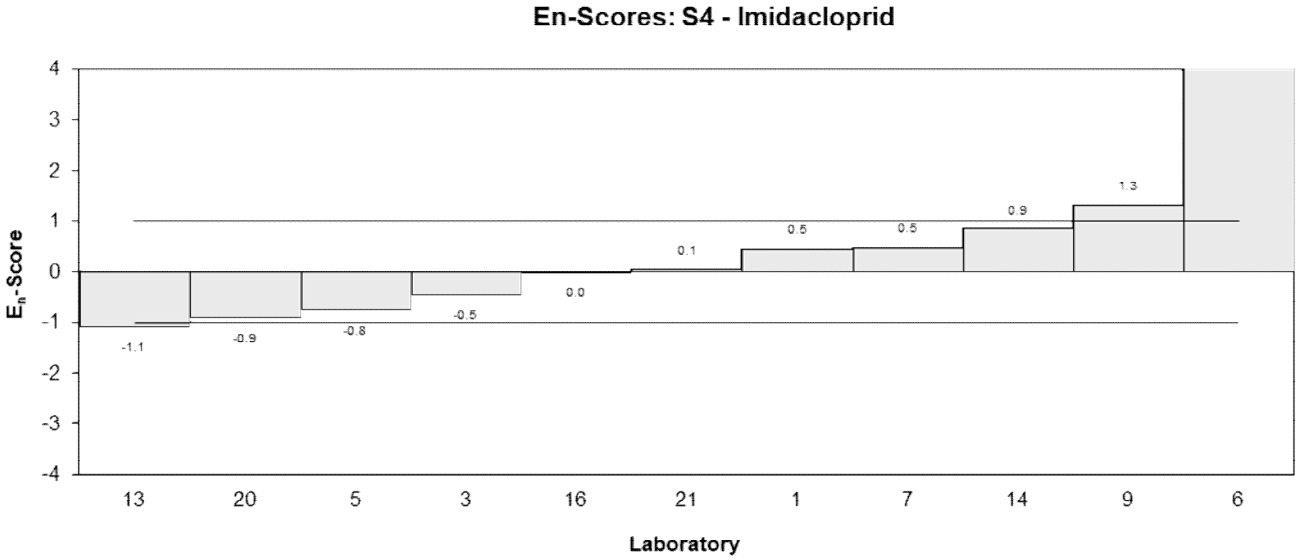

En-Scores: S4 - Imidacloprid
En-Score
-1.1 -0.9 -0.8 -0.5 0.0 0.1 0.5 0.5 0.9 1.3
13 20 5 3 16 21 1 7 14 9 6
Laboratory

Figure 17

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 13528:2015.[7] Results less than 50% and greater than 150% of the robust average were removed before the calculation of the assigned value.[3,4] The calculation of the expanded uncertainty for a robust average is presented in Appendix 4, using endosulfan sulfate in Sample S1 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 cyfluthrin in Sample S2 as reported numeric results were too variable; the variability of participants' results may have been due to the matrix, mass fraction level, properties of the analyte itself, or a combination of these.

A comparison of the assigned value (or robust average if no assigned value was set) and the spiked value is presented in Table 21. The assigned values were within the range 79% to 102% of the spiked values, providing good support for the assigned values and is further evidence for the stability of these analytes in the test samples.

Table 21 Comparison of Assigned Values (or Robust Averages) and Spiked Values

Sample	Analyte	Assigned Value *(Robust Average)* (mg/kg)	Spiked Value (mg/kg)	Assigned Value *(Robust Average)* / Spiked Value (%)
S1	Cyhalothrin	0.0363	0.0458	79
	Dimethoate	0.0534	0.0548	97
	Endosulfan sulfate	0.73	0.865	84
	Omethoate	2.30	2.28	101
S2	Cyfluthrin	*(0.79)*	0.902	*(88)*
	Glyphosate	0.208	0.250	83
	Indoxacarb	2.70	3.01	90
	Pyraclostrobin	1.18	1.20	98
S3	Acetamiprid	0.170	0.172	99
	Carbendazim	0.404	0.496	81
	Pyraclostrobin	0.084	0.0909	92
	Triadimefon	1.87	2.01	93
S4	Acetamiprid	1.93	1.90	102
	Azoxystrobin	5.33	6.05	88
	Cyfluthrin	0.208	0.220	95
	Imidacloprid	2.71	2.79	97

6.2 Measurement Uncertainty Reported by Participants

Participants were asked to report an estimate of the expanded MU associated with their results and the basis of this estimate. It is a requirement of ISO/IEC 17025:2017 that laboratories have procedures to estimate the uncertainty of chemical measurements and to report this in specific circumstances, including when the client's instruction so requires.[9]

Of 217 numerical results for the analytes of interest in this study, 178 (82%) were reported with an associated expanded MU. Participants used a wide variety of procedures to estimate their uncertainty (Table 3).

Laboratory **21** reported their uncertainties as a percentage rather than in units of mg/kg (i.e. uncertainty values were reported as "*x*%"). These values were therefore modified accordingly by the study coordinator for this report.

Laboratories **2**, **6**, **7**, **9**, **10**, **11** and **15** did not provide uncertainties for at least one reported result. Laboratory **10** reported that they were accredited to ISO/IEC 17025, however, they specified that some analytes were not under their laboratory's scope of accreditation, and these were the analytes that they did not provide uncertainties for. Laboratory **6** did not report any uncertainties, while Laboratories **7**, **9**, **11** and **15** each reported a mixture of results with and without uncertainties; these participants all stated that they were accredited to ISO/IEC 17025. Laboratory **2** did not report uncertainties for any of their results; this participant did not provide their accreditation status.

The magnitude of the reported uncertainties for spiked analytes in this study was within the range 0.044% to 150000% relative to the result. In general, an expanded uncertainty of less than 15% relative is likely to be unrealistically small for the routine measurement of a pesticide residue, while over 50% is likely too large. Of the 178 expanded uncertainties, 23 were less than 15% relative and 21 were greater than 50% relative.

Laboratory **4**'s uncertainties were extremely varied, ranging from 0.044% to 55% relative. Laboratory **10** had one reported uncertainty, and this uncertainty was 290% relative to the result. Laboratory **16**'s uncertainties ranged from 440% to 150000% relative; this participant may have reported their uncertainties as relative instead of absolute values, however, values were not reported with a "%" (or equivalent) and therefore no modifications were made.

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

In some cases the 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 2.244 ± 0.7631 mg/kg, it is recommended to report 2.24 ± 0.76 mg/kg.[10]

6.3 z-Scores

Target SDs equivalent to 15% PCV were used to calculate z-scores. CVs predicted by the Thompson-Horwitz equation,[8] target SDs (as PCV), and the between-laboratory CVs obtained in this study for scored analytes are presented for comparison in Table 22.

Table 22 Comparison of Thompson-Horwitz CVs, Target SDs, and Between-Laboratory CV

Sample	Analyte	Assigned value (mg/kg)	Thompson-Horwitz CV (%)	Target SD (as PCV) (%)	Between-Laboratory CV* (%)
S1	Cyhalothrin	0.0363	22	15	18
	Dimethoate	0.0534	22	15	14
	Endosulfan sulfate	0.73	17	15	26
	Omethoate	2.30	14	15	15

Sample	Analyte	Assigned value (mg/kg)	Thompson-Horwitz CV (%)	Target SD (as PCV) (%)	Between-Laboratory CV* (%)
S2	Glyphosate	0.208	20	15	17
	Indoxacarb	2.70	14	15	23
	Pyraclostrobin	1.18	16	15	14
S3	Acetamiprid	0.170	21	15	21
	Carbendazim	0.404	18	15	16
	Pyraclostrobin	0.084	22	15	17
	Triadimefon	1.87	15	15	23
S4	Acetamiprid	1.93	14	15	10
	Azoxystrobin	5.33	12	15	24
	Cyfluthrin	0.208	20	15	27
	Imidacloprid	2.71	14	15	22

* Robust between-laboratory CVs with outliers removed, if applicable.

To account for possible low bias in the consensus value due to laboratories using inefficient analytical or extraction techniques, two z-scores were adjusted for cyhalothrin in Sample S1. A maximum acceptable concentration was set to two target SDs more than the spiked value, and results lower than the maximum acceptable concentration 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 concentration were not adjusted, and z-scores less than 2.0 were left unaltered.

Of 199 results for which z-scores were calculated, 154 (77%) returned $|z| \leq 2.0$, indicating a satisfactory performance.

Laboratories **3**, **5**, **16**, **20** and **21** reported results for all 15 scored analytes.

Satisfactory z-scores were achieved for all scored analytes reported by Laboratories **7** (14), **14** (14) and **12** (9).

The dispersal of participants' z-scores is presented graphically by laboratory in Figure 18 and by analyte in Figure 19. z-Scores greater than 10 have been plotted at 10.

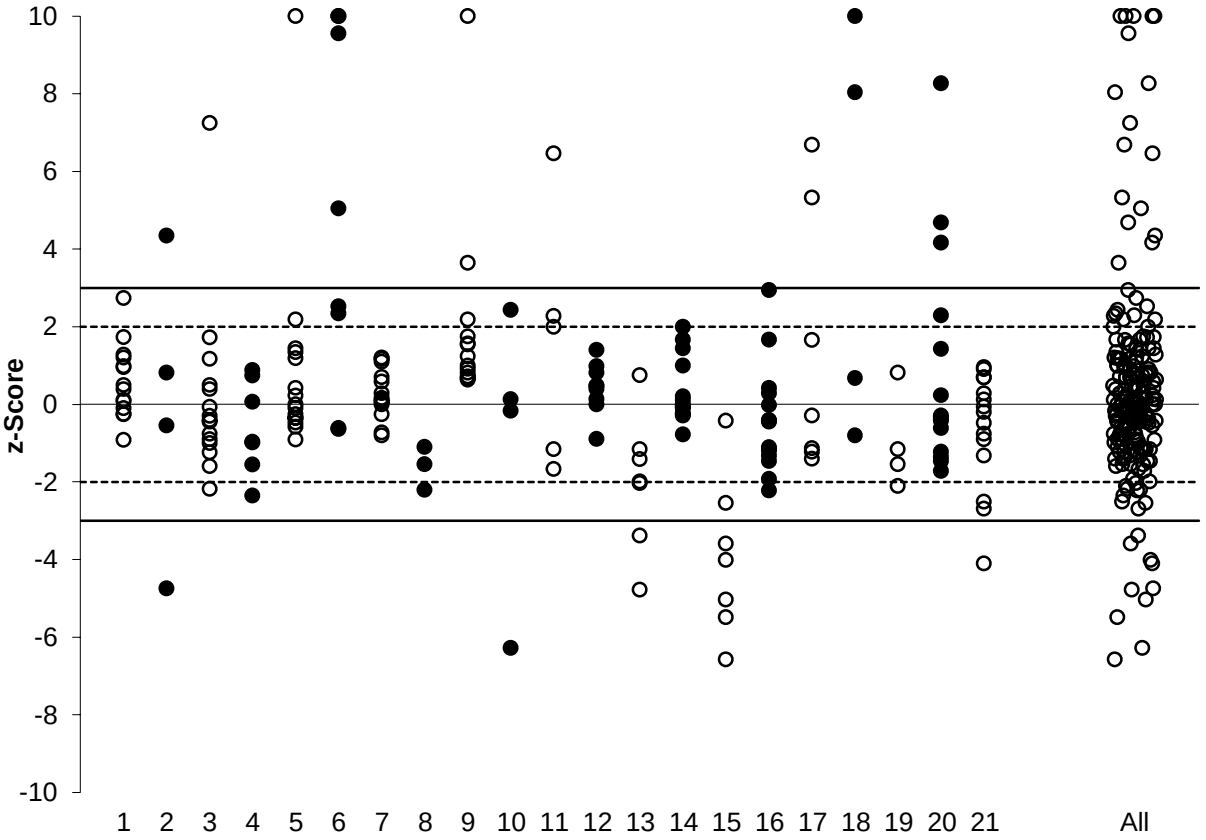


Figure 18 z-Score Dispersal by Laboratory

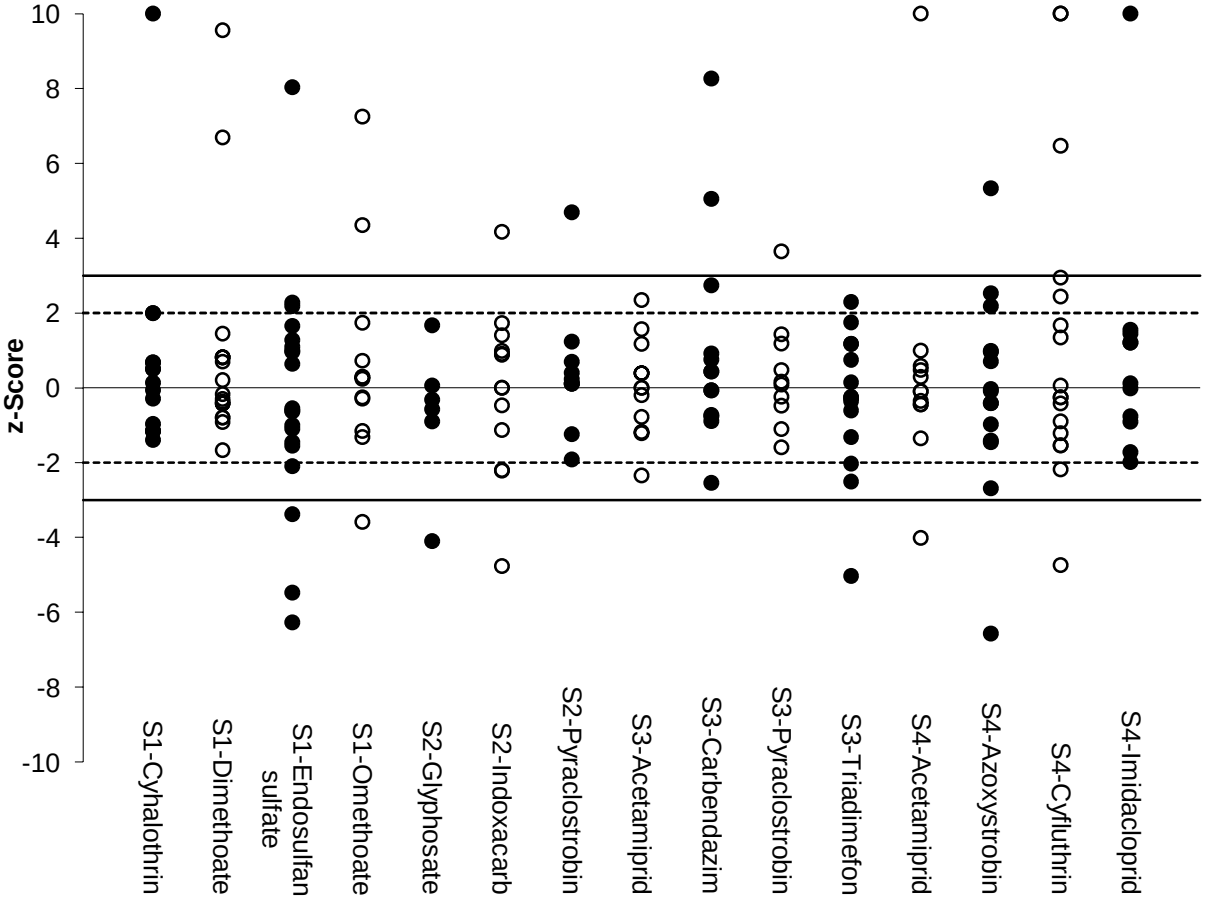
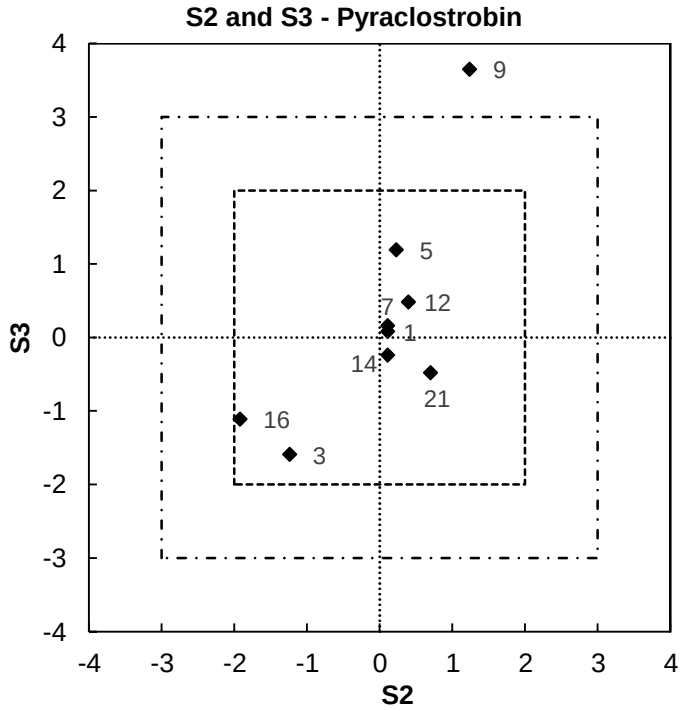


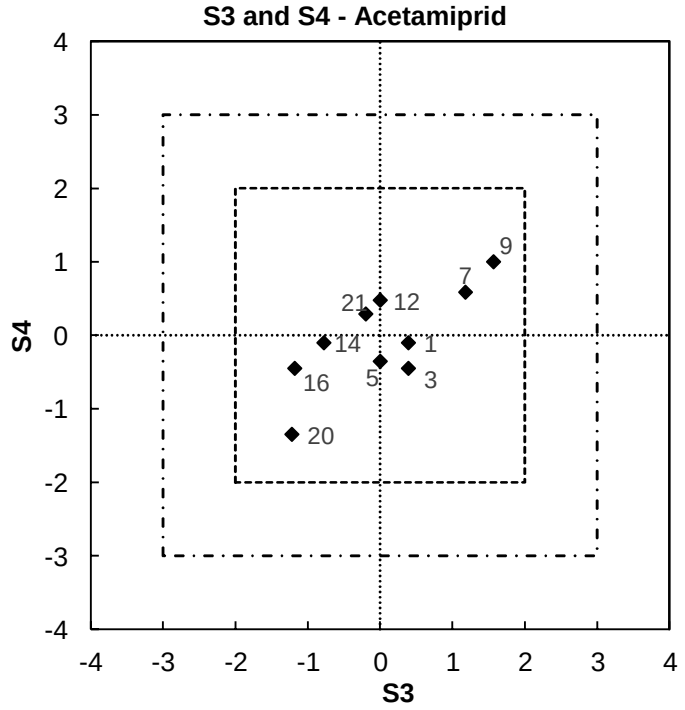
Figure 19 z-Score Dispersal by Analyte

Scatter plots of z-scores for pyraclostrobin and acetamiprid in different samples are presented in Figures 20 and 21. Scores are predominantly in the upper right and lower left quadrants, indicating that laboratory bias is the major contributor to the variability of results. Points close to the diagonal axis demonstrate excellent repeatability, while points close to the zero demonstrate excellent repeatability and accuracy.



Laboratory 20 is off-scale.

Figure 20 z-Score Scatter Plot – Pyraclostrobin in Samples S2 and S3



Laboratory 6 is off-scale.

Figure 21 z-Score Scatter Plot – Acetamiprid in Samples S3 and S4

6.4 E_n-Scores

Where a laboratory did not report an expanded uncertainty with a result, an expanded uncertainty of zero (0) was used to calculate the E_n-score. E_n-scores greater than 1.0 were set to 1.0 for results with z-scores that were adjusted as discussed in Section 6.3 z-Scores.

Of 199 results for which E_n-scores were calculated, 149 (75%) returned $|E_n| \leq 1.0$, indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

Satisfactory E_n-scores were achieved for all scored analytes reported by Laboratories **1** (14), **7** (14), **14** (14), **12** (9) and **8** (3).

Laboratory **16** returned E_n-scores with $|E_n| \leq 1.0$ for all 15 scored analytes, though this participant reported unrealistically large uncertainties (ranging from 440% to 150000% relative to their results).

