EURL-PROFICIENCY TEST-T01, 2013

Pesticide Residues in Tea Homogenate

Final Report

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EURL-EUROPEAN UNION PROFICIENCY TEST 01
FOR THE DETERMINATION OF PESTICIDES IN TEA USING MULTIRESIDUE METHODS
2013

According to Article 28 of Regulation 396/2005/EC (23rd February, 2005) of the European Parliament and of the Council, concerning maximum residue levels for pesticides in or on food and feed of plant and animal origin[1], all laboratories analysing samples for the official control of pesticide residues shall participate in the European Union Proficiency Tests (EUPTs) for pesticide residues organised by the European Union. These proficiency tests are carried out on an annual basis in order to continuously improve the quality, accuracy and comparability of the residue data reported by EU Member States to the European Union, as well as other Member States, within the framework of the EU Multi-annual co-ordinated control programme and national monitoring programmes.

Regulation (EC) No 882/2004[2] lays down the general tasks, duties and requirements for European Union Reference Laboratories (EURLs)[3] for Food, Feed and Animal Health. Among these tasks is the provision for independently-organised comparative tests. The European Union Proficiency Test of pesticides in Tea 01 has been organised by the EURL in Fruit and Vegetables at the University of Almería, Spain[4].

Participation in this European Union Proficiency Test in Tea 01 was on a purely voluntary basis for EU laboratories. Nevertheless, all FV-NRLs and FV-Official laboratories involved in the determination of pesticide residues in fruit and vegetables were invited to take part. Additionally, laboratories from China, Egypt, Israel, Saudi Arabia, Serbia and Uruguay participated.

This report will be presented to the European Union Standing Committee for Animal Health and the Food Chain. In addition, DG-SANCO will have full access to all data from the EUPTs including the lab-code/lab-name key.

[1] Regulation (EC) No 396/2005, published in the OJ of the EU L70 on 16.03.2005, last amended by Regulation 839/2008 published in the OJ of the EU L234 on 30.08.2008.

[2] Regulation (EC) No 882/2004 of the European Parliament and of the Council on official controls performed to ensure compliance verification with feed and food law, animal health and animal welfare rules. Published in the OJ of the EU L191 on 28.05.2004.

[3] The Community Reference Laboratory (CRL) changed its name to the European Union Reference Laboratory (EURL) on 1st December 2009 as a result of the Treaty of Lisbon. OJ of the EU C306 on 17.12.2007.

[4] Commission Regulation (EC) No 776/2006 of 23rd May 2006 - amending Annex VII to Regulation (EC) No 882/2004 of the European Parliament and of the Council as regards European Union Reference Laboratories.

1. INTRODUCTION

In 2012, there were many more notifications in the Rapid Alert System for Food and Feed (RASFF)[5] concerning tea and herbal tea matrices than in previous years. In 2010 and 2011, there were only two alerts each year, but in 2012 there were a total of 41. Of these, one was an alert coming from Lithuania, 37 were border rejections (the majority originating from China) and 3 were for information and follow up. The maximum number of pesticides found in a single sample was 7 with concentrations ranging from 0.011 to 1.13 mg/kg; and out of a total of 82 positive findings, 6 were for unauthorised substances.
So far in 2013, there have been 27 border rejections (up to August), and in one of them there were 14 positives findings with the highest residue level being 1.63 mg/kg.

Taking into account that tea and other herbal teas have become more and more popular due to the health benefits associated with their consumption, and the contamination levels in these kinds of matrices, it is very important to know how efficient the analytical methods used by the laboratories are for reporting official results.

Fifty-four laboratories agreed to participate in European Union Proficiency Test in Tea 01.

The proficiency test was performed in 2013 using a tea homogenate. This proficiency test was based on the analysis of tea samples from China containing incurred pesticide residues. The tea was bought from a specialised shop for Chinese products, in Almería, Spain, containing incurred pesticides. Participating laboratories were not provided with a 'blank' tea homogenate.

The test item, 15 g of tea homogenate containing pesticide residues, was shipped to participants on 10th June 2013. The deadline for results submission to the Organiser was 28th June 2013. The participants were provided with a list of one hundred and seventy-five target pesticide residues (Annex I) and were informed that any of these pesticides might be present in the test item. They were asked to determine the residue levels of all the pesticides that they detected and report the concentrations. This list of target pesticides also contained the Minimum Required Reporting Level (MRRL) for each pesticide fixed between 0.005 and 0.02 mg/Kg.

Pesticides considered as positives were those which were reported by the organiser and the majority of the participants. The median values of the results submitted by participants were used to obtain the assigned (true) values for each of the pesticide residues present. A fit-for-purpose relative target standard deviation (FFP RSD) of 25 % was chosen to calculate the target standard deviations (σ) as well as the z-scores for each pesticide.

For the assessment of overall laboratory performance, only the Average of the squared z-scores (AZ^2) has been used. Laboratories that have 'sufficient scope' and are able to detect at least

[5] http://ec.europa.eu/food/food/rapidalert/rasff_portal_database_en.htm

90 % of the pesticides present in the test item and report no false positives will be classified into Category A. Within this category, the laboratories have also been subclassified as 'good', 'satisfactory' or 'unsatisfactory', in relation to the overall accuracy of the results that they reported.

All the other laboratories have been classified into Category B because they have demonstrated 'insufficient scope'. For laboratories in Category B, individual z-scores have been calculated but their overall accuracy of their results has not been assessed. They have been listed in order of the number of pesticides sought and the number of acceptable z-scores achieved. In addition, the laboratories in the Category B table have been ranked according to the number of pesticides detected from the total number of pesticides taken into account for the statistical evaluation.

Laboratories that did not report results have not been classified into any category and are subsequently indicated in Annex 2 with the rest of laboratories that agreed to participate in EUPT-T01.

2. TEST ITEMS

2.1 Analytical method

The analytical method described briefly below was performed by the EURL-FV in order to conduct the homogeneity and stability tests. This was:

- Modified QuEChERS method[6]: The sample is extracted with acetonitrile using the same salts as for citrate QuEChERS, but firstly the tea was hydrated. In the clean-up step, calcium chloride was added instead of magnesium sulphate. The extract obtained was injected into both GC-MS and LC-MS based instruments.

Acetamiprid, buprofezin, carbendazim, difenoconazole, imidacloprid, methomyl, tebuconazole and thiophanate-methyl were determined using LC-QQQ-MS/MS. All other pesticides (chlorpyrifos, cypermethrin, endosulfan beta, etofenprox, fenpropathrin, lambda cyhalothrin, parathion ethyl and pyridaben) were analysed using GC-QQQ-MS/MS. For confirmation purposes, MS/MS spectra were used.

2.2 Preparation of the test item

One kilogram three hundred grams of dried green tea containing incurred pesticide residues was bought in a local shop in Almería (Spain). A subsample was taken and analysed to ascertain the pesticides present and to determine their concentrations. Following this, the entire sample was processed using a mill and then sieved through a mesh size of 0.5 mm. The milled tea was mixed in a constantly-spinning container for 20 hours to attain a homogeneous material. 15 g portions of the well-mixed homogenate were weighed into previously-labelled sealed plastic bags and stored in a fridge at 4 °C prior to distribution to participants.

[6] A. Lozano, Ł. Rajski, N .Belmonte-Valles, A. Uclés, S. Uclés, M. Mezcua, A. R. Fernández-Alba. Pesticide analysis in teas and chamomile by liquid chromatography and gas chromatography tandem mass spectrometry using a modified QuEChERS method: Validation and pilot survey in real samples. J. Chromatogr. A, 1268 (2012), 109-122.

2.3 Homogeneity test

Ten bags of the test item were randomly chosen from those stored in the fridge and analyses were performed on duplicate portions taken from each bag. The sequence of analyses was determined using a table of randomly-generated numbers. The injection sequence of the twenty extracts that were analysed by GC and LC was also randomly chosen. The quantification by GC and LC was performed using three points for standard addition in triplicate constructed from the test item.

The statistical evaluation was performed according to the International Harmonized Protocol published by IUPAC, ISO and AOAC[7]. The individual residue data from the homogeneity tests are given in Appendix 1. The results of the statistical analyses are given in Table 2.1. The acceptance criteria for the test item to be sufficiently homogenous for the proficiency test were that: $Ss^2 < c$, where Ss is the between-bottle sampling standard deviation and $c = F_1\sigma^2_{all} + F_2S^2_{an}$; F_1 and F_2 being constant values of 1.88 and 1.01, respectively, from the ten samples taken, and σ^2_{all} = 0.3 x FFP RSD(25 %) x the analytical sampling mean for all the pesticides.

Table 2.1. Statistical evaluation of the homogeneity test data (n = 20 analyses)

Pesticide	Mean Conc. (mg/Kg)	Ss^2	c	$Ss^2 < c$ Pass/Fail
Acetamiprid	0.148	2.14×10^{-4}	3.74×10^{-4}	Pass
Buprofezin	0.213	0	5.74×10^{-3}	Pass
Carbendazim	0.271	1.35×10^{-3}	2.94×10^{-3}	Pass
Chlorpyrifos	0.466	2.16×10^{-3}	1.02×10^{-2}	Pass
Cypermethrin	0.145	2.0×10^{-4}	2.40×10^{-4}	Pass
Difenoconazole	0.380	2.08×10^{-3}	6.02×10^{-3}	Pass
Endosulfan beta	0.096	0	5.38×10^{-4}	Pass
Ethofenprox	0.234	0	1.64×10^{-3}	Pass
Fenpropathrin	0.258	0	2.54×10^{-3}	Pass
Imidacloprid	0.108	9.60×10^{-5}	2.17×10^{-4}	Pass
Lambda cyhalothrin	0.175	0	1.13×10^{-3}	Pass
Methomyl	0.113	1.52×10^{-4}	4.11×10^{-4}	Pass
Parathion ethyl	0.436	2.82×10^{-4}	7.16×10^{-3}	Pass
Pyridaben	0.338	0	3.00×10^{-3}	Pass
Tebuconazole	0.423	2.57×10^{-3}	8.29×10^{-3}	Pass
Thiophanate methyl	0.259	4.70×10^{-3}	4.05×10^{-3}	Fail

S_s: Between-Sampling Standard Deviation

[7] M. Thompson, S. L. R. Ellison, and R.Wood. The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories. Pure Appl. Chem., 2006, 78 (1), 145–196.

As can be seen from Table 2.1., all the incurred pesticides in tea matrix passed the homogeneity test, except thiophanate-methyl. Statistical data for this compound are shown only for informative purpose.

2.4 Stability tests

The analytical method described briefly in section 2.1 was also used for the stability tests.

The tests were performed on two occasions. On each occasion, a single bag stored in the fridge at 4°C was chosen randomly and duplicate analyses were performed.

The two occasions were:
- Day 1: coinciding with the first test item shipments, which took place on 10th June 2013.
- Day 2: shortly after the deadline for reporting results, on 28th June 2013.

The individual results are given in Table 2.2. In general, these tests did not show any significant decrease in the pesticide concentratons. This demonstrates that, for the duration of the proficiency test, and provided that the storage conditions prescribed were followed, the time elapsed until the participants performed the analysis would not have influenced their results.

Table 2.2. Statistical test for analytical precision and to demonstrate pesticides stability after a time-elapse interval.

Pesticide	Concentration (mg/kg)							
	Day 1 (1st analysis)	Day 1 (2nd analysis)	Mean 1	Day 2 (1st analysis)	Day 2 (2nd analysis)	Mean 2	(M2-M1) M1	%
Acetamiprid	0.116	0.138	0.127	0.143	0.142	0.143	0.122	12
Buprofezin	0.176	0.153	0.165	0.160	0.165	0.163	-0.012	-1
Carbendazim	0.228	0.281	0.255	0.227	0.242	0.235	-0.079	-8
Chlorpyrifos	0.427	0.455	0.441	0.431	0.410	0.421	-0.046	-5
Cypermethrin	0.135	0.143	0.139	0.149	0.153	0.151	0.086	9
Difenoconazole	0.377	0.394	0.386	0.407	0.377	0.392	0.017	2
Endosulfan beta	0.079	0.086	0.083	0.090	0.106	0.098	0.188	19
Ethofenprox	0.209	0.238	0.224	0.279	0.216	0.248	0.107	11
Fenpropathrin	0.241	0.234	0.238	0.232	0.226	0.229	-0.036	-4
Imidacloprid	0.120	0.142	0.131	0.097	0.118	0.108	-0.179	-18
λ-cyhalothrin	0.206	0.196	0.201	0.182	0.172	0.177	-0.119	-12
Methomyl	0.114	0.118	0.116	0.093	0.102	0.098	-0.159	-16
Parathion ethyl	0.379	0.358	0.369	0.364	0.393	0.379	0.027	3
Pyridaben	0.280	0.341	0.311	0.352	0.312	0.332	0.069	7
Tebuconazole	0.355	0.429	0.392	0.324	0.373	0.349	-0.111	-11
Thiophanate Me*	0.150	0.165	0.158	0.166	0.181	0.174	0.102	10

*Only for informative purpose (homogeneity test fails).

2.5 Distribution of test item and protocol to participants

One bag of of the test item was shipped to each participant in boxes at ambient temperature. The samples were sent on 10th June 2013.

Before test item shipment, the laboratories received full instructions (The Specific Protocol) for the receipt, storage and analysis of the test items although they were encouraged to use their normal sample receipt procedure and method(s) of analysis. These instructions were uploaded onto the open site of the EURL-FV webpage as part of the Specific Protocol. The Application Form was sent to the participants by e-mail as an excel spreadsheet. The Target Pesticide List and the Minimum Required Reporting Levels (MRRLs), as established by the Organiser, were uploaded onto the EURL-FV open website.

3. STATISTICAL METHODS

3.1 False positives and negatives

3.1.1 False positives

These are results above the MRRLs that show the apparent presence of any pesticide that was listed in the Target Pesticide List, but which was: (i) not detected by the Organiser, even after repeated analyses, and (ii) not detected by most of the participating laboratories that had targeted that specific pesticide.

Results reported which were lower than the MRRL, have been disregarded and have not therefore been considered to be false positives.

No z-score values have been calculated for false positive results. Any laboratory reporting a false positive, even when reporting the necessary number of pesticides to obtain sufficient scope, has been classified into Category B.

3.1.2 False negatives

These are results for any pesticide reported by the laboratories as "analysed" but reported without numerical values, although they were detected by the Organiser and the majority of the participants that had targeted this specific pesticide, at, or above, the MRRL.

z-Scores have been calculated for all pesticides detected and reported at levels at, or above, the MRRL, including false negatives. However, these z-scores were not taken into account in assessing the 90 %, or more, of pesticides present in the sample needed to be classified into Category A.

3.2 Estimation of the assigned values

The assigned values for each pesticide were based on the median level of all the reported results, excluding outliers. Individual results without any numerical values reported, such as detected (D), were not considered. The spread of results for each pesticide was tested for multimodality.

Taking into account the regulation for robust analysis in ISO 13528[8], an uncertainty accompanied the assigned value for each pesticide, which was calculated according to the following equation:

$$u = \frac{1.25 \cdot \frac{QnRSD}{100} \cdot Median}{\sqrt{n}}$$

[8] ISO 13528:2005 "Statistical methods for use in proficiency testing by interlaboratory comparisons"

Where:

- u is the uncertainty in mg/Kg.
- Qn RSD is the robust standard deviation.
- n is the total number of laboratories reporting a result for each pesticide, excluding outliers.

3.3 Fixed target standard deviations

Based on the experience gained from previous EU proficiency tests and recommendations from the Advisory Group, a fixed relative standard deviation (FFP RSD) of 25 % was chosen[9]. This is in line with the internationally-accepted Target Measurement Uncertainty of 50 % for multiresidue analysis of pesticides[10], which is derived from, and linked to, the EUPTs.
The same target RSD has been applied to all the pesticides, independent of concentration. The target standard deviation (σ) for each individual pesticide was calculated by multiplying this FFP RSD by the assigned value. The FFP-RSD for each pesticide was compared to Qn RSD[11].

3.4 z-Scores

A z-score for each laboratory/pesticide combination was calculated according to the following equation:

$$z = (x-X) / \sigma$$

Where:

- x is the result reported by the participant, the MRRL or the RL (whichever one is lower) for those labs not having detected the presence of the pesticide in the sample.
- X is the assigned value.
- σ is the target standard deviation (the FFP-RSD of 25 % multiplied by the assigned value).

z-Score classification is as follows:

$|z| \leq 2$ Acceptable

$2 < |z| \leq 3$ Questionable

$|z| > 3$ Unacceptable

- Any z-score values of $|z| > 5$ have been reported as '5'.
- No z-score calculations have been performed for false positive results.

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[9] P. Medina-Pastor, C. Rodriguez-Torreblanca, A. Andersson, A. R. Fernandez-Alba, European Commission proficiency tests for pesticide residues in fruits and vegetables, Trends in Analytical Chemistry, 2010, 29 (1), 70-83.

[10] P.Medina Pastor, A. Valverde, T. Pihlström, S. Masselter, M. Gamón, M. Mezcua, C. Rodríguez Torreblanca, A. R. Fernández-Alba, Comparative Study of the Main Top-down Approaches for the Estimation of Measurement Uncertainty in Multiresidue Analysis of Pesticides in Fruits and Vegetables, J. Agric. Food Chem., 2011, 59 (14), 7609-7619.

[11] C. H. Muller and S. Uhlig, Estimation of variance components with high breakdown points and high efficiency, Biometrijka, 2001, 88, 353-336.

- For false negative results, the MRRL (or RL) has been used to calculate the z-score. These z-scores have also been included in the graphical representation, and are marked with an asterisk.

3.5 Combined z-scores

In order to evaluate each laboratory's overall performance according to the quality of its results and its scope, two classifications - Category A and B - were used. To be classified into Category A, laboratories had to detect (that is *sought and detected*) 90 % or more of the total number of pesticides present in the test item and report no false positives. If these two requirements were met, then the combined z-scores were calculated as the 'Average of the Squared z-scores' (AZ^2)[12].

3.5.1 The Average of the Squared z-Scores (AZ^2)

The 'Average of the Squared z-scores' was introduced for the first time in EUPT 12. This formula, analogous to the SWZ, also consists of a weighting factor ω defined as follows:

$$\omega(Z_i) = Z_i$$

But now the resultant Average of the Squared z-scores formula (AZ^2) is:

$$AZ^2 = \frac{\sum_{i=1}^{n} |Z_i| \omega(Z_i)}{n}$$

The resultant formula is the sum of the z-scores value, multiplied by itself and divided by the number of z-scores (n) detected by each laboratory, including those from false negatives.

This formula is subsequently used to produce an overall classification of laboratories with three sub-classifications: 'good', 'satisfactory' and 'unsatisfactory'.

$$|AZ^2| \leq 2 \quad \text{Good}$$
$$2 < |AZ^2| \leq 3 \quad \text{Satisfactory}$$
$$|AZ^2| > 3 \quad \text{Unsatisfactory}$$

In this way, a simple, single, combined value is also achieved, as with the previous formula. However, this time, it is more mathematically justifiable as it uses the actual z-score value rather than the factors 1, 3 and 5. Again, the aim is to encourage laboratories to not only improve the accuracy of their results but also to analyse a greater number of pesticides.

[12] P . Medina-Pastor, M. Mezcua, C. Rodríguez-Torreblanca, A. R. Fernández-Alba, Laboratory assessment by combined z-score values in proficiency tests: experience gained through the European Union proficiency tests for pesticide residues in fruits and vegetables, Anal. Bioanal. Chem., 2010, 397, 3061–3070.

Laboratories that did not detect sufficient pesticides, or reported a false positive, have been placed in Category B and no combined z-score has been calculated.

In Appendices 5 and 6, only results of laboratories in Category A have been presented, along with their graphical representations.

4. RESULTS

4.1 Summary of reported results

Fifty-four laboratories agreed to participate in this proficiency test and all but one submitted results. One laboratory (lab038) had problems with the sample reception and its results were not considered for the statistical treatment. The results reported by all the laboratories are presented in this report. However, only results reported by laboratories from EU-countries and EFTA-countries (Norway and Switzerland) have been included in the statistical treatment. The results submitted by laboratories in China, Egypt, Israel, Saudi Arabia, Serbia and Uruguay have not been included. This last group totals six laboratories, one from each country. Twenty pesticides were present in the test sample. For all of them, except for bifenthrin, endosulfan alpha, endosulfan sulphate and omethoate, statistical results have been calculated and presented in this report. In the case of bifenthrin, endosulfan alpha, endosulfan sulphate and omethoate, the pesticide MRRL was 0.020 mg/klg and the achieved concentration medians were 0.018, 0.045, 0.079 and 0.027 mg/kg, repectively. As stated in the general protocol, "In cases of the assigned value being less than a factor of 4 times the MRRL, false negatives will not be assigned as this is not statistically justifiable". For this reason, those pesticides will not be used for the laboratory evaluation. However, for informative purposes only, their histogram will be included in the Final Report. In addition, as thiophanate methyl did not pass the homogeneity test, statistical results are shown only for informative purpose. A summary of the reported results can be seen below in Table 4.1.

Table 4.1. Summary of Reported Results

Pesticides	No. of Reported Results	No. of False Negative Results	No. of Not Analysed Results	Percentage of Reported Results (out of 46)*
Acetamiprid	36	4	6	78
Bifenthrin**	19	22	5	41
Buprofezin	41	3	2	89
Carbendazim	42	0	4	91
Chlorpyrifos	43	1	2	93
Cypermethrin	31	6	9	67
Difenoconazole	40	1	5	87
Endosulfan alpha**	36	5	5	78
Endosulfan beta	38	3	5	83
Endosulfan sulfate**	39	2	5	85
Ethofenprox	36	2	8	78
Fenpropathrin	40	1	5	87
Imidacloprid	33	7	6	72
Lambda cyhalothrin	44	0	2	96
Methomyl	29	9	8	63
Omethoate**	20	19	7	43
Parathion ethyl	40	2	4	87
Pyridaben	44	1	1	96
Tebuconazole	42	1	3	91
Thiophanate methyl**	35	2	9	76

* The % of Reported Results comes from 46 laboratories.
**Only for informative purpose (median < 4MRRL or homogeneity test fails).

The laboratories that agreed to participate are listed in Annex 2. All analytical results reported by the participants are given in Appendix 3, whilst the analytical methods used are given in Appendix 7.

4.1.1 False positives

Eight laboratories reported results for additional pesticides that were not present in the test item. These pesticides, and the residue levels reported, are presented in Table 4.2. together with the MRRL. Where the reported concentration of the erroneously-detected pesticide was higher than, or equal to, the assigned MRRL value in the Target Pesticide List (Annex 1), the result has been considered as a false positive.

Two out of these eight laboratories reporting a false positive result have not been classified into Category A despite achieving sufficient scope.

Table 4.2. Laboratories that reported as 'official concentration' results for pesticides which were not present in the test item

Lab. Code	Pesticide	Concentration (mg/kg)	Determination Technique	RL (mg/Kg)	MRRL (mg/Kg)
07	Orthophenylphenol	0.020	LC-MS/MS (QQQ)	0.01	0.02
09	Penconazole	0.071	LC-MS/MS (QQQ)	0.01	0.02
11	Amitraz	0.126	LC-MS/MS (QQQ)	0.02	0.02
12	Methidathion	0.041	GC-MS/MS (QQQ)	0.01	0.02
13	Carbaryl	0.042	Other	0.02	0.02
34	Folpet	0.056	GC-MS/MS (QQQ), FPD, ECD	0.02	0.02
35	Ethoprophos	0.155	LC and GC MS/MS (QQQ)	0.01	0.02
43	Ethoprophos	0.177	LC-MS/MS (QQQ)	0.01	0.02
43	Triazophos	0.196	LC-MS/MS (QQQ)	0.01	0.02

False positives from China, Egypt and Serbia have not been included in this table.

If the residue levels reported were below the MRRLs, or if the pesticides did not appear in the pesticide target list included in Annex I, then they were not considered to be false positives.

4.1.2 False negatives

Table 4.3. summarises the results from laboratories that reported false negatives.

Table 4.3. Laboratories that failed to report pesticides which were present in the test item.

Laboratory Code	Acetamiprid	Buprofezin	Chlorpyrifos	Cypermethrin (cypermethrin incl. other mixtures of constituent isomers (sum of isomers))	Difenoconazole	Endosulfan beta	Etofenprox	Fenpropathrin	Imidacloprid	Methomyl	Parathion-ethyl	Pyridaben	Tebuconazole	Thiophanate-methyl*	False negatives in total by laboratory
03									ND	ND					2
06	ND														1
07				ND						ND				ND	3
10		ND													1
11									ND	ND					2
13			ND												1
16				ND		ND			ND	ND					4
18				ND											1
20									ND						1
22											ND	ND	ND		3
25				ND						ND					2
26										ND					1
27	ND								ND	ND	ND				4
33		ND													1
34		ND						ND							2
35							ND								1
39				ND											1
43				ND		ND	ND								3
47						ND									1
50														ND	1
51	ND								ND	ND					3
54	ND				ND				ND	ND					4
False negatives in total by pesticide	4	3	1	6	1	3	2	1	7	9	2	1	1	2	

False negatives from China, Egypt, Israel, Serbia and Uruguay have not been included in this table.
*Only for informative purpose (homogeneity test fails).

4.1.3 Distribution of data

The distributions of the concentrations of the pesticides reported by the laboratories have been plotted as histograms after removing results that were distant from the main population (results that gave rise to z-scores above 5.0 in the first round calculation) in Appendix 2.

4.2 Assigned values and target standard deviations

The assigned values were based on the median values calculated using all the reported results, but excluding those values that were far from the median, i.e. outliers. The assigned values and the uncertainty for the fifteen pesticides are presented in Table 4.4.

The target standard deviation was calculated using a fixed FFP RSD value of 25 %. For comparison, a robust standard deviation (Qn) was also calculated for informative purposes. These RSDs can be seen in Table 4.4.

Table 4.4. Median values, uncertainty and %RSDs for all pesticides present in the test item.

Pesticides	MRRL (mg/Kg)	Median (mg/Kg)	U (mg/kg)	FFP RSD (%)	Qn RSD (%)
Acetamiprid	0.02	0.108	0.007	25	31
Buprofezin	0.02	0.180	0.014	25	39
Carbendazim	0.02	0.250	0.021	25	44
Chlorpyrifos	0.02	0.364	0.021	25	35
Cypermethrin	0.02	0.112	0.011	25	44
Difenoconazole	0.02	0.367	0.018	25	25
Endosulfan beta	0.02	0.081	0.007	25	44
Ethofenprox	0.01	0.170	0.012	25	34
Fenpropathrin	0.02	0.188	0.008	25	22
Imidacloprid	0.02	0.081	0.006	25	36
Lambda cyhalothrin	0.02	0.141	0.009	25	33
Methomyl	0.02	0.089	0.007	25	32
Parathion ethyl	0.02	0.372	0.021	25	29
Pyridaben	0.02	0.268	0.017	25	32
Tebuconazole	0.02	0.338	0.017	25	26
Thiophanate methyl*	0.02	0.190	0.020	25	47

* Only for informative purpose (homogeneity test fails).

4.3 Assessment of laboratory performance

4.3.1 z-Scores

z-Scores were calculated using the FFP RSD of 25 % for all the pesticides present. In Appendix 3, the individual z-scores are presented for each laboratory, together with the median values for each pesticide. The z-scores for China, Egypt, Israel, Saudi Arabia, Serbia and Uruguay have been included in Appendix 3 but have not been considered in the following table.

Table 4.5. Classification of z-scores for the pesticides reported

Pesticides	Acceptable (%)	Questionable (%)	Unacceptable (%)
Acetamiprid	72.5	5.0	22.5
Buprofezin	79.6	6.8	13.6
Carbendazim	57.1	23.8	19.1
Chlorpyrifos	88.6	4.6	6.8
Cypermethrin	62.2	16.2	21.6
Difenoconazole	90.2	4.9	4.9
Endosulfan beta	70.7	22.0	7.3
Ethofenprox	81.6	7.9	10.5
Fenpropathrin	90.3	2.4	7.3
Imidacloprid	72.5	15.0	12.5
Lambda cyhalothrin	86.4	9.1	4.5
Methomyl	65.8	5.3	28.9
Parathion ethyl	81.0	14.2	4.8
Pyridaben	84.4	6.7	8.9
Tebuconazole	88.4	2.3	9.3
Thiophanate methyl*	59.5	5.4	35.1

*Only for informative purpose (homogeneity test fails).

z-Scores for false negative results have been calculated using the MRRL value given in the Target Pesticide List (Annex 1) or the RL value from the laboratory (whichever was lower).

In Appendix 4, graphical representations of the z-scores are presented. No z-scores have been calculated for false positive results. z-Scores for false negative results have been included on the chart and are indicated by an asterisk. The charts have been constructed using different colour bars according to the determination technique and according to the extraction method used for each particular pesticide. The z-scores for the sum of carbendazim and thiophanate methyl expressed as carbendazim have been calculated and ploted to know if the degradation of thiopahante methyl into carbendazim in diferent degrees has any repercussion on the results.

4.3.2 Combined z-scores

As previously mentioned in Section 3.5, the AZ^2 formula alone has been applied to categorise laboratories into Category A and B.

The table in Appendix 5 shows the values of individual z-scores for each pesticide and the combined 'Average of the Squared z-scores' (AZ^2) for those laboratories in Category A. In this category are the laboratories that sought and detected fourteen or more compounds and did not report any false positive results. A graphical representation of the results for these laboratories can also be found in Appendix 6.

Twenty-four of the forty-six EU and EFTA laboratories that submitted results have been classified into Category A (52 %).
From the AZ^2, 75.0 percent were classed as 'good', 20.8 percent as 'satisfactory' and 4.2 percent as 'unsatisfactory'.
Of the twenty-two laboratories in Category B, two would have been in Category A had they not reported a false positive result.

Table 4.6.1. shows the laboratories in Category A, the number of pesticides reported, the AZ^2 values and their subclassifications. Laboratories that reported false negative results in Category A are marked with an asterisk and laboratories with AZ^2 values greater than 3.0 have been marked with an '↑'.

Table 4.6.2. shows the laboratories in Category B, the number of results reported, and the number of acceptable z-scores. Laboratories reporting a false negative are marked with an asterisk and laboratories reporting a false positive are marked with a '+'.

The AZ^2 graphical representations for laboratories classified into Category A can be seen in Appendix 6. The National Reference Laboratories (NRLs) for Fruit and Vegetables have been plotted using a different colour. Also the extraction methods used are distinguished by colour. There is an extra graph showing the classification for the AZ^2 using the sum of carbendazim and thiophanate methyl expressed as carbendazim.

Table 4.6.1. Performance and Classification of laboratories in Category A using the AZ^2 formula

Lab Code	No. of z-scores achieved in total (n)	AZ^2	Classification
Lab017	15	0.2	Good
Lab001	15	0.3	Good
Lab052	15	0.3	Good
Lab008	15	0.4	Good
Lab046	15	0.5	Good
Lab004	15	0.5	Good
Lab002	15	0.8	Good
Lab032	15	1.0	Good
Lab049	15	1.0	Good

Lab Code	No. of z-scores achieved in total (n)	AZ^2	Classification
Lab023	15	1.0	Good
Lab021	15	1.1	Good
Lab026*	15	1.2	Good
Lab050	15	1.2	Good
Lab005	15	1.2	Good
Lab039*	15	1.6	Good
Lab006*	15	1.7	Good
Lab015	14	1.8	Good
Lab018*	15	2.0	Good
Lab020*	15	2.6	Satisfactory
Lab033*	15	2.8	Satisfactory
Lab024	15	2.8	Satisfactory
Lab047*	15	3.0	Satisfactory
Lab029	15	3.0	Satisfactory
Lab030↑	15	5.0	Unsatisfactory

* Laboratories reporting a false negative result.
↑ Laboratories with AZ^2 values > 3

Table 4.6.2. Performance of laboratories in Category B.

Lab Code	No. of acceptable z-scores	No. of pesticides detected	No. of total z-scores	% No. of detected z-scores / No. of pesticides present
Lab009+	13	15	15	100
Lab035*+	13	14	15	93
Lab011*+	11	13	15	87
Lab012+	10	13	13	87
Lab025*	12	13	15	87
Lab034*+	13	13	15	87
Lab007*+	9	12	14	80
Lab043*+	8	12	15	80
Lab051*	0	12	15	80
Lab016*	3	11	15	73
Lab019	8	11	11	73
Lab027*	10	11	15	73
Lab036	11	11	11	73
Lab054*	6	11	15	73
Lab013*+	8	9	10	60
Lab003*	4	9	11	60
Lab041	9	9	9	60
Lab042	5	8	8	53
Lab010*	4	6	7	40
Lab048	4	6	6	40
Lab014	3	5	5	20
Lab022*	2	4	7	13

* Laboratories reporting a false negative result.
+ Laboratories reporting a false positive result.

5. CONCLUSIONS

Fifty-four laboratories agreed to participate in EUPT-T01. Out of these, only one did not submit results. One laboratory had problems with the sample reception and its results were not considered for the statistical treatment. Six of those submitting results were not from EU or EFTA countries; therefore no statistical analysis was performed on their results.

The pesticides present in the tea test item were all incurred in the bought tea sample. Pesticides considered as positives were those which were reported by both the Organiser and the majority of participants.

For each laboratory/pesticide combination, z-scores based on the FFP RSD of 25 % have been calculated. The different chromatographic techniques used by the participant laboratories, whether gas or liquid, as well as the extraction method used, are shown in the z-score graphs. Asterisks have been used to mark each bar of the chart to represent a false negative result reported as 'ND' by a laboratory. Classification of z-score values into 'acceptable', 'questionable' or 'unacceptable' has also been undertaken.

The criterion of using the Average of Squared z-Scores formula has been used for the evaluation of the participant laboratories. Laboratories reporting fourteen or more quantitative results, and no false positive results, were considered to have sufficient scope and were therefore classified into Category A. Laboratories in Category A were also classed as 'good', 'satisfactory' or 'unsatisfactory'. Laboratories reporting false negatives were marked with an asterisk and those obtaining an AZ^2 value greater than 3 were marked with a '↑'.

Those laboratories that reported less than fourteen results were considered as having insufficient scope and were automatically classified into Category B, together with those reporting one, or more, false positive results. These laboratories have been categorised depending on the number of pesticides detected and quantified out of the total (fifteen). Laboratories reporting false negatives were marked with an asterisk. Laboratories having reported a false positive have been marked with a '+'.

The median value for each pesticide was used as the assigned value or "true" concentration, which was also used to calculate the z-scores. Results were required from the laboratories not only for the pesticides, as defined by the MRL definition, but also for all the individual components that are included in the MRL definition.

The difficulties of this matrix type have been evaluated as a consequence of the large amount of coextractive natural components provoking higher dispersion (Qn) than in most fruits and vegetables. For carbendazim, cypermetryn, endosulfan beta and thiophanate methyl (the Qn %RSDs of 44, 44, 44 and 47%, respectively) are notable. Forcarbendazim and thiophanate methyl, the results were influenced by the different degrees of degradation obtained by participants during the sample handling - as is shown in the z-scores graphs. The values obtained by

combining carbendazim and thiophanate methyl results give rise to a lower Qn and less unacceptable results than for the individual pesticides (figure on page 63).

The Qn %RSDs and the median values for the results reported by QuEChERS, without calcium chloride addition (50% of the laboratories used QuEChERS), have been calculated separately. The results were compared with the Qn and median values obtained from all the data (the whole data set received from this PT). Both medians are quite similar, but the dispersion accounted for as Qn is higher in the QuEChERS method (the average Qn for QuEChERS was 41% whereas for the combined data, it was 34%).

One compound did not pass the homogeneity test (thiophanate methyl). This fact is clearly as a consequence of some degradation to carbendazim.

Overall, the results can be considered to be good with regard to the z-scores for each pesticide present in the test item. For the majority of the pesticides, a low number of unacceptable results were obtained in terms of z-scores, except for acetamiprid, carbendazim, cypermethrin, methomyl and thiophanate-methyl (22.5, 19.1, 21.6, 28.9 and 35.1%, respectively).

It would appear that multiresidue methods such as QuEChERS have improved following modification. However, the small population of those results obtained from modified QuEChERS with calcium chloride and the large population of laboratories employing QuECHERS methodologies does not allow us to draw definitive conclusions. On the other hand, all of the laboratories applying the miniLuke method obtained acceptable results in terms of z-scores, except for carbendazim.

6. ACKNOWLEDGEMENTS

The Organiser is most grateful to the European Commission for funding this European Proficiency Test in Tea 01.

The Organiser wishes to thank the members of the Quality Control Group and the Scientific Committee for their invaluable expert advice. Many thanks also to the Statistical Group for their cooperation.

The Organiser wishes to give a special thank-you to the University of Almeria for the use of their facilities.

APPENDIX 1. Homogeneity data.

Acetamiprid (mg/Kg)		Buprofezin (mg/Kg)		Carbendazim (mg/Kg)		Chlorpyrifos (mg/Kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.140	0.166	0.151	0.389	0.264	0.362	0.415	0.220
0.150	0.150	0.254	0.388	0.315	0.270	0.596	0.415
0.149	0.157	0.195	0.173	0.257	0.360	0.410	0.540
0.146	0.179	0.211	0.191	0.300	0.310	0.363	0.436
0.135	0.162	0.224	0.207	0.219	0.338	0.471	0.589
0.125	0.136	0.173	0.274	0.189	0.225	0.516	0.325
0.142	0.094	0.188	0.133	0.231	0.206	0.439	0.447
0.213	0.148	0.182	0.200	0.360	0.324	0.428	0.461
0.162	0.124	0.122	0.243	0.203	0.229	0.502	0.580
0.148	0.143	0.189	0.170	0.201	0.252	0.618	0.546

Cypermethrin (mg/Kg)		Difenoconazole (mg/Kg)		Endosulfan beta (mg/Kg)		Ethofenprox (mg/Kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.158	0.155	0.385	0.403	0.072	0.089	0.219	0.265
0.125	0.122	0.375	0.272	0.118	0.098	0.276	0.214
0.157	0.155	0.320	0.481	0.121	0.109	0.257	0.219
0.140	0.155	0.496	0.343	0.086	0.126	0.196	0.257
0.157	0.156	0.462	0.371	0.079	0.093	0.244	0.198
0.122	0.128	0.311	0.387	0.097	0.082	0.258	0.281
0.146	0.149	0.190	0.268	0.100	0.087	0.214	0.211
0.125	0.130	0.375	0.385	0.115	0.085	0.228	0.195
0.157	0.159	0.433	0.469	0.078	0.070	0.232	0.259
0.154	0.157	0.478	0.393	0.142	0.071	0.194	0.268

Fenpropathrin (mg/Kg)		Imidacloprid (mg/Kg)		Lambda Cyhalothrin (mg/Kg)		Methomyl (mg/Kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.204	0.287	0.110	0.098	0.144	0.177	0.111	0.123
0.251	0.206	0.105	0.119	0.217	0.153	0.143	0.125
0.251	0.233	0.124	0.112	0.164	0.183	0.134	0.113
0.218	0.283	0.129	0.121	0.180	0.163	0.083	0.096
0.229	0.218	0.093	0.083	0.175	0.140	0.104	0.129
0.293	0.255	0.100	0.109	0.208	0.205	0.079	0.084
0.233	0.265	0.123	0.105	0.169	0.163	0.127	0.122
0.280	0.265	0.096	0.081	0.197	0.157	0.123	0.074
0.333	0.301	0.113	0.113	0.164	0.234	0.101	0.131
0.206	0.344	0.099	0.124	0.128	0.177	0.114	0.135

APPENDIX 1. Homogeneity data.

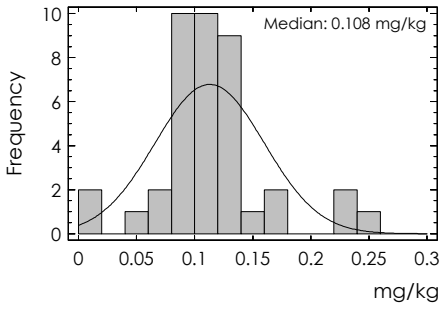
Parathion ethyl (mg/Kg)		Pyridaben (mg/Kg)		Tebuconazole (mg/Kg)		Thiophanate methyl (mg/Kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.315	0.469	0.294	0.361	0.462	0.498	0.151	0.260
0.414	0.395	0.395	0.313	0.412	0.455	0.182	0.143
0.510	0.434	0.349	0.321	0.348	0.577	0.154	0.301
0.362	0.470	0.300	0.365	0.439	0.414	0.165	0.235
0.537	0.384	0.344	0.284	0.326	0.367	0.299	0.328
0.617	0.516	0.398	0.405	0.342	0.360	0.325	0.477
0.447	0.334	0.332	0.317	0.366	0.341	0.321	0.358
0.451	0.403	0.361	0.290	0.466	0.719	0.236	0.212
0.416	0.427	0.316	0.374	0.378	0.388	0.197	0.183
0.353	0.463	0.281	0.365	0.431	0.372	0.335	0.325

The sample numbers used for this test were: 002, 037, 032, 062, 016, 059, 053, 070, 065 and 034.

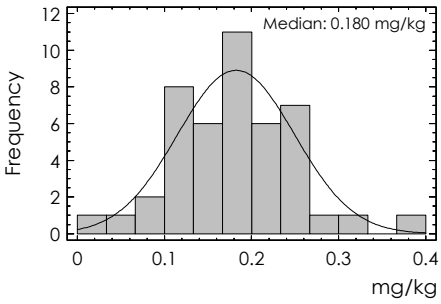
APPENDIX 2. Histograms of residue data for each pesticide from all the laboratories.

Results presented as histograms.

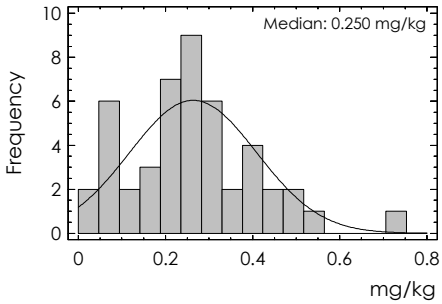
Acetamiprid



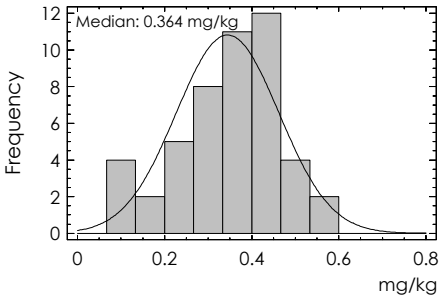
Buprofezin



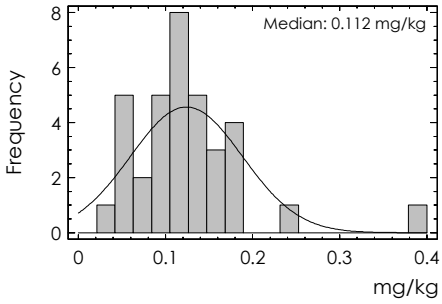
Carbendazim



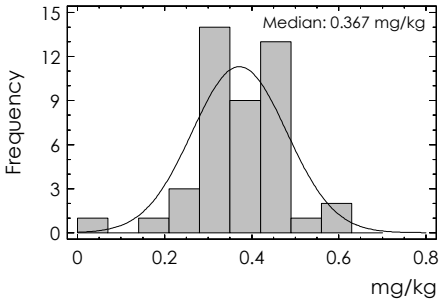
Chlorpyrifos



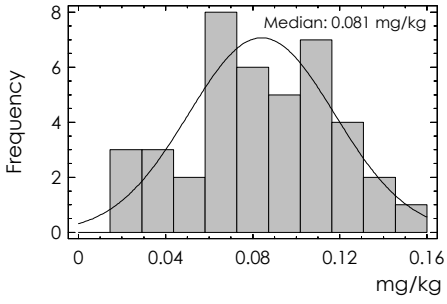
Cypermethrin



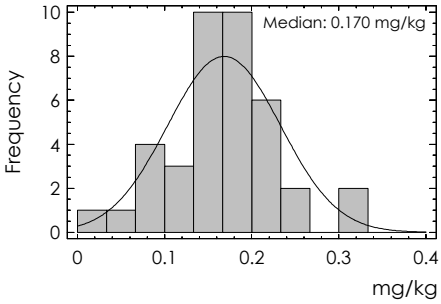
Difenoconazole



Endosulfan beta

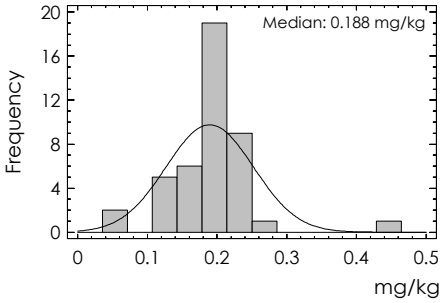


Etofenprox

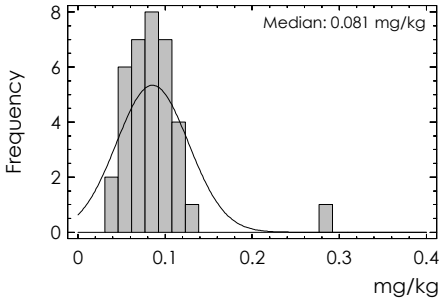


APPENDIX 2. Histograms of residue data for each pesticide from all the laboratories.

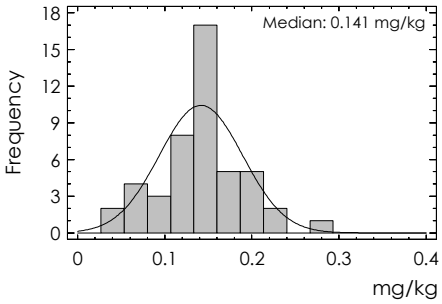
Fenpropathrin



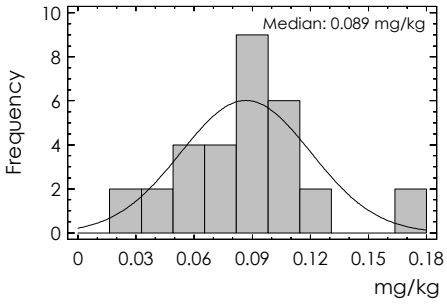
Imidacloprid



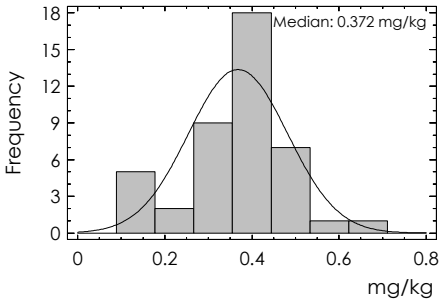
Lambda-Cyhalothrin



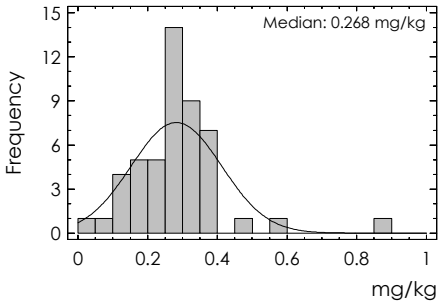
Methomyl



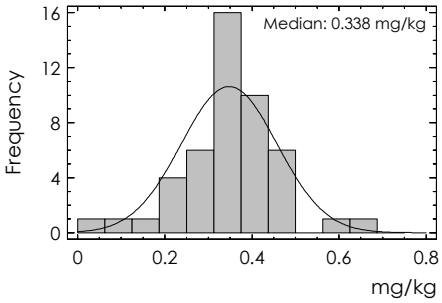
Parathion-ethyl



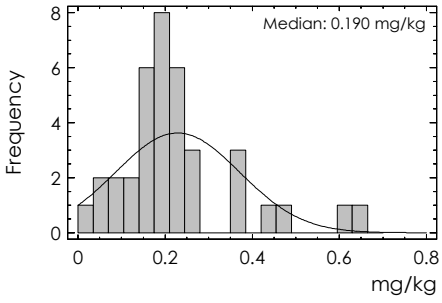
Pyridaben



Tebuconazole



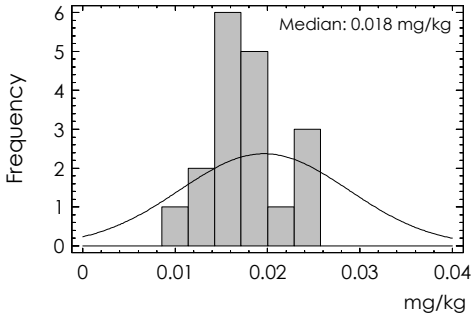
Thiophanate-methyl



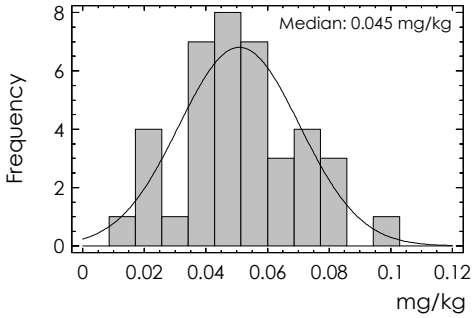
APPENDIX 2. Histograms of residue data for each pesticide from all the laboratories.

For informative purposes only (median < 4MRRL).

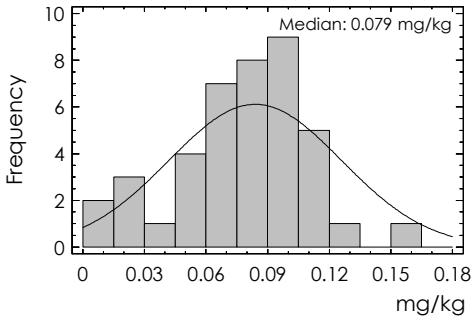
Bifenthrin



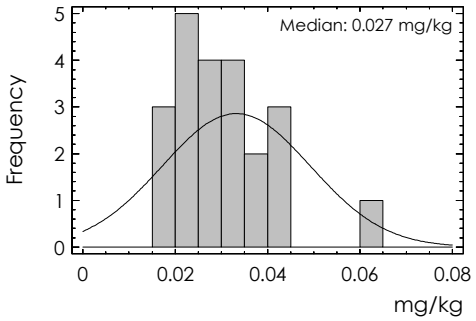
Endosulfan alpha



Endosulfan sulfate



Omethoate



APPENDIX 3. Results (mg/kg) and z-scores for FFP RSD (25 %).

Results given by the laboratories (mg/kg) and their calculated z-score value using FFP RSD 25 %

Lab Code	Acetamiprid	z-Score (FFP RSD 25%)	Buprofezin	z-Score (FFP RSD 25%)	Carbendazim	z-Score (FFP RSD 25%)	Chlorpyrifos	z-Score (FFP RSD 25%)	Cypermethrin	z-Score (FFP RSD 25%)	Difenoconazole	z-Score (FFP RSD 25%)	Endosulfan beta	z-Score (FFP RSD 25%)	Etofenprox	z-Score (FFP RSD 25%)
MRRL (mg/kg)	**0.02**		**0.02**		**0.02**		**0.02**		**0.02**		**0.02**		**0.02**		**0.01**	
Median (mg/kg)	**0.108**		**0.180**		**0.250**		**0.364**		**0.112**		**0.367**		**0.081**		**0.170**	
01	0.120	0.4	0.176	-0.1	0.292	0.7	0.364	0.0	0.095	-0.6	0.362	0.0	0.102	1.1	0.157	-0.3
02	0.133	0.9	0.186	0.1	0.321	1.1	0.356	-0.1	0.109	-0.1	0.427	0.7	0.130	2.5	0.203	0.8
03	0.010	-3.6	0.090	-2.0	0.090	-2.6	0.230	-1.5	0.060	-1.8	NA		0.030	-2.5	NA	
04	0.103	-0.2	0.159	-0.5	0.263	0.2	0.318	-0.5	0.147	1.3	0.300	-0.7	0.062	-0.9	0.170	0.0
05	0.114	0.2	0.144	-0.8	0.266	0.3	0.327	-0.4	0.124	0.4	0.432	0.8	0.079	-0.1	0.311	3.4
06	ND	-3.3	0.120	-1.3	0.267	0.3	0.301	-0.7	0.111	0.0	0.407	0.5	0.055	-1.3	0.154	-0.4
07	0.093	-0.6	0.061	-2.6	0.230	-0.3	0.380	0.2	ND	-3.6	0.250	-1.2	0.025	-2.8	0.200	0.7
08	0.120	0.4	0.180	0.0	0.240	-0.2	0.410	0.5	0.092	-0.7	0.380	0.2	0.065	-0.8	0.150	-0.4
09	0.143	1.3	0.255	1.7	0.330	1.3	0.420	0.6	0.181	2.5	0.450	1.0	0.111	1.6	0.238	1.6
10	NA		ND	-3.6	0.061	-3.0	0.067	-3.3	NA		0.280	-0.9	NA		NA	
11	0.051	-2.1	0.133	-1.0	0.105	-2.3	0.179	-2.0	0.080	-1.1	0.215	-1.6	0.067	-0.7	0.169	0.0
12	0.090	-0.7	0.370	4.2	0.300	0.8	0.385	0.3	NA		0.600	2.6	0.051	-1.5	0.229	1.4
13	NA		0.210	0.7	NA		ND	-3.8	0.160	1.7	0.440	0.9	0.098	0.9	NA	
14	0.243	5.0	0.158	-0.5	0.410	2.6	NA		NA		NA		NA		NA	
15	0.112	0.1	0.220	0.9	0.119	-2.1	0.440	0.9	NA		0.470	1.2	0.123	2.2	0.051	-2.8
16	0.013	-3.5	0.110	-1.6	0.042	-3.3	0.140	-2.5	ND	-3.3	0.340	-0.2	ND	-3.0	NA	
17	0.113	0.2	0.184	0.1	0.247	0.0	0.395	0.4	0.080	-1.1	0.371	0.1	0.075	-0.3	0.155	-0.3
18	0.096	-0.4	0.160	-0.4	0.230	-0.3	0.410	0.5	ND	-3.3	0.250	-1.2	0.110	1.5	0.160	-0.2
19	0.222	4.2	0.241	1.4	0.488	3.8	NA		NA		0.350	-0.1	NA		0.191	0.5
20	0.105	-0.1	0.121	-1.3	0.079	-2.7	0.235	-1.4	0.046	-2.3	0.315	-0.5	0.032	-2.4	0.085	-2.0
21	0.113	0.2	0.118	-1.4	0.309	0.9	0.478	1.3	0.114	0.1	0.404	0.5	0.065	-0.8	0.152	-0.4
22	NA		NA		0.082	-2.7	0.068	-3.2	NA		NA		0.040	-2.0	NA	
23	0.133	0.9	0.222	0.9	0.393	2.3	0.514	1.7	0.106	-0.2	0.475	1.2	0.111	1.6	0.190	0.5
24	0.108	0.0	0.236	1.2	0.519	4.3	0.538	1.9	0.144	1.2	0.467	1.2	0.118	1.9	0.230	1.4
25	0.120	0.4	0.200	0.4	0.260	0.2	0.400	0.4	ND	-3.3	0.280	-0.9	0.120	2.0	0.072	-2.3
26	0.116	0.3	0.185	0.1	0.204	-0.7	0.471	1.2	0.128	0.6	0.412	0.6	0.089	0.5	0.188	0.4
27	ND	-3.6	0.200	0.4	0.078	-2.8	0.360	0.0	0.150	1.4	0.320	-0.5	0.082	0.1	0.180	0.3
28	0.094	-0.5	0.115	-1.4	0.188	-1.0	0.319	-0.5	0.122	0.4	0.307	-0.6	0.101	1.1	0.117	-1.2
29	0.166	2.1	0.274	2.1	0.469	3.5	0.538	1.9	0.189	2.8	0.448	1.0	0.091	0.6	0.205	0.9
30	0.233	4.6	0.325	3.2	0.257	0.1	0.410	0.5	0.242	4.7	0.553	2.1	0.145	3.3	0.264	2.2
31	ND	-3.3	ND	-3.6	NA		0.330	-0.4	ND	-3.3	0.420	0.6	ND	-3.0	NA	
32	0.105	-0.1	0.236	1.2	0.283	0.5	0.459	1.1	0.136	0.9	0.432	0.8	0.083	0.2	0.233	1.5
33	0.085	-0.9	ND	-3.6	0.226	-0.4	0.446	0.9	0.047	-2.3	0.312	-0.6	0.140	3.0	0.103	-1.6
34	0.082	-1.0	ND	-3.6	0.250	0.0	0.427	0.7	0.105	-0.2	0.338	-0.3	0.083	0.2	0.162	-0.2
35	0.087	-0.8	0.158	-0.5	0.402	2.4	0.411	0.5	0.112	0.0	0.338	-0.3	0.104	1.2	ND	-3.8
36	NA		0.120	-1.3	NA		0.290	-0.8	0.061	-1.8	0.320	-0.5	0.065	-0.8	0.120	-1.2
37	No results submitted															
38	NA		0.083	-2.2	NA		0.193	-1.9	0.057	-2.0	0.155	-2.3	0.047	-1.7	0.089	-1.9
39	0.072	-1.3	0.170	-0.2	0.230	-0.3	0.340	-0.2	ND	-3.3	0.280	-0.9	0.110	1.5	0.170	0.0
40	0.170	2.3	0.260	1.8	0.420	2.7	0.430	0.8	0.090	-0.8	0.480	1.3	0.150	3.5	0.330	3.8

APPENDIX 3. Results (mg/Kg) and z-scores for FFP RSD (25%).

Lab Code	Acetamiprid	z-Score (FFP RSD 25%)	Buprofezin	z-Score (FFP RSD 25%)	Carbendazim	z-Score (FFP RSD 25%)	Chlorpyrifos	z-Score (FFP RSD 25%)	Cypermethrin	z-Score (FFP RSD 25%)	Difenoconazole	z-Score (FFP RSD 25%)	Endosulfan beta	z-Score (FFP RSD 25%)	Etofenprox	z-Score (FFP RSD 25%)
MRRL (mg/kg)	**0.02**		**0.02**		**0.02**		**0.02**		**0.02**		**0.02**		**0.02**		**0.01**	
Median (mg/kg)	**0.108**		**0.180**		**0.250**		**0.364**		**0.112**		**0.367**		**0.081**		**0.170**	
41	0.064	-1.6	0.120	-1.3	0.150	-1.6	0.260	-1.1	NA		0.380	0.2	NA		NA	
42	NA		0.260	1.8	NA		0.320	-0.5	NA		NA		0.080	0.0	NA	
43	0.100	-0.3	0.223	1.0	0.043	-3.3	0.428	0.7	ND	-3.3	0.601	2.6	ND	-3.5	ND	-3.8
44	0.124	0.6	0.226	1.0	0.491	3.9	0.484	1.3	0.178	2.4	0.152	-2.3	0.095	0.8	ND	-3.8
45	NA		0.190	0.2	0.150	-1.6	NA		0.380	5.0	NA		NA		NA	
46	0.108	0.0	0.149	-0.7	0.315	1.0	0.340	-0.2	0.149	1.3	0.336	-0.3	0.108	1.4	0.189	0.5
47	0.135	1.0	0.237	1.3	0.732	5.0	0.384	0.2	0.119	0.3	0.472	1.2	ND	-3.0	0.185	0.4
48	NA		NA		NA		0.270	-1.0	NA		NA		NA		0.090	-1.9
49	0.090	-0.7	0.190	0.2	0.190	-1.0	0.260	-1.1	0.180	2.5	0.360	0.0	0.068	-0.6	0.140	-0.7
50	0.123	0.6	0.223	1.0	0.453	3.2	0.428	0.7	0.128	0.6	0.450	1.0	0.072	-0.4	0.231	1.5
51	ND	-3.6	0.027	-3.4	0.052	-3.2	0.110	-2.8	0.026	-3.1	0.019	-3.8	0.018	-3.1	0.029	-3.3
52	0.083	-0.9	0.180	0.0	0.250	0.0	0.350	-0.1	0.100	-0.4	0.360	0.0	0.066	-0.7	0.150	-0.4
53	0.125	0.6	ND	-3.8	0.366	1.9	0.110	-2.8	ND	-3.6	ND	-3.9	ND	-3.5	0.068	-2.4
54	ND	-3.3	0.077	-2.3	0.235	-0.2	0.208	-1.7	0.049	-2.2	ND	-3.8	0.028	-2.6	0.140	-0.7

APPENDIX 3. Results (mg/kg) and z-scores for FFP RSD (25 %).