The dispersal of participants' E_n-scores is presented graphically by laboratory in Figure 22. E_n-scores greater than 10 have been plotted at 10.

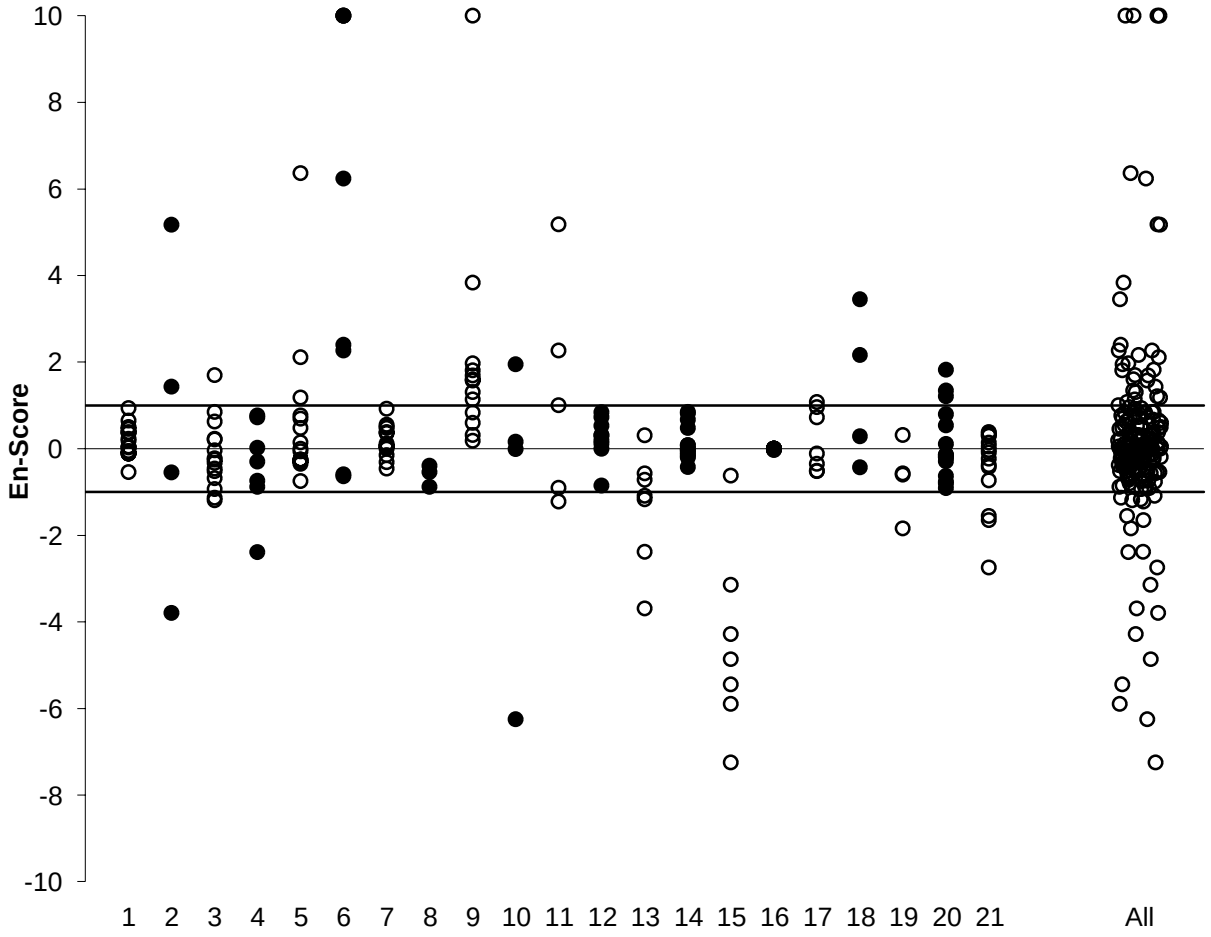


Figure 22 E_n-Score Dispersal by Laboratory

6.5 False Negatives

Table 23 presents false negative results. These are analytes present in the samples which a participant tested for but did not report a result, e.g. the participant reported a 'less-than' result (<*x*) when the assigned and spiked values were higher than their limit of reporting (LOR), or laboratories that didn't report any value.

Table 23 False Negatives

Lab. Code	Sample	Analyte	Assigned Value (mg/kg)	Spiked Value (mg/kg)	Result (mg/kg)*
2	S1	Cyhalothrin	0.0363	0.0458	NR
	S2	Glyphosate	0.208	0.250	NR
		Indoxacarb	2.70	3.01	NR
		Pyraclostrobin	1.18	1.20	NR
	S3	Acetamiprid	0.170	0.172	NR
		Carbendazim	0.404	0.496	NR
		Pyraclostrobin	0.084	0.0909	NR
		Triadimefon	1.87	2.01	NR
	S4	Acetamiprid	1.93	1.90	NR
		Azoxystrobin	5.33	6.05	NR
		Imidacloprid	2.71	2.79	NR
4	S1	Dimethoate	0.0534	0.0548	NR
7	S1	Cyhalothrin	0.0363	0.0458	NR
8	S1	Cyhalothrin	0.0363	0.0458	NR
9	S2	Glyphosate	0.208	0.250	NR
10	S1	Omethoate	2.30	2.28	NR
13	S1	Dimethoate	0.0534	0.0548	<0.01
		Omethoate	2.30	2.28	<0.5
15	S1	Cyhalothrin	0.0363	0.0458	NR
	S2	Indoxacarb	2.70	3.01	NR
	S3	Acetamiprid	0.170	0.172	NR
	S4	Cyfluthrin	0.208	0.220	NR
		Imidacloprid	2.71	2.79	NR

* NR results may or may not be false negatives, depending on the participant's actual LOR.

6.6 Reporting of Additional Analytes

Three laboratories reported at least one pesticide which was not spiked into the test samples. These results are presented in Table 24.

Table 24 Non-Spiked Analytes Reported by Participants

Lab. Code	Sample	Analyte	Result (mg/kg)	Uncertainty (mg/kg)	Recovery (%)
3	S1	p,p'-DDT	0.0064	0.0019	84.62
		beta-Endosulfan*	0.0051	0.0015	102.37
	S3	Dicofol	0.057	0.011	71.40
7	S1	p,p'-DDT	0.049	0.016	37.3
	S2	Terbacil**	0.047	NR	50
19	S2	Deltamethrin	0.04	0.01	NR

* Beta-Endosulfan is likely a minor (<1%) impurity in the endosulfan sulfate standard used to spike Sample S1.

** Laboratory 7 reported also detecting terbacil in the unspiked Sample S2 (0.033 mg/kg).

6.7 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, 13 different analytes were spiked into the samples for this study, with 3 analytes being spiked into multiple samples. 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 for participants' testing of the spiked pesticides is presented in Table 25.

Laboratories **2**, **3**, **5**, **7**, **16**, **20** and **21** reported that they tested for all spiked analytes. All participants tested for at least one of the spiked analytes, with the proportion of analytes being tested for by each participant ranging from 31% to 100%. Laboratory **4** tested for acetamiprid in Sample S3 (apple) but not in Sample S4 (orange).

Out of the spiked analytes in this study, dimethoate and endosulfan sulfate were tested for by the highest proportion of participants (95% for both). The proportion of participants testing for each analyte in this study ranged from 38% to 95%.

Table 25 Summary of Pesticides Analysed by Participants

Lab. Code / Analyte	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	Proportion of Participants (%)
Acetamiprid	✓	✓	✓	S3: ✓ S4: NT	✓	✓	✓	NT	✓	NT	NT	✓	NT	✓	✓	✓	NT	NT	NT	✓	✓	64
Azoxystrobin	✓	✓	✓	✓	✓	✓	✓	NT	✓	NT	NT	✓	✓	✓	✓	✓	✓	NT	NT	✓	✓	76
Carbendazim	✓	✓	✓	NT	✓	✓	✓	NT	✓	NT	NT	✓	✓	✓	✓	✓	NT	NT	NT	✓	✓	67
Cyfluthrin	✓	✓	✓	✓	✓	NT	✓	✓	✓	✓	✓	NT	NT	✓	✓	✓	✓	✓	✓	✓	✓	86
Cyhalothrin	✓	✓	✓	✓	✓	NT	✓	✓	✓	✓	✓	NT	✓	✓	✓	✓	✓	✓	✓	✓	✓	90
Dimethoate	✓	✓	✓	✓	✓	✓	✓	NT	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	95
Endosulfan sulfate	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	NT	✓	✓	✓	✓	✓	✓	✓	✓	✓	95
Glyphosate	NT	✓	✓	NT	✓	NT	✓	NT	✓	NT	NT	NT	NT	NT	NT	✓	NT	NT	NT	✓	✓	38
Imidacloprid	✓	✓	✓	NT	✓	✓	✓	NT	✓	NT	NT	NT	✓	✓	✓	✓	NT	NT	NT	✓	✓	62
Indoxacarb	✓	✓	✓	✓	✓	NT	✓	✓	NT	NT	NT	✓	✓	✓	✓	✓	✓	NT	NT	✓	✓	71
Omethoate	✓	✓	✓	NT	✓	NT	✓	NT	✓	✓	✓	NT	✓	✓	✓	✓	NT	NT	NT	✓	✓	67
Pyraclostrobin	✓	✓	✓	NT	✓	NT	✓	NT	✓	NT	NT	✓	NT	✓	NT	✓	NT	NT	NT	✓	✓	52
Triadimefon	✓	✓	✓	✓	✓	✓	✓	NT	✓	NT	NT	✓	✓	✓	✓	✓	✓	NT	NT	✓	✓	76
Proportion of Analytes (%)	92	100	100	57	100	54	100	31	92	38	38	54	69	92	85	100	54	31	31	100	100	72

6.8 Fitness for Purpose of Pesticide Results

The Australia New Zealand Food Standards (ANZFS) Code specifies the MRLs for various pesticides in different food products.[5] Laboratories should be able to identify if a sample is compliant or not with the relevant MRL. In particular, a laboratory should not classify a sample as compliant if the pesticide level is actually greater than the MRL, and vice versa. In this study, 13 analytes had assigned values (with uncertainty) that indicated either compliance or non-compliance with the relevant MRL. Figures 23 to 35 show comparisons of the assigned values (A.V.), participants' results, and the MRLs for these analytes. Where no numeric result or LOR was reported, and the participant did not report that the analyte was not tested for, these results been plotted as zero (0). In some cases, the MRL refers to the sum of a number of different permitted residues (Table 2), and not only the named analyte given here.

The majority of participants' results correctly identified compliance or non-compliance. Of the 184 results assessed, 132 (72%) gave the correct compliance status (inclusive of uncertainty), while 31 (17%) gave conditionally correct compliance statuses (i.e. the result gave the correct compliance status but the uncertainty spanned the MRL). Laboratories **14** (12), **12** (9), **11** (3) and **18** (2) returned the correct compliance status for all reported analytes assessed, while Laboratories **3** (13), **5** (13), **16** (13), **20** (13), **1** (12), **17** (5) and **19** (2) returned correct or conditionally correct compliance statuses for all reported analytes assessed.

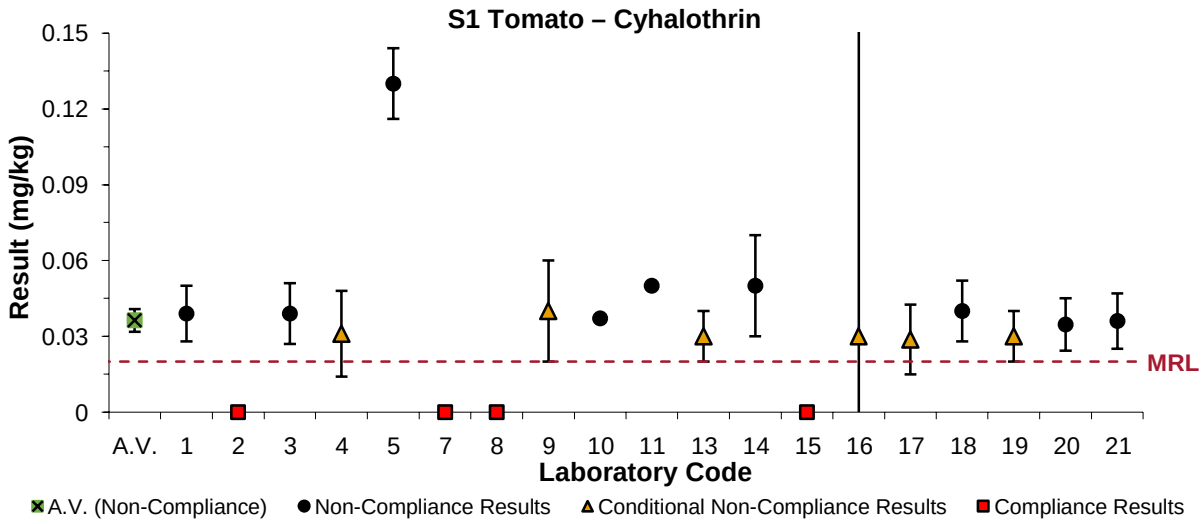


Figure 23 Sample S1 Tomato Cyhalothrin Assigned Value, Participant Results and MRL

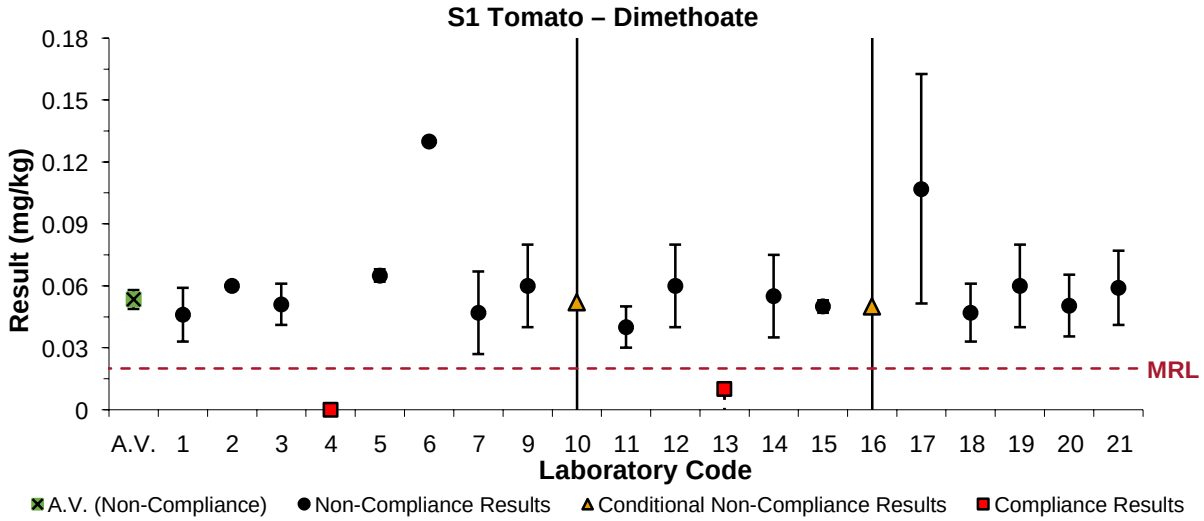


Figure 24 Sample S1 Tomato Dimethoate Assigned Value, Participant Results and MRL

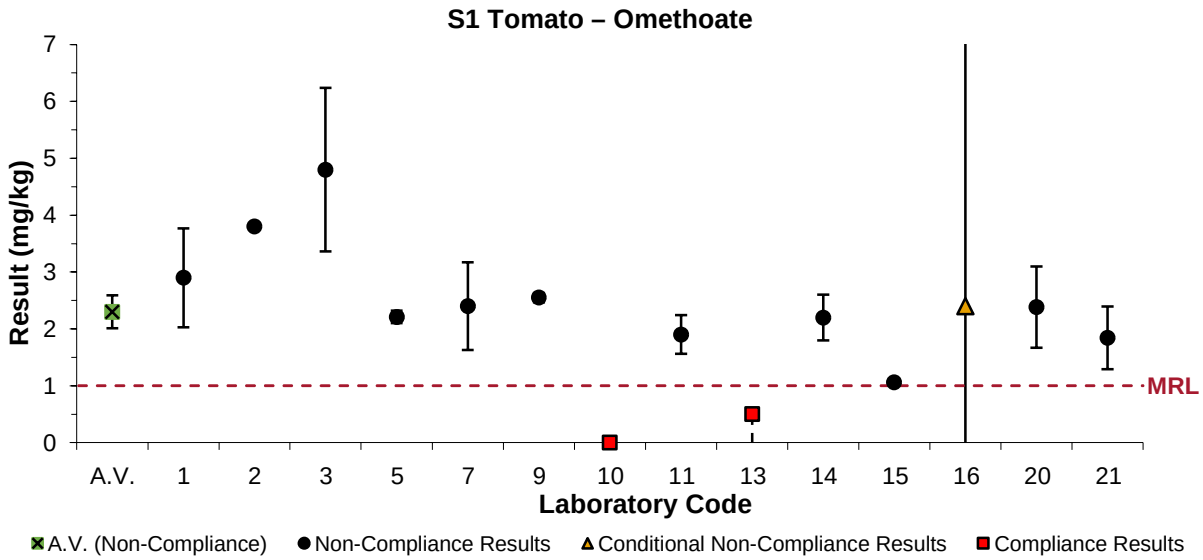


Figure 25 Sample S1 Tomato Omethoate Assigned Value, Participant Results and MRL

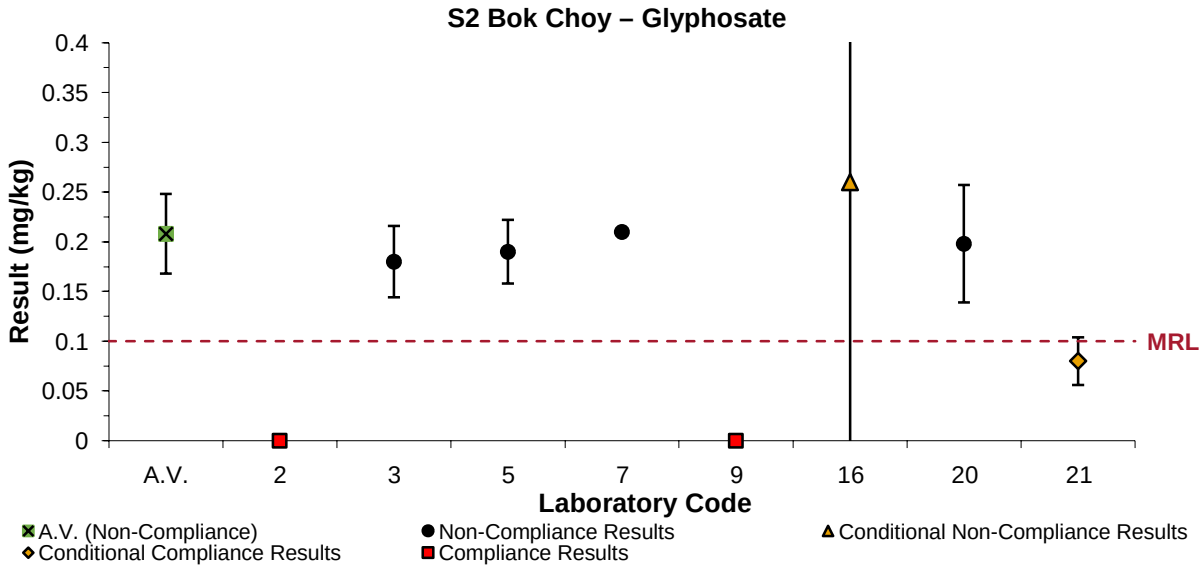


Figure 26 Sample S2 Bok Choy Glyphosate Assigned Value, Participant Results and MRL