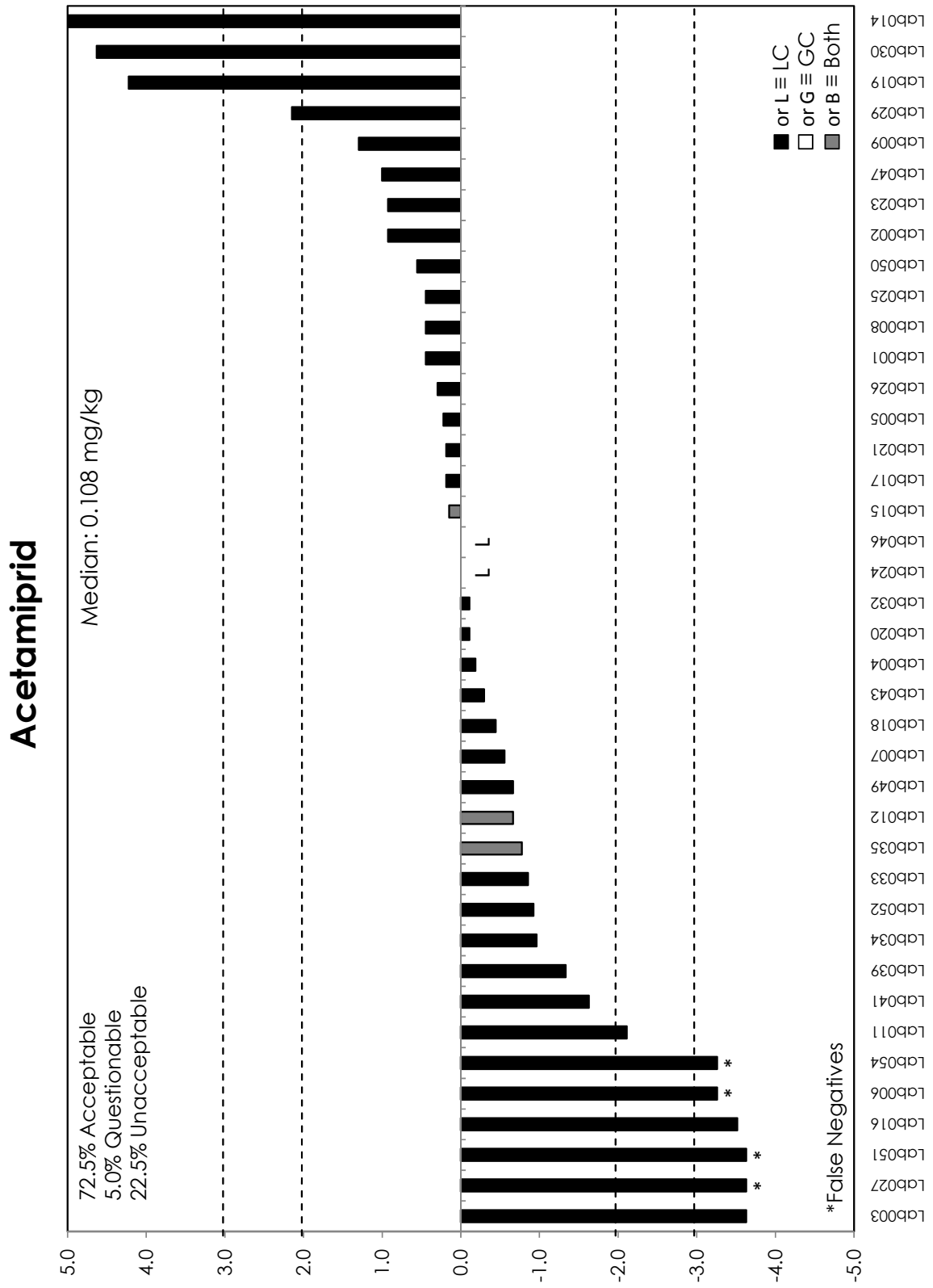
Lab Code	Fenpropathrin	z-Score (FFP RSD 25%)	Imidacloprid	z-Score (FFP RSD 25%)	Lambda Cyhalothrin	z-Score (FFP RSD 25%)	Methomyl	z-Score (FFP RSD 25%)	Parathion ethyl	z-Score (FFP RSD 25%)	Pyridaben	z-Score (FFP RSD 25%)	Tebuconazole	z-Score (FFP RSD 25%)	Thiophanate methyl*	z-Score (FFP RSD 25%)
MRRL (mg/kg)	**0.02**		**0.02**		**0.02**		**0.02**		**0.02**		**0.02**		**0.02**		**0.02**	
Median (mg/kg)	**0.188**		**0.081**		**0.141**		**0.089**		**0.372**		**0.268**		**0.338**		**0.190**	
01	0.159	-0.6	0.108	1.4	0.136	-0.1	0.089	0.0	0.397	0.3	0.259	-0.1	0.364	0.3	0.178	-0.2
02	0.211	0.5	0.101	1.0	0.137	-0.1	0.109	0.9	0.358	-0.1	0.306	0.6	0.400	0.8	0.229	0.8
03	NA		ND	-3.5	0.080	-1.7	ND	-3.6	0.150	-2.4	0.120	-2.2	NA		NA	
04	0.203	0.4	0.095	0.7	0.134	-0.2	0.066	-1.0	0.238	-1.4	0.246	-0.3	0.280	-0.7	0.132	-1.2
05	0.179	-0.2	0.102	1.1	0.134	-0.2	0.089	0.0	0.328	-0.5	0.388	1.8	0.419	1.0	0.377	4.0
06	0.147	-0.8	0.054	-1.3	0.142	0.1	0.083	-0.3	0.235	-1.5	0.175	-1.4	0.184	-1.8	0.238	1.0
07	0.190	0.1	0.035	-2.3	0.160	0.6	ND	-3.6	0.360	-0.1	NA		0.300	-0.4	ND	-3.8
08	0.110	-1.6	0.083	0.1	0.140	0.0	0.110	0.9	0.400	0.3	0.270	0.0	0.350	0.2	0.190	0.0
09	0.220	0.7	0.124	2.2	0.188	1.4	0.084	-0.2	0.450	0.8	0.333	1.0	0.430	1.1	0.260	1.5
10	NA		NA		0.120	-0.6	NA		NA		0.150	-1.8	0.320	-0.2	NA	
11	0.135	-1.1	ND	-3.0	0.102	-1.1	ND	-3.1	0.296	-0.8	0.227	-0.6	0.195	-1.7	0.168	-0.5
12	0.215	0.6	0.050	-1.5	0.055	-2.4	0.077	-0.5	NA		0.304	0.5	0.203	-1.6	0.450	5.0
13	0.180	-0.1	NA		0.160	0.6	NA		0.550	1.9	0.460	2.9	0.420	1.0	NA	
14	NA		0.048	-1.6	NA		NA		NA		0.268	0.0	NA		0.175	-0.3
15	0.150	-0.8	0.076	-0.2	0.129	-0.3	0.058	-1.4	0.522	1.6	0.297	0.4	0.340	0.0	0.660	5.0
16	0.045	-3.0	ND	-3.5	0.028	-3.2	ND	-3.6	0.160	-2.3	0.058	-3.1	0.320	-0.2	0.110	-1.7
17	0.176	-0.2	0.096	0.8	0.138	-0.1	0.105	0.7	0.324	-0.5	0.265	0.0	0.360	0.3	0.193	0.1
18	0.200	0.3	0.052	-1.4	0.220	2.3	0.028	-2.7	0.400	0.3	0.260	-0.1	0.290	-0.5	0.170	-0.4
19	0.182	-0.1	0.286	5.0	0.162	0.6	NA		0.424	0.6	0.294	0.4	0.374	0.5	0.356	3.5
20	0.185	0.0	ND	-3.0	0.111	-0.8	0.095	0.3	0.361	-0.1	0.193	-1.1	0.395	0.7	0.175	-0.3
21	0.175	-0.2	0.066	-0.7	0.157	0.5	0.020	-3.1	0.414	0.5	0.316	0.7	0.331	-0.1	0.174	-0.3
22	NA		NA		0.087	-1.5	NA		ND	-3.8	ND	-3.7	ND	-3.8	NA	
23	0.183	-0.1	0.099	0.9	0.119	-0.6	0.102	0.6	0.363	-0.1	0.283	0.2	0.400	0.8	0.262	1.5
24	0.250	1.4	0.110	1.5	0.183	1.2	0.109	0.9	0.419	0.5	0.397	1.9	0.353	0.2	0.086	-2.2
25	0.230	0.9	0.050	-1.5	0.076	-1.8	ND	-3.1	0.400	0.3	0.360	1.4	0.250	-1.0	0.150	-0.8
26	0.213	0.6	0.090	0.5	0.194	1.5	ND	-3.1	0.492	1.3	0.307	0.6	0.313	-0.3	0.218	0.6
27	0.190	0.1	ND	-3.5	0.160	0.6	ND	-3.8	ND	-3.9	0.300	0.5	0.285	-0.6	0.020	-3.6
28	0.180	-0.1	0.084	0.2	0.117	-0.7	0.169	3.6	0.400	0.3	0.275	0.1	0.441	1.3	0.194	0.1
29	0.242	1.2	0.112	1.6	0.183	1.2	0.100	0.5	0.450	0.8	0.361	1.4	0.491	1.8	0.089	-2.1
30	0.279	2.0	0.095	0.7	0.214	2.1	0.164	3.4	0.324	-0.5	0.316	0.7	0.447	1.3	0.620	5.0
31	NA		NA		0.150	0.3	NA		NA		NA		0.290	-0.5	NA	
32	0.225	0.8	0.091	0.5	0.204	1.8	0.116	1.2	0.439	0.7	0.345	1.1	0.408	0.9	0.231	0.9
33	0.192	0.1	0.039	-2.1	0.142	0.1	0.096	0.3	0.168	-2.2	0.207	-0.9	0.279	-0.7	0.039	-3.2
34	ND	-3.6	0.091	0.5	0.131	-0.3	0.095	0.3	0.402	0.3	0.301	0.5	0.336	0.0	0.482	5.0
35	0.228	0.9	0.078	-0.1	0.142	0.1	0.075	-0.6	0.334	-0.4	0.181	-1.3	0.313	-0.3	0.189	0.0
36	0.130	-1.2	NA		0.069	-2.0	NA		0.380	0.1	0.160	-1.6	0.190	-1.7	NA	
37	No results submitted															
38	0.081	-2.3	NA		0.056	-2.4	NA		0.156	-2.3	0.143	-1.9	0.153	-2.2	NA	
39	0.210	0.5	0.067	-0.7	0.190	1.4	0.038	-2.3	0.330	-0.4	0.250	-0.3	0.330	-0.1	0.180	-0.2
40	0.240	1.1	0.097	0.8	0.270	3.7	ND	-3.8	ND	-3.8	0.590	4.8	0.440	1.2	0.180	-0.2
41	NA		0.063	-0.9	NA		0.130	1.8	NA		0.260	-0.1	0.320	-0.2	NA	
42	0.450	5.0	NA		0.100	-1.1	NA		0.510	1.5	0.860	5.0	0.650	3.7	NA	
43	0.188	0.0	0.048	-1.6	0.280	4.0	0.048	-1.8	0.492	1.3	0.359	1.4	0.605	3.2	6.200	5.0

APPENDIX 3. Results (mg/Kg) and z-scores for FFP RSD (25%).

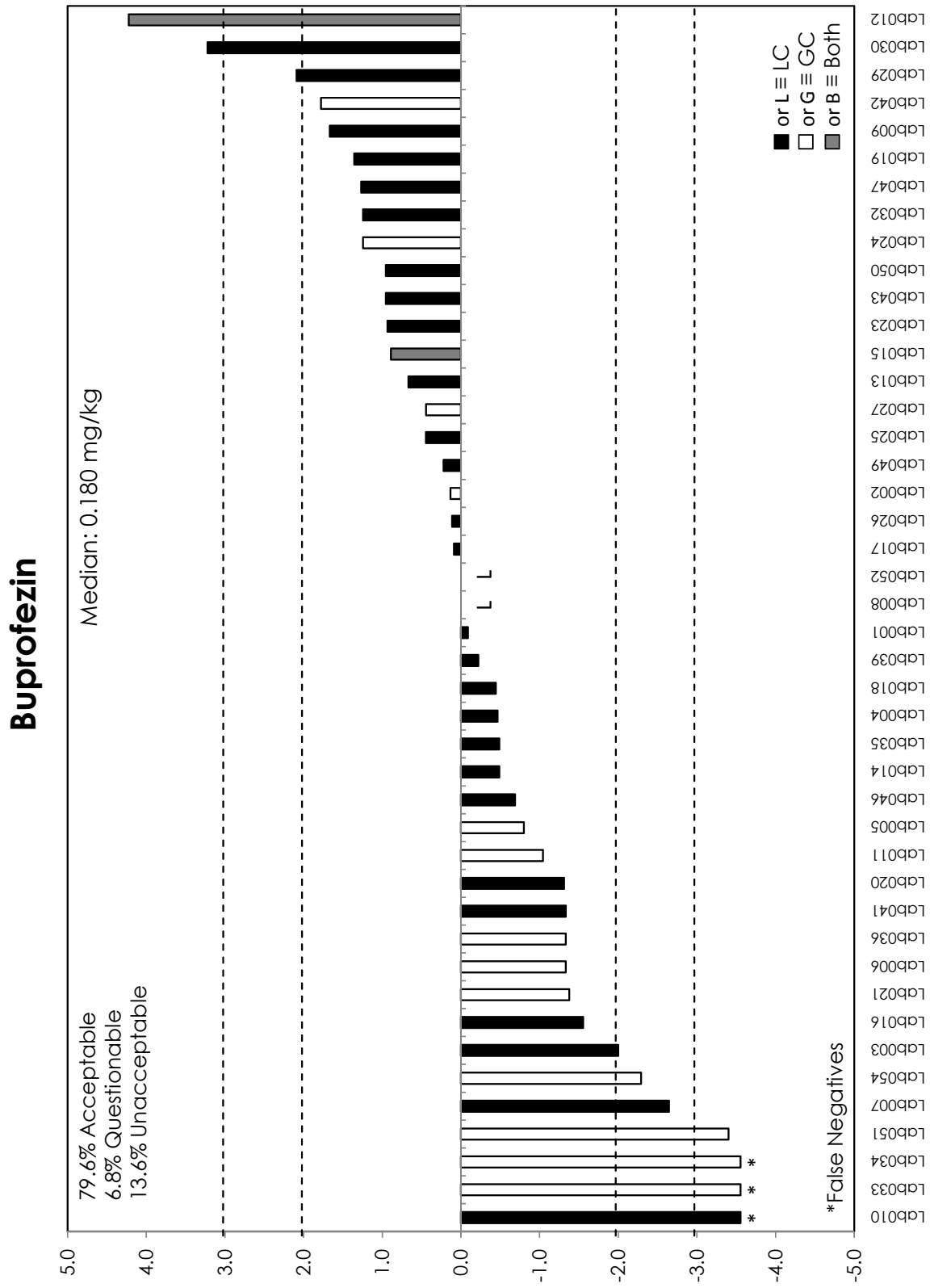
Lab Code	Fenpropathrin	z-Score (FFP RSD 25%)	Imidacloprid	z-Score (FFP RSD 25%)	Lambda Cyhalothrin	z-Score (FFP RSD 25%)	Methomyl	z-Score (FFP RSD 25%)	Parathion ethyl	z-Score (FFP RSD 25%)	Pyridaben	z-Score (FFP RSD 25%)	Tebuconazole	z-Score (FFP RSD 25%)	Thiophanate methyl*	z-Score (FFP RSD 25%)
MRRL (mg/kg)	**0.02**		**0.02**		**0.02**		**0.02**		**0.02**		**0.02**		**0.02**		**0.02**	
Median (mg/kg)	**0.188**		**0.081**		**0.141**		**0.089**		**0.372**		**0.268**		**0.338**		**0.190**	
44	0.214	0.6	0.091	0.5	0.209	2.0	ND	-3.1	0.510	1.5	0.362	1.4	0.482	1.7	ND	-3.6
45	NA		NA		0.520	5.0	0.060	-1.3	0.410	0.4	0.270	0.0	0.390	0.6	0.210	0.4
46	0.189	0.1	0.088	0.4	0.171	0.9	0.091	0.1	0.307	-0.7	0.257	-0.2	0.331	-0.1	0.214	0.5
47	0.170	-0.4	0.109	1.4	0.148	0.2	0.093	0.2	0.418	0.5	0.376	1.6	0.438	1.2	0.380	4.0
48	0.130	-1.2	NA		0.120	-0.6	NA		0.650	3.0	0.120	-2.2	NA		NA	
49	0.190	0.1	0.067	-0.7	0.180	1.1	0.060	-1.3	0.280	-1.0	0.320	0.8	0.380	0.5	0.190	0.0
50	0.228	0.9	0.064	-0.8	0.150	0.3	0.076	-0.6	0.340	-0.3	0.268	0.0	0.400	0.8	ND	-3.8
51	0.039	-3.2	ND	-3.5	0.039	-2.9	ND	-3.6	0.130	-2.6	0.039	-3.4	0.018	-3.8	NA	
52	0.180	-0.1	0.070	-0.5	0.150	0.3	0.054	-1.6	0.380	0.1	0.230	-0.6	0.360	0.3	0.250	1.3
53	ND	-3.8	ND	-3.5	0.660	5.0	ND	-3.6	ND	-3.9	0.128	-2.1	ND	-3.9	ND	-3.8
54	0.115	-1.5	ND	-3.0	0.121	-0.5	ND	-3.1	0.135	-2.5	0.168	-1.5	0.124	-2.5	0.043	-3.1

*For informative purpose only (homogeneity test fails).

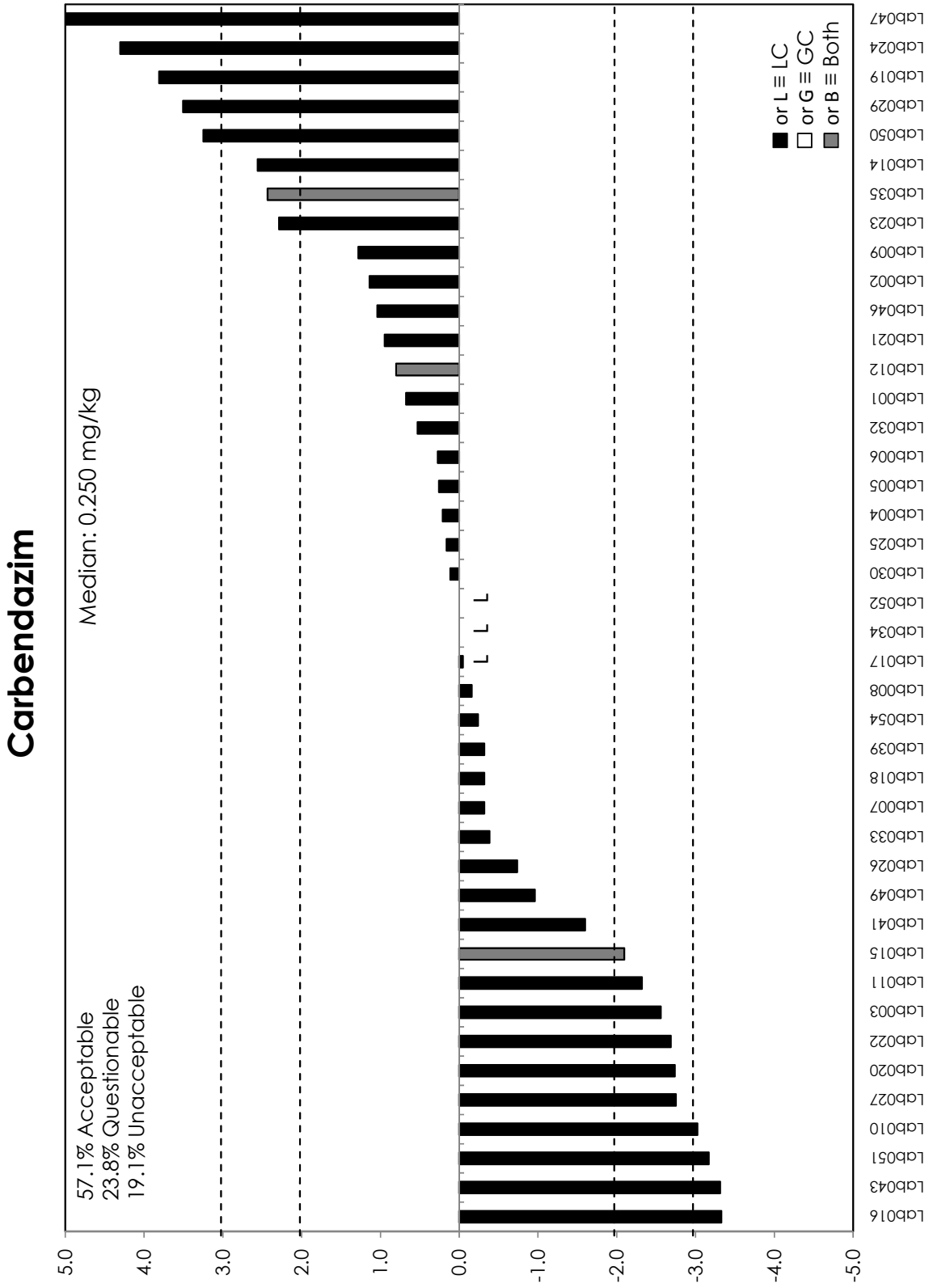
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



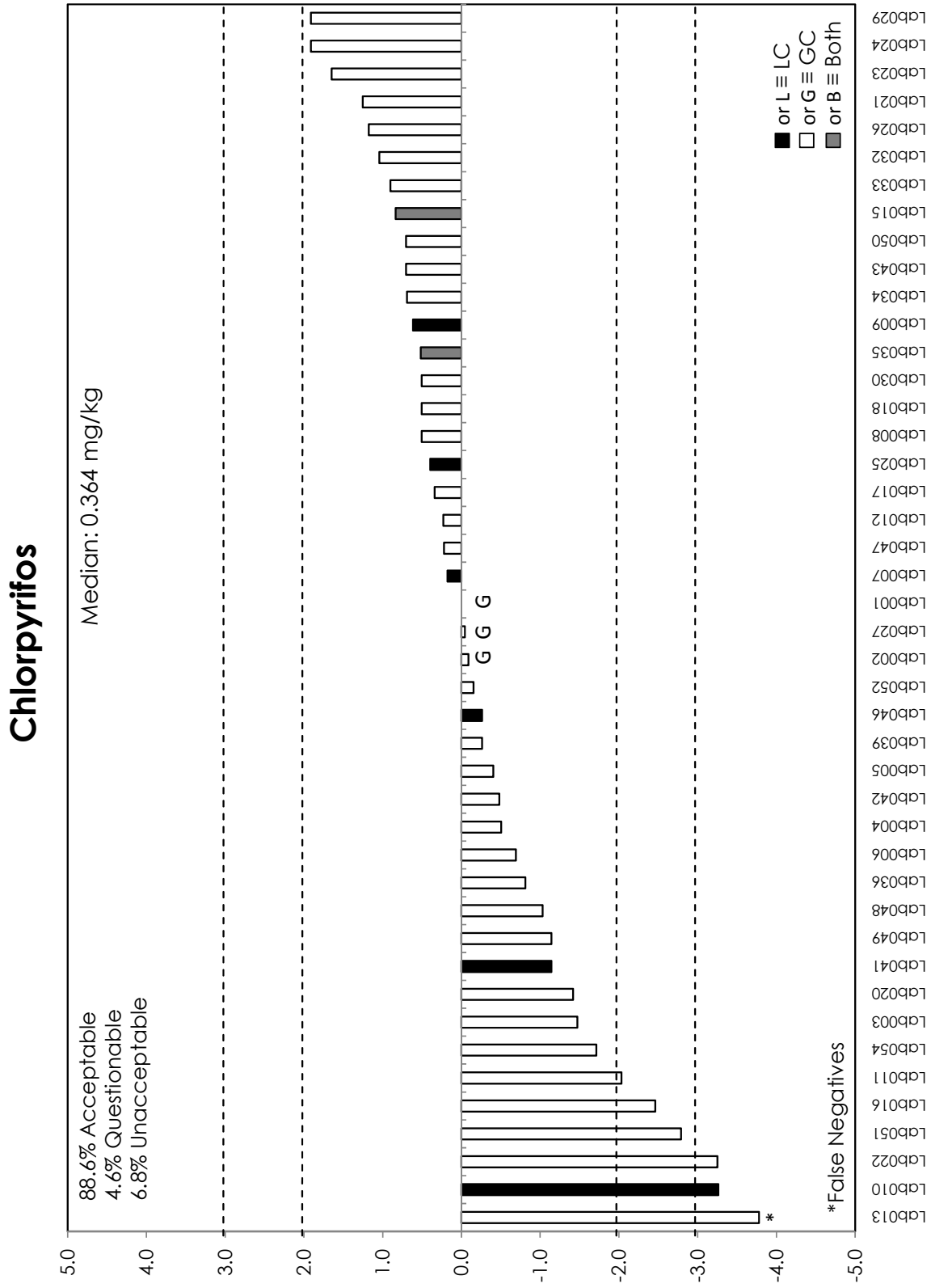
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



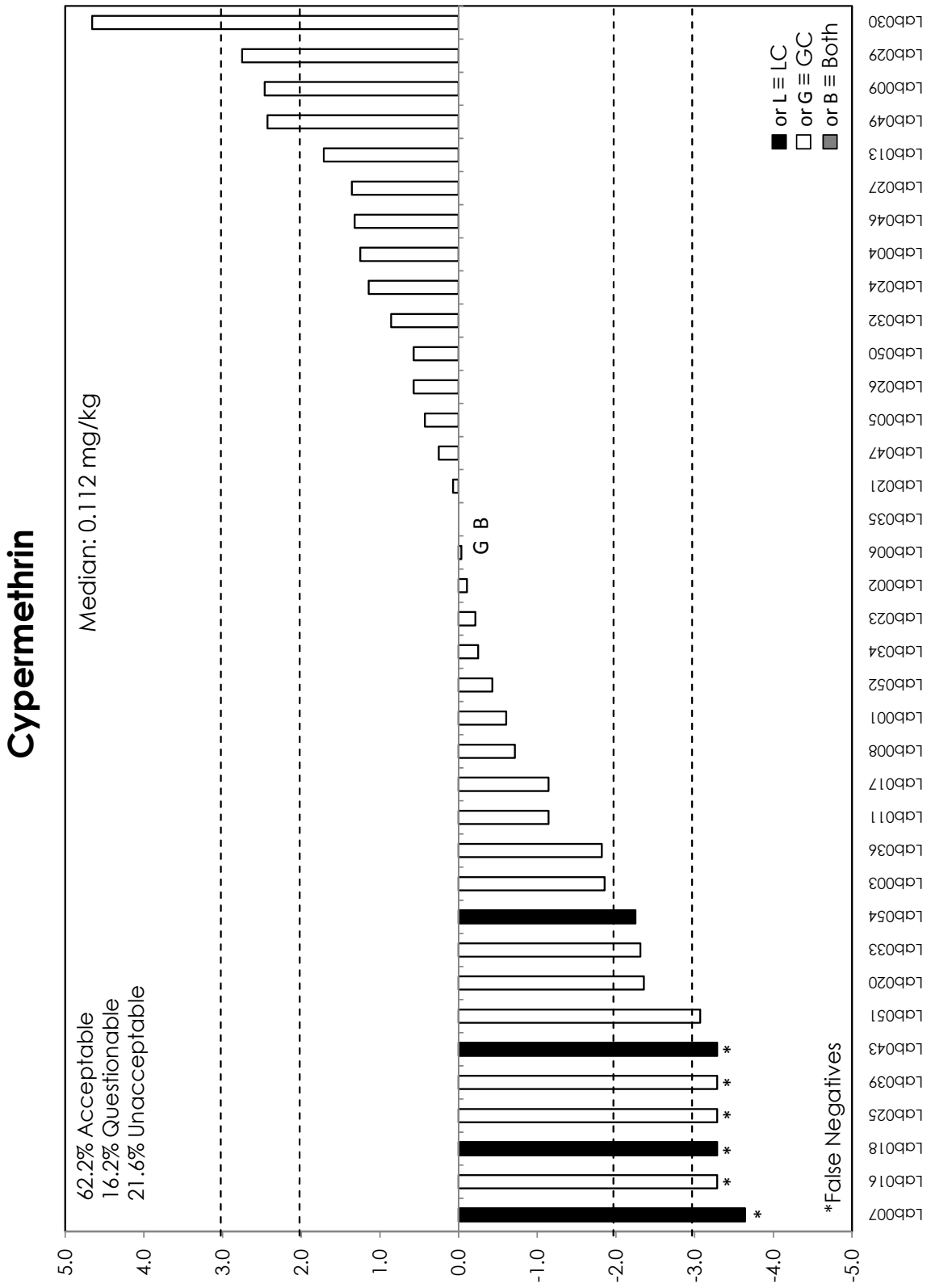
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



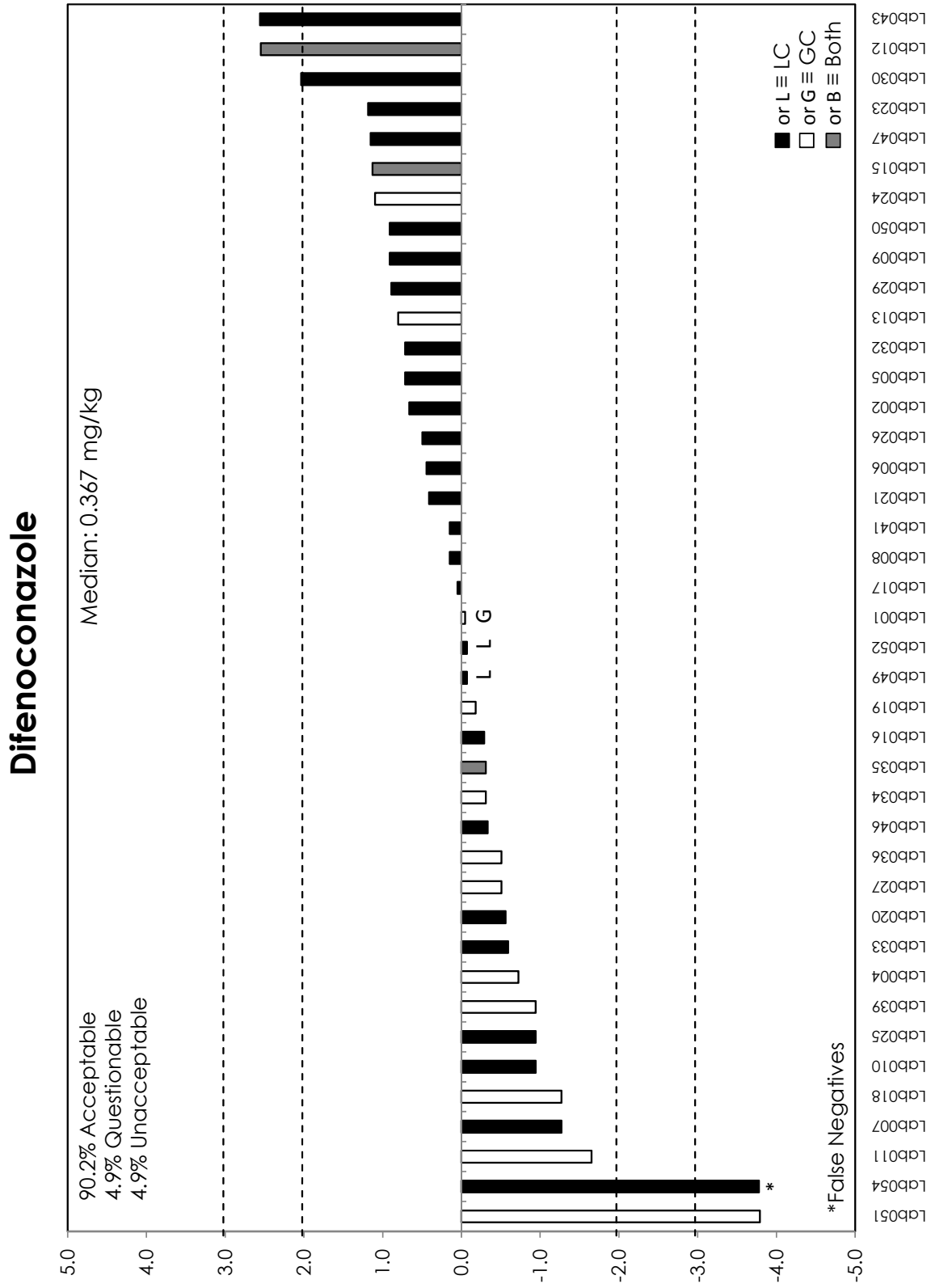
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



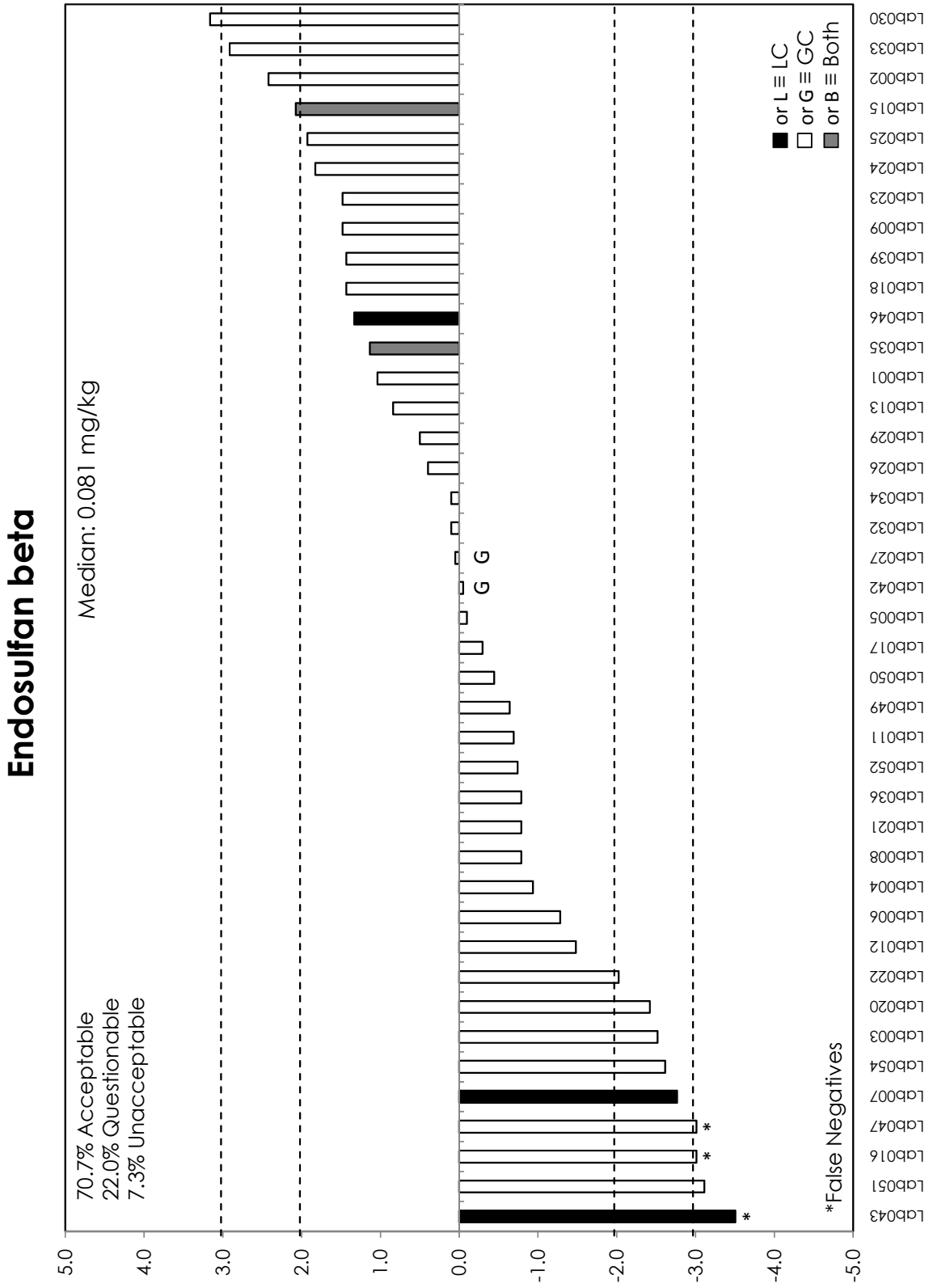
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



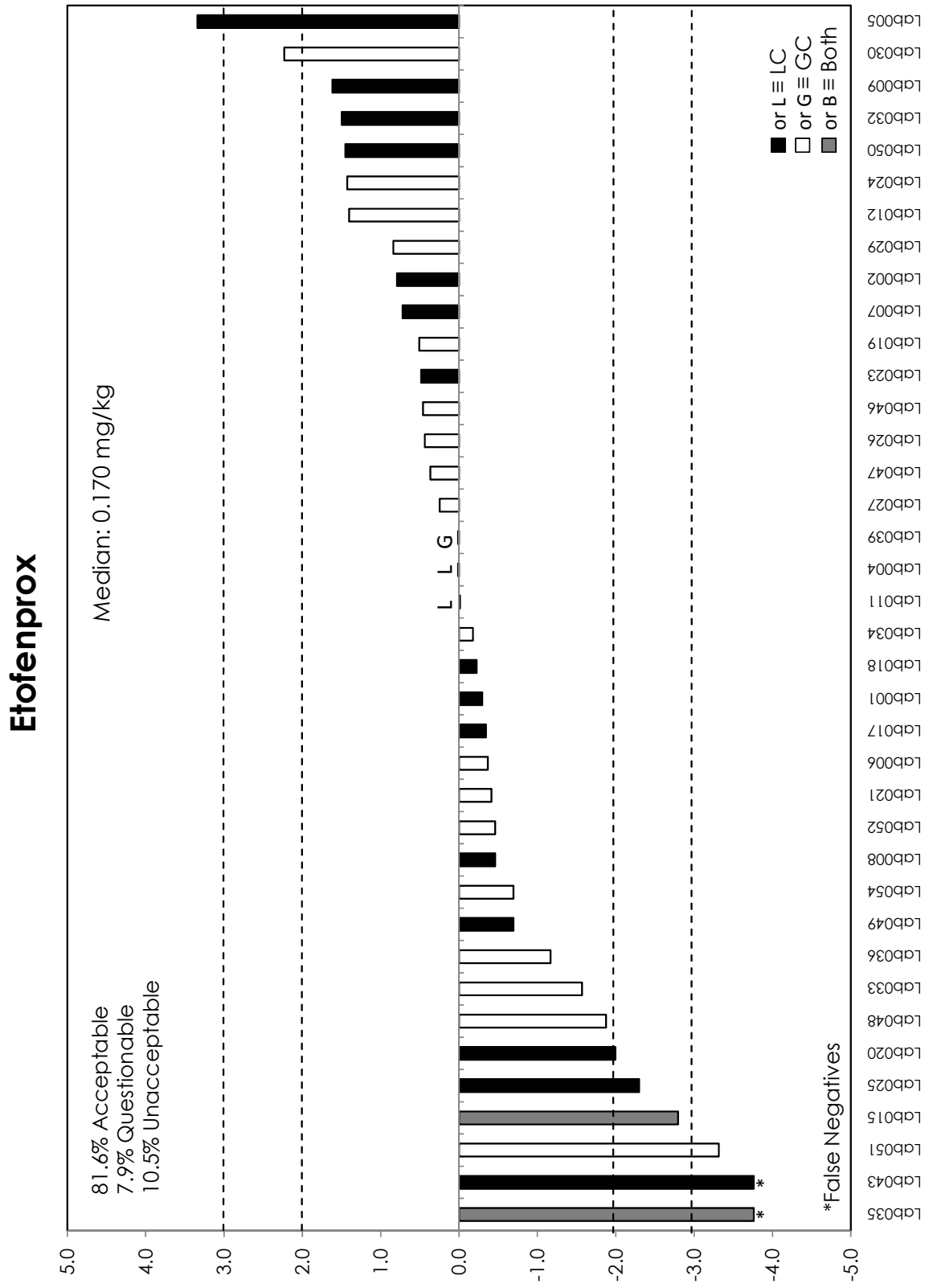
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



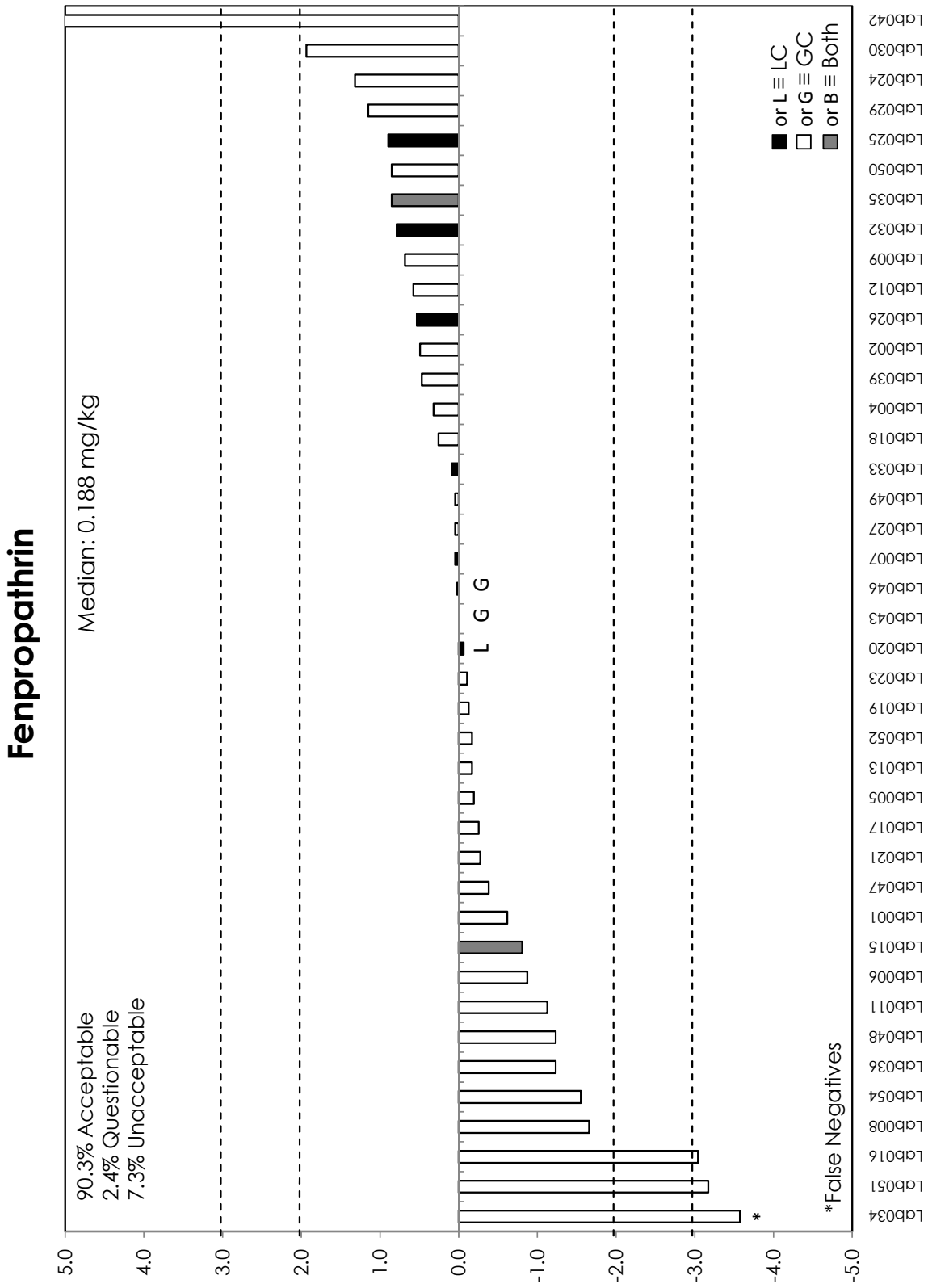
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



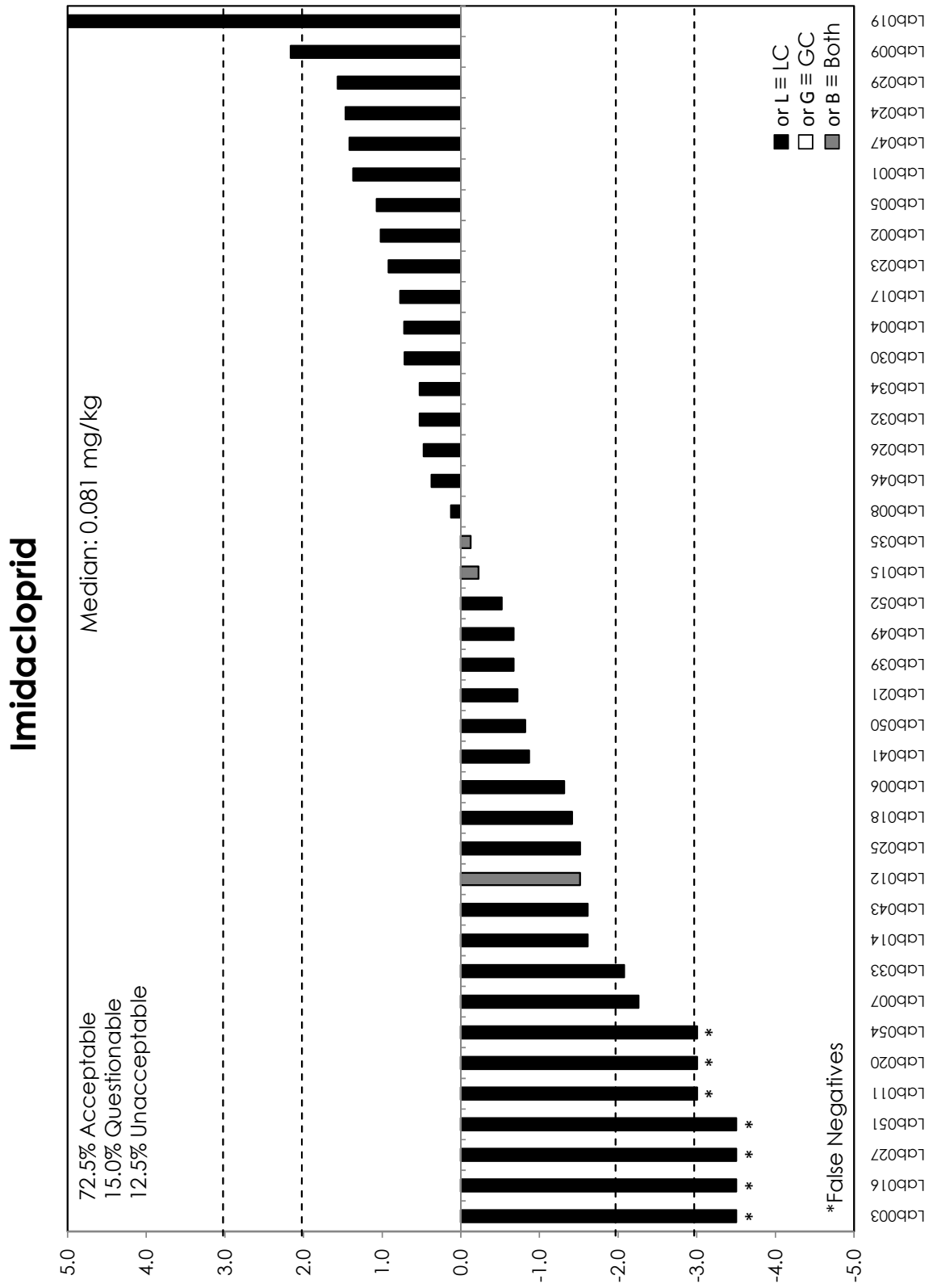
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



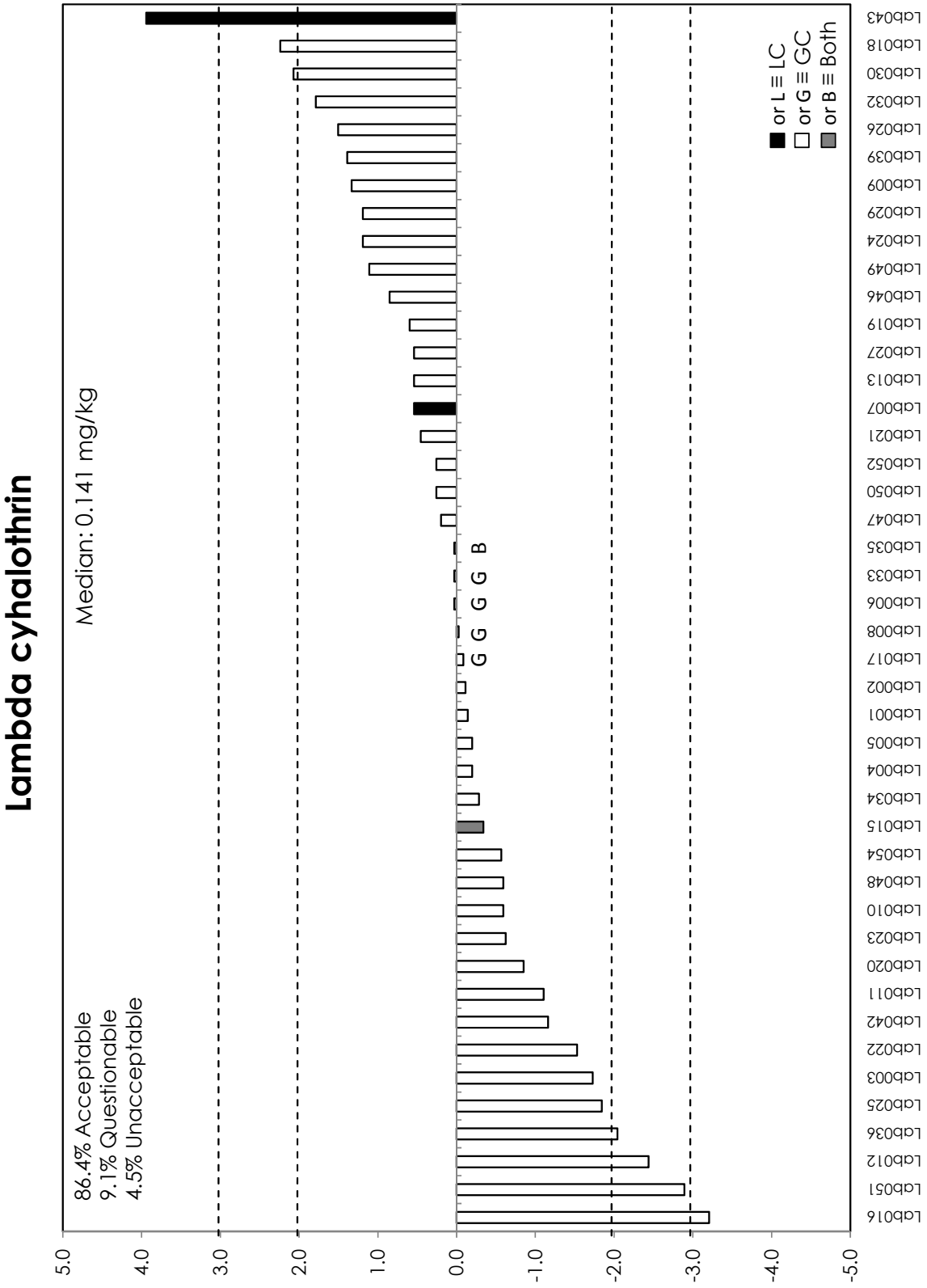
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



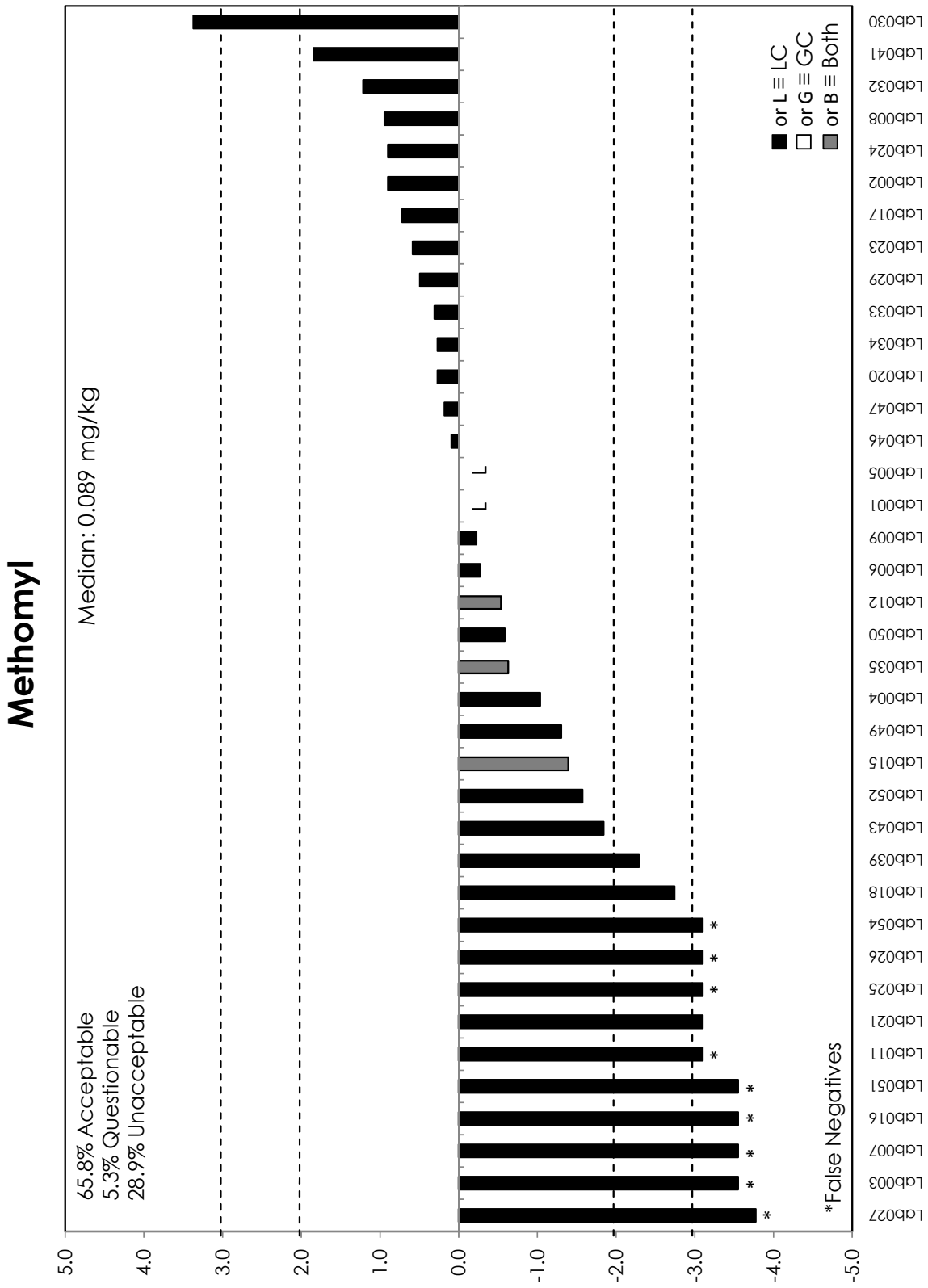
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



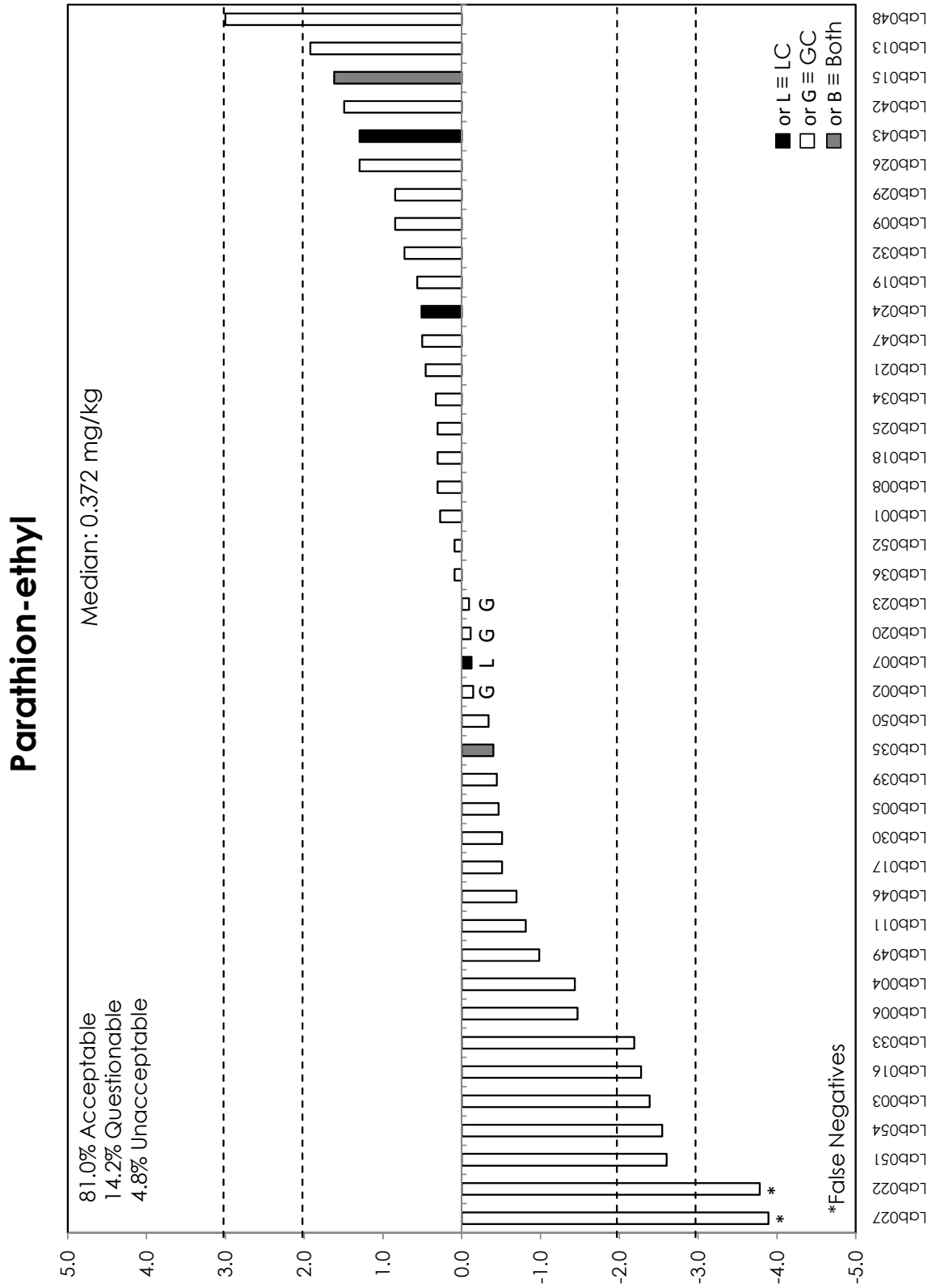
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