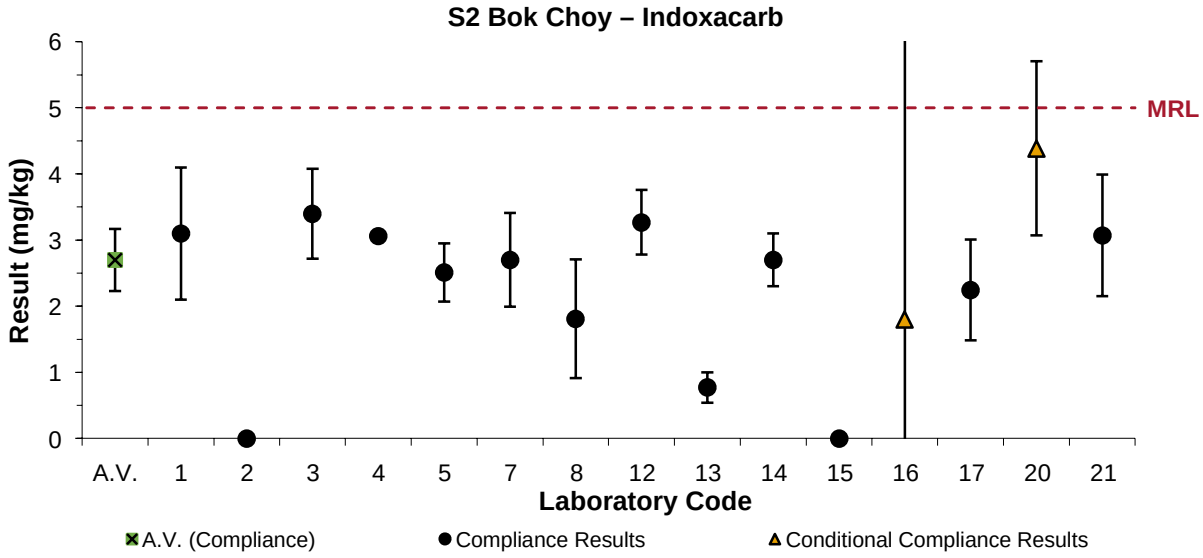


Figure 27 Sample S2 Bok Choy Indoxacarb Assigned Value, Participant Results and MRL

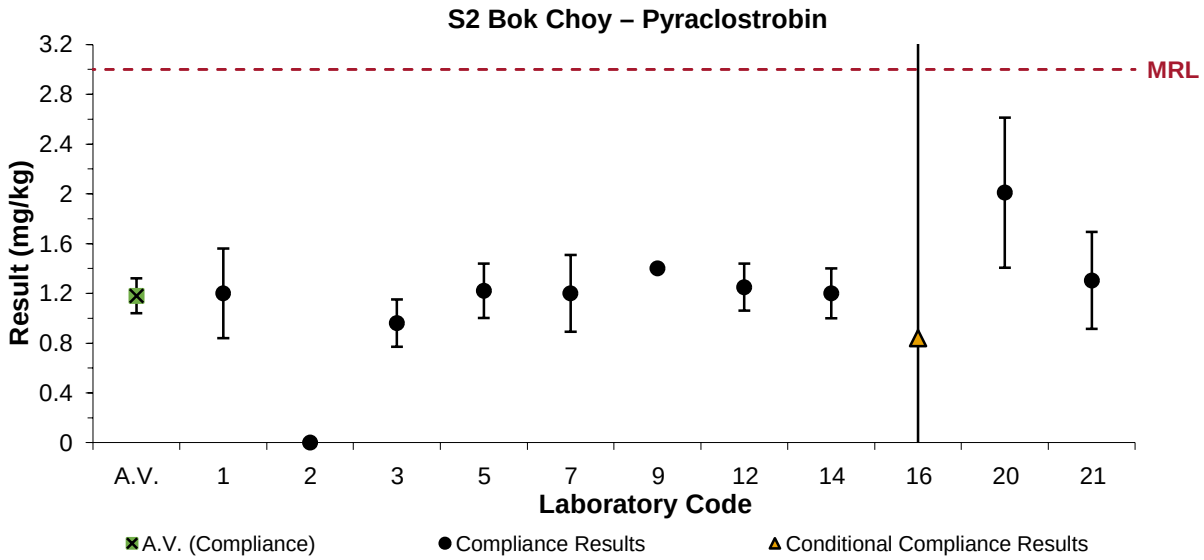


Figure 28 Sample S2 Bok Choy Pyraclostrobin Assigned Value, Participant Results and MRL

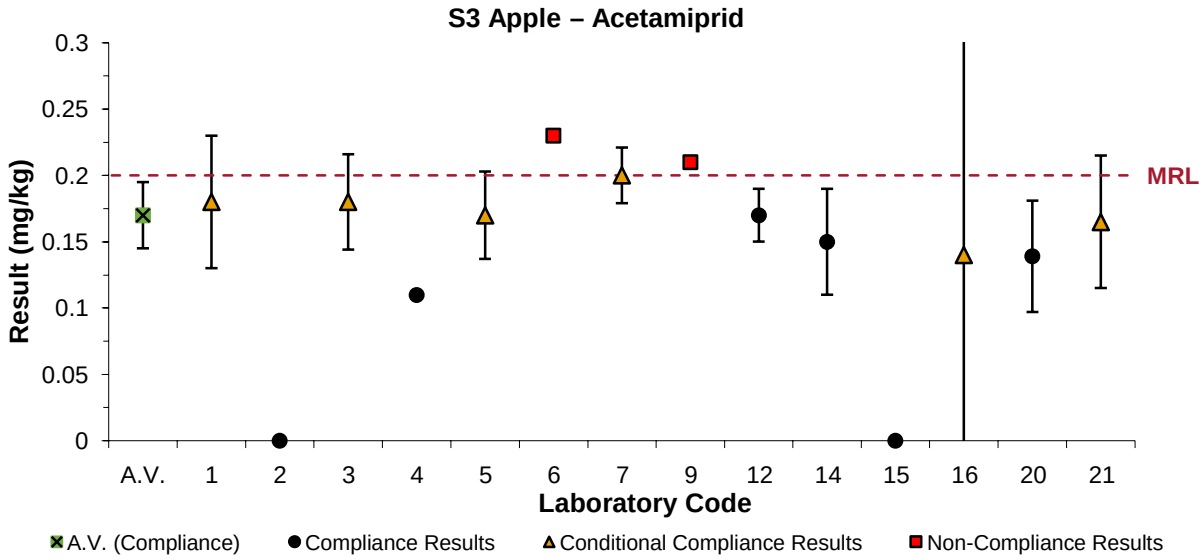


Figure 29 Sample S3 Apple Acetamiprid Assigned Value, Participant Results and MRL

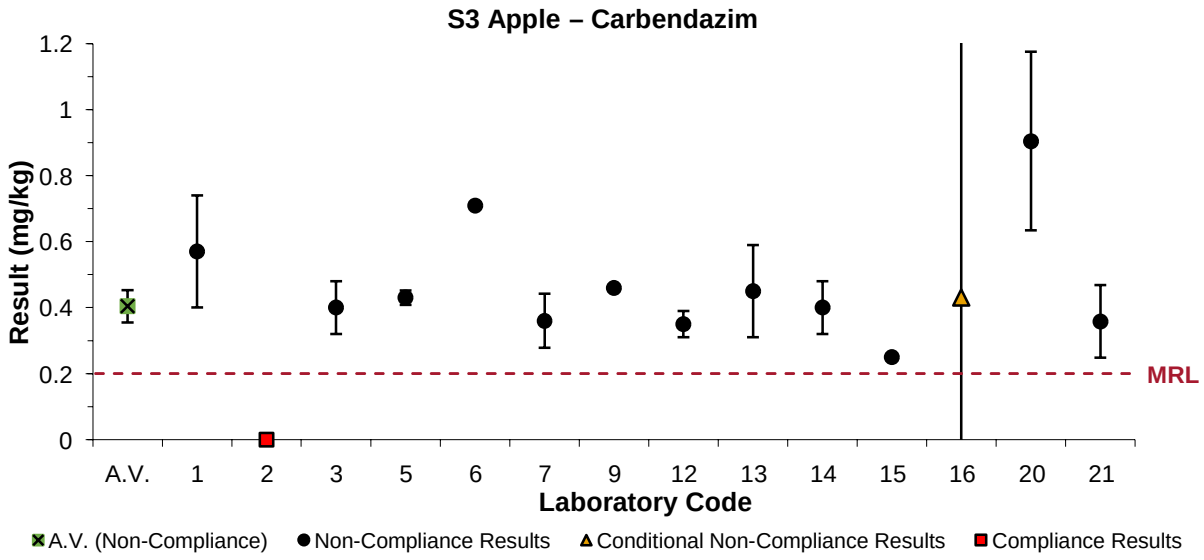


Figure 30 Sample S3 Apple Carbendazim Assigned Value, Participant Results and MRL

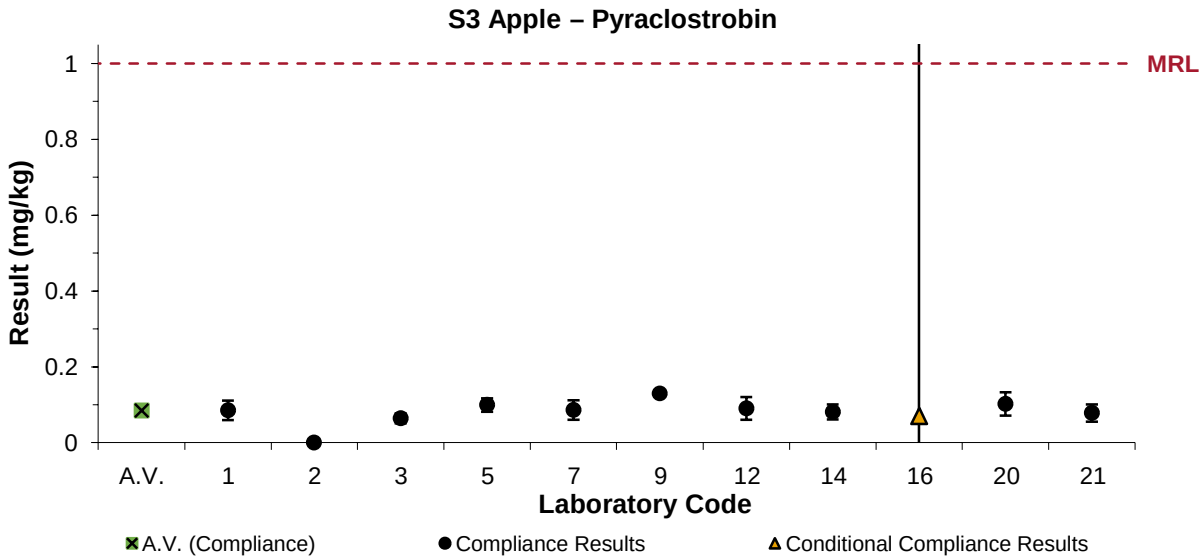


Figure 31 Sample S3 Apple Pyraclostrobin Assigned Value, Participant Results and MRL

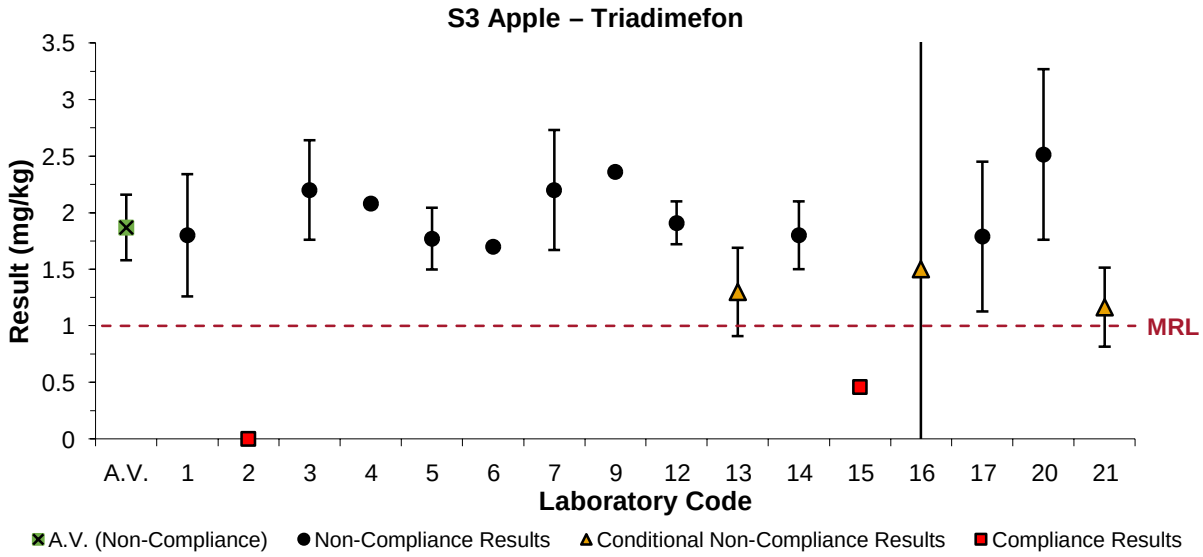


Figure 32 Sample S3 Apple Triadimefon Assigned Value, Participant Results and MRL

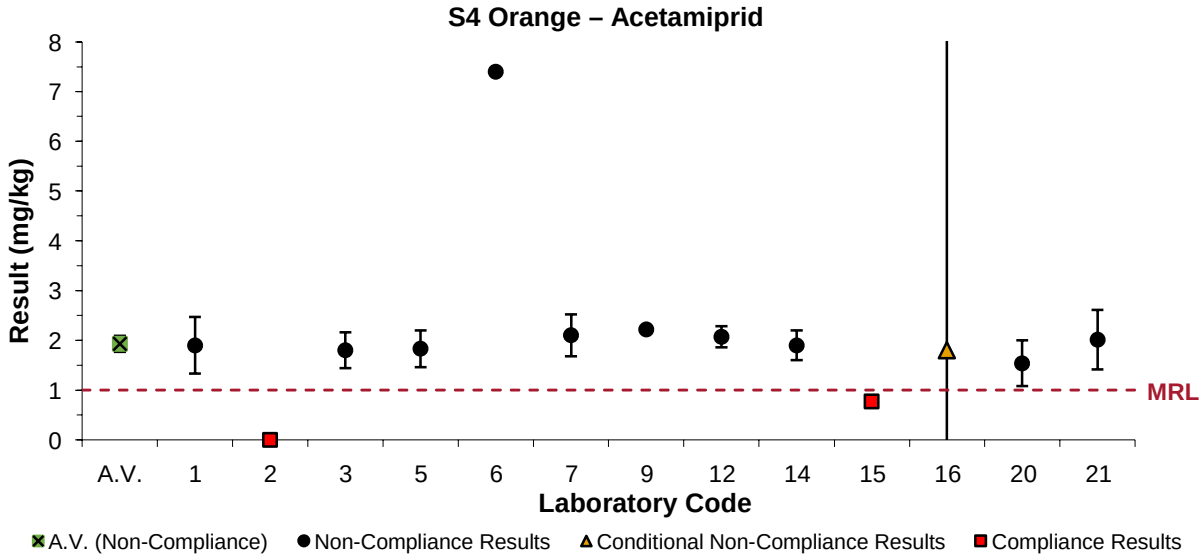


Figure 33 Sample S4 Orange Acetamiprid Assigned Value, Participant Results and MRL

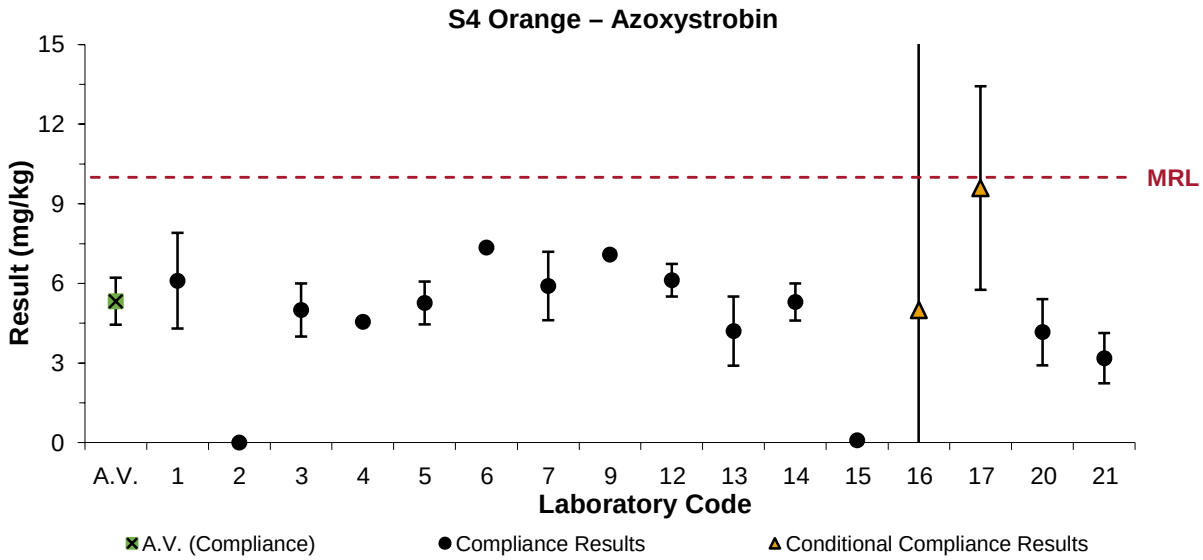


Figure 34 Sample S4 Orange Azoxystrobin Assigned Value, Participant Results and MRL

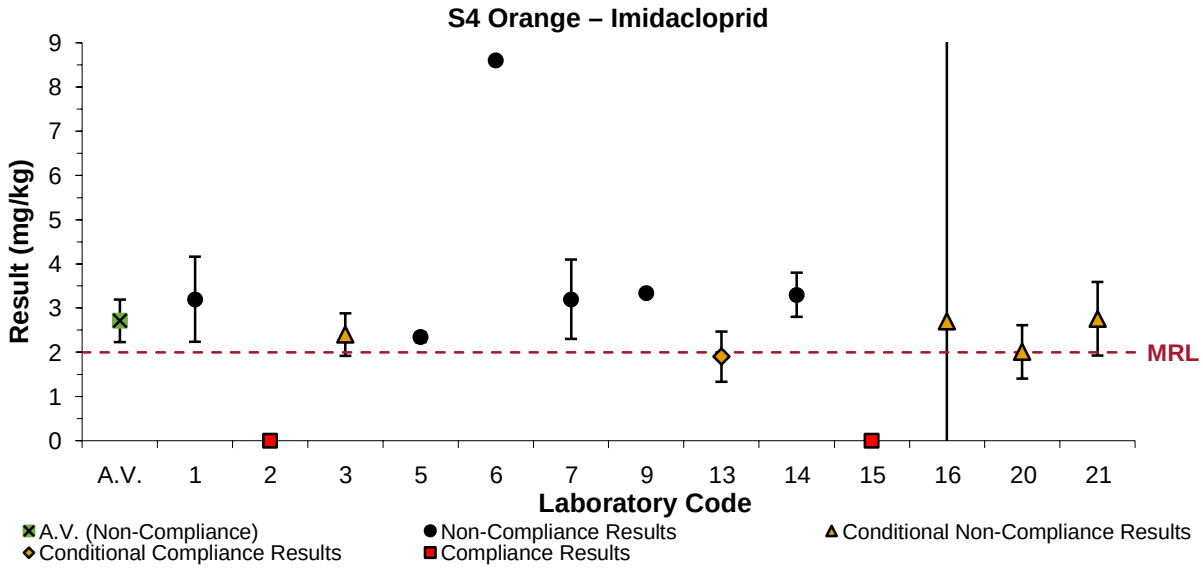
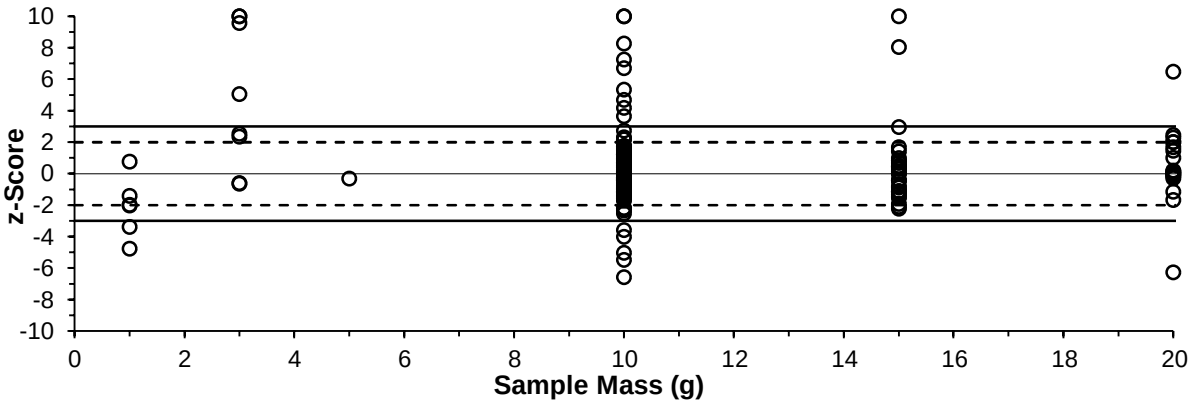


Figure 35 Sample S4 Orange Imidacloprid Assigned Value, Participant Results and MRL

6.9 Participants' Analytical Methods

A variety of analytical methods were used by participants in this study (Appendix 3).

Figure 36 shows z-scores obtained as compared to the sample masses used for analysis. Participants reported using sample sizes between 1 g and 20 g per analysis, with the majority of participants using around 10 g. Results in this study reported by participants using small sample sizes for analysis were in general mostly biased high or low. Caution should be exercised when a small sample size is taken for analysis as this may not be a suitable representation of the whole sample. There was no evident correlation between the results obtained and sample mass when larger sample masses (e.g. ≥ 10 g) were used.



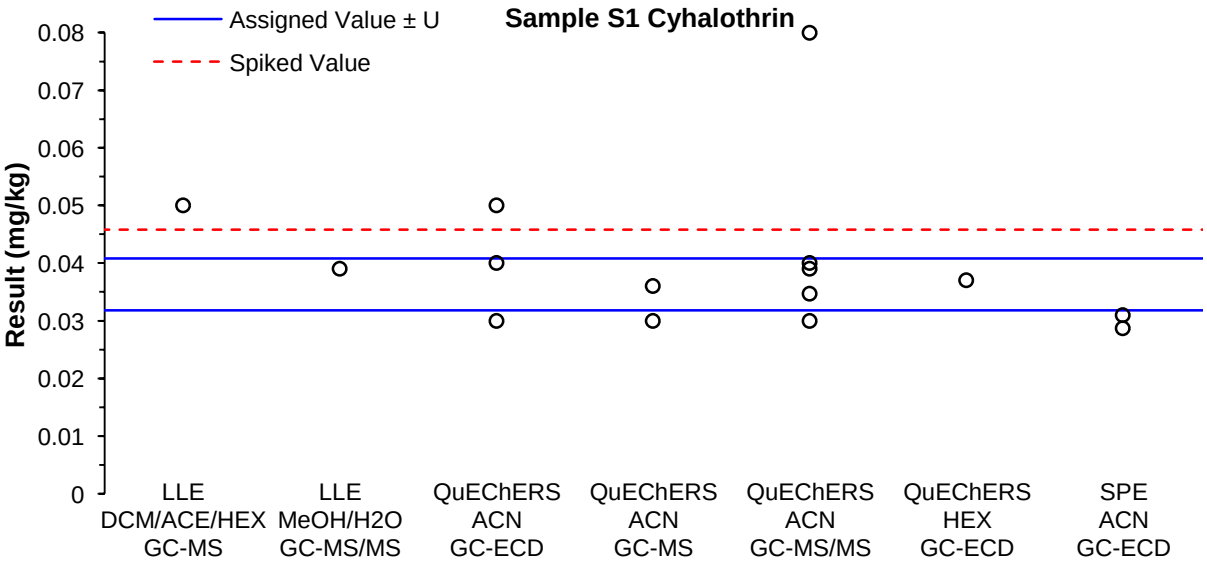
z-Scores greater than 10 have been plotted at 10.

Figure 36 z-Score vs Sample Mass Used for Analysis

Participants reported using a variety of extraction techniques including QuEChERS, liquid-liquid, and solid phase extraction, using acetonitrile, acetone, hexane, ethyl acetate, dichloromethane, methanol, water, acid(s), and combinations of these as the extraction solvent. The majority of participants used a clean-up step for analysis, with the use of PSA, C18, $MgSO_4$, carbon (e.g. Envicarb, GCB), silica gel (e.g. Florisil) and ChemElut being reported. A variety of instruments were used for analysis, including LC-MS(/MS), GC-MS(/MS) and GC-(ECD/FPD/NPD).

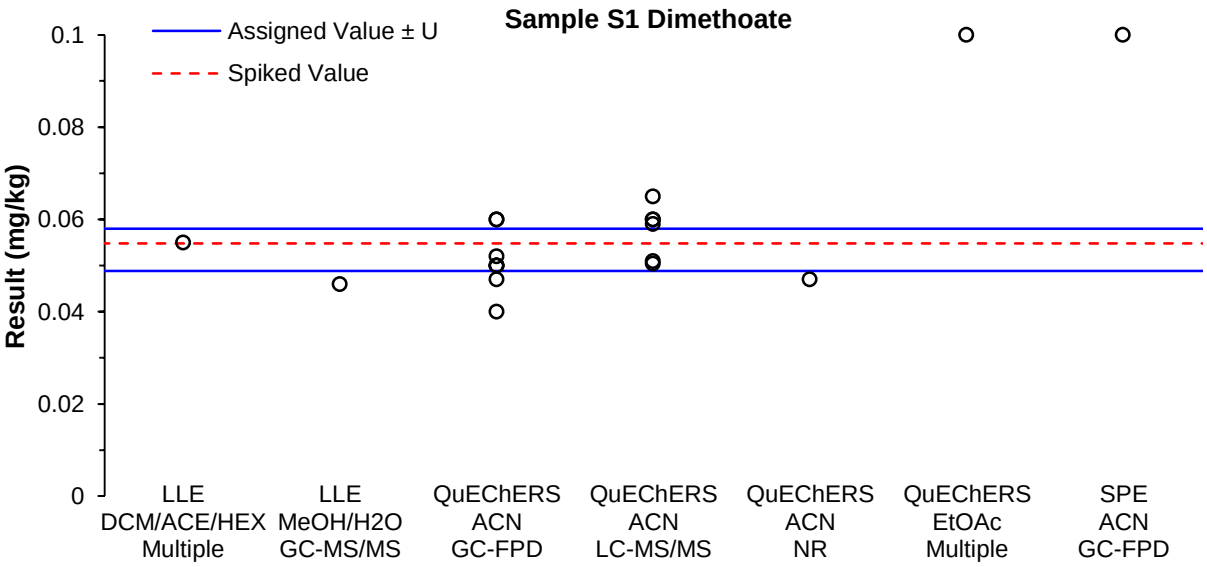
Participants used a wide variety of methodologies, and no trend with respect to results was observed. The most common methodology used was extraction using the QuEChERS procedure,[11] with acetonitrile as the extraction solvent and using LC-MS/MS for analysis.

Results compared to methodology used are presented in Figures 37 to 51. Solvent abbreviations used: ACE = Acetone; ACN = Acetonitrile; DCM = Dichloromethane; EtOAc = Ethyl Acetate; HEX = Hexane; MeOH = Methanol. Extraction method abbreviations used: LLE = Liquid-Liquid Extraction; SPE = Solid-Phase Extraction; QuEChERS = Quick, Easy, Cheap, Effective, Rugged and Safe Extraction. Instrument abbreviations used: GC = Gas Chromatography; HPLC = High Performance Liquid Chromatography; LC = Liquid Chromatography; ECD = Electron Capture Detector; FPD = Flame Photometric Detector; MS = Mass Spectrometry; MS/MS = Tandem Mass Spectrometry. Where a participant did not report the method, this has been labelled as “NR”.



Results greater than 0.08 mg/kg have been plotted at 0.08 mg/kg.

Figure 37 Sample S1 Tomato Cyhalothrin Result vs Methodology



Results greater than 0.1 mg/kg have been plotted at 0.1 mg/kg.