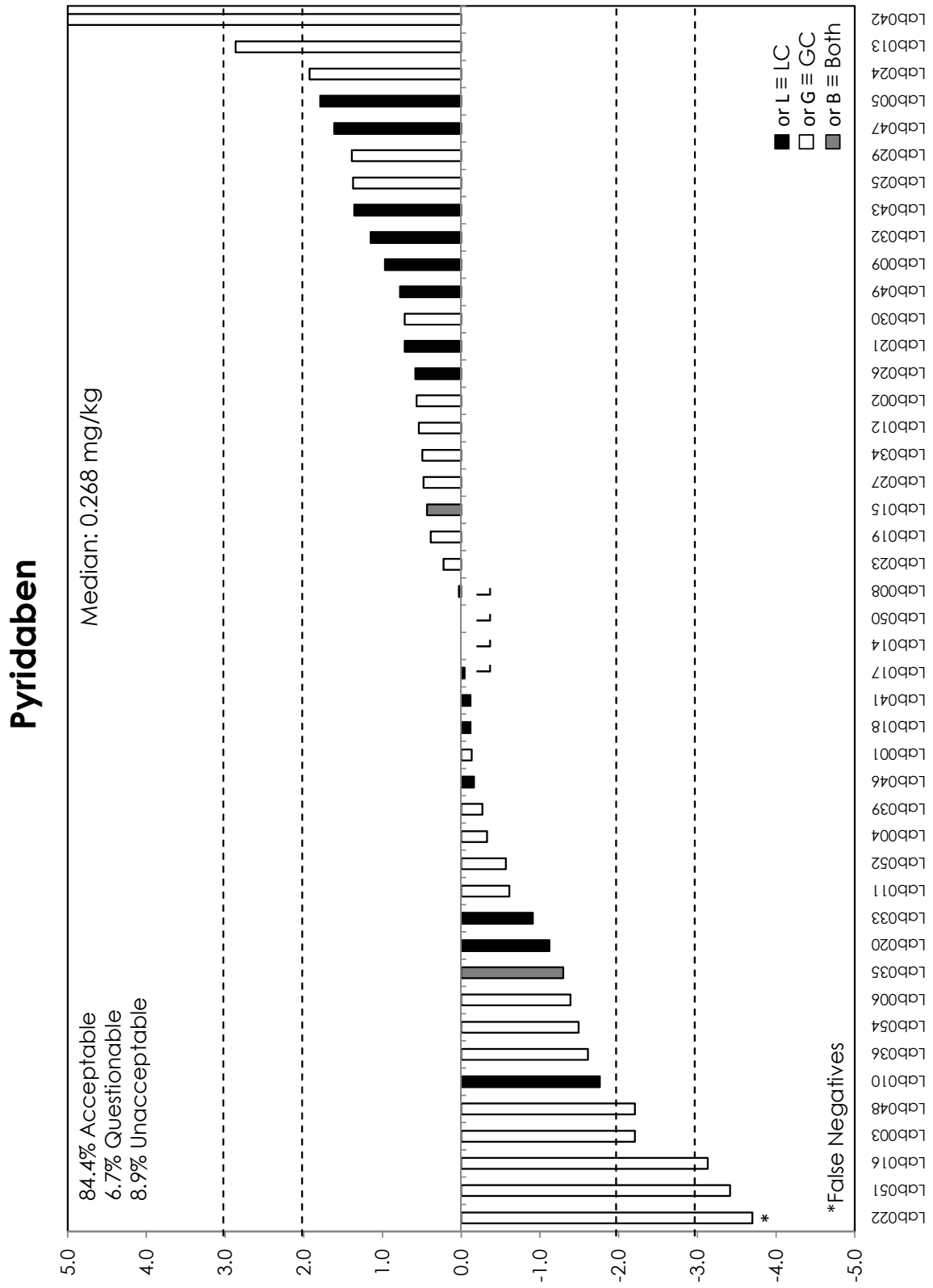
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



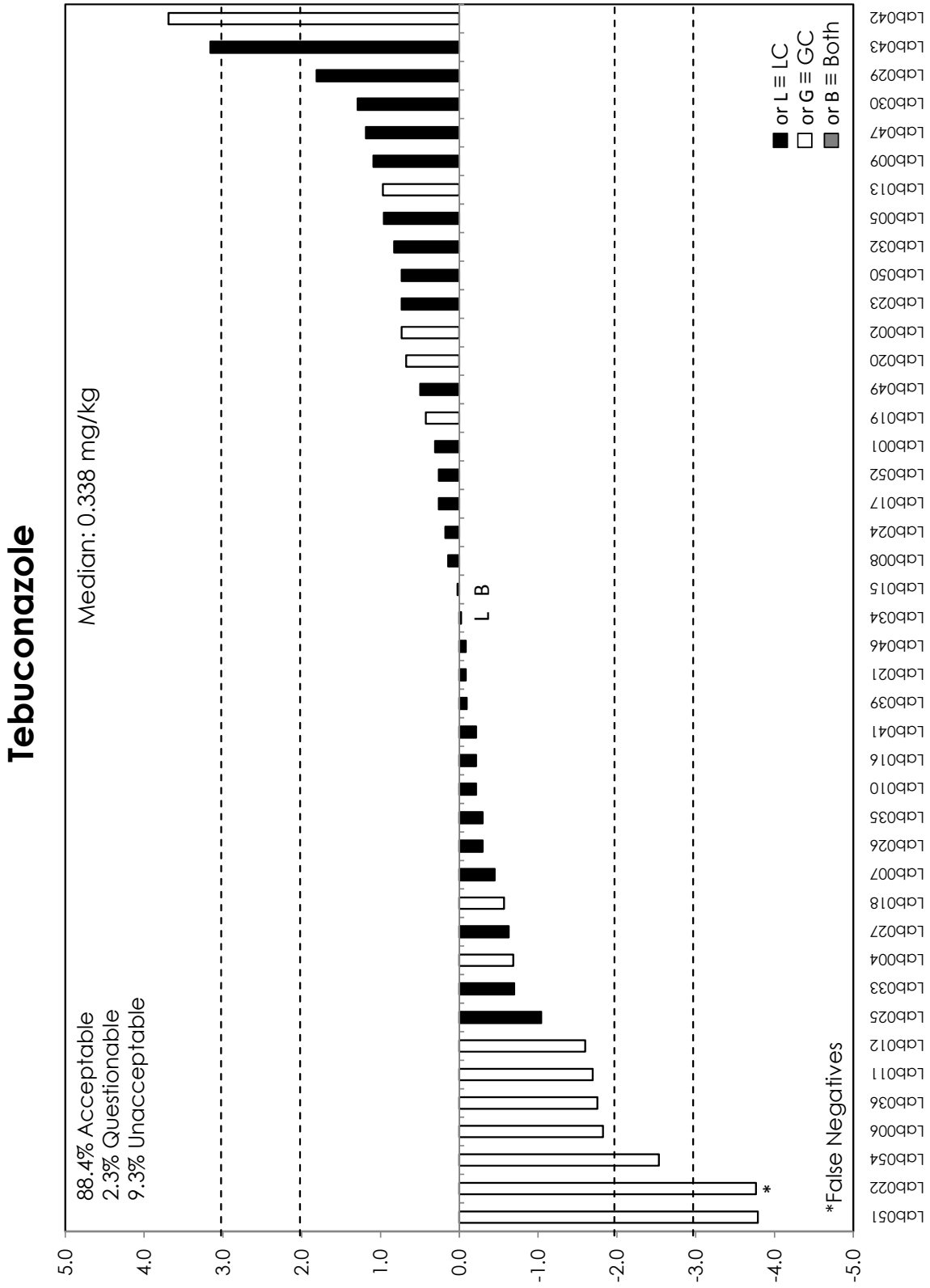
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



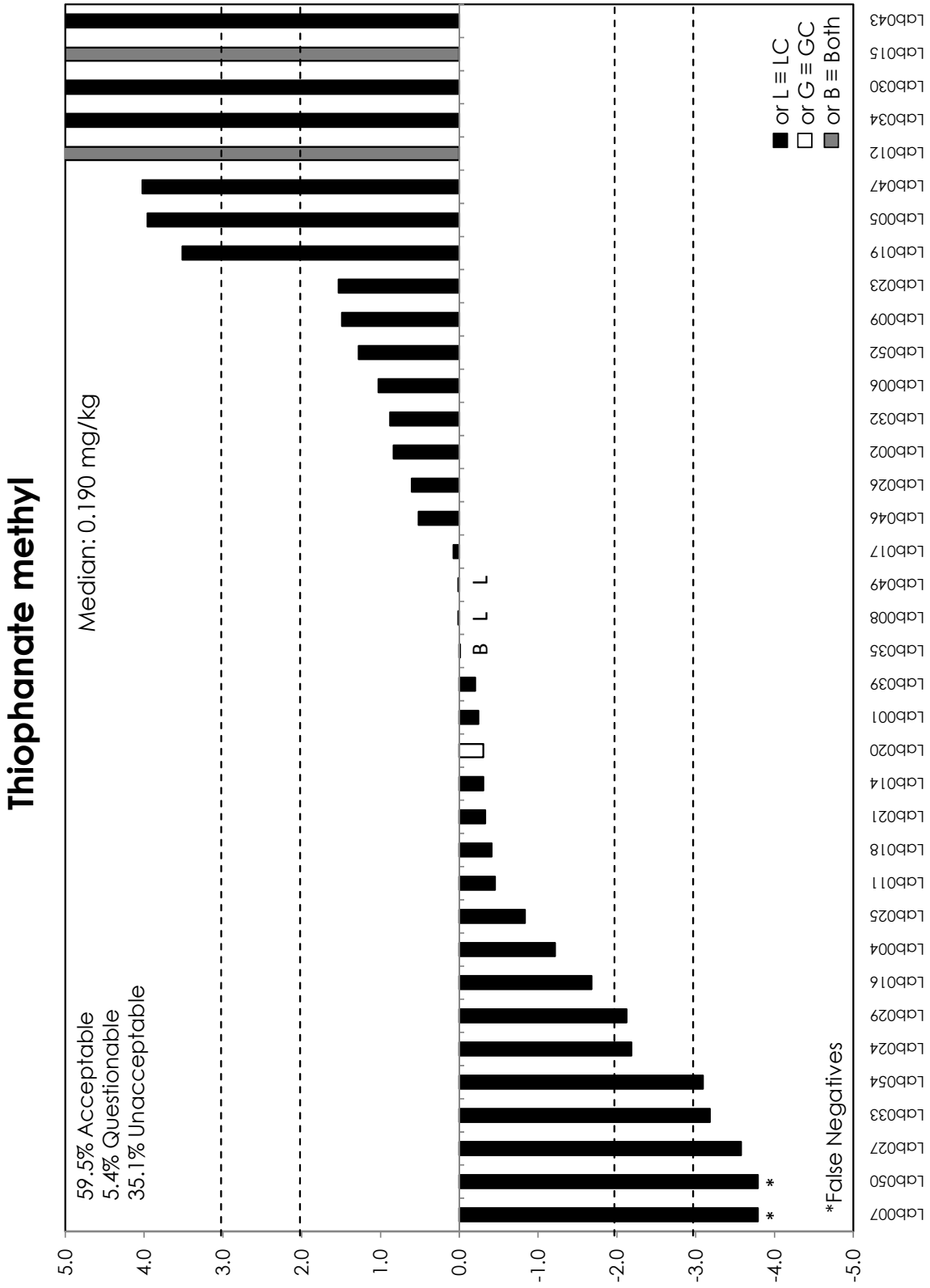
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



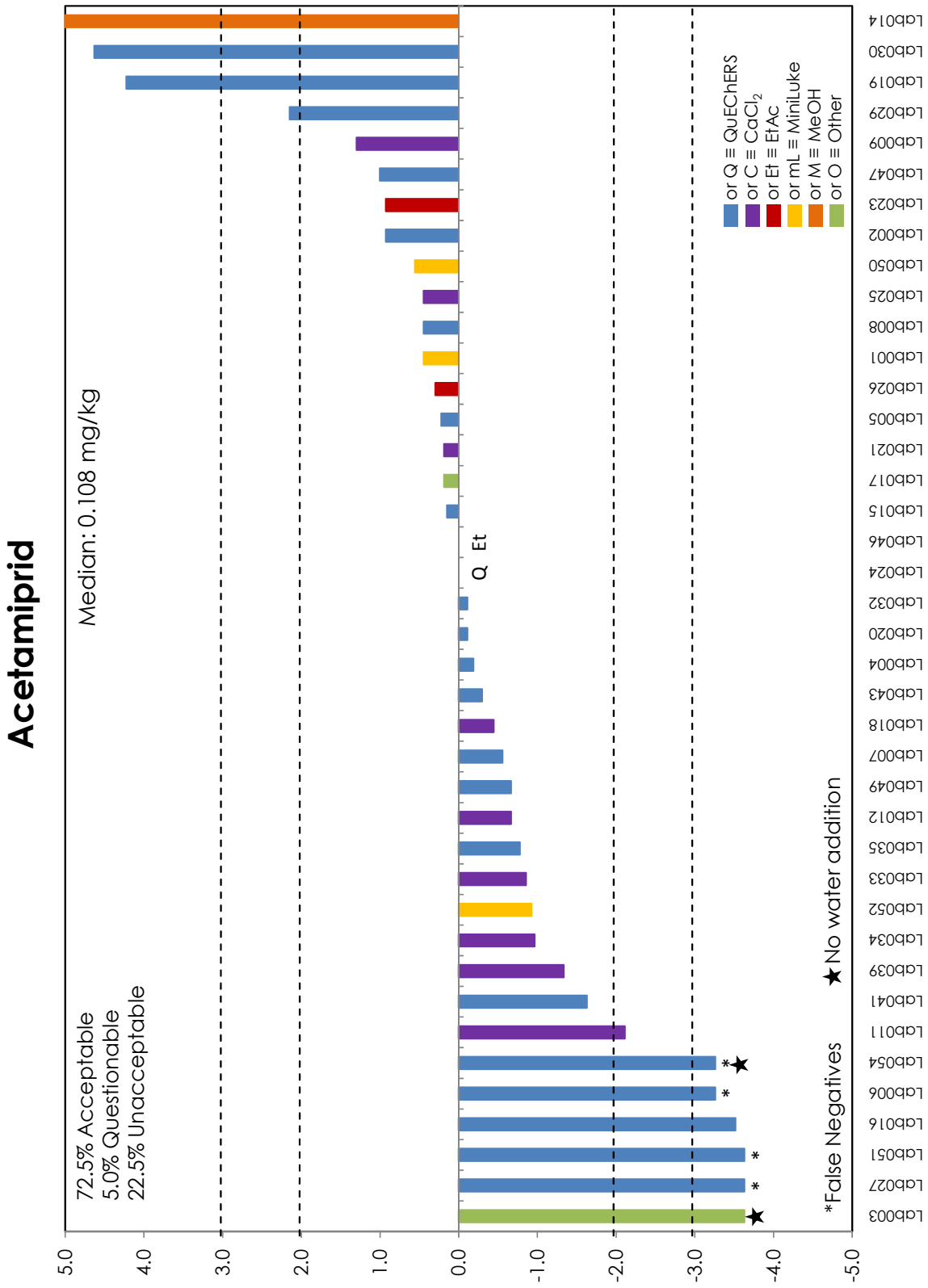
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



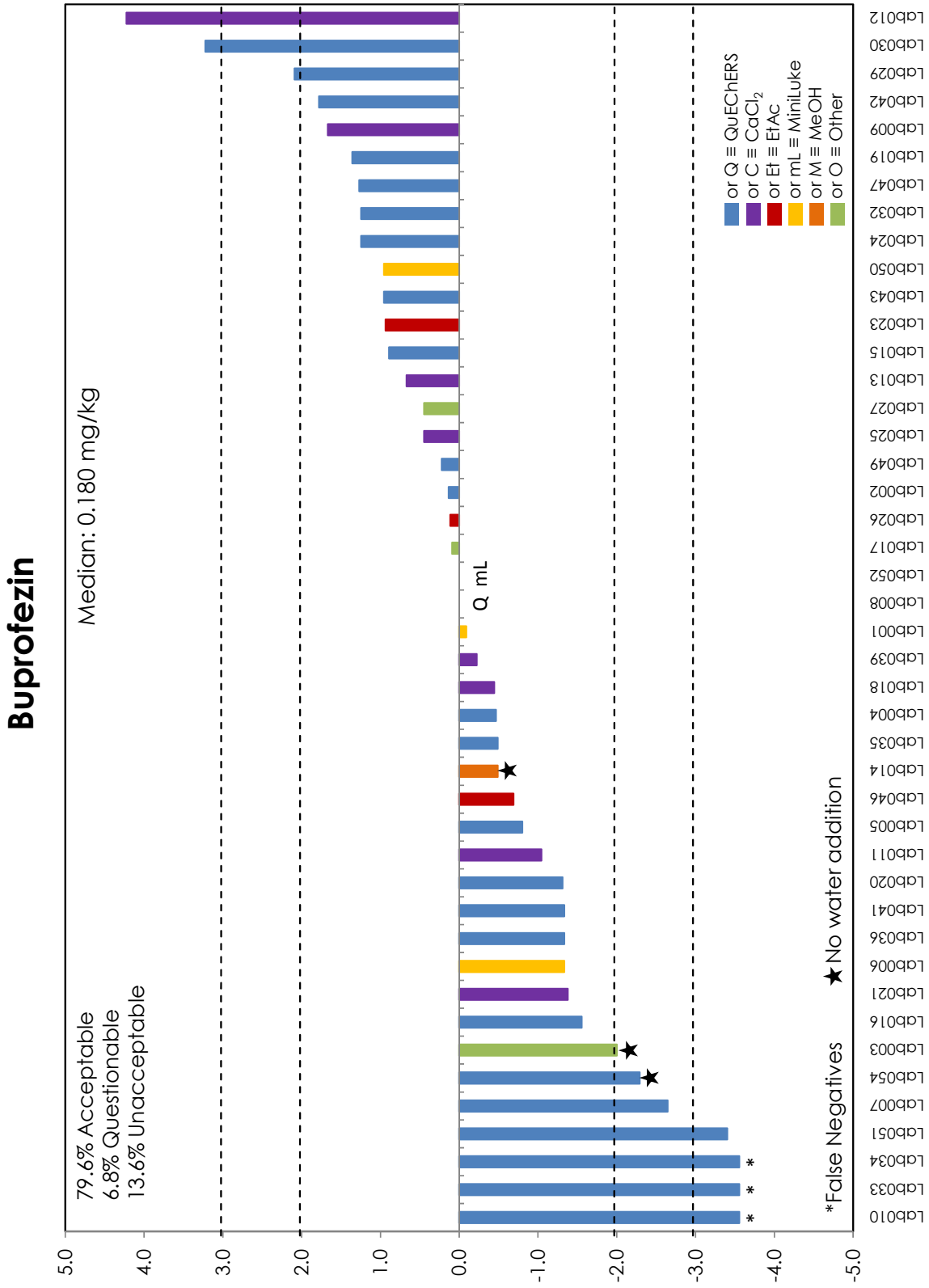
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



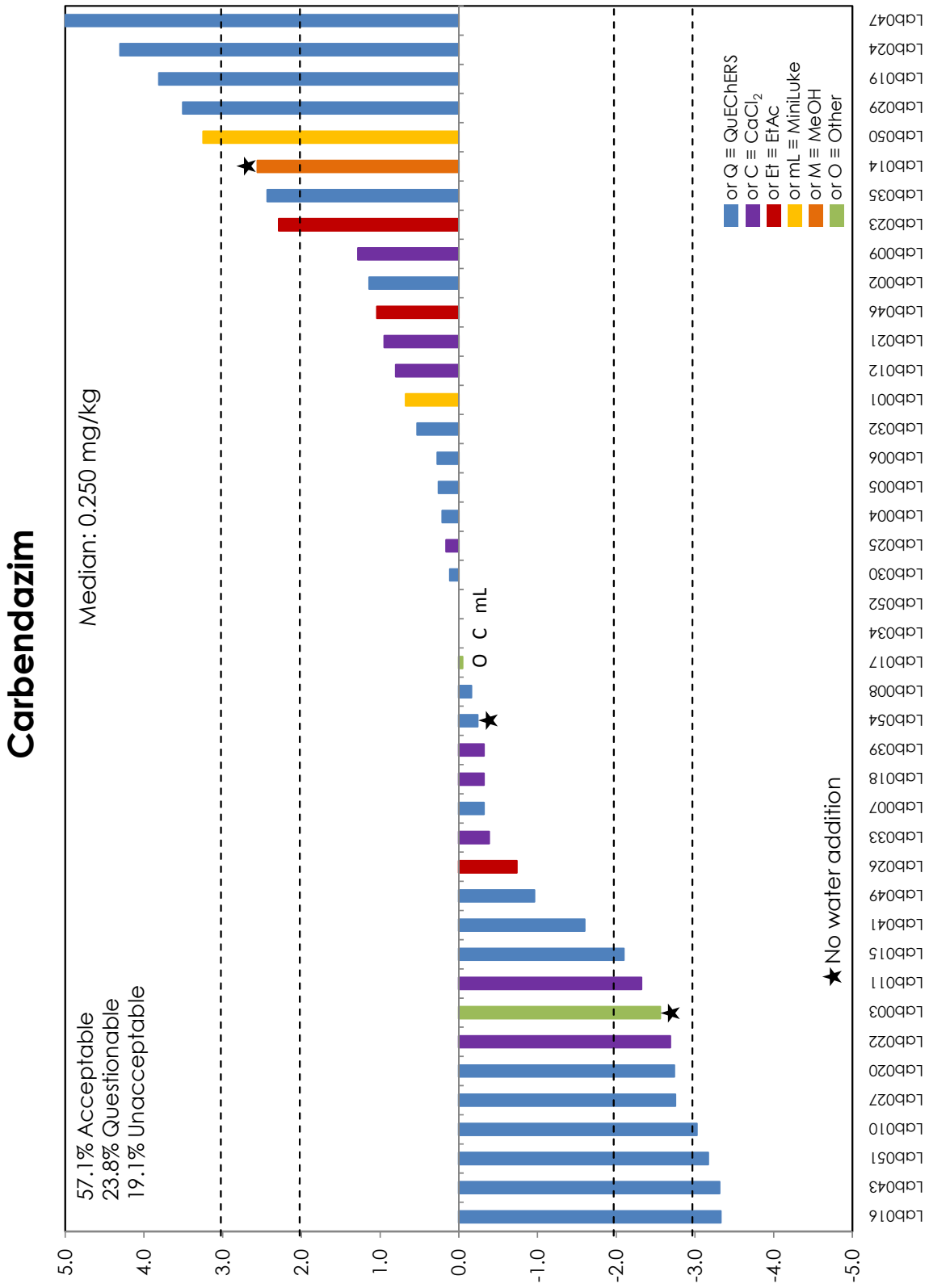
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



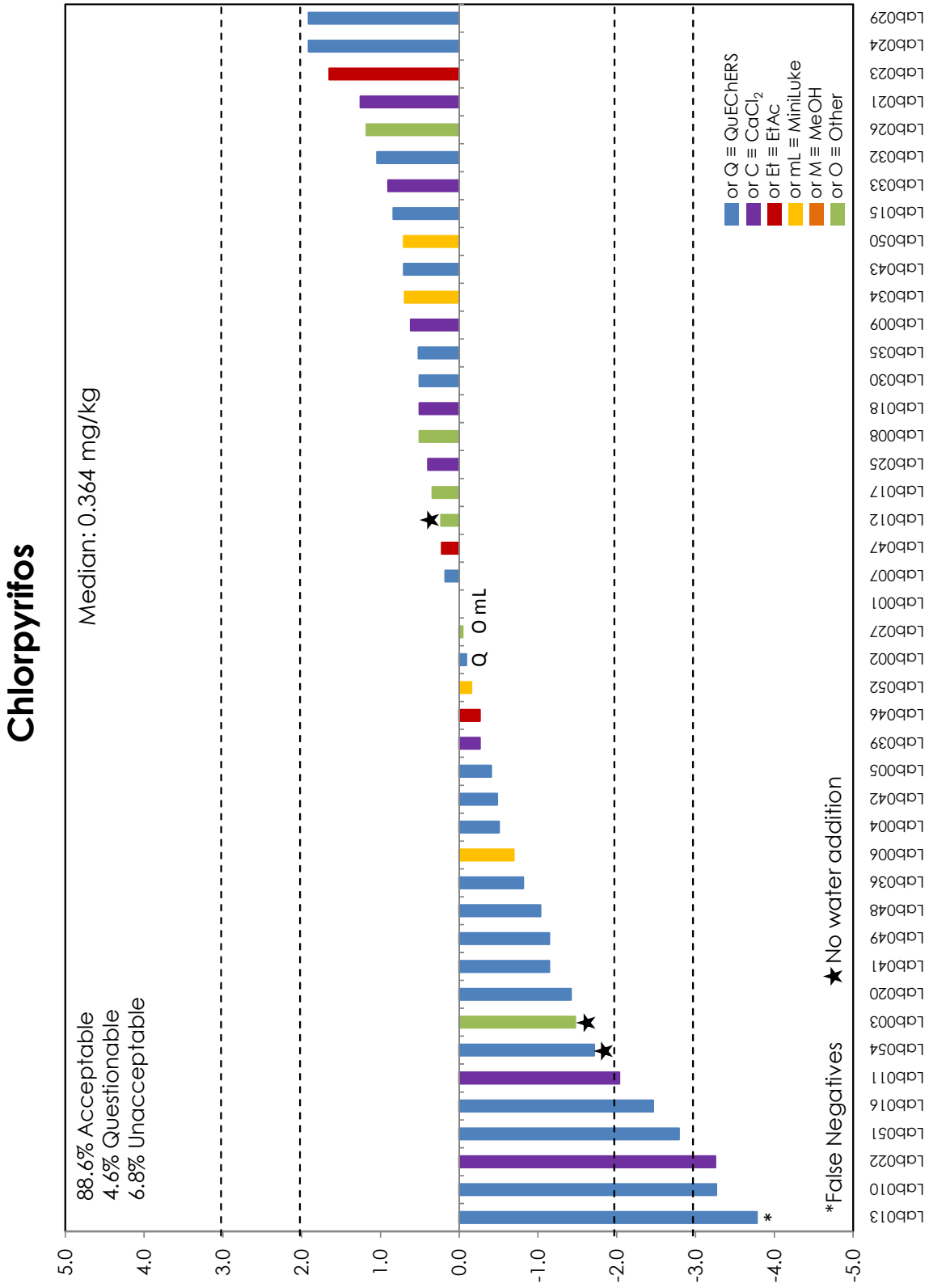
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



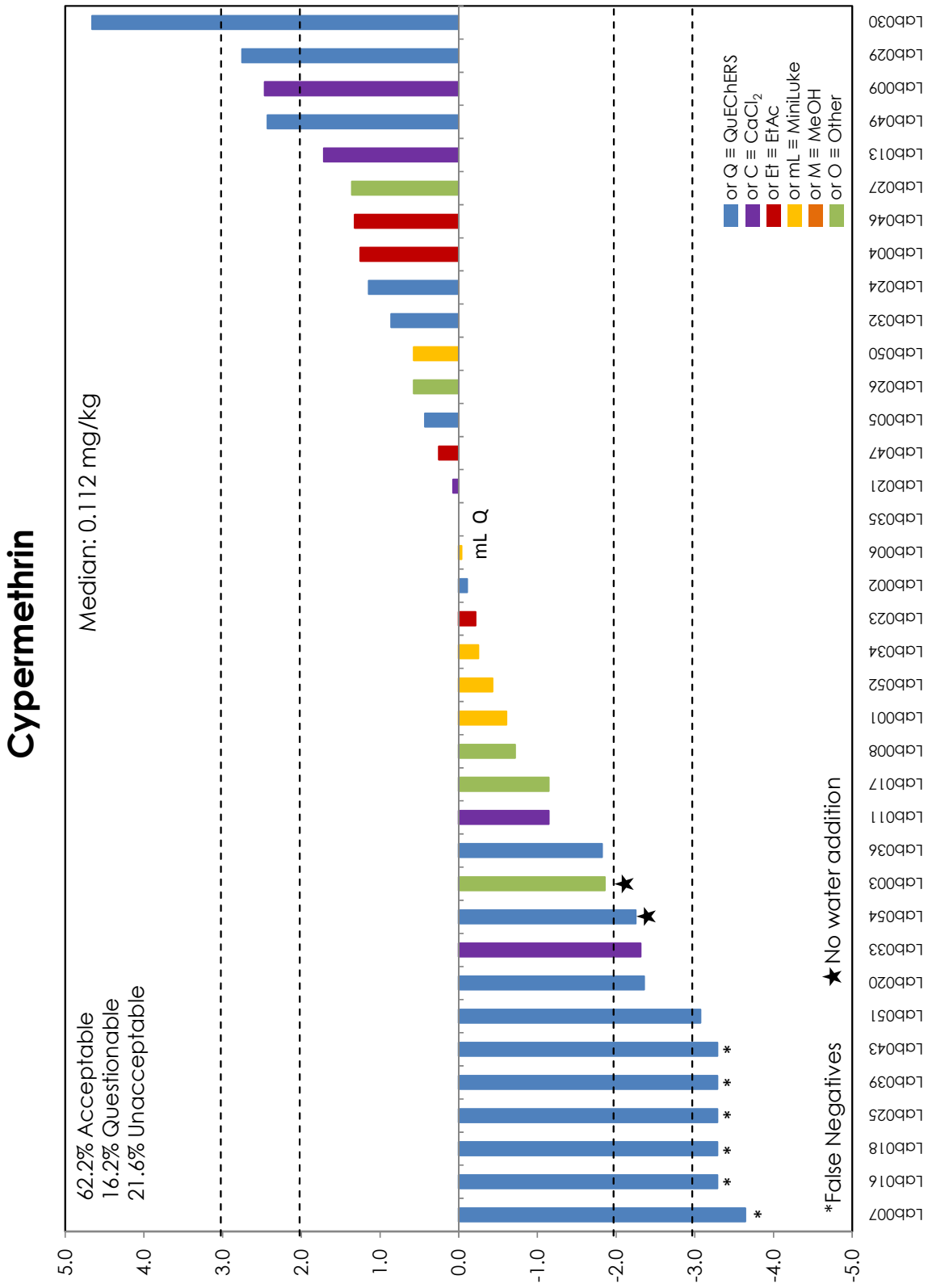
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