Figure 38 Sample S1 Tomato Dimethoate Result vs Methodology

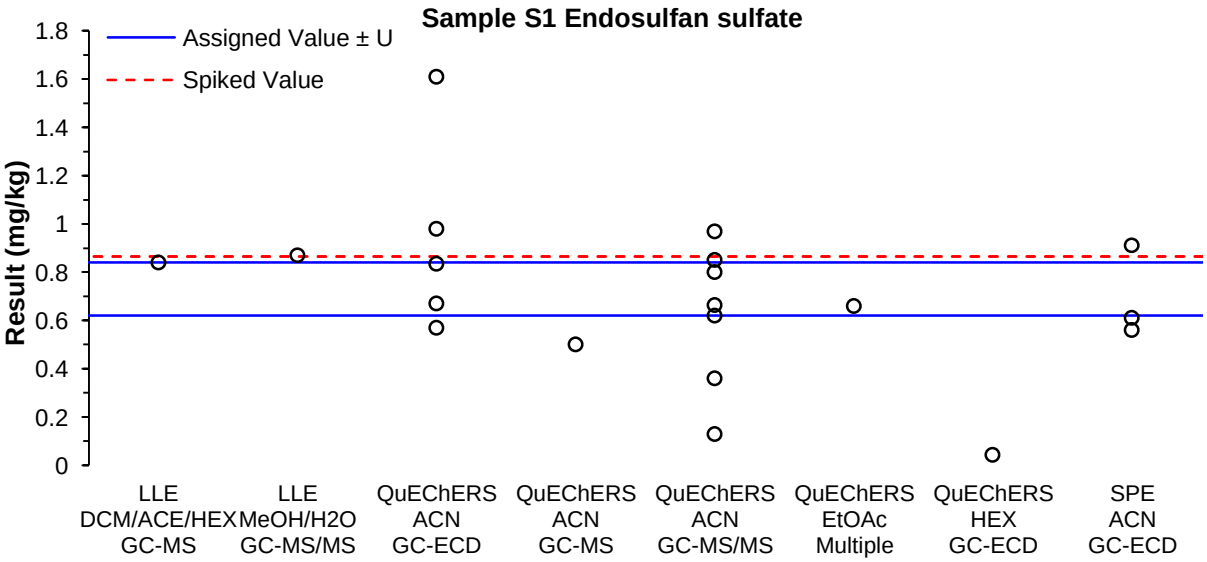


Figure 39 Sample S1 Tomato Endosulfan Sulfate Result vs Methodology

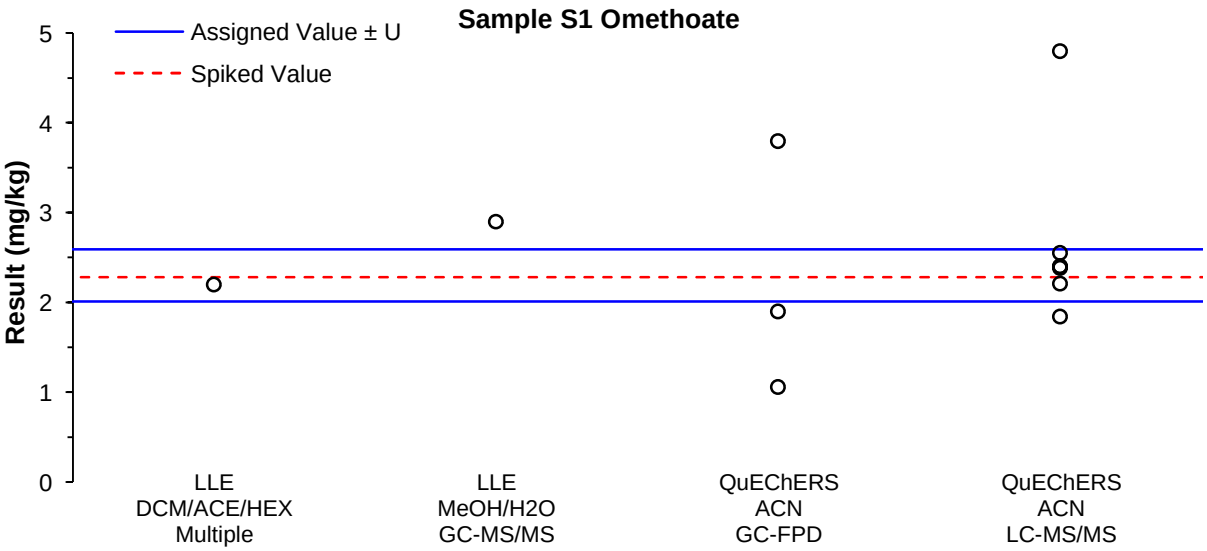


Figure 40 Sample S1 Tomato Omethoate Result vs Methodology

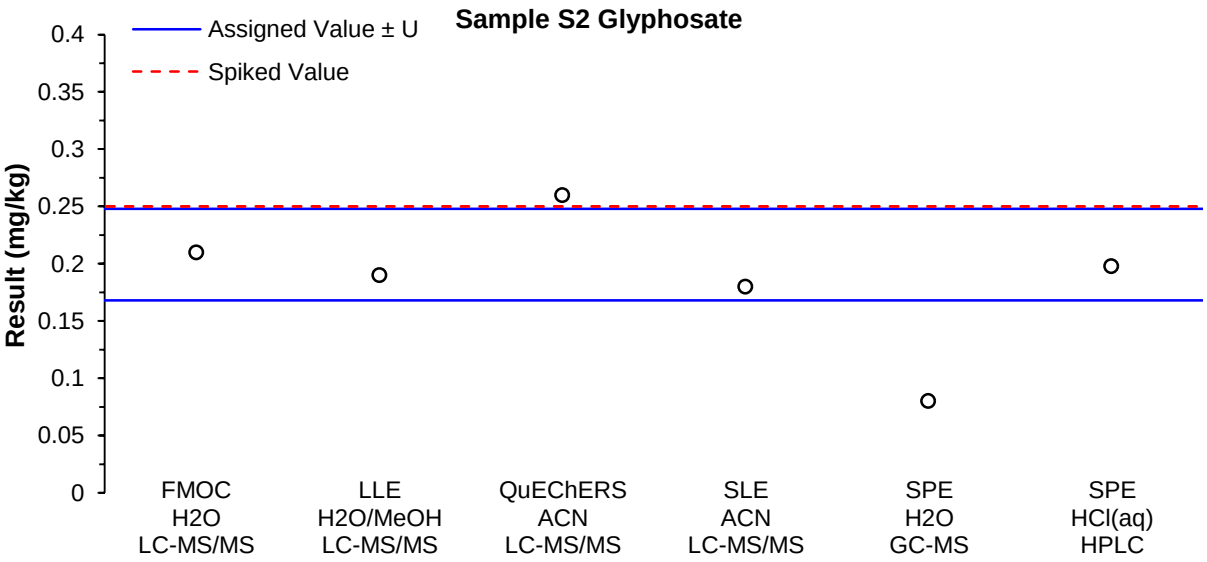


Figure 41 Sample S2 Bok Choy Glyphosate Result vs Methodology

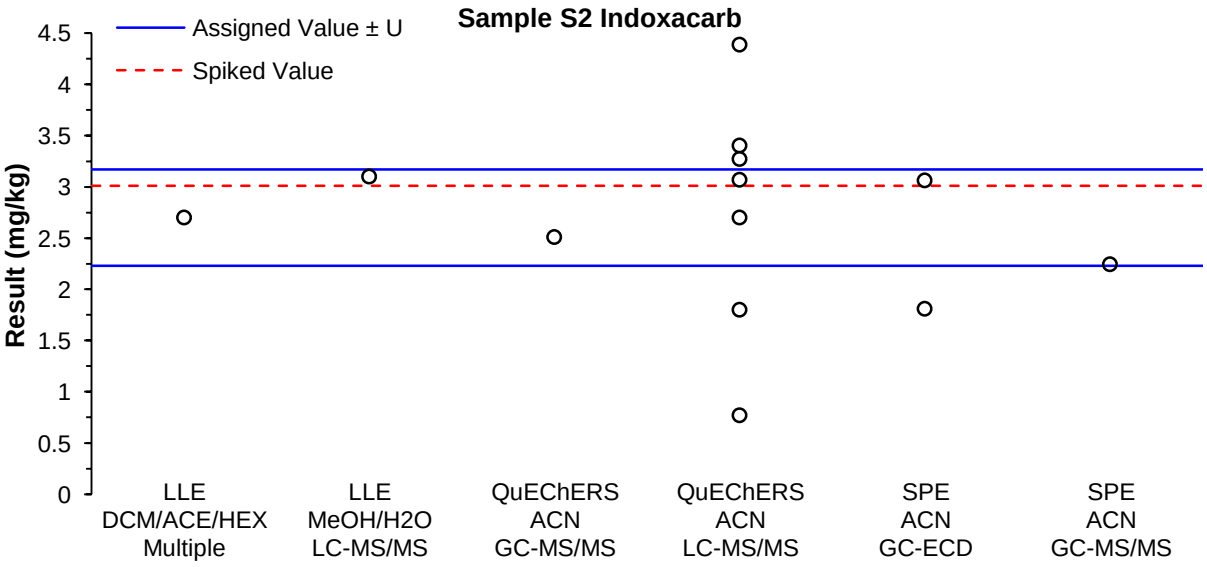


Figure 42 Sample S2 Bok Choy Indoxacarb Result vs Methodology

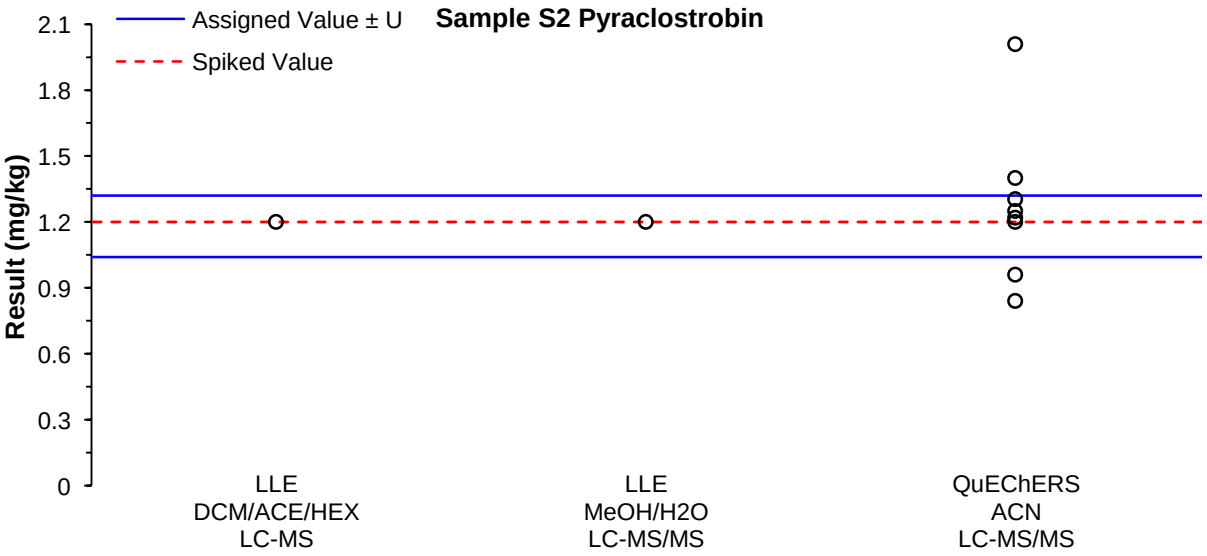


Figure 43 Sample S2 Bok Choy Pyraclostrobin Result vs Methodology

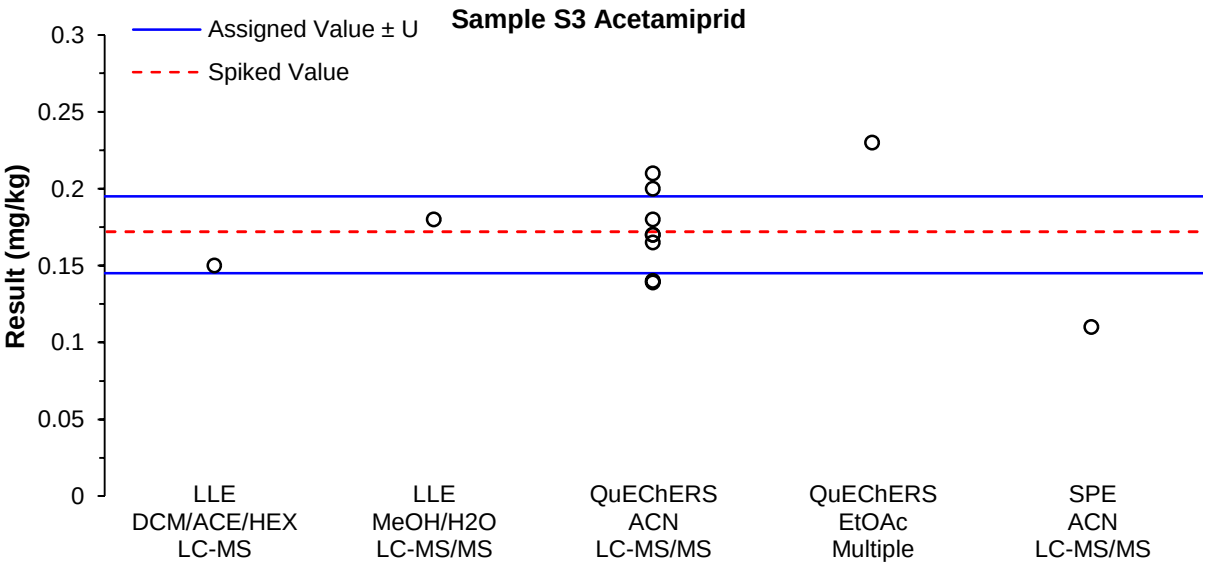


Figure 44 Sample S3 Apple Acetamiprid Result vs Methodology

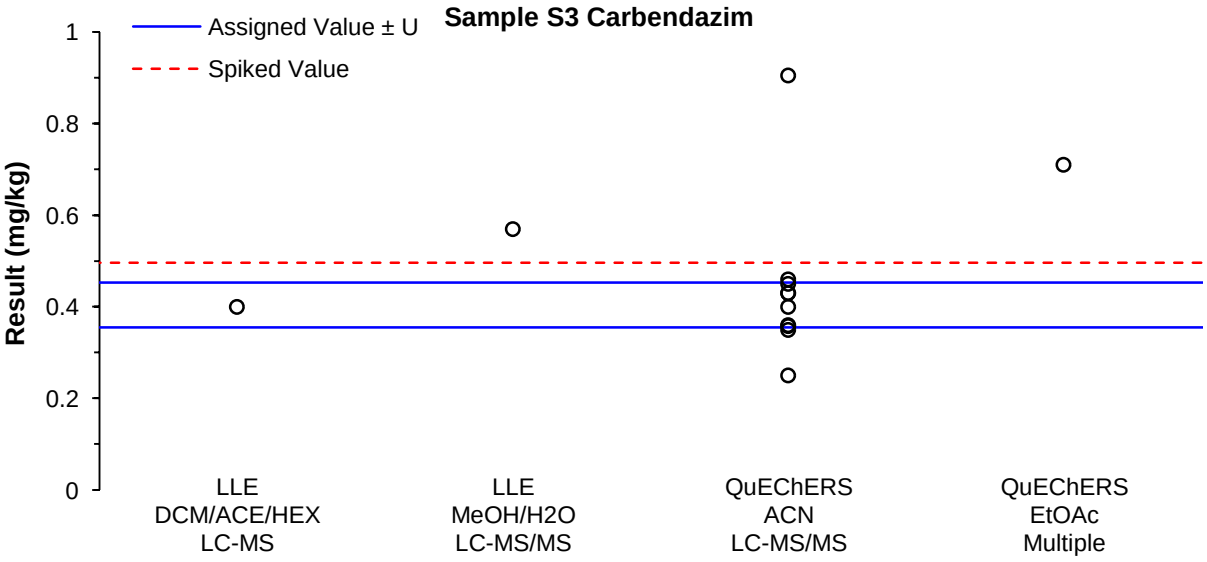


Figure 45 Sample S3 Apple Carbendazim Result vs Methodology

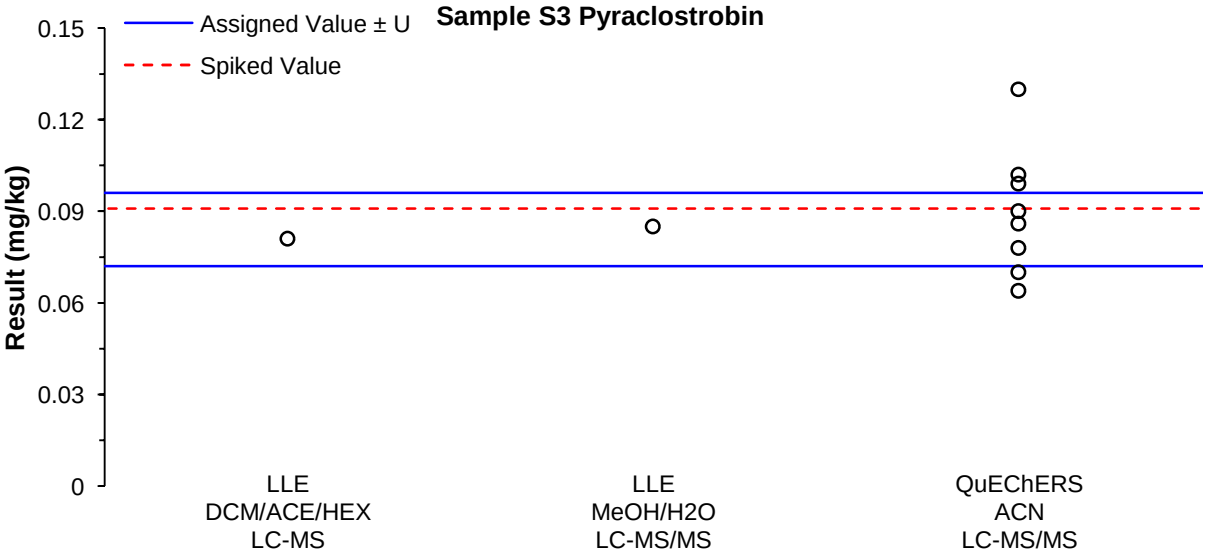


Figure 46 Sample S3 Apple Pyraclostrobin Result vs Methodology

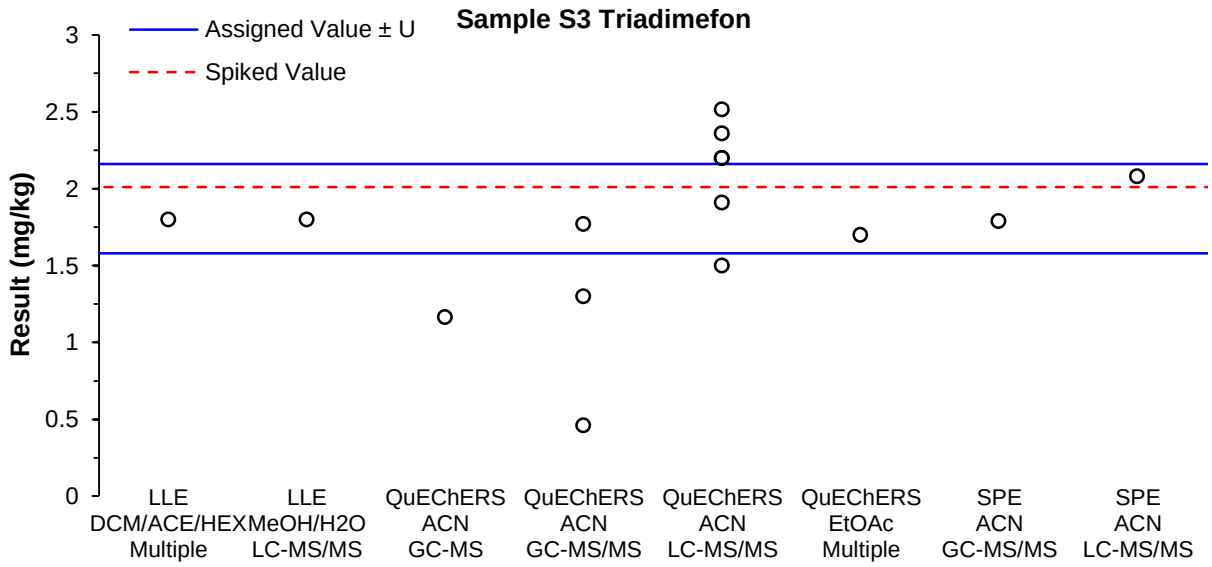
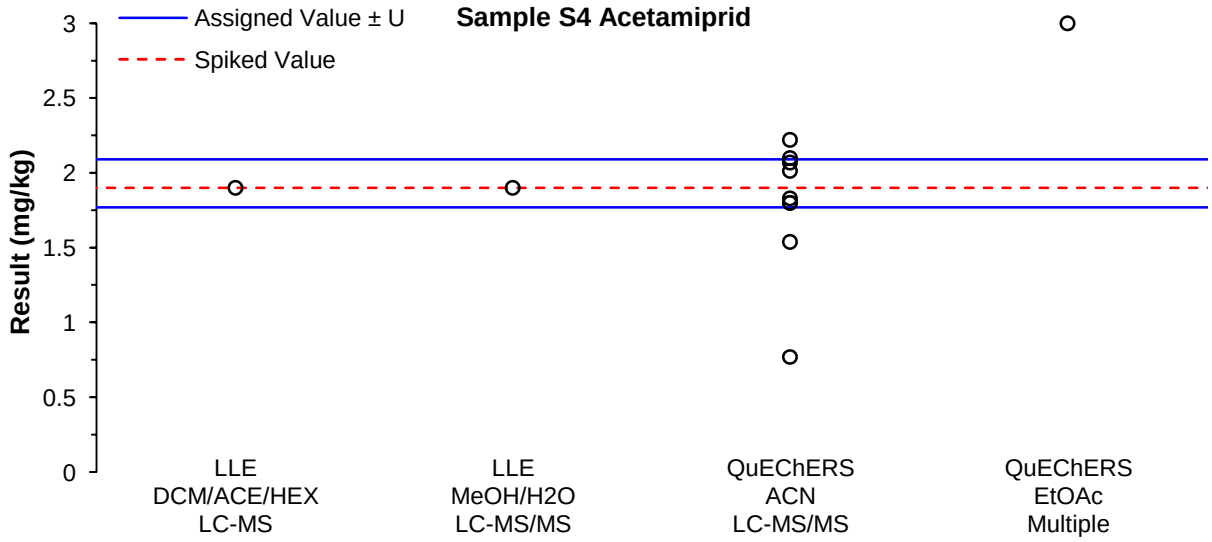


Figure 47 Sample S3 Apple Triadimefon Result vs Methodology



Results greater than 3 mg/kg have been plotted at 3 mg/kg.

Figure 48 Sample S4 Orange Acetamiprid Result vs Methodology

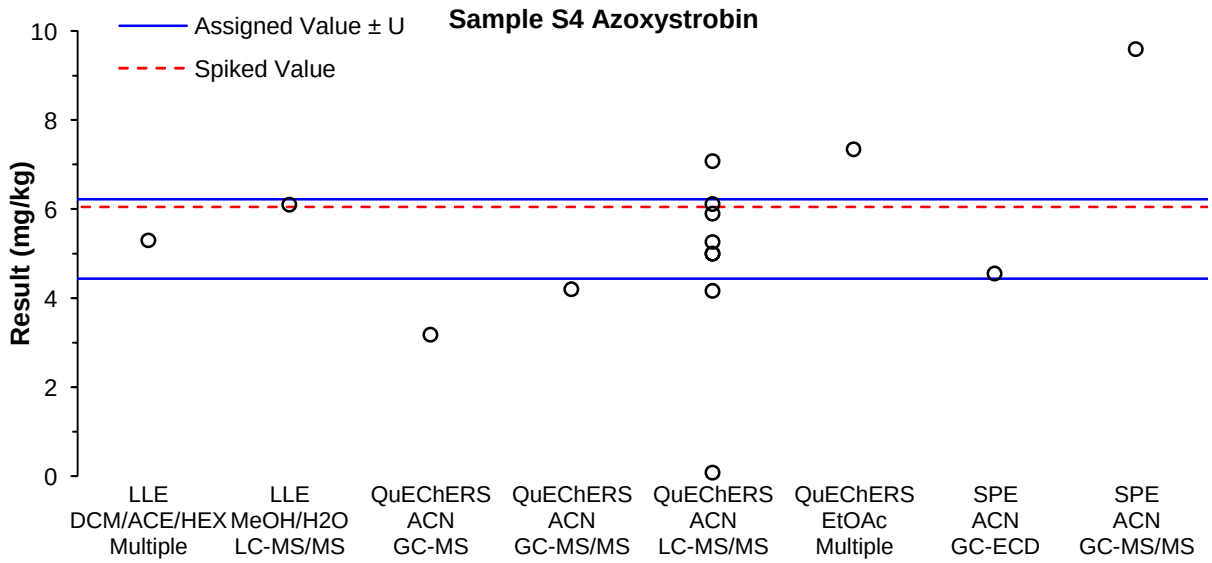
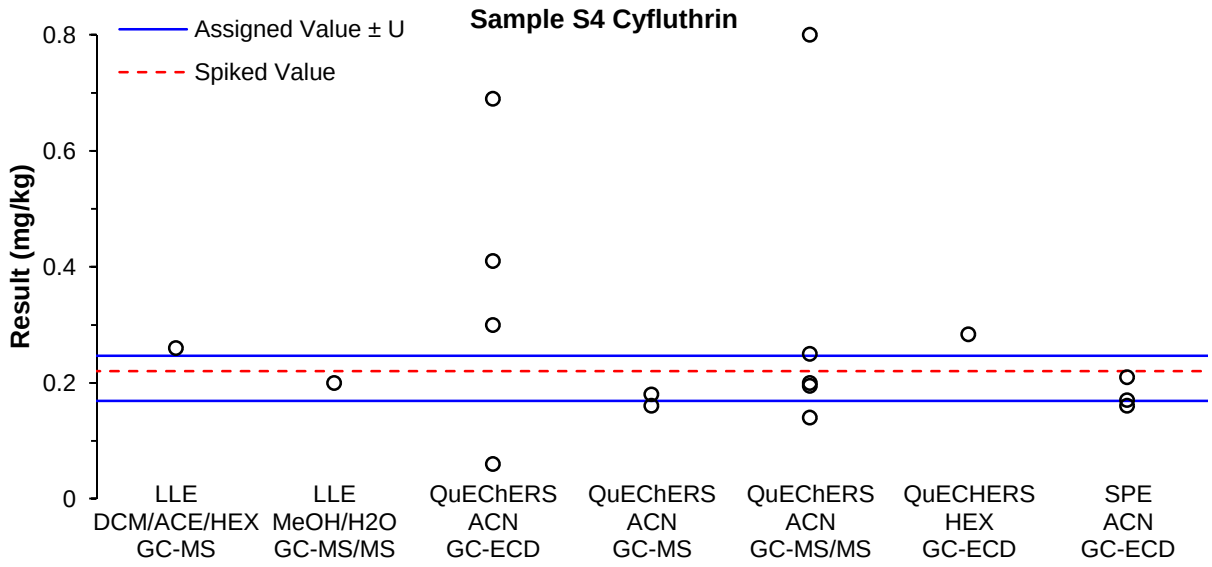
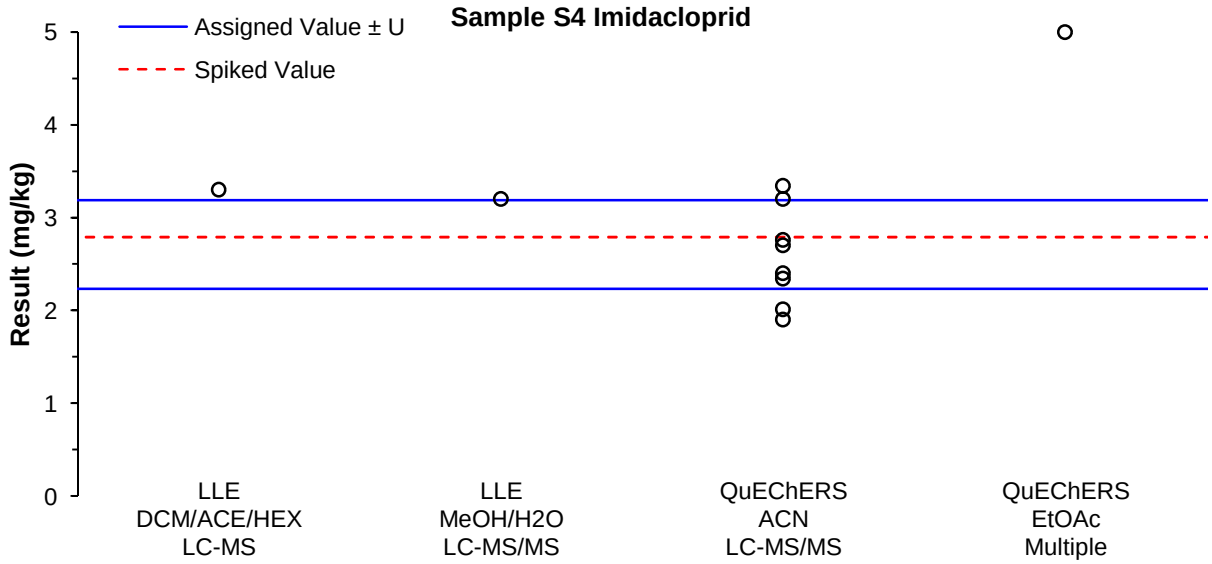


Figure 49 Sample S4 Orange Azoxystrobin Result vs Methodology



Results greater than 0.8 mg/kg have been plotted at 0.8 mg/kg.

Figure 50 Sample S4 Orange Cyfluthrin Result vs Methodology



Results greater than 5 mg/kg have been plotted at 5 mg/kg.

Figure 51 Sample S4 Orange Imidacloprid Result 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**, **3**, **4**, **5**, **7**, **8**, **9**, **10**, **11**, **12**, **14**, **15**, **16**, **17**, **18**, **20** and **21** reported recoveries for at least one analyte of interest in this study, and the recoveries reported were within the range of 42% to 158%. Laboratories **1**, **9**, **17** and **18** reported that they corrected results for recovery.

Participants were also provided with blank samples to be analysed if part of their routine procedures. Laboratories **1**, **3**, **4**, **6**, **7**, **8**, **9**, **10**, **12**, **13**, **14**, **15**, **16**, **17**, **18** and **20** reported analysing the blank samples.

6.10 Certified Reference Materials (CRM)

Participants were requested to report whether certified standards or matrix reference materials had been used as part of the quality assurance for their analysis. Thirteen participants reported using certified standards. The following were listed:

- Dr. Ehrenstorfer
- AccuStandards
- ISO 17034 certified standards
- Certified or reference compounds from other suppliers
- Laboratory control samples

These materials may or may not meet the internationally recognised definition of a Certified Reference Material:

> *'**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]

6.11 Effect of Sample Matrix

The samples in this study were purees of tomatoes (S1), bok choy (S2), apples (S3) and oranges (S4). A summary of the results reported and z-scores obtained for each matrix is presented in Table 26.

The proportion of results reported relative to expected number of results ranged from 56% to 77%, and the proportion of satisfactory z-scores obtained ranged from 73% to 80%. Sample S1 tomato had both the highest proportion of results reported and satisfactory z-scores.

Table 26 Result Comparison by Matrix

Sample	Matrix	Expected Number of Results	Numeric Results Reported	z-Scores	Satisfactory z-Scores
S1	Tomato	84	65 (77%)	65	52 (80%)
S2	Bok Choy	84	47 (56%)	29	23 (79%)
S3	Apple	84	50 (60%)	50	39 (78%)
S4	Orange	84	55 (65%)	55	40 (73%)

6.12 Summary of Participants' Results and Performances

Summaries of participants' results and performances for scored analytes in this PT study are presented in Tables 27 and 28, and Figure 52.