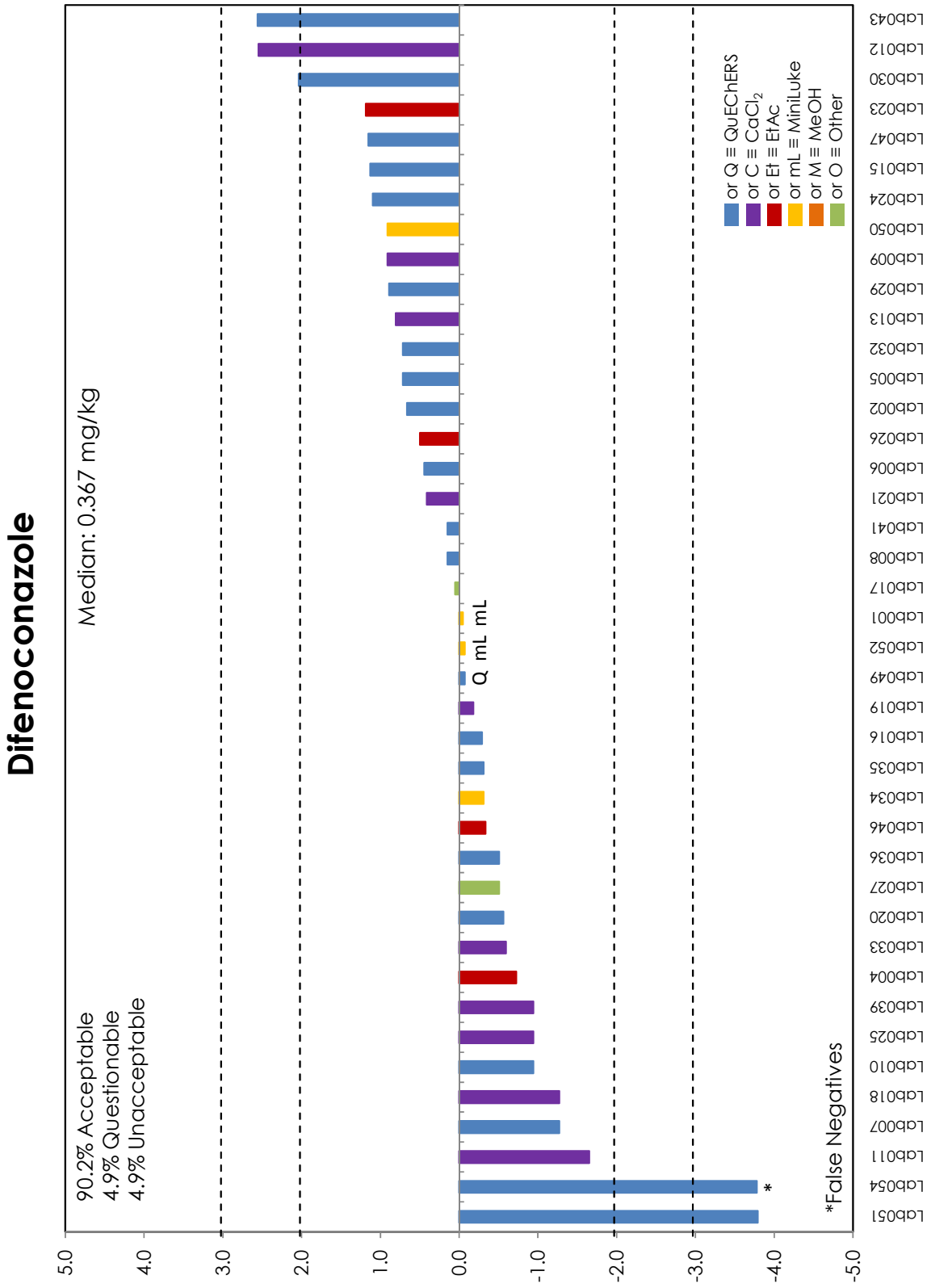
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



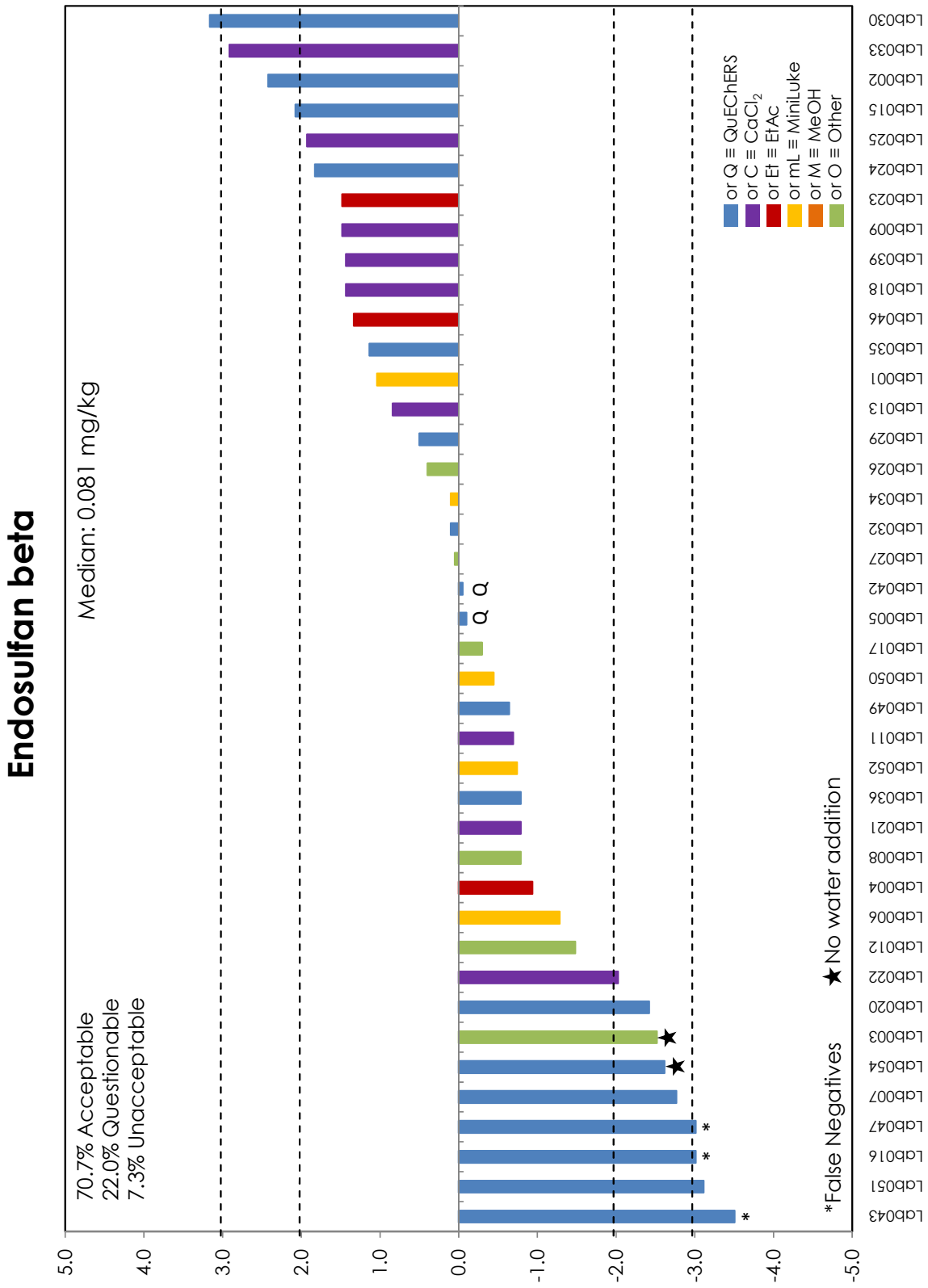
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



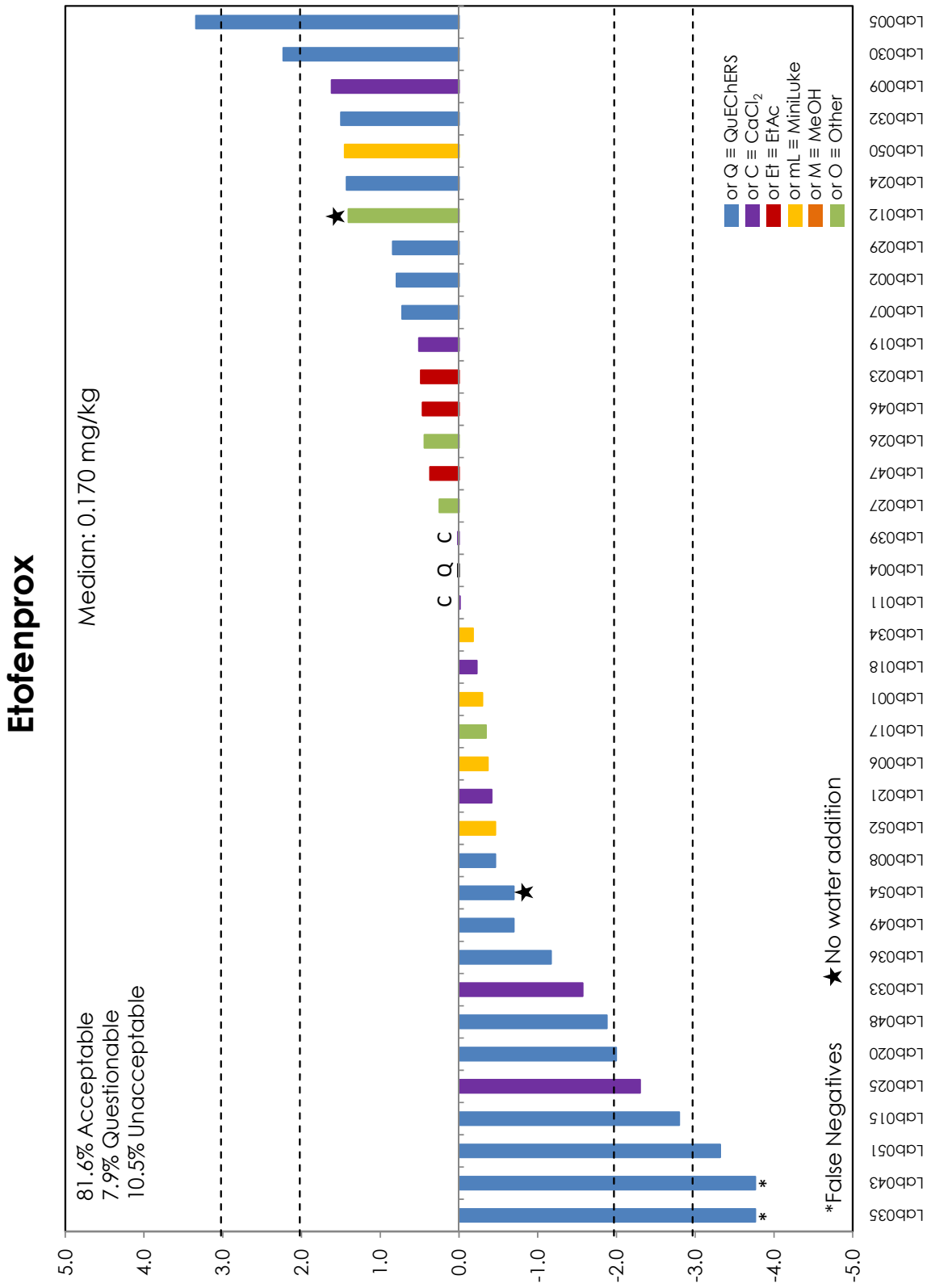
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



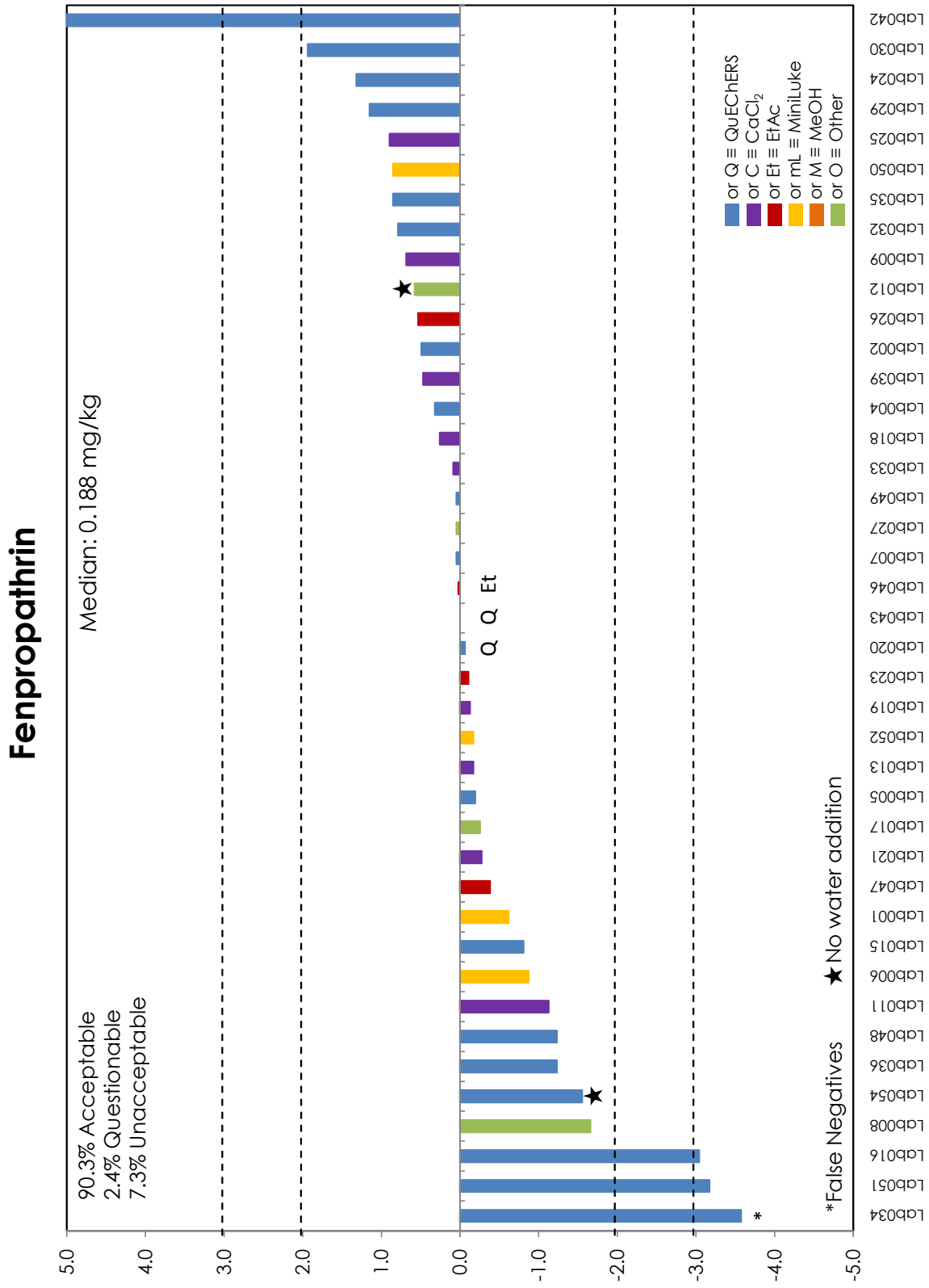
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



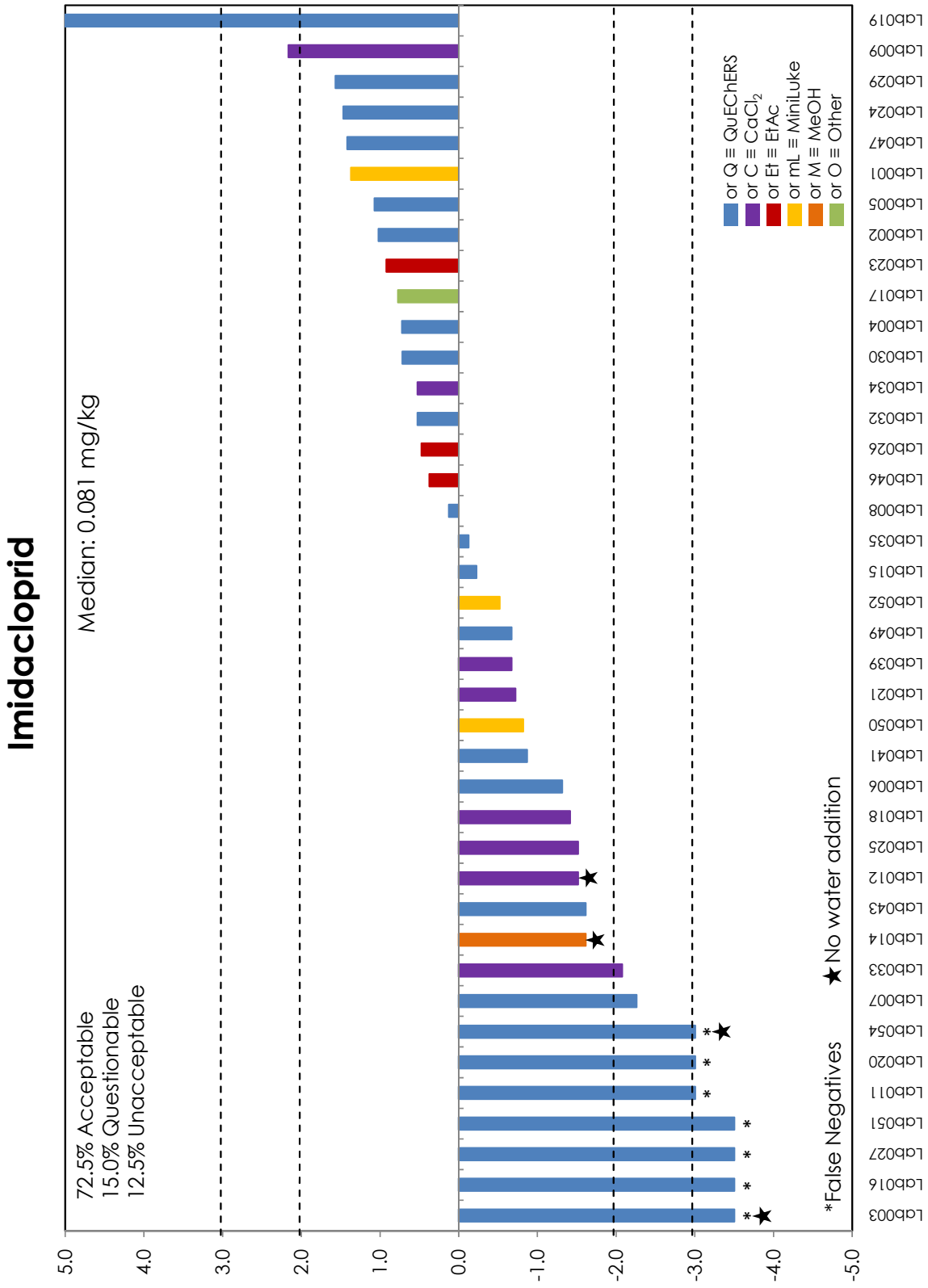
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



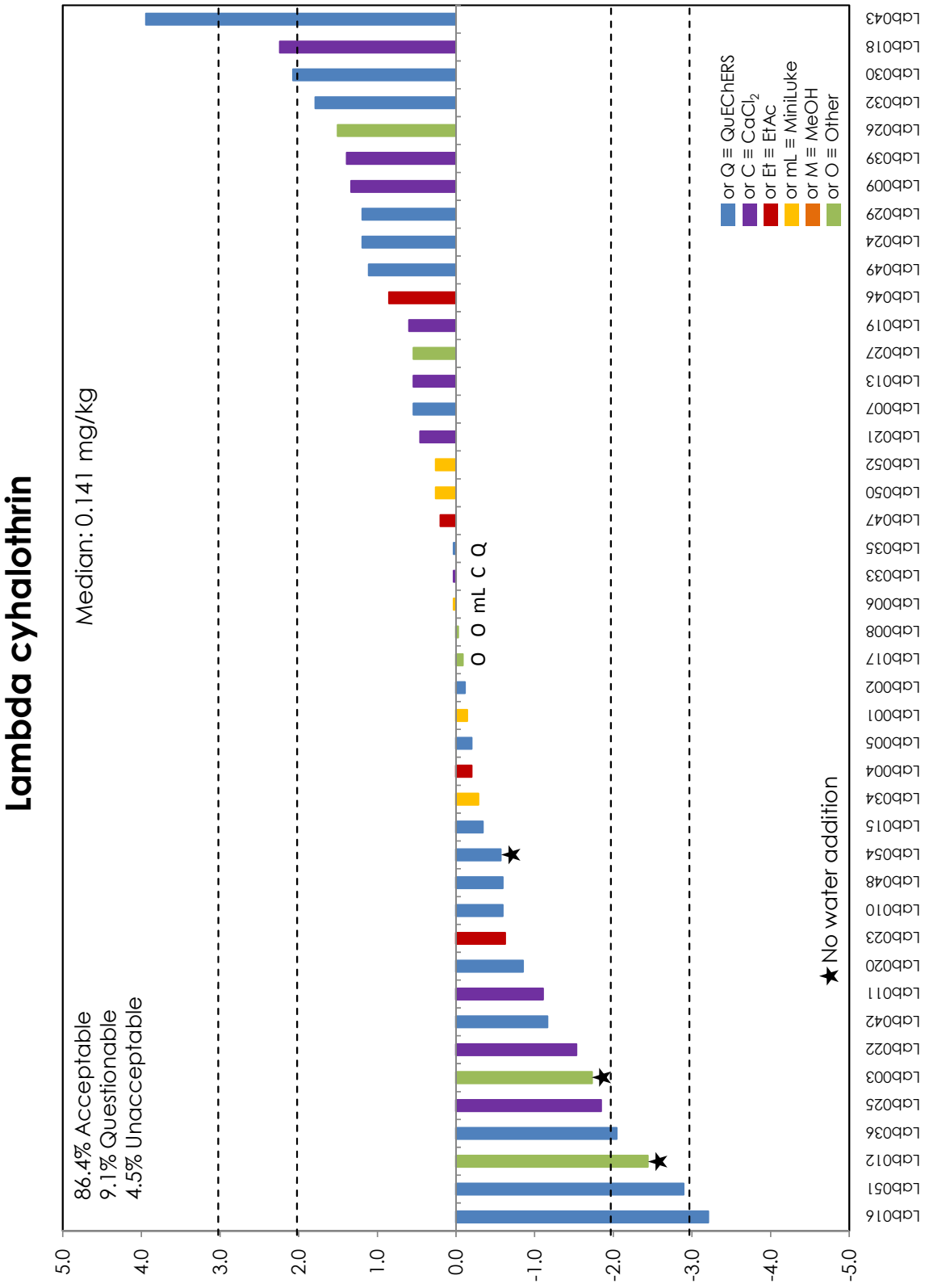
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



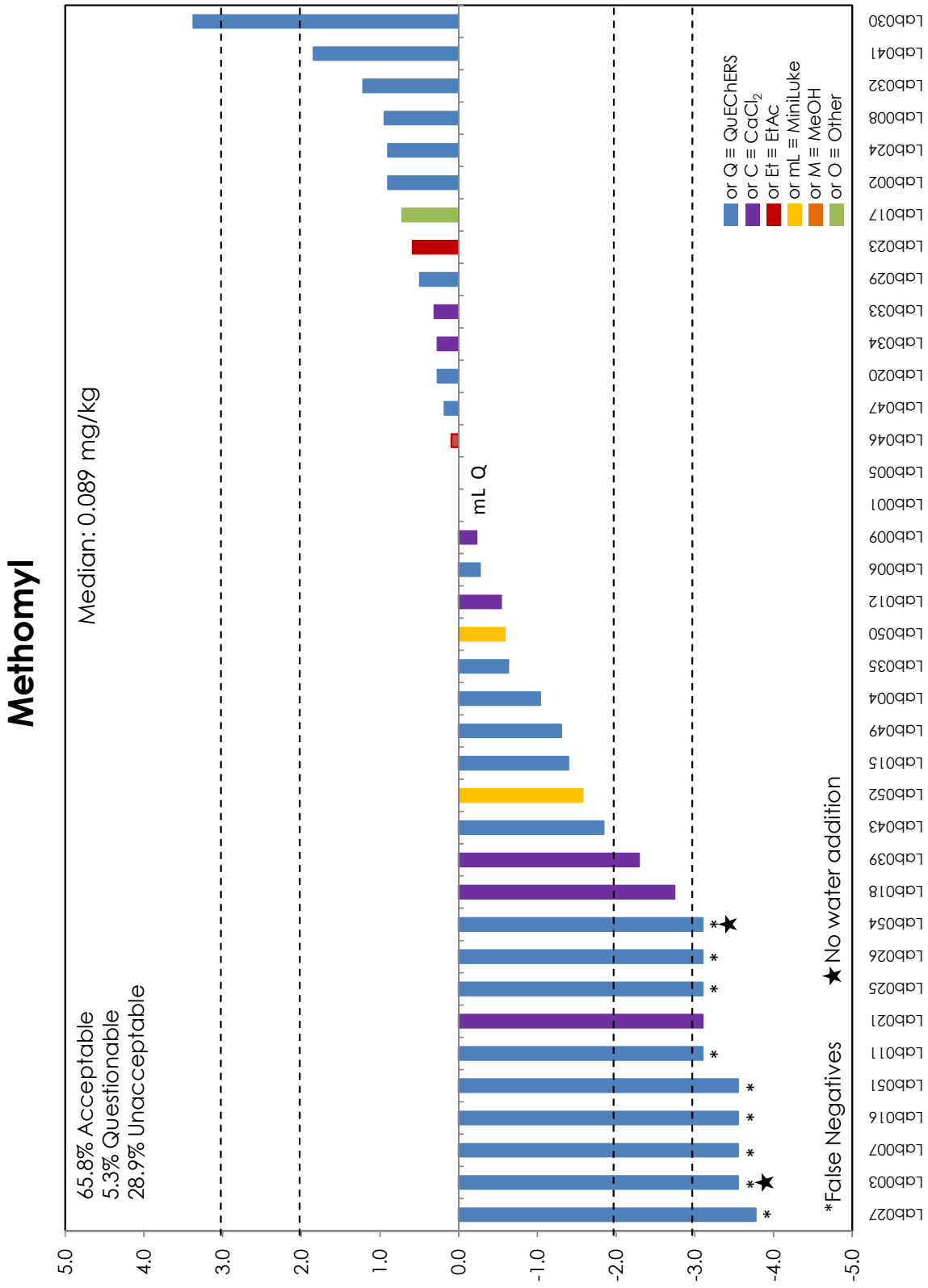
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



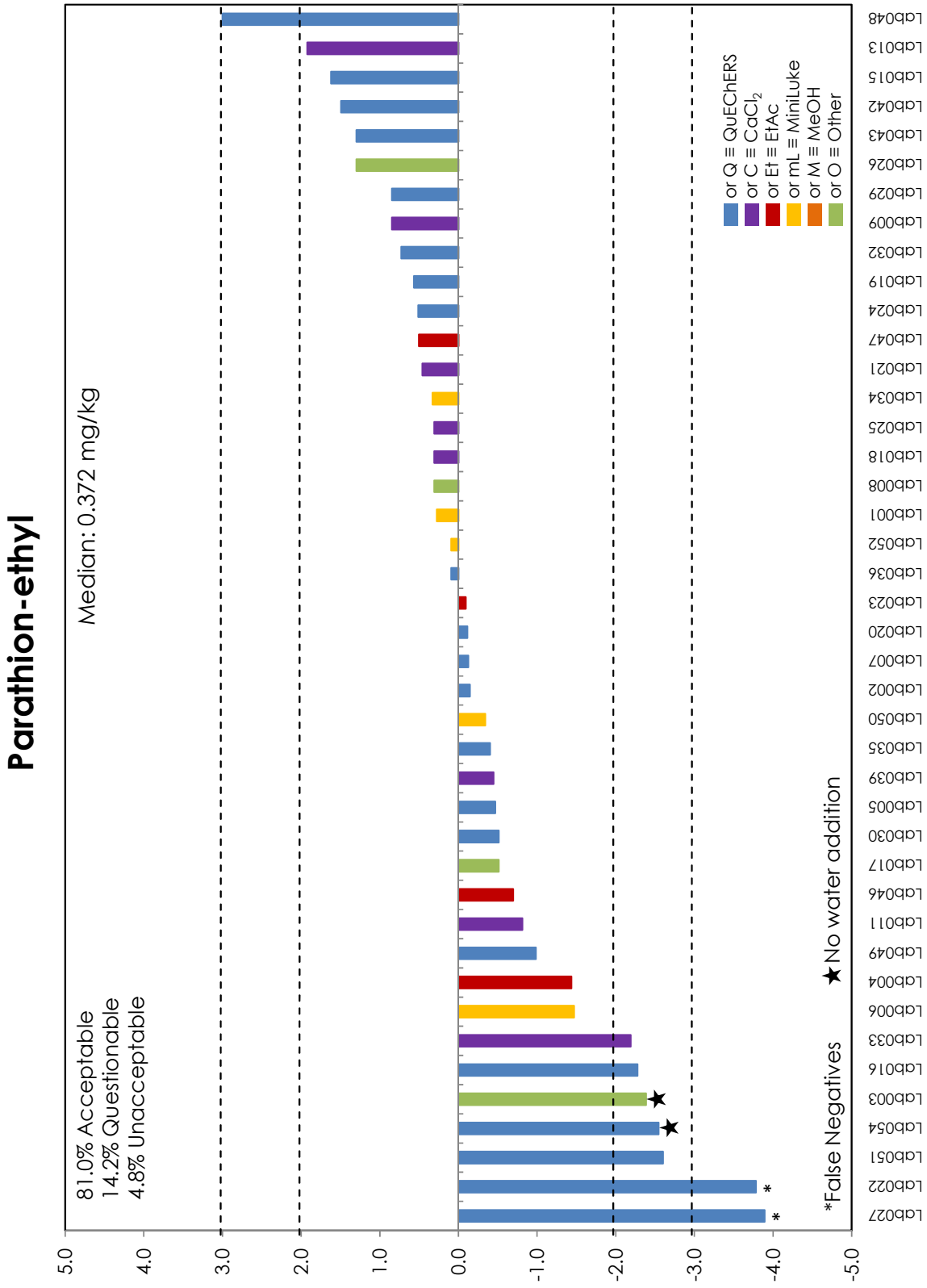
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



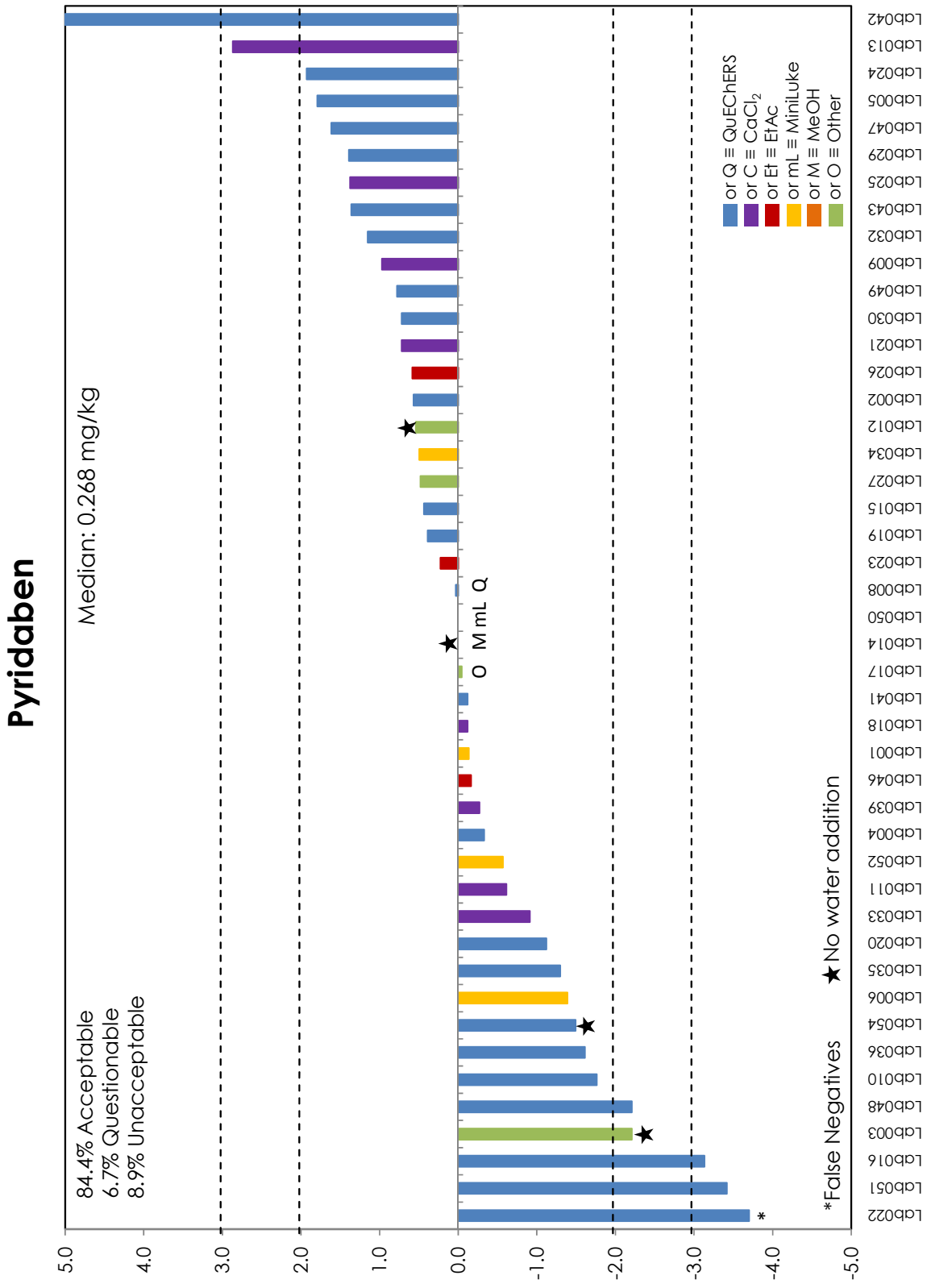
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



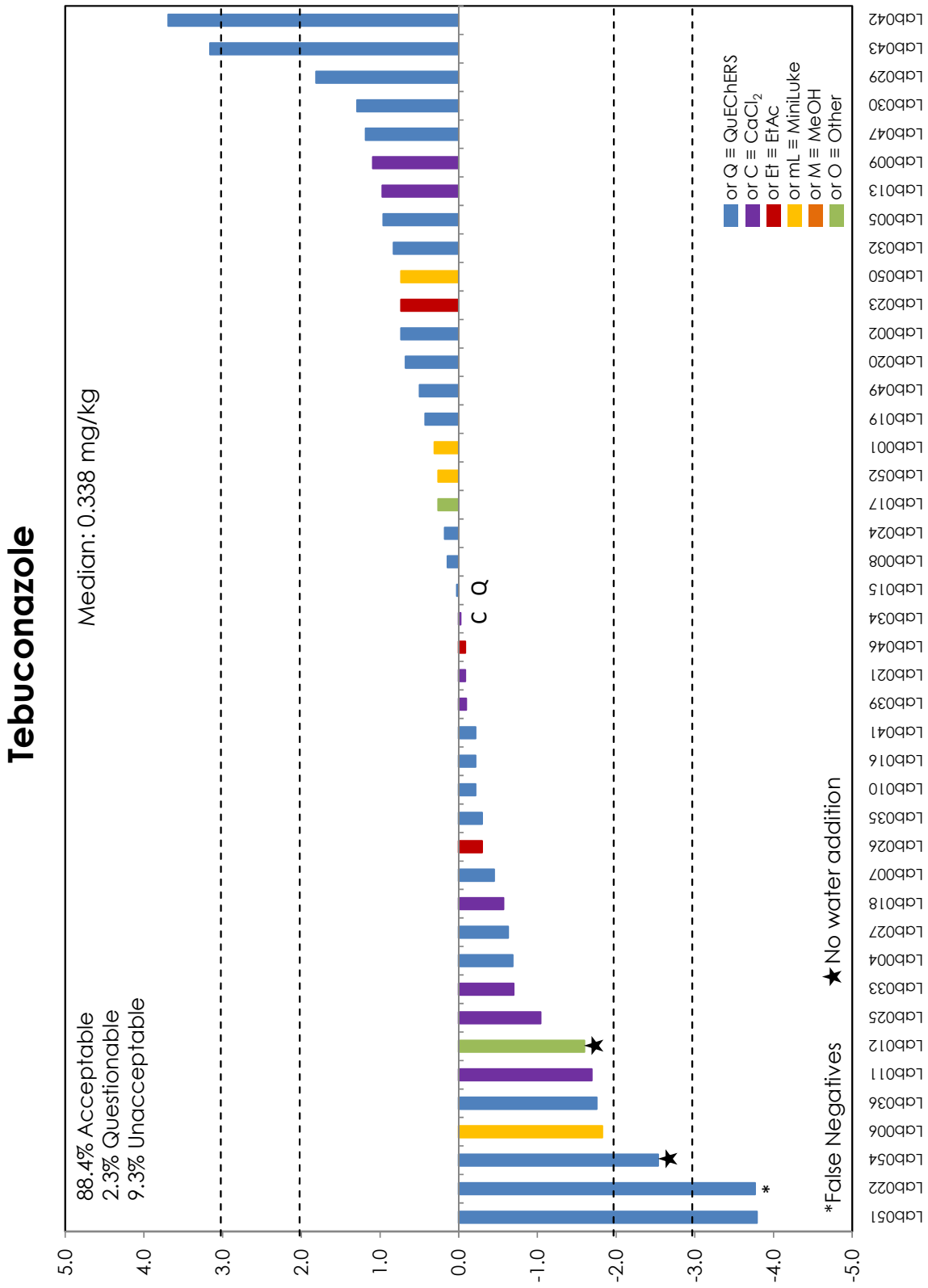
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



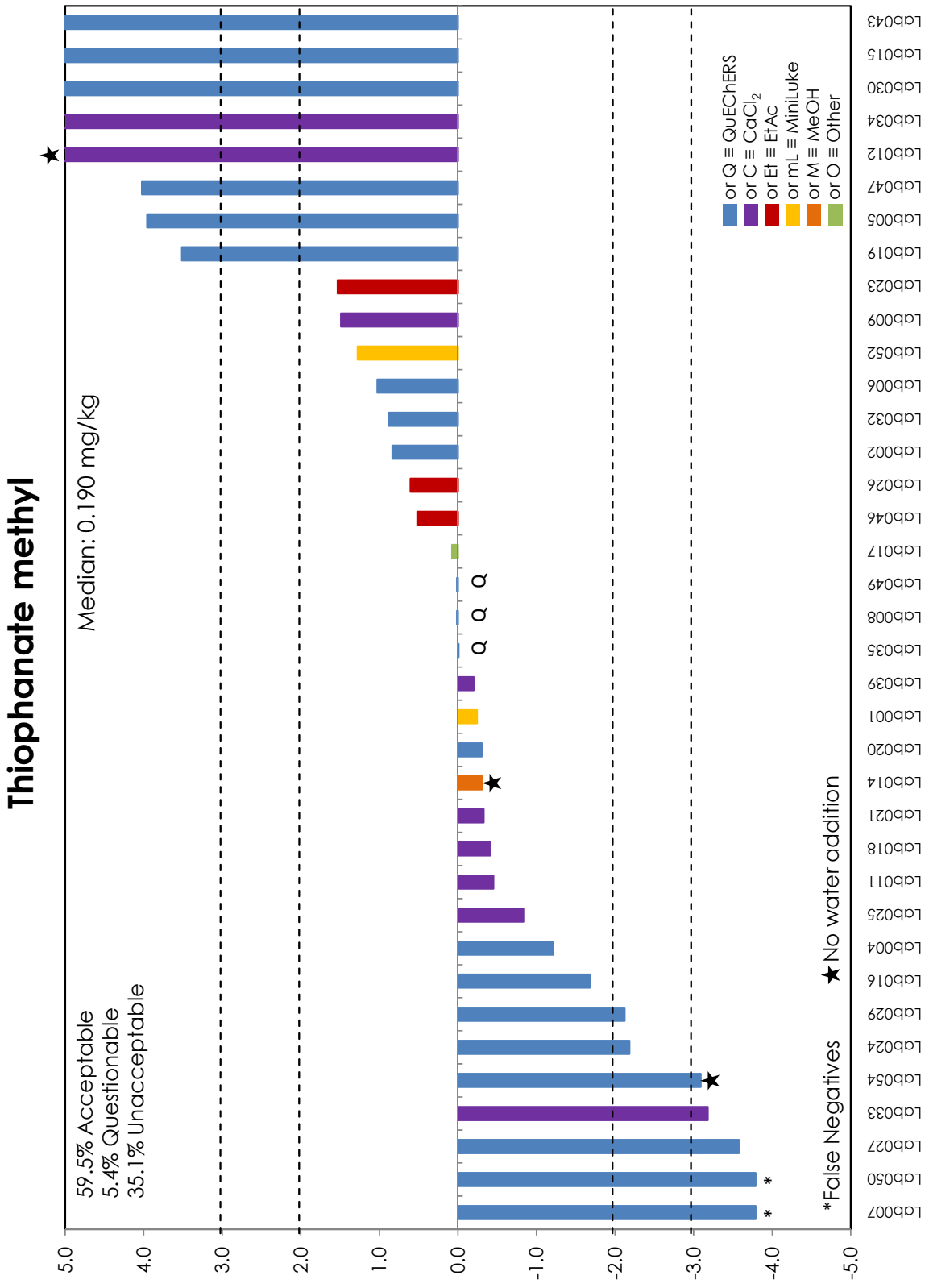
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



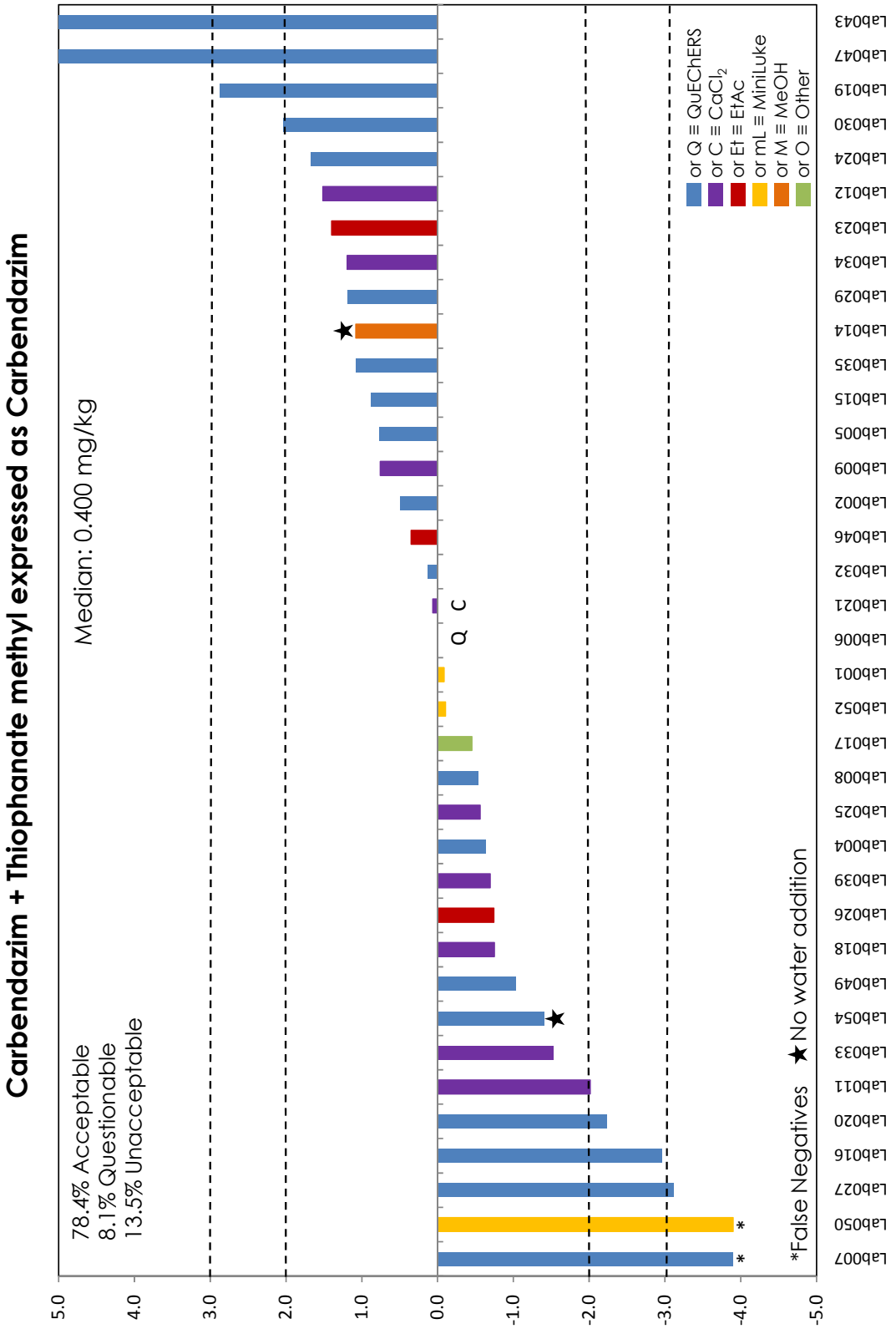
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



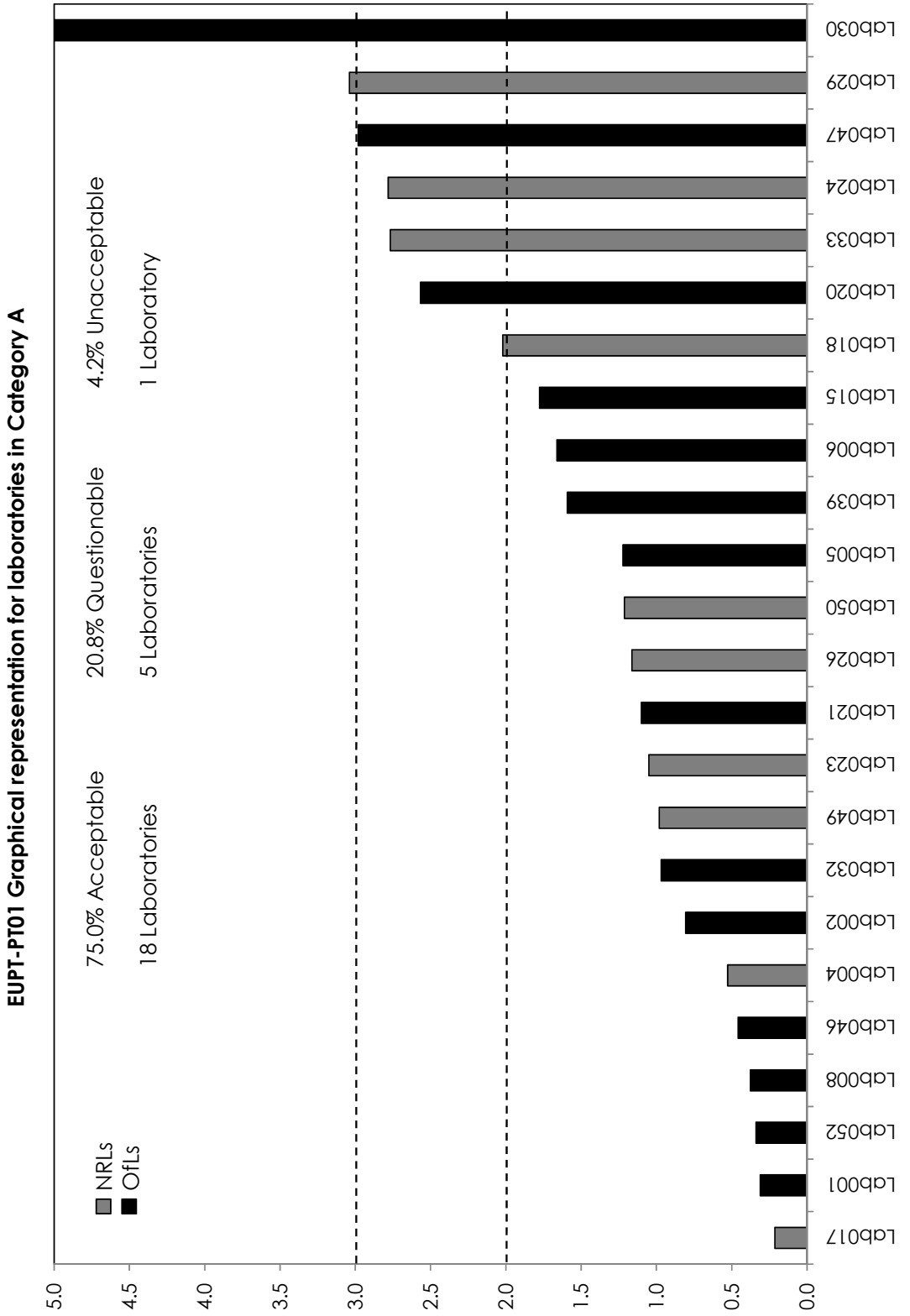
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



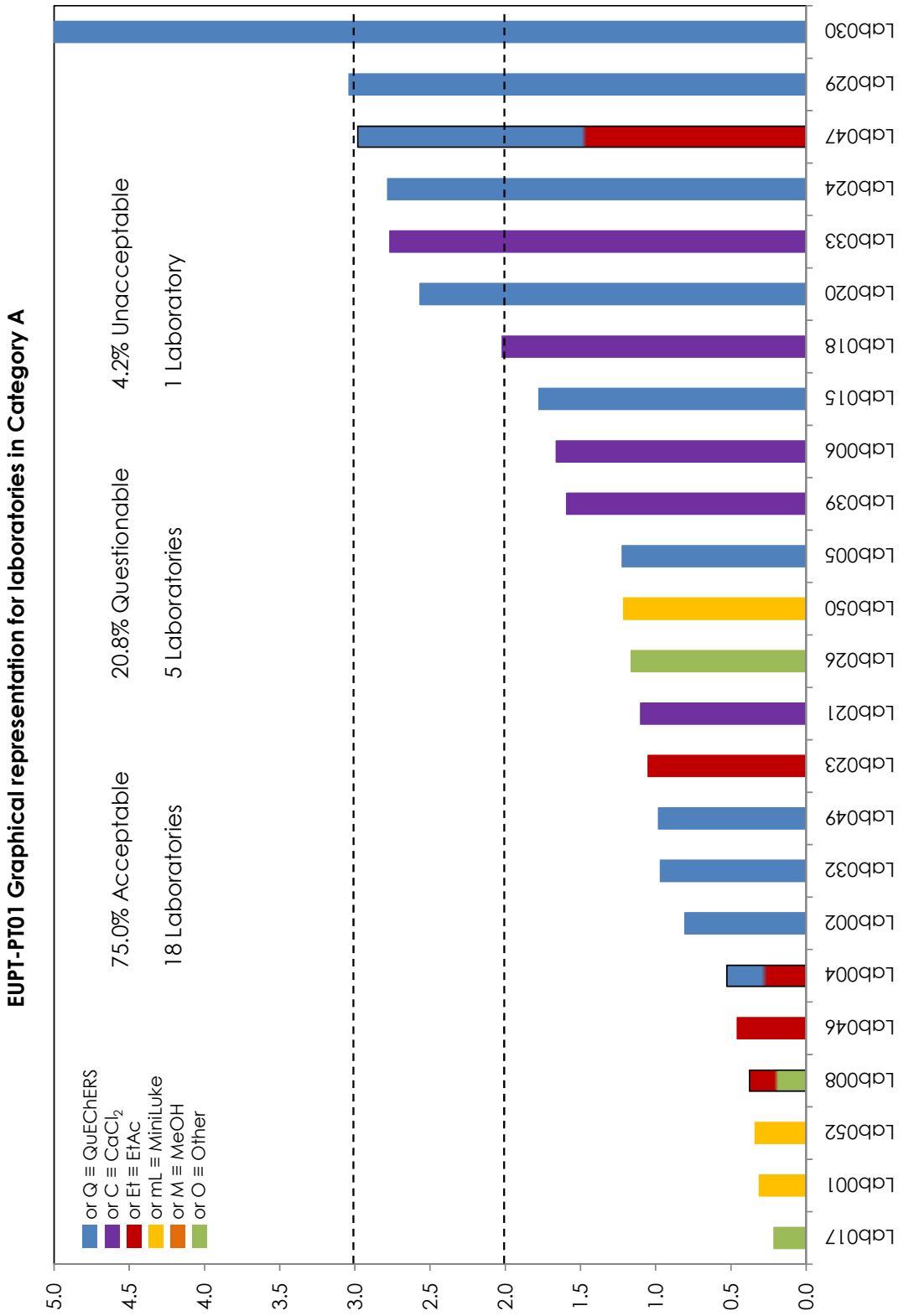
APPENDIX 5. 'Average Sum of z-Scores' (AZ^2) for laboratories in Category A.

Lab Code	Acetamiprid	Buprofezin	Carbendazim	Chlorpyrifos	Cypermethrin	Difenoconazole	Endosulfan beta	Etofenprox	Fenpropathrin	Imidacloprid	Lambda-Cyhalothrin	Methomyl	Parathion-ethyl	Pyridaben	Tebuconazole	No. of Pesticides	AZ^2
	z-score																
01	0.4	-0.1	0.7	0.0	-0.6	0.0	1.0	-0.3	-0.6	1.4	-0.1	0.0	0.3	-0.1	0.3	15	0.3
02	0.9	0.1	1.1	-0.1	-0.1	0.7	2.4	0.8	0.5	1.0	-0.1	0.9	-0.1	0.6	0.7	15	0.8
04	-0.2	-0.5	0.2	-0.5	1.3	-0.7	-0.9	0.0	0.3	0.7	-0.2	-1.0	-1.4	-0.3	-0.7	15	0.5
05	0.2	-0.8	0.3	-0.4	0.4	0.7	-0.1	3.3	-0.2	1.1	-0.2	0.0	-0.5	1.8	1.0	15	1.2
06	-3.3	-1.3	0.3	-0.7	0.0	0.4	-1.3	-0.4	-0.9	-1.3	0.0	-0.3	-1.5	-1.4	-1.8	15	1.7
08	0.4	0.0	-0.2	0.5	-0.7	0.1	-0.8	-0.5	-1.7	0.1	0.0	0.9	0.3	0.0	0.1	15	0.4
15	0.1	0.9	-2.1	0.8		1.1	2.1	-2.8	-0.8	-0.2	-0.3	-1.4	1.6	0.4	0.0	14	1.8
17	0.2	0.1	0.0	0.3	-1.1	0.0	-0.3	-0.3	-0.3	0.8	-0.1	0.7	-0.5	0.0	0.3	15	0.2
18	-0.4	-0.4	-0.3	0.5	-3.3	-1.3	1.4	-0.2	0.3	-1.4	2.2	-2.7	0.3	-0.1	-0.6	15	2.0
20	-0.1	-1.3	-2.7	-1.4	-2.4	-0.6	-2.4	-2.0	-0.1	-3.0	-0.9	0.3	-0.1	-1.1	0.7	15	2.6
21	0.2	-1.4	0.9	1.3	0.1	0.4	-0.8	-0.4	-0.3	-0.7	0.5	-3.1	0.5	0.7	-0.1	15	1.1
23	0.9	0.9	2.3	1.6	-0.2	1.2	1.5	0.5	-0.1	0.9	-0.6	0.6	-0.1	0.2	0.7	15	1.0
24	0.0	1.2	4.3	1.9	1.1	1.1	1.8	1.4	1.3	1.5	1.2	0.9	0.5	1.9	0.2	15	2.8
26	0.3	0.1	-0.7	1.2	0.6	0.5	0.4	0.4	0.5	0.5	1.5	-3.1	1.3	0.6	-0.3	15	1.2
29	2.1	2.1	3.5	1.9	2.8	0.9	0.5	0.8	1.1	1.6	1.2	0.5	0.8	1.4	1.8	15	3.0
30	4.6	3.2	0.1	0.5	4.7	2.0	3.2	2.2	1.9	0.7	2.1	3.4	-0.5	0.7	1.3	15	5.0
32	-0.1	1.2	0.5	1.0	0.9	0.7	0.1	1.5	0.8	0.5	1.8	1.2	0.7	1.1	0.8	15	1.0
33	-0.9	-3.6	-0.4	0.9	-2.3	-0.6	2.9	-1.6	0.1	-2.1	0.0	0.3	-2.2	-0.9	-0.7	15	2.8
39	-1.3	-0.2	-0.3	-0.3	-3.3	-0.9	1.4	0.0	0.5	-0.7	1.4	-2.3	-0.4	-0.3	-0.1	15	1.6
46	0.0	-0.7	1.0	-0.3	1.3	-0.3	1.3	0.5	0.0	0.4	0.9	0.1	-0.7	-0.2	-0.1	15	0.5
47	1.0	1.3	5.0	0.2	0.3	1.2	-3.0	0.4	-0.4	1.4	0.2	0.2	0.5	1.6	1.2	15	3.0
49	-0.7	0.2	-1.0	-1.1	2.4	-0.1	-0.6	-0.7	0.0	-0.7	1.1	-1.3	-1.0	0.8	0.5	15	1.0
50	0.6	1.0	3.2	0.7	0.6	0.9	-0.4	1.5	0.9	-0.8	0.3	-0.6	-0.3	0.0	0.7	15	1.2
52	-0.9	0.0	0.0	-0.2	-0.4	-0.1	-0.7	-0.5	-0.2	-0.5	0.3	-1.6	0.1	-0.6	0.3	15	0.3

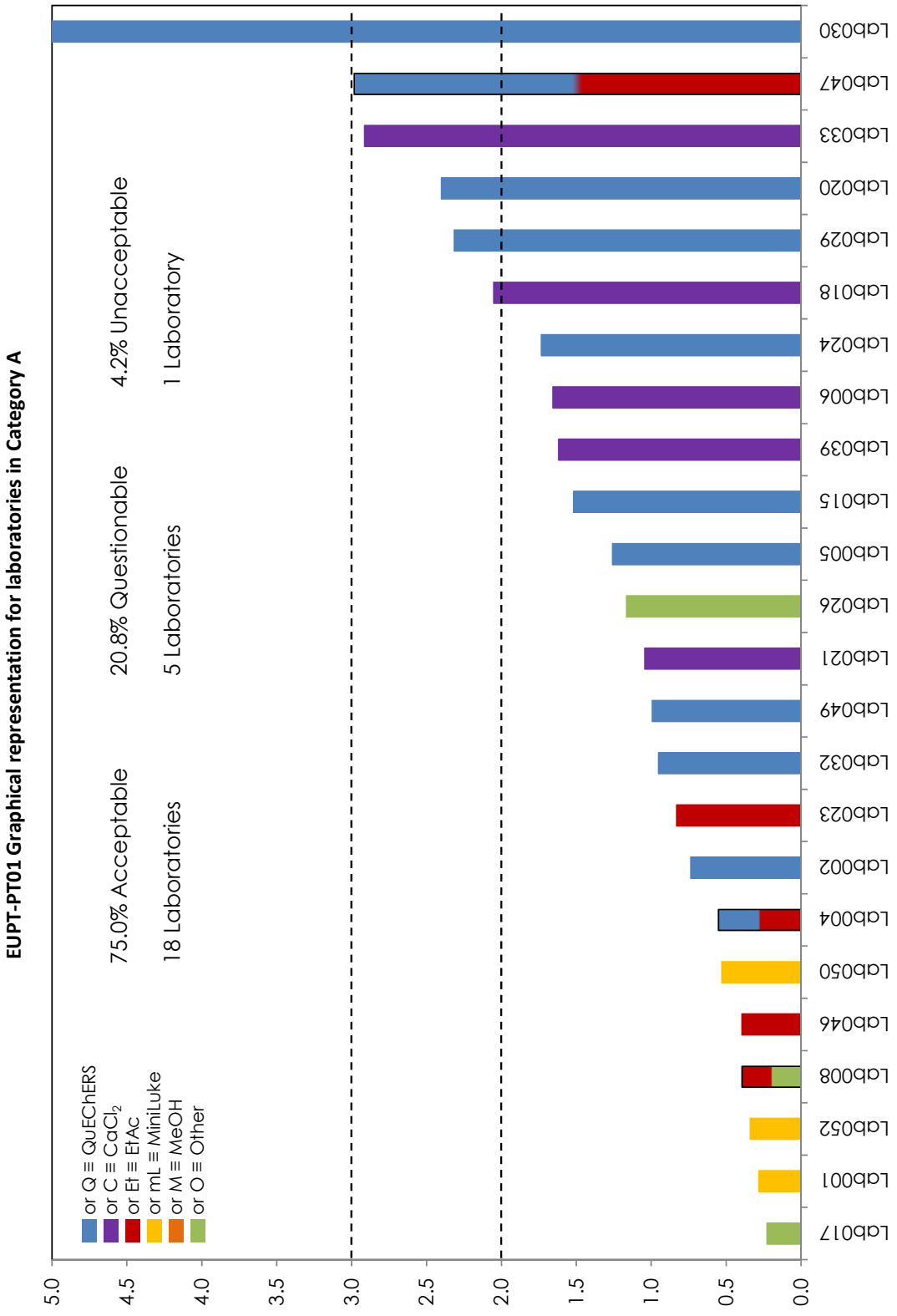
APPENDIX 6. EUPT-T01. AZ² Graphical representation for laboratories in Category A



AZ² using individual results for carbendazim and thiophanate methyl



AZ² using the sum of carbendazim and thiophanate methyl.