Table 27 Summary of Participants' Sample S1 and S2 Results*

Lab. Code	S1 Cyhalothrin	S1 Dimethoate	S1 Endosulfan sulfate	S1 Omethoate	S2 Glyphosate	S2 Indoxacarb	S2 Pyraclostrobin
A.V.	0.0363	0.0534	0.73	2.30	0.208	2.70	1.18
S.V.	0.0458	0.0548	0.865	2.28	0.250	3.01	1.20
1	0.039	0.046	0.87	2.9	NT	3.1	1.2
2	NR	0.06	0.67	3.80	NR	NR	NR
3	0.039	0.051	0.62	4.8	0.18	3.4	0.96
4	0.031	NR	0.56	NT	NT	3.06	NT
5	0.13	0.065	0.97	2.21	0.19	2.51	1.22
6	NT	0.13	0.66	NT	NT	NT	NT
7	NR	0.047	0.85	2.4	0.21	2.7	1.2
8	NR	NT	0.61	NT	NT	1.81	NT
9	0.04	0.06	0.8	2.55	NR	NT	1.4
10	0.037	0.052	0.043	NR	NT	NT	NT
11	0.05	0.04	0.98	1.9	NT	NT	NT
12	NT	0.06	NT	NT	NT	3.27	1.25
13	0.03	<0.01	0.36	<0.5	NT	0.77	NT
14	0.050	0.055	0.84	2.2	NT	2.7	1.2
15	NR	0.05	0.13	1.06	NT	NR	NT
16	0.03	0.05	0.57	2.4	0.26	1.8	0.84
17	0.0287	0.107	0.912	NT	NT	2.244	NT
18	0.040	0.047	1.61	NT	NT	NT	NT
19	0.03	0.06	0.5	NT	NT	NT	NT
20	0.0347	0.0504	0.663	2.384	0.198	4.388	2.010
21	0.036	0.059	0.835	1.843	0.08	3.069	1.304

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

Table 28 Summary of Participants' Sample S3 and S4 Results*

Lab. Code	S3 Acetamiprid	S3 Carbendazim	S3 Pyraclostrobin	S3 Triadimefon	S4 Acetamiprid	S4 Azoxystrobin	S4 Cyfluthrin	S4 Imidacloprid
A.V.	0.170	0.404	0.084	1.87	1.93	5.33	0.208	2.71
S.V.	0.172	0.496	0.0909	2.01	1.90	6.05	0.220	2.79
1	0.18	0.57	0.085	1.8	1.9	6.1	0.20	3.2
2	NR	NR	NR	NR	NR	NR	0.06	NR
3	0.18	0.4	0.064	2.2	1.8	5.0	0.14	2.4
4	0.11	NT	NT	2.08	NT	4.55	0.21	NT
5	0.17	0.43	0.099	1.77	1.83	5.26	0.25	2.34
6	0.23	0.71	NT	1.7	7.4	7.35	NT	8.6
7	0.2	0.36	0.086	2.2	2.1	5.9	0.20	3.2
8	NT	NT	NT	NT	NT	NT	0.16	NT
9	0.21	0.46	0.13	2.36	2.22	7.08	1.37	3.34
10	NT	NT	NT	NT	NT	NT	0.284	NT
11	NT	NT	NT	NT	NT	NT	0.41	NT
12	0.17	0.35	0.09	1.91	2.07	6.12	NT	NT
13	NT	0.45	NT	1.3	NT	4.2	NT	1.9
14	0.15	0.40	0.081	1.8	1.9	5.3	0.26	3.3
15	NR	0.25	NT	0.46	0.77	0.08	NR	NR
16	0.14	0.43	0.07	1.5	1.8	5.0	0.30	2.7
17	NT	NT	NT	1.789	NT	9.595	0.17	NT
18	NT	NT	NT	NT	NT	NT	0.69	NT
19	NT	NT	NT	NT	NT	NT	0.16	NT
20	0.139	0.905	0.102	2.515	1.540	4.165	0.195	2.010
21	0.165	0.358	0.078	1.165	2.014	3.181	0.18	2.758

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

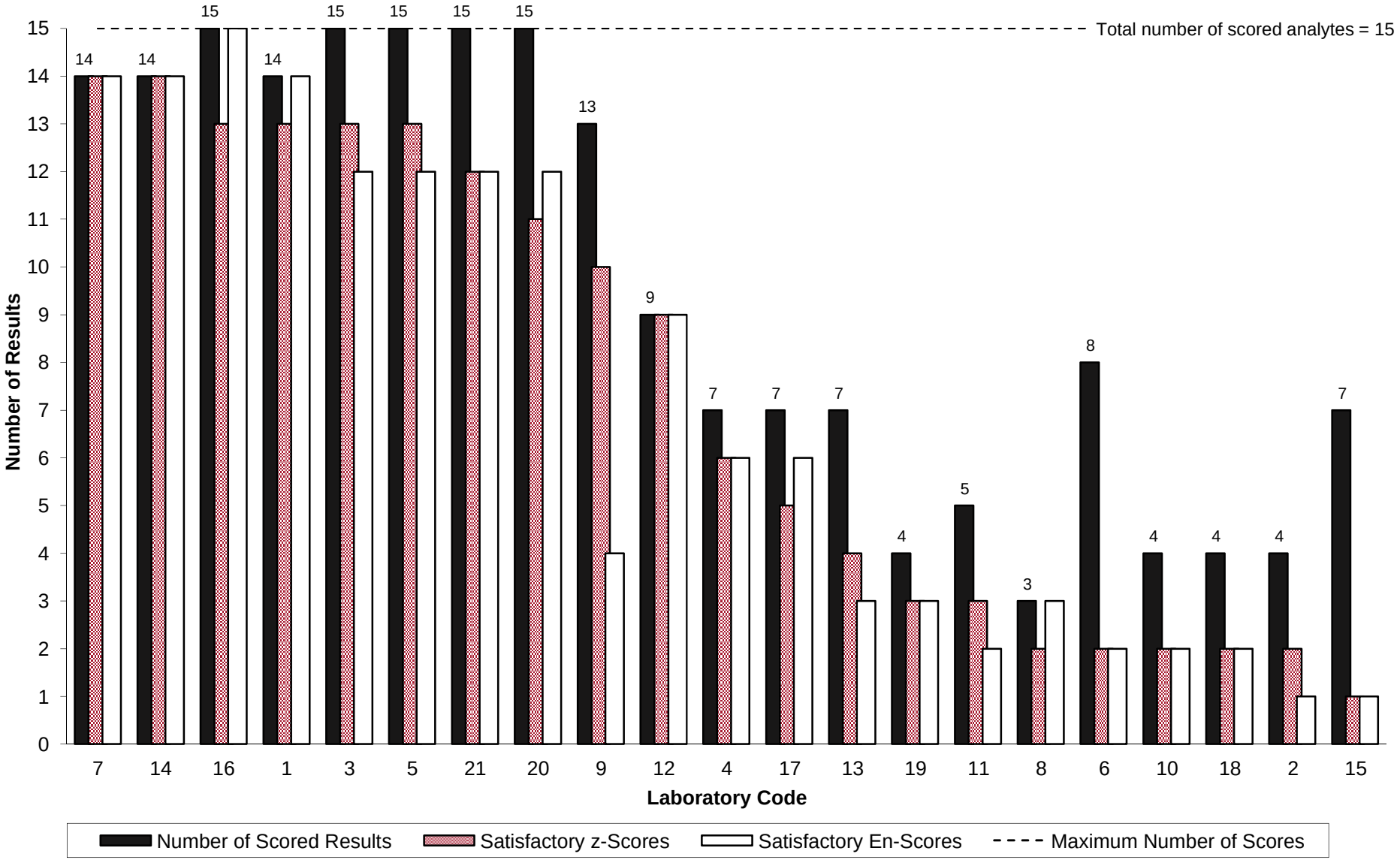


Figure 52 Summary of Participants' Performance

6.13 Comparison with Previous Pesticides in Fruit & Vegetables PT Studies

A summary of participation and reported results rates in Pesticides in Fruit & Vegetables PT studies over the last 10 studies (2014 to 2021) is presented in Figure 53. While the number of spiked analytes per study has increased, the numeric results reported by participants has remained fairly steady over this period.

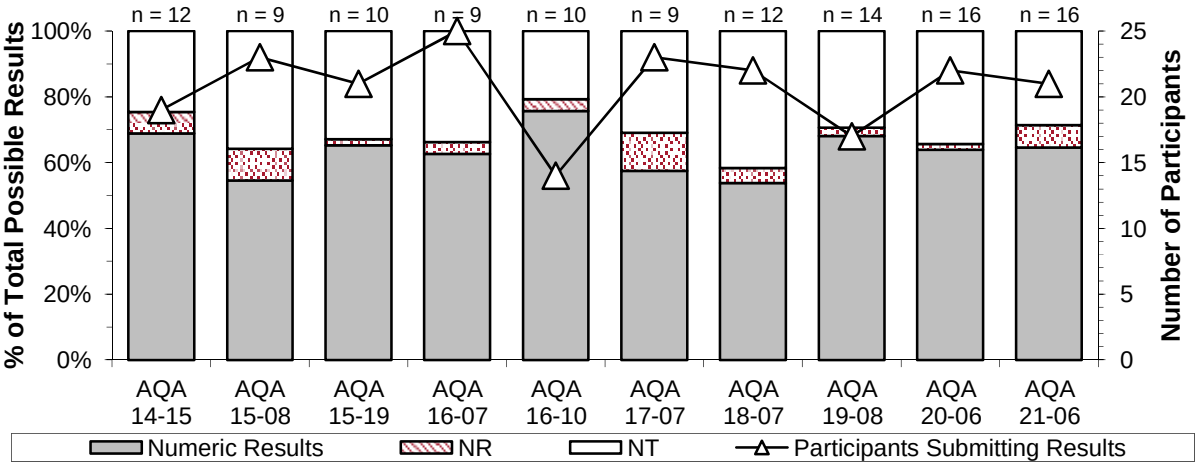


Figure 53 Summary of Participation and Reported Results in Pesticides in Fruit & Vegetables PT Studies (n = number of spiked analytes)

A summary of the satisfactory performance (presented as a percentage of the total number of scores for each study) in Pesticides in Fruit & Vegetables PT studies over the last 10 studies (2014 to 2021) is presented in Figure 54. 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 scores was 76% for z-scores and 69% for E_n-scores. While each PT study has a different sample set and a different group of participants, taken as a group, the performance over this period has been improving.

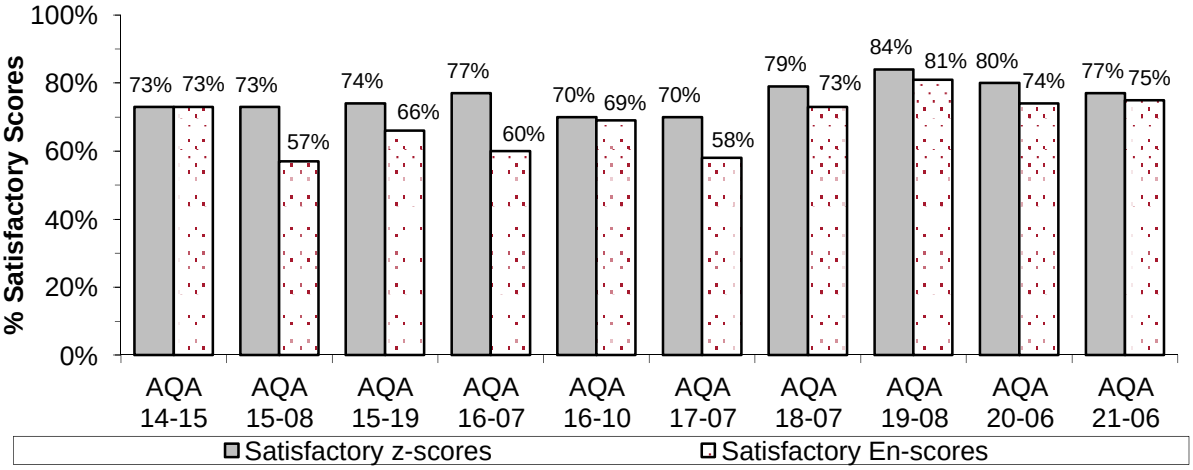


Figure 54 Summary of Participants' Performance in Pesticides in Fruit & Vegetables PT Studies

Individual performance history reports are emailed to participants at the end of each PT study; the consideration of z-scores over time provides much more useful information than a single z-score. Over time, laboratories should expect at least 95% of their z-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.

7 REFERENCES

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[12] BIPM, JCGM 200:2012, *International vocabulary of metrology – Basic and general concepts and associated terms (VIM)*, 3rd edition, viewed August 2021, <https://www.bipm.org/utils/common/documents/jcgm/JCGM_200_2012.pdf>.

[13] Federal Register of Legislation, 2021, *Australia New Zealand Food Standards Code – Schedule 22 – Foods and classes of foods*, viewed August 2021, <https://www.legislation.gov.au/Details/F2021C00757>.

APPENDIX 1 – SAMPLE PREPARATION

Test Sample Preparation

Tomatoes, bok choy, apples and oranges were bought from a Sydney organic fruit and vegetable wholesaler. The portion of the fruit prepared was in accordance with the Australian New Zealand Food Standards Code – Schedule 22 – Foods and classes of foods.[13]

Preparation of Sample S1 (Tomato)

The tomatoes were rinsed using tap water and allowed to air dry. Whole tomatoes, including the peel, was chopped, pureed and passed through an 850 µm sieve. The sieved puree was continuously stirred while 50 aliquots of at least 100 g were dispensed into 200 mL amber bottles to provide unspiked samples. The remaining puree was spiked with aliquots of each pesticide standard solution. The spiked puree was stirred for at least two hours and bottled. Each bottle was then labelled, shrink-wrapped in plastic film and placed in a freezer.

Preparation of Sample S2 (Bok Choy)

The bok choy was rinsed using tap water and allowed to air dry. It was then chopped, placed in a stainless steel drum, pureed with a stick mixer and passed through an 850 µm sieve. The puree was continuously stirred while 50 aliquots of at least 100 g were dispensed into 200 mL amber bottles to provide unspiked samples. The remaining puree was spiked with aliquots of each pesticide standard solution. The spiked puree was stirred for at least two hours and bottled. Each bottle was then labelled, shrink-wrapped in plastic film and placed in a freezer.

Preparation of Sample S3 (Apple)

The apples were rinsed with tap water and allowed to dry. Whole apples, excluding stems and seeds, were chopped, placed into a stainless steel drum and blended using a stick mixer to form a puree which was passed through an 850 µm sieve. The puree was continuously stirred while 50 aliquots of at least 100 g were dispensed into 200 mL amber bottles to provide unspiked samples. The remaining puree was spiked with aliquots of each pesticide standard solution. The spiked puree was stirred for at least two hours and bottled. Each bottle was then labelled, shrink-wrapped in plastic film and placed in a freezer.

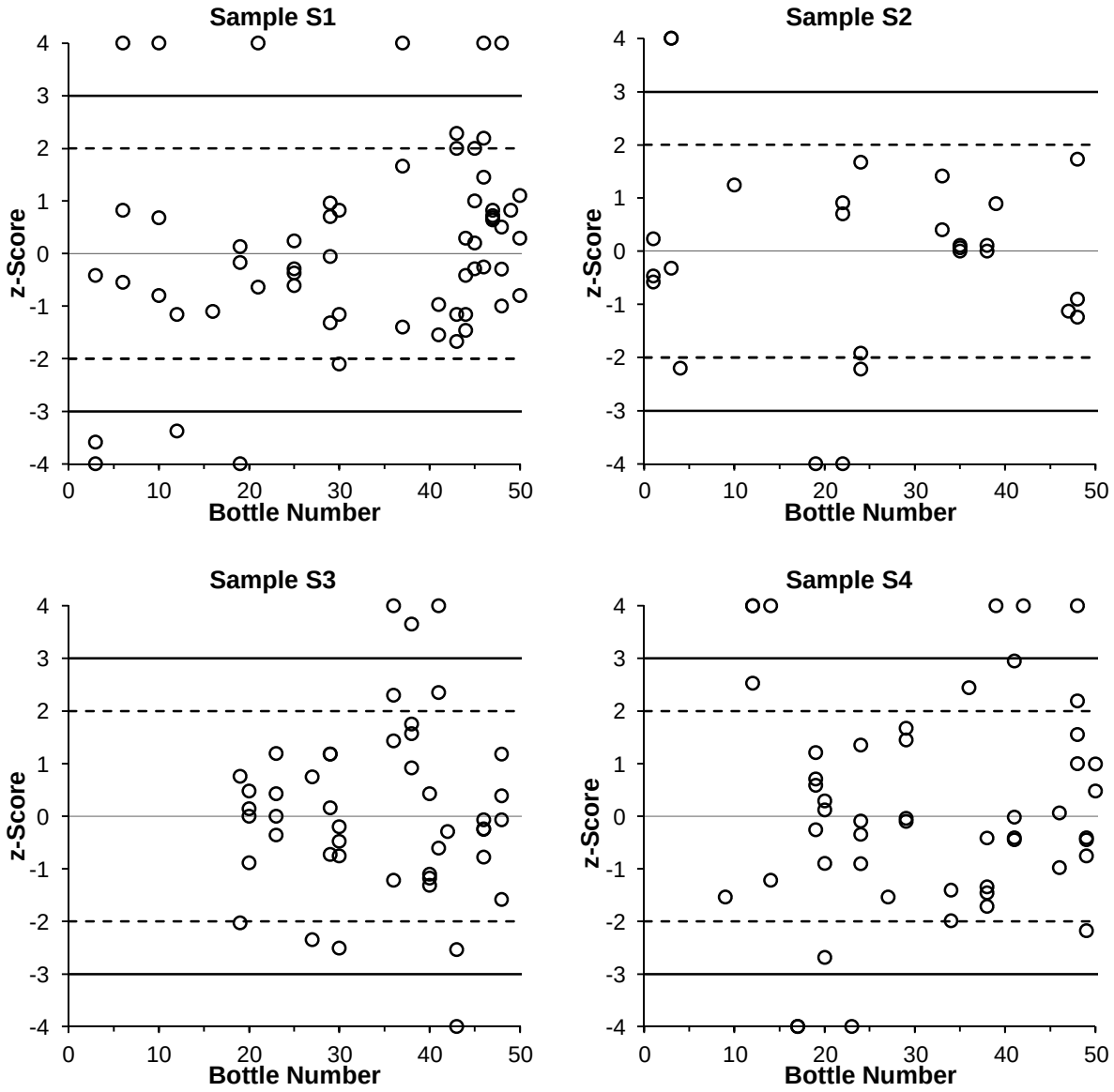
Preparation of Sample S4 (Orange)

The oranges were rinsed with tap water and allowed to dry. Whole oranges, including the peel, were chopped, placed in a stainless steel drum, pureed with a stick mixer and passed through an 850 µm sieve. After sieving the oranges, water was added to enable mixing. The resultant puree was continuously stirred while 50 aliquots of at least 100 g were dispensed into 200 mL amber bottles to provide unspiked samples. The remaining puree was spiked with aliquots of each pesticide standard solution. The spiked puree was stirred for at least two hours and bottled. Each bottle was then labelled, shrink-wrapped and placed in a freezer.

APPENDIX 2 – ASSESSMENT OF HOMOGENEITY AND TRANSPORTATION STABILITY

A2.1 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 z-scores obtained for all scored analytes to bottle number analysed by participants are presented in Figure 55, and no significant trend was observed.

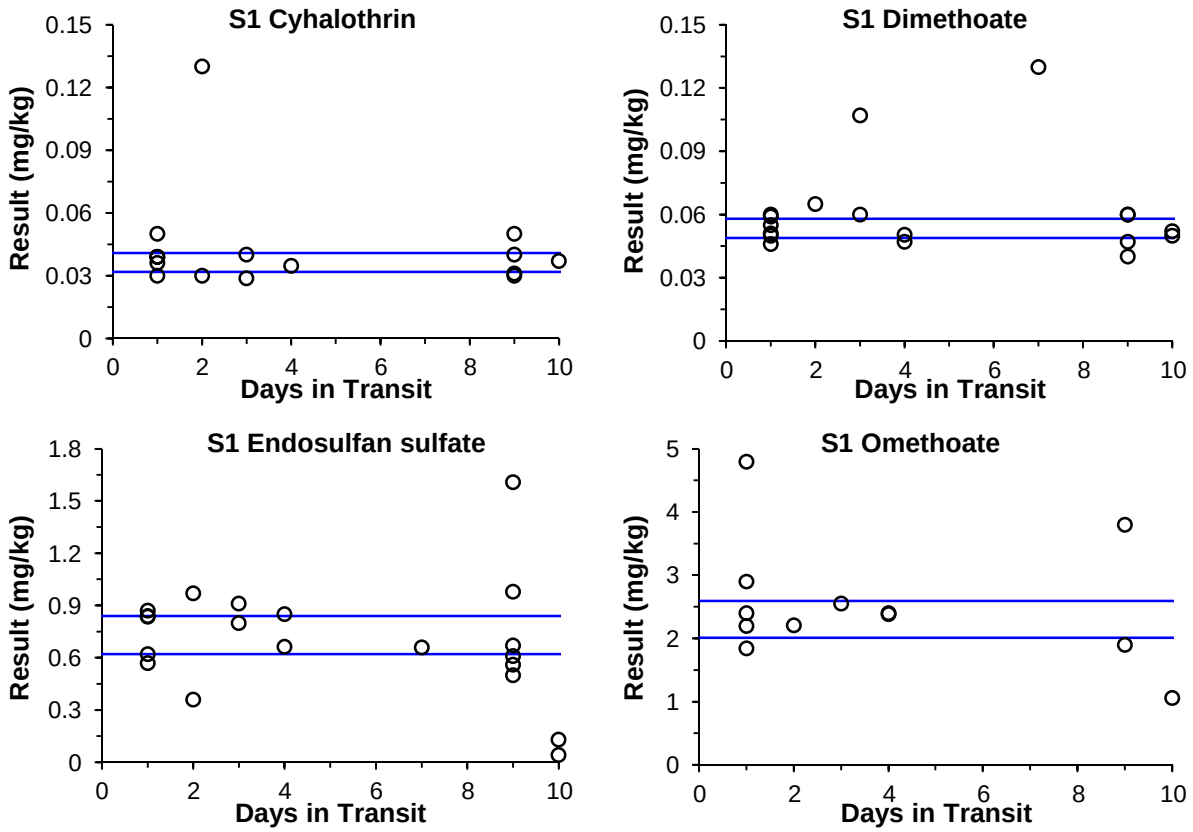


z-Scores greater than 4 or less than -4 have been plotted at 4 and -4 respectively.

Figure 55 z-Score vs Bottle Number for Samples S1, S2, S3 and S4

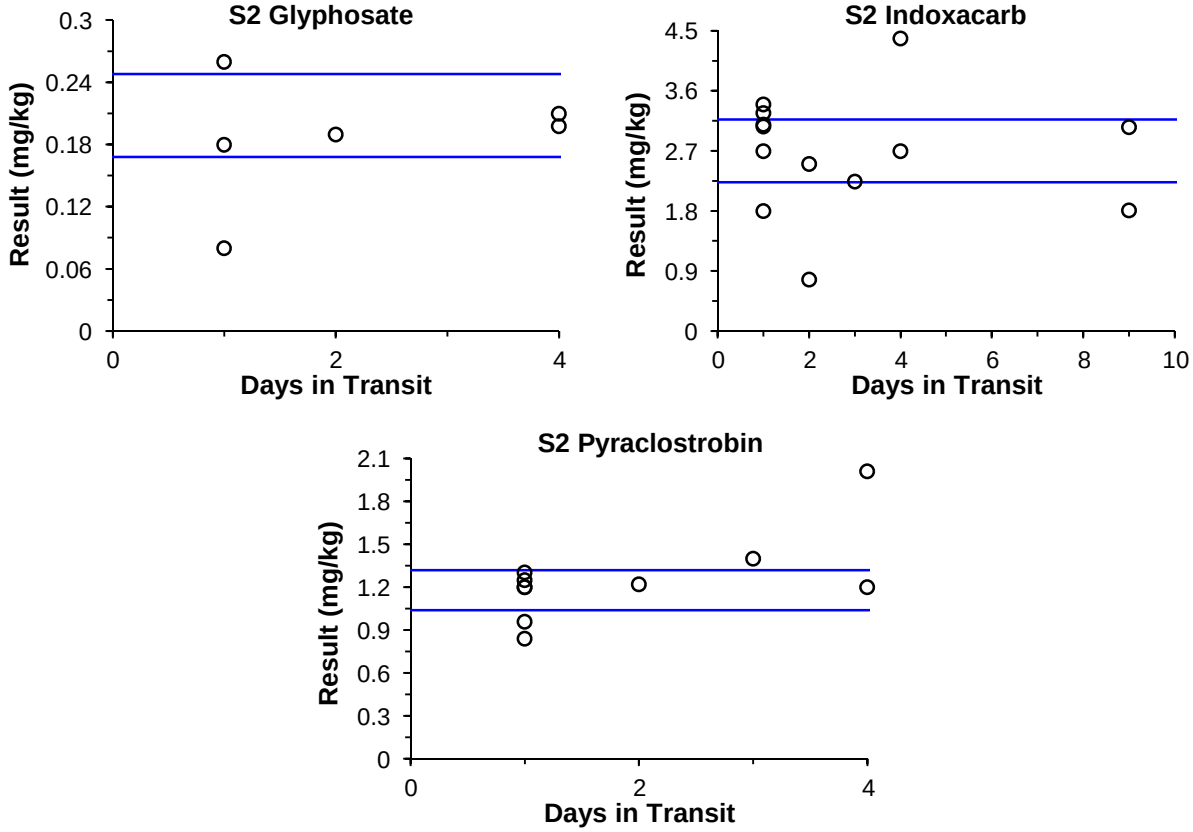
A2.2 Stability

No stability testing was conducted for this study, though previous use of these or similar analytes gave some assurance that they were stable in frozen produce. The samples were stored in the freezer at approximately -20 °C after preparation and prior to dispatch. For dispatch, samples were packaged into insulated foam boxes with cooler bricks. Comparisons of results obtained to days spent in transit for scored analytes are presented in Figures 56 to 59. No evidence of analyte degradation with respect to the amount of time spent in transit was observed.



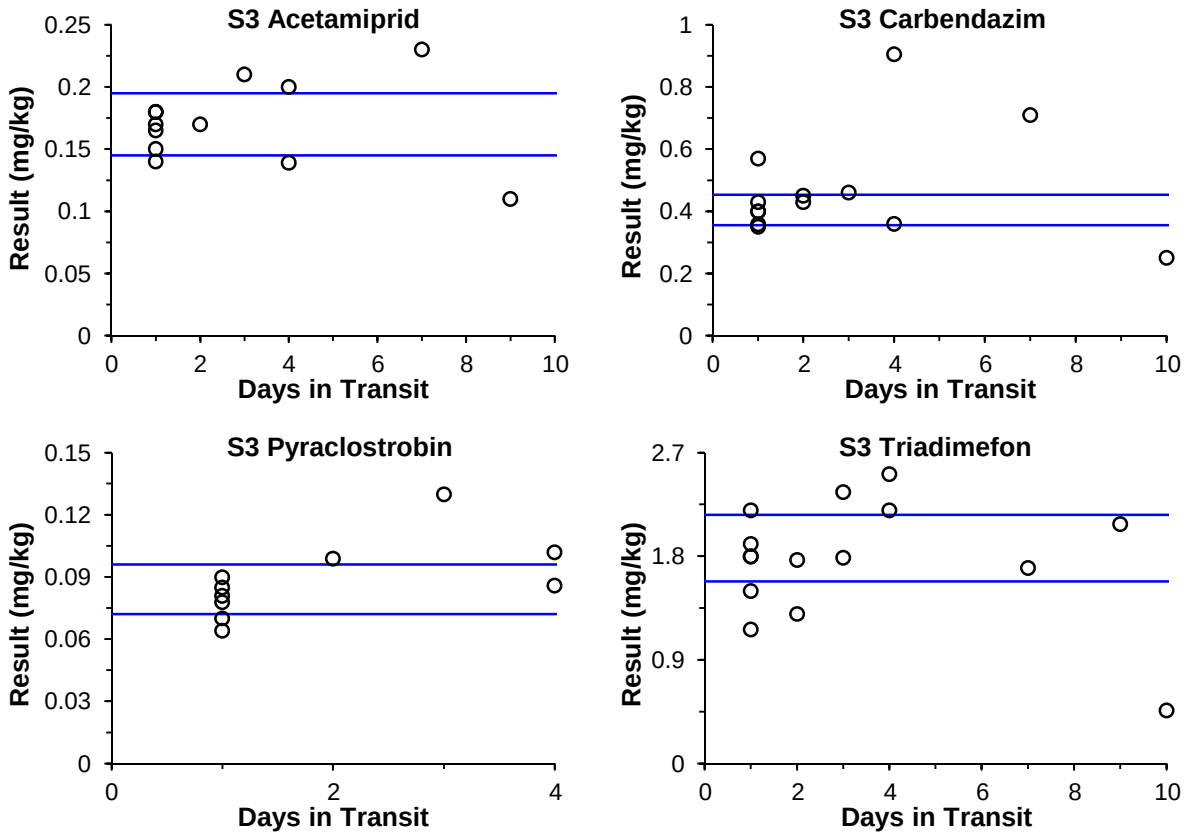
The solid blue lines correspond to the assigned value ± U for each analyte.

Figure 56 Result vs Days in Transit for Sample S1 Analytes



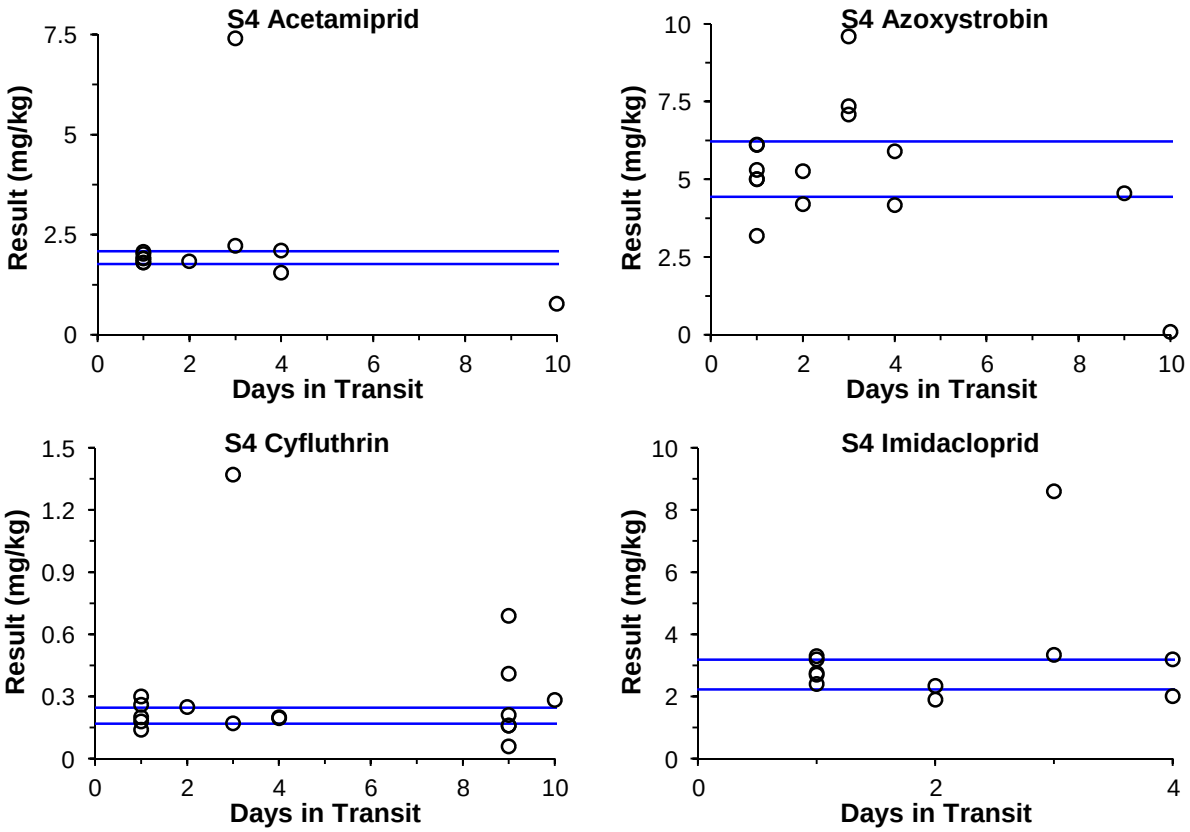
The solid blue lines correspond to the assigned value ± U for each analyte.

Figure 57 Result vs Days in Transit for Sample S2 Analytes



The solid blue lines correspond to the assigned value ± U for each analyte.

Figure 58 Result vs Days in Transit for Sample S3 Analytes



The solid blue lines correspond to the assigned value ± U for each analyte.

Figure 59 Result vs Days in Transit for Sample S4 Analytes

APPENDIX 3 – PARTICIPANTS' TEST METHODS

Participants were requested to provide information about their test methods. Responses are presented in Tables 29 to 45. Some responses may be modified so that the participant cannot be identified.

Table 29 Analysis of Blank Sample and Sample Mass Used

Lab. Code	Blank Analysed?	S1 Sample Mass (g)	S2 Sample Mass (g)	S3 Sample Mass (g)	S4 Sample Mass (g)
1	Yes	10	10	10	10
2	NR		20	20	
3	Yes	10	10	10	10
4	Yes	10	10	10	10
5	No	10	10	10	10
6	Yes	3	3	3	3
7	Yes	10	10	10	10
8	Yes	10	10	10	10
9	Yes	10	10	10	10
10	Yes	20	20	20	20
11	No	20	20	20	20
12	Yes	15	15	15	15
13	Yes	10 (1 g of sample extracted for endosulfan sulfate)	10 (1 g of sample extracted for indoxacarb)	10 (1 g of sample extracted for carbendazim and triadimefon)	10 (1 g of sample extracted for azoxystrobin and imidacloprid)
14	Yes	20	20		20
15	Yes	10	10	10	10
16	Yes	15	15	15	15
17	Yes	10	10	10	10
18	Yes	15	15	15	15
19	No	15	15	15	15
20	Yes	10	10 (5 g for glyphosate)	10	10
21	NR	5 & 10	5 & 10	5 & 10	5 & 10

Table 30 Sample S1 Tomato Cyhalothrin Methodology

Lab. Code	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument	Comments
1	Liquid-Liquid	Methanol/Water	ChemElut	GC-MS/MS	
2					
3	QuEChERS	Acetonitrile	PSA	GC-MS/MS	
4	SPE	acetonitrile	C18, Envicarb,Florisil	GC-ECD	
5	QuEChERS	Acetonitrile	PSA	GC-MS/MS	
6	NT				
7	QuEChERS	Acetonitrile	PSA	GC-MS/MS	
8	SPE	Acetonitrile	C18, carbon, florisil	GC-ECD	
9	QuEChERS	ACN	d-SPE	GC-MS/MS	
10	QuEChERS	Hexane	deactivate silica gel	GC-ECD	
11	QuEChERS	Acetonitrile	dispersive-SPE	GC-ECD	
12	NT				
13	QuEChERS	acetonitrile	dSPE(PSA)	GC-MS/MS	solvent exchanged to EtOAc prior to analysis
14	Liquid-Liquid	DCM, Acetone, Hexane		GCMS	
15					
16	QuEChERS	Acetonitrile (0.1% acetic acid)	C18/PSA	GC-ECD	
17	SPE	Acetonitrile	C-18-Gce-Florisil	GC-ECD	
18	QuEChERS	Acetonitrile	C18, PSA-GCB	GC-ECD	
19	QuEChERS	Acetonitrile	dSPE	GC-MS	
20	QuEChERS	Acetonitrile	Dispersive SPE	GC-MS/MS	
21	QuEChERS	Acetonitrile	Dispersive SPE:- PSA, C18, MgSO4.	GC-MS	

Table 31 Sample S1 Tomato Dimethoate Methodology

Lab. Code	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument	Comments
1	Liquid-Liquid	Methanol/Water	ChemElut	GC-MS/MS	
2	QuEChERS	Acetonitrile	Florisil	GC-FPD	
3	QuEChERS	Acetonitrile	PSA	LC-MS/MS	
4	SPE	acetonitrile	C18, Envicarb,Florisil	GC-NPD	
5	QuEChERS	Acetonitrile	PSA	LC-MS/MS	
6	QuEChERS	Acidic ethyl acetate		GC-MS/MS or LC MS/MS	
7	QuEChERS	Acetonitrile			
8	NT				
9	QuEChERS	ACN	d-SPE	LC-MS/MS	
10	Quechers	Acetonitrile		GC-FPD	
11	QuEChERS	Acetonitrile	dispersive-SPE	GC-FPD	
12	QuEChERS	CAN	PSA	LC-MS/MS	
13	QuEChERS	acetonitrile	dSPE(PSA)	GC-MS/MS	solvent exchanged to EtOAc prior to analysis
14	Liquid-Liquid	DCM, Acetone, Hexane		GCMS, LCMS	
15	QuEChERS	acetonitrile	Dispersive Solid Phase Extraction(d-spe)	GC-FPD	
16	QuEChERS	Acetonitrile (0.1% acetic acid)	C18/PSA	GC-FPD	
17	SPE	Acetonitrile	C-18-Gce-Florisil	GC-FPD	
18	QuEChERS	Acetonitrile	C18, PSA-GCB	GC-FPD	
19	QuEChERS	Acetonitrile	dSPE	GC-FPD	
20	QuEChERS	Acetonitrile	Dispersive SPE	LC-MS/MS	
21	QuEChERS	Acetonitrile	Dispersive SPE:- PSA, C18, MgSO4.	LC-MS/MS	

Table 32 Sample S1 Tomato Endosulfan Sulfate Methodology

Lab. Code	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument	Comments
1	Liquid-Liquid	Methanol/Water	ChemElut	GC-MS/MS	
2	QuEChERS	Acetonitrile	Florisil	GC-ECD	
3	QuEChERS	Acetonitrile	PSA	GC-MS/MS	
4	SPE	acetonitrile	C18, Envicarb,Florisil	GC-ECD	
5	QuEChERS	Acetonitrile	PSA	GC-MS/MS	
6	QuEChERS	Acidic ethyl acetate		GC-MS/MS or LC MS/MS	
7	QuEChERS	Acetonitrile	PSA	GC-MS/MS	
8	SPE	Acetonitrile	C18, carbon, florisil	GC-ECD	
9	QuEChERS	ACN	d-SPE	GC-MS/MS	
10	QuEChERS	Hexane	deactivate silica gel	GC-ECD	
11	QuEChERS	Acetonitrile	dispersive-SPE	GC-ECD	
12	NT				
13	QuEChERS	acetonitrile	dSPE(PSA)	GC-MS/MS	solvent exchanged to EtOAc prior to analysis. 1g of sample extracted
14	Liquid-Liquid	DCM, Acetone, Hexane		GCMS	
15	QuEChERS	acetonitrile	Dispersive Solid Phase Extraction(d-spe)	GC-MS/MS	
16	QuEChERS	Acetonitrile (0.1% acetic acid)	C18/PSA	GC-ECD	
17	SPE	Acetonitrile	C-18-Gce-Florisil	GC-ECD	
18	QuEChERS	Acetonitrile	C18, PSA-GCB	GC-ECD	
19	QuEChERS	Acetonitrile	dSPE	GC-MS	
20	QuEChERS	Acetonitrile	Dispersive SPE	GC-MS/MS	
21	QuEChERS	Acetonitrile	Dispersive SPE:- PSA, C18, MgSO4.	GC-ECD	

Table 33 Sample S1 Tomato Omethoate Methodology

Lab. Code	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument	Comments
1	Liquid-Liquid	Methanol/Water	ChemElut	GC-MS/MS	
2	QuEChERS	Acetonitrile	Florisil	GC-FPD	
3	QuEChERS	Acetonitrile	PSA	LC-MS/MS	
4	NT				
5	QuEChERS	Acetonitrile	PSA	LC-MS/MS	
6	NT				
7	QuEChERS	Acetonitrile		LC-MS/MS	
8	NT				
9	QuEChERS	ACN	d-SPE	LC-MS/MS	
10	Quechers	Acetonitrile		GC-FPD	
11	QuEChERS	Acetonitrile	dispersive-SPE	GC-FPD	
12	NT				
13	QuEChERS	acetonitrile	dSPE(PSA)	GC-MS/MS	solvent exchanged to EtOAc prior to analysis
14	Liquid-Liquid	DCM, Acetone, Hexane		GCMS, LCMS	
15	QuEChERS	acetonitrile	Dispersive Solid Phase Extaction(d-spe)	GC-FPD	
16	QuEChERS	Acetonitrile (0.1% acetic acid)	C18/PSA	LC-MS/MS	
17	NT				
18	NT				
19	NT				
20	QuEChERS	Acetonitrile	Dispersive SPE	LC-MS/MS	
21	QuEChERS	Acetonitrile	Dispersive SPE:- PSA, C18, MgSO4.	LC-MS/MS	

Table 34 Sample S2 Bok Choy Cyfluthrin Methodology

Lab. Code	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument
1	Liquid-Liquid	Methanol/Water	ChemElut	GC-MS/MS
2	QuEChERS	Acetonitrile	Florisil	GC-ECD
3	QuEChERS	Acetonitrile	PSA	GC-MS/MS
4	SPE	acetonitrile	C18, Envicarb,Florisil	GC-ECD
5	QuEChERS	Acetonitrile	PSA	GC-MS/MS
6	NT			
7	QuEChERS	Acetonitrile	PSA	GC-MS/MS
8	SPE	Acetonitrile	C18, carbon, florisil	GC-ECD
9	QuEChERS	ACN	d-SPE	GC-MS/MS
10	QuEChERS	Hexane	deactivate silica gel	GC-ECD
11	QuEChERS	Acetonitrile	dispersive-SPE	GC-ECD
12	NT			
13	NT			
14	Liquid-Liquid	DCM, Acetone, Hexane		GCMS
15	QuEChERS	acetonitrile	Dispersive Solid Phase Extraction(d-spe)	GC-MS/MS
16	QuEChERS	Acetonitrile (0.1% acetic acid)	C18/PSA	GC-ECD
17	SPE	Acetonitrile	C-18-Gce-Florisil	GC-ECD
18	QuEChERS	Acetonitrile	C18, PSA-GCB	GC-ECD
19	QuEChERS	Acetonitrile	dSPE	GC-MS
20	QuEChERS	Acetonitrile	Dispersive SPE	GC-MS/MS
21	QuEChERS	Acetonitrile	Dispersive SPE:- PSA, C18, MgSO4.	GC-MS

Table 35 Sample S2 Bok Choy Glyphosate Methodology

Lab. Code	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument	Comments
1	NT				
2					
3	Solid-Liquid	Acetonitrile		LC-MS/MS	
4	NT				
5	Liquid-Liquid	Water/MEOH		LC-MS/MS	
6	NT				
7	FMOC Derivatization	Water		LC-MS/MS	
8	NT				
9	NT	NT	NT	NT	
10	NT				
11	NT				
12	NT				
13	NT				
14	NT				
15	NT				
16	QuEChERS	Acetonitrile (0.1% acetic acid)	C18/PSA	LC-MS/MS	
17	NT				
18	NT				
19	NT				
20	SPE	HCL 0.1 N	Resin	HPLC Post-column	Sample weight 5 g
21	SPE	Water	Florisil	GC-MS	

Table 36 Sample S2 Bok Choy Indoxacarb Methodology

Lab. Code	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument	Comments
1	Liquid-Liquid	Methanol/Water	ChemElut	LC-MS/MS	
2					
3	QuEChERS	Acetonitrile	PSA	LC-MS/MS	
4	SPE	acetonitrile	C18, Envicarb,Florisil	GC-ECD	
5	QuEChERS	Acetonitrile	PSA	GC-MS/MS	
6	NT				
7	QuEChERS	Acetonitrile		LC-MS/MS	
8	SPE	Acetonitrile	C18, carbon, florisil	GC-ECD	
9	NT				
10	NT				
11	NT				
12	QuEChERS	CAN	PSA	LC-MS/MS	
13	QuEChERS	acetonitrile	dSPE(PSA)	LC-MS/MS	1 g of sample extracted
14	Liquid-Liquid	DCM, Acetone, Hexane		GCMS, LCMS	
15					
16	QuEChERS	Acetonitrile (0.1% acetic acid)	C18/PSA	LC-MS/MS	
17	SPE	Acetonitrile	C-18-Gce-Florisil	GC-MS/MS	
18	NT				
19	NT				
20	QuEChERS	Acetonitrile	Dispersive SPE	LC-MS/MS	
21	QuEChERS	Acetonitrile	Dispersive SPE:- PSA, C18, MgSO4.	LC-MS/MS	

Table 37 Sample S2 Bok Choy Pyraclostrobin Methodology

Lab. Code	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument
1	Liquid-Liquid	Methanol/Water	ChemElut	LC-MS/MS
2				
3	QuEChERS	Acetonitrile	PSA	LC-MS/MS
4	NT			
5	QuEChERS	Acetonitrile	PSA	LC-MS/MS
6	NT			
7	QuEChERS	Acetonitrile		LC-MS/MS
8	NT			
9	QuEChERS	ACN	d-SPE	LC-MS/MS
10	NT			
11	NT			
12	QuEChERS	CAN	PSA	LC-MS/MS
13	NT			
14	Liquid-Liquid	DCM, Acetone, Hexane		LCMS
15	NT			
16	QuEChERS	Acetonitrile (0.1% acetic acid)	C18/PSA	LC-MS/MS
17	NT			
18	NT			
19	NT			
20	QuEChERS	Acetonitrile	Dispersive SPE	LC-MS/MS
21	QuEChERS	Acetonitrile	Dispersive SPE:- PSA, C18, MgSO4.	LC-MS/MS

Table 38 Sample S3 Apple Acetamiprid Methodology

Lab. Code	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument
1	Liquid-Liquid	Methanol/Water	ChemElut	LC-MS/MS
2				
3	QuEChERS	Acetonitrile	PSA	LC-MS/MS
4	SPE	acetonitrile	C18, Envicarb,Florisil	LC-MS/MS
5	QuEChERS	Acetonitrile	PSA	LC-MS/MS
6	QuEChERS	Acidic ethyl acetate		GC-MS/MS or LC MS/MS
7	QuEChERS	Acetonitrile		LC-MS/MS
8	NT			
9	QuEChERS	ACN	d-SPE	LC/MS/MS
10	NT			
11	NT			
12	QuEChERS	CAN	PSA	LC-MS/MS
13	NT			
14	Liquid-Liquid	DCM, Acetone, Hexane		LCMS
15				
16	QuEChERS	Acetonitrile (0.1% acetic acid)	C18/PSA	LC-MS/MS
17	NT			
18	NT			
19	NT			
20	QuEChERS	Acetonitrile	Dispersive SPE	LC-MS/MS
21	QuEChERS	Acetonitrile	Dispersive SPE:- PSA, C18, MgSO4.	LC-MS/MS

Table 39 Sample S3 Apple Carbendazim Methodology

Lab. Code	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument	Comments
1	Liquid-Liquid	Methanol/Water	ChemElut	LC-MS/MS	
2					
3	QuEChERS	Acetonitrile	PSA	LC-MS/MS	
4	NT				
5	QuEChERS	Acetonitrile	PSA	LC-MS/MS	
6	QuEChERS	Acidic ethyl acetate		GC-MS/MS or LC MS/MS	
7	QuEChERS	Acetonitrile		LC-MS/MS	
8	NT				
9	QuEChERS	ACN	d-SPE	LC-MS/MS	
10	NT				
11	NT				
12	QuEChERS	CAN	PSA	LC-MS/MS	
13	QuEChERS	acetonitrile	dSPE(PSA)	LC-MS/MS	1 g of sample extracted
14	Liquid-Liquid	DCM, Acetone, Hexane		, LCMS	
15	QuEChERS	acetonitrile	Dispersive Solid Phase Extraction(d-spe)	LC-MS/MS	
16	QuEChERS	Acetonitrile (0.1% acetic acid)	C18/PSA	LC-MS/MS	
17	NT				
18	NT				
19	NT				
20	QuEChERS	Acetonitrile	Dispersive SPE	LC-MS/MS	
21	QuEChERS	Acetonitrile	Dispersive SPE:- PSA, C18, MgSO4.	LC-MS/MS	

Table 40 Sample S3 Apple Pyraclostrobin Methodology

Lab. Code	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument
1	Liquid-Liquid	Methanol/Water	ChemElut	LC-MS/MS
2				
3	QuEChERS	Acetonitrile	PSA	LC-MS/MS
4	NT			
5	QuEChERS	Acetonitrile	PSA	LC-MS/MS
6	NT			
7	QuEChERS	Acetonitrile		LC-MS/MS
8	NT			
9	QuEChERS	ACN	d-SPE	LC-MS/MS
10	NT			
11	NT			
12	QuEChERS	CAN	PSA	LC-MS/MS
13	NT			
14	Liquid-Liquid	DCM, Acetone, Hexane		LCMS
15	NT			
16	QuEChERS	Acetonitrile (0.1% acetic acid)	C18/PSA	LC-MS/MS
17	NT			
18	NT			
19	NT			
20	QuEChERS	Acetonitrile	Dispersive SPE	LC-MS/MS
21	QuEChERS	Acetonitrile	Dispersive SPE:- PSA, C18, MgSO4.	LC-MS/MS

Table 41 Sample S3 Apple Triadimefon Methodology

Lab. Code	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument	Comments
1	Liquid-Liquid	Methanol/Water	ChemElut	LC-MS/MS	
2					
3	QuEChERS	Acetonitrile	PSA	LC-MS/MS	
4	SPE	acetonitrile	C18, Envicarb,Florisil	LC-MS/MS	
5	QuEChERS	Acetonitrile	PSA	GC-MS/MS	
6	QuEChERS	Acidic ethyl acetate		GC-MS/MS or LC MS/MS	
7	QuEChERS	Acetonitrile		LC-MS/MS	
8	NT				
9	QuEChERS	ACN	d-SPE	LC-MS/MS	
10	NT				
11	NT				
12	QuEChERS	CAN	PSA	LC-MS/MS	
13	QuEChERS	acetonitrile	dSPE(PSA)	GC-MS/MS	solvent exchanged to EtOAc prior to analysis. 1 g of sample extracte
14	Liquid-Liquid	DCM, Acetone, Hexane		GCMS, LCMS	
15	QuEChERS	acetonitrile	Dispersive Solid Phase Extraction(d-spe)	GC-MS/MS	
16	QuEChERS	Acetonitrile (0.1% acetic acid)	C18/PSA	LC-MS/MS	
17	SPE	Acetonitrile	C-18-Gce-Florisil	GC-MS/MS	
18	NT				
19	NT				
20	QuEChERS	Acetonitrile	Dispersive SPE	LC-MS/MS	
21	QuEChERS	Acetonitrile	Dispersive SPE:- PSA, C18, MgSO4.	GC-MS	

Table 42 Sample S4 Orange Acetamiprid Methodology

Lab. Code	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument
1	Liquid-Liquid	Methanol/Water	ChemElut	LC-MS/MS
2				
3	QuEChERS	Acetonitrile	PSA	LC-MS/MS
4	NT			
5	QuEChERS	Acetonitrile	PSA	LC-MS/MS
6	QuEChERS	Acidic ethyl acetate		GC-MS/MS or LC MS/MS
7	QuEChERS	Acetonitrile		LC-MS/MS
8	NT			
9	QuEChERS	ACN	d-SPE	LC/MS/MS
10	NT			
11	NT			
12	QuEChERS	CAN	PSA	LC-MS/MS
13	NT			
14	Liquid-Liquid	DCM, Acetone, Hexane		LCMS
15	QuEChERS	acetonitrile	Dispersive Solid Phase Extraction(d-spe)	LC-MS/MS
16	QuEChERS	Acetonitrile (0.1% acetic acid)	C18/PSA	LC-MS/MS
17	NT			
18	NT			
19	NT			
20	QuEChERS	Acetonitrile	Dispersive SPE	LC-MS/MS
21	QuEChERS	Acetonitrile	Dispersive SPE:- PSA, C18, MgSO4.	LC-MS/MS

Table 43 Sample S4 Orange Azoxystrobin Methodology

Lab. Code	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument	Comments
1	Liquid-Liquid	Methanol/Water	ChemElut	LC-MS/MS	
2					
3	QuEChERS	Acetonitrile	PSA	LC-MS/MS	
4	SPE	acetonitrile	C18, Envicarb,Florisil	GC-ECD	
5	QuEChERS	Acetonitrile	PSA	LC-MS/MS	
6	QuEChERS	Acidic ethyl acetate		GC-MS/MS or LC MS/MS	
7	QuEChERS	Acetonitrile		LC-MS/MS	
8	NT				
9	QuEChERS	ACN	d-SPE	LC-MS/MS	
10	NT				
11	NT				
12	QuEChERS	CAN	PSA	LC-MS/MS	
13	QuEChERS	acetonitrile	dSPE(PSA)	GC-MS/MS	solvent exchanged to EtOAc prior to analysis. 1 g of sample extracted
14	Liquid-Liquid	DCM, Acetone, Hexane		GCMS, LCMS	
15	QuEChERS	acetonitrile	Dispersive Solid Phase Extraction(d-spe)	LC-MS/MS	
16	QuEChERS	Acetonitrile (0.1% acetic acid)	C18/PSA	LC-MS/MS	
17	SPE	Acetonitrile	C-18-Gce-Florisil	GC-MS/MS	
18	NT				
19	NT				
20	QuEChERS	Acetonitrile	Dispersive SPE	LC-MS/MS	
21	QuEChERS	Acetonitrile	Dispersive SPE:- PSA, C18, MgSO4.	GC-MS	

Table 44 Sample S4 Orange Cyfluthrin Methodology

Lab. Code	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument
1	Liquid-Liquid	Methanol/Water	ChemElut	GC-MS/MS
2	QuEChERS	Acetonitrile	Florisil	GC-ECD
3	QuEChERS	Acetonitrile	PSA	GC-MS/MS
4	SPE	acetonitrile	C18, Envicarb,Florisil	GC-ECD
5	QuEChERS	Acetonitrile	PSA	GC-MS/MS
6	NT			
7	QuEChERS	Acetonitrile	PSA	GC-MS/MS
8	SPE	Acetonitrile	C18, carbon, florisil	GC-ECD
9	QuEChERS	ACN	d-SPE	GC-MS/MS
10	QuEChERS	Hexane	deactivate silica gel	GC-ECD
11	QuEChERS	Acetonitrile	dispersive-SPE	GC-ECD
12	NT			
13	NT			
14	Liquid-Liquid	DCM, Acetone, Hexane		GCMS
15				
16	QuEChERS	Acetonitrile (0.1% acetic acid)	C18/PSA	GC-ECD
17	SPE	Acetonitrile	C-18-Gce-Florisil	GC-ECD
18	QuEChERS	Acetonitrile	C18, PSA-GCB	GC-ECD
19	QuEChERS	Acetonitrile	SPE	GC-MS
20	QuEChERS	Acetonitrile	Dispersive SPE	GC-MS/MS
21	QuEChERS	Acetonitrile	Dispersive SPE:- PSA, C18, MgSO4.	GC-MS

Table 45 Sample S4 Orange Imidacloprid Methodology

Lab. Code	Extraction	Extraction Solvent	Clean-Up	Measurement Instrument	Comments
1	Liquid-Liquid	Methanol/Water	ChemElut	LC-MS/MS	
2					
3	QuEChERS	Acetonitrile	PSA	LC-MS/MS	
4	NT				
5	QuEChERS	Acetonitrile	PSA	LC-MS/MS	
6	QuEChERS	Acidic ethyl acetate		GC-MS/MS or LC MS/MS	
7	QuEChERS	Acetonitrile		LC-MS/MS	
8	NT				
9	QuEChERS	ACN	d-SPE	LC-MS/MS	
10	NT				
11	NT				
12	NT				
13	QuEChERS	acetonitrile	dSPE(PSA)	LC-MS/MS	1 g of sample extracted
14	Liquid-Liquid	DCM, Acetone, Hexane		LCMS	
15					
16	QuEChERS	Acetonitrile (0.1% acetic acid)	C18/PSA	LC-MS/MS	
17	NT				
18	NT				
19	NT				
20	QuEChERS	Acetonitrile	Dispersive SPE	LC-MS/MS	
21	QuEChERS	Acetonitrile	Dispersive SPE:- PSA, C18, MgSO4.	LC-MS/MS	

APPENDIX 4 – ROBUST AVERAGE AND ASSOCIATED UNCERTAINTY, z-SCORE AND E_n-SCORE CALCULATIONS

A4.1 Robust Average and Associated Uncertainty

Robust averages were calculated using the procedure described in ISO 13528:2015.[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 is set out below in Table 46.

Table 46 Uncertainty of Robust Average for Sample S1 Endosulfan Sulfate

No. results (p)	20
Robust Average	0.70 mg/kg
$S_{rob\ av}$	0.25 mg/kg
$u_{rob\ av}$	0.07 mg/kg
k	2
$U_{rob\ av}$	0.14 mg/kg

Therefore, the robust average for endosulfan sulfate in Sample S1 is 0.70 ± 0.14 mg/kg.

A4.2 z-Score and E_n-Score Calculation

For each participant's result, a z-score and E_n-score are calculated according to Equations 2 and 3 respectively (Sections 4.7 and 4.8).

A worked example for is set out below in Table 47.

Table 47 z-Score and E_n-Score for Sample S1 Cyhalothrin Result Reported by Laboratory 1

Participant Result (mg/kg)	Assigned Value (mg/kg)	Target Standard Deviation	z-Score	E_n-Score
0.039 ± 0.011	0.0363 ± 0.0045	15% as CV, or: 0.15 × 0.0363 = 0.005445 mg/kg	z-Score = $\frac{0.039-0.0363}{0.005445}$ = 0.50	E_n-Score = $\frac{0.039-0.0363}{\sqrt{0.011^2+0.0045^2}}$ = 0.23

APPENDIX 5 – ACRONYMS AND ABBREVIATIONS

2,4-D	2,4-Dichlorophenoxyacetic acid
A.V.	Assigned Value
ACE	Acetone
ACN	Acetonitrile
AMPA	Aminomethylphosphonic acid
ANZFS	Australia New Zealand Food Standards
CITAC	Cooperation on International Traceability in Analytical Chemistry
CRM	Certified Reference Material
CV	Coefficient of Variation
DCM	Dichloromethane
dSPE	Dispersive Solid Phase Extraction
ECD	Electron Capture Detector
EtOAc	Ethyl Acetate
FAO	Food and Agriculture Organization of the United Nations
FPD	Flame Photometric Detector
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
JMPR	Joint FAO/WHO Meeting on Pesticide Residues
LC	Liquid Chromatography
LLE	Liquid-Liquid Extraction
LOR	Limit of Reporting
Max.	Maximum
Md	Median
MeOH	Methanol
Min.	Minimum
MRL	Maximum Residue Limit
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
NPD	Nitrogen Phosphorus Detector
NR	Not Reported
NT	Not Tested
p,p'-DDT	Dichlorodiphenyltrichloroethane
PCV	Performance Coefficient of Variation
PSA	Primary/Secondary Amine
PT	Proficiency Test
QuEChERS	Quick, Easy, Cheap, Effective, Rapid and Safe extraction technique
R.A.	Robust Average
RM	Reference Material
S.V.	Spiked Value (or the formulated concentration) of a PT sample
SANTE	Directorate-General for Health and Food Safety
SD	Standard Deviation
SI	International System of Units
SPE	Solid Phase Extraction
SS	Spiked Samples
WHO	World Health Organization

END OF REPORT