APPENDIX 7.Methods used by participants for determining pesticides.

Acetamiprid																			
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
001	D	0.01	0.12	71.1	5	Yes (10ml)	Acetone	Petroleum ether	Dichloromethane	No	None	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Via Standard Addition	No		ESI (Electrospray Ionisation)	Positive
002	D	0.02	0.133		2	Yes (10 mL)	Acetonitrile			Yes	DSPE, citrate buffered, dSPE with PSA	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Via Standard Addition	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
003	D	< 0,01	0.01	85	10	No	Acetone	Dichloromethane		No	None	Pure solvent-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
004	D	0.05	0.103	100	3	Yes (15 mL)	Acetonitrile			No	Freezing out	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
005	D	0.02	0.114	90	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition		MS (QQQ)	None	Via Standard Addition	No		ESI (Electrospray Ionisation)	Positive
006	ND	0.02	0.02		2	4	Acetonitrile			Yes	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
007	D	0.01	0.093	101	2	4	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)		Rec. from validation data	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
008	D	0.01	0.12	87	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)	LC-MS (QQQ)	Other untreated green tea was spiked	Yes: Other	no calculation, only to check extraction efficiency	ESI (Electrospray Ionisation)	Negative
009	D	0.01	0.143	92.5	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	triphenylphosphate	ESI (Electrospray Ionisation)	Positive
010	NA																			
011	D	0.02	0.051	70	2	4	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
012	D	0.01	0.09	80	2	10	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (QQQ)	MS (QQQ)	None	Rec. from validation data	Yes: Other	TFP	ESI (Electrospray Ionisation)	Positive
013	NA																			
014	D	0.01	0.243	142	2	no	Methanol	Water		no	Filter	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
015	D	0.02	0.112	74	2	Yes - 10ml	Acetonitrile			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (IT)	MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP (LC) / Bromophos methyl - (GC)	ESI (Electrospray Ionisation)	Positive
016	D	0.01	0.013	109	2	Yes (10mL)	Acetonitrile			No	DSPE, Graphitized Carbon Black	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
017	D	0.01	0.113	98.8	2.5	7.5	Acetonitrile			No	DSPE, DSPE with graphitised carbon and PSA	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Acetamiprid																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
018	D	0.02	0.096	119.2	2	10	Acetonitrile			Yes	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	Yes: Other	Triphenyl phosphate	ESI (Electrospray Ionisation)	Positive
019	D	0.01	0.222	95.8	2	10	Acetonitrile			No	SPE, solid phase extraction column	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch			ESI (Electrospray Ionisation)	Positive
020	D	0.02	0.105	103	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level		MS (Orbitrap)	LC-MS (Orbitrap)	Rec. from validation data	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
021	D	0.02	0.113	99	2	10	Acetonitrile			No	DSPE, CaCl2 and -NH2 (instead of PSA)	Matrix matched-Single Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
022	NA																			
023	D	0.01	0.133	91	2	4	Ethyl acetate			Yes	Other (filtration)	Standard Addition		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
024	D	0.02	0.108	100	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	None	Other - via procedural matrix calibration	Yes: Other	Triphenylphosphate	ESI (Electrospray Ionisation)	Positive
025	D	0.05	0.12	93.7	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
026	D	0.04	0.116	105	2.5	7.5	Methanol	Ammonium acetate		No	Other: dilution by 10 and filtration	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	Oxfendazole	ESI (Electrospray Ionisation)	Positive
027	ND	0.01	0.01		3	5	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from validation data	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
028	D	0.01	0.094	74	2	Yes (4 mL)	Acetonitrile			No	None	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
029	D	0.02	0.166	91.9	2	10	Acetonitrile	Acetonitrile		Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TRIS	ESI (Electrospray Ionisation)	Positive
030	D	0.02	0.233	90	2	Yes 10 ml	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from validation data	No		ESI (Electrospray Ionisation)	Positive
031	ND	0.10	0.02	78	2	Yes (8 mL)	Ethyl acetate			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (Q)		GC-MS (Q)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	Positive
032	D	0.02	0.105	94	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Isotopically labelled	Linuron-D6	ESI (Electrospray Ionisation)	Positive
033	D	0.02	0.0849	83.4	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction, then CaCl2	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
034	D	0.02	0.082	70	2	Yes 4mL	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Acetamiprid																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
035	D	0.01	0.087	81.3	2g	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level	MS (QQQ)	MS (QQQ)		Via Standard Addition	Yes: Other	tris(2-chloro-1(chloromethyl)ethyl)phosphate	EI (Electron Ionisation)	Positive
036	NA																			
037	No Results Submited																			
038	No Results Submited																			
039	D	0.02	0.072	101.7	2	Yes (10 mL)	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
040	D	0.005	0.17	Blank with large amount of particular pesticide	2	Yes,10	Acetonitrile+ water(1:1)			Yes	DSPE, dispersive solid phase extraction	Standard Addition		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
041	D	0.01	0.064	85	2.5	Yes (10 ml)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from validation data	No		ESI (Electrospray Ionisation)	Positive
042	NA																			
043	D	0.01	0.1	99	5	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Other	TDCPP		
044	D	0.02	0.124	94.2	1	Yes (2.5 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction(PSA)	Matrix matched-Multiple Level	Other	MS (Orbitrap)+MSMS(QQQ)	LC-MS (Orbitrap) +LC-MS（QQQ）	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
045	NA																			
046	D	0.02	0.108	92	2	10	Ethyl acetate			No	Filtration	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
047	D	0.02	0.135	50	0.5	10	Acetonitrile			No	DSPE, PSA	Pure solvent-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
048	NA																			
049	D	0.025	0.09	90	2	4	Acetonitrile			Yes	DSPE, dispersive solid phase extraction (PSA/CaCl2)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
050	D	0.01	0.123	94	2	Yes (13 mL)	Acetone	Dichloromethane	Petroleum ether (PE)	No	None	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
051	ND	0.01	0.01		2	10	Acetonitrile			No	DSPE, C18 y PSA	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
052	D	0.01	0.083	70	2.5	Yes (5 mL)	Acetone	Dichloromethane	Petroleum ether (PE)	No	Other (Na2SO4)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
053	D	0.01	0.125	75	2	Yes,	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition		MS (QQQ)	LC-MS (QQQ)	Via Standard Addition	Yes: Other	tris-(1,3-dichlorisopropyle)-phosphate	ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Acetamiprid																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
054	ND	0.03	0.02		2	No	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level with complete sample preparation		MS (QQQ)	LC-MS (QQQ)		Yes: Isotopically labelled	Atrazin D5	ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Buprofezin

Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
001	D	0.01	0.176	96.3	5	Yes (10ml)	Acetone	Other (Petroleum ether)	Dichloromethane	No	None	Pure solvent-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Via Standard Addition	No		ESI (Electrospray Ionisation)	Positive
002	D	0.02	0.186	98	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, citrate buffered, dSPE with PSA	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP, Tributylphosphat	EI (Electron Ionisation)	Positive
003	D	< 0,02	0.09	89		No	Acetone	Dichloromethane		No	None	Matrix matched-Multiple Level	MS (IT)		None	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
004	D	0.05	0.159	94	3	Yes (15 mL)	Acetonitrile			No	Freezing out	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
005	D	0.02	0.144	76	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition	MS (QQQ)		None	Via Standard Addition	Yes: Other	Triphenylphosphate	EI (Electron Ionisation)	Positive
006	D	0.02	0.12	102			Acetone	Dichloromethane	Petroleum ether	No	None	Matrix matched-Multiple Level	MS (QQQ)			Rec. from the same batch	No		EI (Electron Ionisation)	
007	D	0.01	0.061		2	4	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)		Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	Positive
008	D	0.02	0.18	89	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)	LC-MS (QQQ)	Other untreated green tea was spiked	Yes: Other	no calculation, only to check extraction efficiency	ESI (Electrospray Ionisation)	Negative
009	D	0.01	0.255	74	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	triphenylphosphate	ESI (Electrospray Ionisation)	Positive
010	ND	0.05	0.02		5	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from validation data	No		ESI (Electrospray Ionisation)	Positive
011	D	0.02	0.133	60	2	4	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
012	D	0.01	0.37	110	2	10	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (QQQ)	MS (QQQ)	None	Rec. from validation data	Yes: Other	TFP	ESI (Electrospray Ionisation)	Positive
013	D	0.05	0.21	60	1	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level	Other		Other					
014	D	0.01	0.158	102	2	no	Methanol	Water		no	Filter	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
015	D	0.02	0.22	115	2	Yes - 10ml	Acetonitrile			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (IT)	MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: TPP (LC) / Bromophos methyl - (GC)		ESI (Electrospray Ionisation)	Positive
016	D	0.01	0.11	100	2	Yes (10mL)	Acetonitrile			No	DSPE, Graphitized Carbon Black	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Buprofezin																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
017	D	0.01	0.184	85	2.5	7.5	Acetonitrile			No	DSPE, DSPE with graphitised carbon and PSA	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
018	D	0.02	0.16	89.7	2	10	Acetonitrile			Yes	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Rec. from the same batch	Yes: Other	Triphenyl phosphate	ESI (Electrospray Ionisation)	Positive
019	D	0.01	0.241	91.4	2	10	Acetonitrile			No	SPE, solid phase extraction column	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch			ESI (Electrospray Ionisation)	Positive
020	D	0.02	0.121	98	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level		MS (Orbitrap)	LC-MS (Orbitrap)	Rec. from validation data	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
021	D	0.02	0.118	70	2	10	Acetonitrile			No	DSPE, CaCl2 and -NH2 (instead of PSA)	Matrix matched-Single Level	MS (QQQ)		GC-MS (Q)	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
022	NA																			
023	D	0.01	0.222	105	2	4	Ethyl acetate			Yes	Other (filtration)	Standard Addition		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
024	D	0.02	0.236	100	2	10	Acetonitrile	n-Hexane		No	DSPE, LLE with n-hexane	Matrix matched-Multiple Level	MS (QQQ)		LC-MS (QQQ)	Other - via procedural matrix calibration	Yes: Other	Triphenylphosphate	EI (Electron Ionisation)	
025	D	0.05	0.2	104	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
026	D	0.04	0.185	84	2.5	7.5	Methanol	Other: ammonium acetate		No	Other: dilution by 10 and filtration	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	Oxfendazole	ESI (Electrospray Ionisation)	Positive
027	D	0.01	0.2	89.1	2	5	acetone/hexane/ethyl acetate			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (Q)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	
028	D	0.01	0.115	97	2	Yes (4 mL)	Acetonitrile			No	None	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
029	D	0.02	0.274	88.1	2	10	Acetonitrile	Acetonitrile		Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TRIS	ESI (Electrospray Ionisation)	Positive
030	D	0.02	0.325	85	2	Yes 10 ml	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from validation data	No		ESI (Electrospray Ionisation)	Positive
031	ND	0.06	0.02		2	Yes (8 mL)	Ethyl acetate			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (Q)		GC-MS (Q)	Rec. from validation data	Yes: Other (TPP)		EI (Electron Ionisation)	Positive
032	D	0.02	0.236	84	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Isotopically labelled	Linuron-D6	ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Buprofezin																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
033	ND	0.02	0.02		2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction, then CaCl2	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
034	ND	0.02	0.02	98	3	Yes 6ml	Acetone	Dichloromethane	Petroleum ether	No	None	Matrix matched-Multiple Level	MS (QQQ) FPD ECD		None	Rec. from the same batch	No			
035	D	0.01	0.158	63.2	2g	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level	MS (QQQ)	MS (QQQ)		Via Standard Addition	Yes: Other	tris(2-chloro-1(chloromethyl)ethyl)phosphate	EI (Electron Ionisation)	Positive
036	D	0.01	0.12	96	2	10	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TRISCP	EI (Electron Ionisation)	Positive
037	No Results Submited																			
038	No Results Submited																			
039	D	0.02	0.17	85.6	2	Yes (10 mL)	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level		MS (QQQ)	GC-MS (IT)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
040	D	0.01	0.26	64.2	15	Yes, 9ml	ACETONE with 30% water	Dichloromethane	Petroleum Ether	No	Liquid-liquid partitioning	Standard Addition	MS (Q)		GC-MS (Q)	Rec. from the same batch	No		EI (Electron Ionisation)	
041	D	0.01	0.12	75	2.5	Yes (10 ml)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from validation data	No		ESI (Electrospray Ionisation)	Positive
042	D	0.05	0.26	70	2	10 mL	Acetonitrile	isoottano		No	Liquid-liquid partitioning	Pure solvent-Single Level	NPD		GC-MS (Q)	Rec. from validation data				
043	D	0.01	0.223	56	5	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Other	TDCPP		
044	D	0.01	0.226	88.5	1	Yes (2.5 mL)	Acetonitrile			No	SPE, solid phase extraction column(Carb/PSA)+onlineGPC	Matrix matched-Single Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
045	D	0.01	0.19	90.5	2	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
046	D	0.02	0.149	92	2	10	Ethyl acetate			No	Filtration	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
047	D	0.02	0.237	78	0.5	10	Acetonitrile			No	DSPE, PSA	Pure solvent-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
048	NA																			
049	D	0.025	0.19	101	2	4	Acetonitrile			Yes	DSPE, dispersive solid phase extraction (PSA/CaCl2)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
050	D	0.01	0.223	91	2	Yes (13 mL)	Acetone	Dichloromethane	Petroleum ether (PE)	No	None	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Buprofezin																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
051	D	0.01	0.027	60	2	10	Acetonitrile			No	DSPE, C18 y PSA	Matrix matched-Multiple Level	MS (QQQ)			Rec. from the same batch	No		EI (Electron Ionisation)	
052	D	0.01	0.18	100	2.5	Yes (5 mL)	Acetone	Dichloromethane	Other (PE)	No	Other (Na2SO4)	Standard Addition		MS (QQQ)	LC-MS (QQQ)	Via Standard Addition	No		ESI (Electrospray Ionisation)	Positive
053	ND	0.01	0.01		2	Yes (please, specify mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition	MS (Q)		GC-MS (Q)	Via Standard Addition	Yes: Other	PCB 31	EI (Electron Ionisation)	Negative
054	D	0.03	0.077		2	No	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (Q)		GC-MS (Q)		No		EI (Electron Ionisation)	

APPENDIX 7.Methods used by participants for determining pesticides.

Carbendazim																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
001	D	0.01	0.292	82.9	5	Yes (10ml)	Acetone	Other (Petroleum ether)	Dichloromethane	No	None	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Via Standard Addition	No		ESI (Electrospray Ionisation)	Positive
002	D	0.02	0.321		2	Yes (10 mL)	Acetonitrile			Yes	DSPE, citrate buffered, dSPE with PSA	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Via Standard Addition	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
003	D	< 0,01	0.09	94	10	No	Acetone	Dichloromethane		No	None	Pure solvent-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
004	D	0.05	0.263	83	3	Yes (15 mL)	Acetonitrile			No	Freezing out	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
005	D	0.02	0.266	79	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition		MS (QQQ)	None	Via Standard Addition	No		ESI (Electrospray Ionisation)	Positive
006	D	0.02	0.267	83	2	4	Acetonitrile			Yes	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
007	D	0.01	0.23		2	4	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)		Rec. from validation data	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
008	D	0.02	0.24	87	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)	LC-MS (QQQ)	Other untreated green tea was spiked	Yes: Other	no calculation, only to check extraction efficiency	ESI (Electrospray Ionisation)	Negative
009	D	0.01	0.33	110	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	triphenylphosphate	ESI (Electrospray Ionisation)	Positive
010	D	0.02	0.061	101	5	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from validation data	No		ESI (Electrospray Ionisation)	Positive
011	D	0.02	0.105	80	2	4	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
012	D	0.01	0.3	75	2	10	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (QQQ)	MS (QQQ)	None	Rec. from validation data	Yes: Other	TFP	ESI (Electrospray Ionisation)	Positive
013	NA																			
014	D	0.01	0.41	288	2	no	Methanol	Water		no	Filter	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
015	D	0.02	0.119	117	2	Yes - 10ml	Acetonitrile			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (IT)	MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP (LC) / Bromophos methyl - (GC)	ESI (Electrospray Ionisation)	Positive
016	D	0.01	0.042	66	2	Yes (10mL)	Acetonitrile			No	DSPE, Graphitized Carbon Black	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive

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Carbendazim																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
017	D	0.01	0.247	98.8	2.5	7.5	Acetonitrile			No	DSPE, DSPE with graphitised carbon and PSA	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
018	D	0.02	0.23	068.2	2	10	Acetonitrile			Yes	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	Yes: Other	Triphenyl phosphate	ESI (Electrospray Ionisation)	Positive
019	D	0.01	0.488	90.1	2	10	Acetonitrile			No	SPE, solid phase extraction column	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch			ESI (Electrospray Ionisation)	Positive
020	D	0.02	0.079	89	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level		MS (Orbitrap)	LC-MS (Orbitrap)	Rec. from validation data	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
021	D	0.02	0.309	90	2	10	Acetonitrile			No	DSPE, CaCl2 and -NH2 (instead of PSA)	Matrix matched-Single Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
022	D	0.02	0.082	70	2	Yes (4 mL)	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level		MS (IT)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
023	D	0.01	0.393	72	2	4	Ethyl acetate			Yes	Other (filtration)	Standard Addition		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
024	D	0.02	0.519	100	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	None	Other - via procedural matrix calibration	Yes: Other	Triphenylphosphate	ESI (Electrospray Ionisation)	Positive
025	D	0.1	0.26	37.8	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
026	D	0.04	0.204	104	2.5	7.5	Methanol	Other: ammonium acetate		No	Other: dilution by 10 and filtration	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	Oxfendazole	ESI (Electrospray Ionisation)	Positive
027	D	0.01	0.078	71.3	3	5	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from validation data	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
028	D	0.01	0.188	80	2	Yes (4 mL)	Acetonitrile			No	None	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
029	D	0.02	0.469	86.9	2	10	Acetonitrile	Acetonitrile		Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TRIS	ESI (Electrospray Ionisation)	Positive
030	D	0.02	0.257	117	2	Yes 10 ml	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from validation data	No		ESI (Electrospray Ionisation)	Positive
031	NA																			
032	D	0.02	0.283	83	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Isotopically labelled	Linuron-D6	ESI (Electrospray Ionisation)	Positive

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Carbendazim																			
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
033	D	0.02	0.226	64.1	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction, then CaCl2	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
034	D	0.02	0.25	70	2	Yes 4mL	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
035	D	0.01	0.402	75	2g	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level	MS (QQQ)	MS (QQQ)		Via Standard Addition	Yes: Other	tris(2-chloro-1(chloromethyl)ethyl)phosphate	EI (Electron Ionisation)	Positive
036	NA																			
037		No Results Submited																		
038		No Results Submited																		
039	D	0.02	0.23	73.0	2	Yes (10 mL)	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
040	D	0.005	0.42	60	2	Yes,10	Acetonitrile+ water(1:1)			Yes	DSPE, dispersive solid phase extraction	Standard Addition		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
041	D	0.01	0.15	75	2.5	Yes (10 ml)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from validation data	No		ESI (Electrospray Ionisation)	Positive
042	NA																			
043	D	0.025	0.043	99	5	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Other	TDCPP		
044	D	0.02	0.491	92.5	1	Yes (2.5 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction(PSA)	Matrix matched-Multiple Level	Other	MS (Orbitrap)+MSMS(QQQ)	LC-MS (Orbitrap) +LC-MS （QQQ）	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
045	D	0.01	0.15	67.3	2	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
046	D	0.02	0.315	58	2	10	Ethyl acetate			No	Filtration	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
047	D	0.02	0.732	43	0.5	10	Acetonitrile			No	DSPE, PSA	Pure solvent-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
048	NA																			
049	D	0.025	0.19	107	2	4	Acetonitrile			Yes	DSPE, dispersive solid phase extraction (PSA/CaCl2)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
050	D	0.01	0.453	81	2	Yes (13 mL)	Acetone	Dichloromethane	Petroleum ether (PE)	No	None	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Carbendazim																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
051	D	0.01	0.052	60	2	10	Acetonitrile			No	DSPE, C18 y PSA	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
052	D	0.01	0.25	86	2.5	Yes (5 mL)	Acetone	Dichloromethane	Other (PE)	No	Other (Na2SO4)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
053	D	0.01	0.366	71	2	Yes	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition		MS (QQQ)	LC-MS (QQQ)	Via Standard Addition	Yes: Other	tris-(1,3-dichlorisopropyle)-phosphate	ESI (Electrospray Ionisation)	Positive
054	D	0.05	0.235		2	No	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level with complete sample preparation		MS (QQQ)	LC-MS (QQQ)		Yes: Isotopically labelled	Atrazin D5	ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Chlorpyrifos																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
001	D	0.01	0.364	99.1	5	Yes (10ml)	Acetone	Other (Petroleum ether)	Dichloromethane	No	None	Other (Apple matrix multiple level)	MS (QQQ)		GC-MS (QQQ)	Via Standard Addition	Yes: Other	PCB 153	EI (Electron Ionisation)	Positive
002	D	0.02	0.356	105	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, citrate buffered, dSPE with PSA	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP, Tributylphosphat	EI (Electron Ionisation)	Positive
003	D	< 0,01	0.23	101		No	Acetone	Dichloromethane		No	None	Matrix matched-Multiple Level	MS (IT)		None	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
004	D	0.02	0.318	82	3	Yes (15 mL)	Acetonitrile			No	Liquid-liquid partitioning, isooctane with the adition of 20% NaCL	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Isotopically labelled	TPP	EI (Electron Ionisation)	
005	D	0.02	0.327	83	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition	MS (QQQ)		None	Via Standard Addition	Yes: Other	Triphenylphosphate	EI (Electron Ionisation)	Positive
006	D	0.02	0.301	104			Acetone	Dichloromethane	Petroleum ether	No	None	Matrix matched-Multiple Level	MS (QQQ)			Rec. from the same batch	No		EI (Electron Ionisation)	
007	D	0.01	0.38	95	2	4	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)		Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	Positive
008	D	0.02	0.41	91	5		Accelerated Solvent	Cyclohexane	Ethyl acetate	No	Accelerated Solvent, GPC, gel permeation chromatography Mini-Silicagel column	Pure solvent-Single Level	Other FPD and ECD		Two columns	Other untreated green tea was spiked	Yes: Other	no calculation, only to check extraction efficiency		
009	D	0.01	0.42	120	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	triphenylphosphate	ESI (Electrospray Ionisation)	Positive
010	D	0.02	0.067	79	5	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from validation data	No		ESI (Electrospray Ionisation)	Positive
011	D	0.02	0.179	75	2	4	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
012	D	0.01	0.385	99	4	No	Acetonitrile	Dichloromethane	Petroleum ether	No	Liquid-liquid partitioning	Matrix matched-Multiple Level	MS (QQQ)		None	Rec. from validation data	Yes: Other	TFP	EI (Electron Ionisation)	
013	ND	0.05	0.02		1	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level	Other		Other					
014	NA																			
015	D	0.02	0.44	122	2	Yes - 10ml	Acetonitrile			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (IT)	MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: TPP (LC) / Bromophos methyl - (GC)		EI (Electron Ionisation)	

APPENDIX 7.Methods used by participants for determining pesticides.

Chlorpyrifos																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
016	D	0.01	0.14	70	2	Yes (10mL)	Acetonitrile			No	DSPE, Graphitized Carbon Black	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	
017	D	0.01	0.395	93.3	2.5	7.5	Acetonitrile			No	DSPE + Liquid-liquid partitioning with hexane	Matrix matched-Multiple Level	MS (QQQ)		LC-MS (QQQ)	Rec. from the same batch	Yes: Other	Bromophos methyl	EI (Electron Ionisation)	
018	D	0.02	0.41	119.0	2	10	Acetonitrile			Yes	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	FPD		GC-MS (Q)	Rec. from the same batch	Yes: Other	Triphenyl phosphate	no	
019	NA																			
020	D	0.02	0.235	108	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	Negative
021	D	0.02	0.478	103	2	10	Acetonitrile			No	DSPE, CaCl2 and -NH2 (instead of PSA)	Matrix matched-Single Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	No		CI (Chemical Ionisation)	Negative
022	D	0.02	0.068	80	2	Yes (4 mL)	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level	MS (IT)		None	Rec. from the same batch	Yes: Isotopically labelled	fenclorfos	EI (Electron Ionisation)	Positive
023	D	0.01	0.514	77	2	4	Ethyl acetate			Yes	Other (filtration)	Standard Addition	MS (QQQ)		None	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
024	D	0.02	0.538	100	2	10	Acetonitrile	n-Hexane		No	DSPE, LLE with n-hexane	Matrix matched-Multiple Level	MS (QQQ)		LC-MS (QQQ)	Other - via procedural matrix calibration	Yes: Other	Triphenylphosphate	EI (Electron Ionisation)	
025	D	0.05	0.4	107.2	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
026	D	0.02	0.471	93.1	2.5	7.5	Acetone			No	Liquid-liquid partitioning	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
027	D	0.01	0.36	88	2	5	acetone/hexane/ethyl acetate			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (Q)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	
028	D	0.01	0.319	118	2	Yes (4 mL)	Acetonitrile			No	None	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
029	D	0.02	0.538	110.9	2	10	Acetonitrile	n-Hexan		No	Liquid-liquid partitioning	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	PCB 209	EI (Electron Ionisation)	Positive
030	D	0.02	0.41	100	2	Yes 10 ml	Acetonitrile	Ethyl acetate		No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (Q)	Rec. from validation data	No		EI (Electron Ionisation)	Positive
031	D	0.03	0.33	73	2	Yes (8 mL)	Ethyl acetate			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (Q)		GC-MS (Q)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	Positive
032	D	0.02	0.459	96	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (Q)			Rec. from the same batch	Yes: Other	Triphenylmethan	EI (Electron Ionisation)	Positive

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Chlorpyrifos																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
033	D	0.02	0.446	35	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction, then CaCl2	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
034	D	0.02	0.427	95	3	Yes 6ml	Acetone	Dichloromethane	Petroleum ether	No	None	Matrix matched-Multiple Level	MS (QQQ) FPD ECD		None	Rec. from the same batch	No			
035	D	0.01	0.411	118.9	2g	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level	MS (QQQ)	MS (QQQ)		Via Standard Addition	Yes: Other	tris(2-chloro-1(chloromethyl)ethyl)phosphate	EI (Electron Ionisation)	Positive
036	D	0.01	0.29	92	2	10	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TRISCP	EI (Electron Ionisation)	Positive
037	No Results Submited																			
038	No Results Submited																			
039	D	0.02	0.34	98.1	2	Yes (10 mL)	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		Other (GC-ECD)	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
040	D	0.01	0.43	77.6	15	Yes, 9ml	ACETONE with 30% water	Dichloromethane	Petroleum Ether	No	Liquid-liquid partitioning	Standard Addition	FPD		GC-MS (Q)	Rec. from the same batch	No		EI (Electron Ionisation)	
041	D	0.02	0.26	71	2.5	Yes (10 ml)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from validation data	No		ESI (Electrospray Ionisation)	Positive
042	D	0.05	0.32	85	2	10 mL	Acetonitrile	isoottano		No	Liquid-liquid partitioning	Pure solvent-Single Level	NPD		GC-MS (Q)	Rec. from validation data				
043	D	0.01	0.428	100	5	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (Q)			Rec. from the same batch	Yes: Other	TDCPP		
044	D	0.02	0.484	94.9	1	Yes (2.5 mL)	Acetonitrile			No	SPE, solid phase extraction column(Carb/PSA)+onlineGPC	Matrix matched-Single Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
045	NA																			
046	D	0.02	0.34	100	2	10	Ethyl acetate			No	Filtration	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
047	D	0.02	0.384	101	2.5	10	Ethyl acetate			No	None	Standard Addition	MS (QQQ)		None	Rec. from the same batch	Yes: Other	PCB-28	EI (Electron Ionisation)	Positive
048	D	0.05	0.27	110	2	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (IT)		GC-MS (Q)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	
049	D	0.025	0.26	75	2	4	Acetonitrile			Yes	DSPE, dispersive solid phase extraction(PSA/CaCl2)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP		

APPENDIX 7.Methods used by participants for determining pesticides.

Chlorpyrifos																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
050	D	0.01	0.428	96	2	Yes (13 mL)	Acetone	Dichloromethane	Petroleum ether (PE)	No	None	Matrix matched-Multiple Level	MS (ITD)		GC-MS (QQQ)	Rec. from the same batch	Yes: Isotopically labelled (target pesticide)		EI (Electron Ionisation)	Positive
051	D	0.01	0.11	70	2	10	Acetonitrile			No	DSPE, C18 y PSA	Matrix matched-Multiple Level	MS (QQQ)			Rec. from the same batch	No		EI (Electron Ionisation)	
052	D	0.005	0.35	85	2.5	Yes (5 mL)	Acetone	Dichloromethane	Other (PE)	No	Other (Na2SO4)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
053	D	0.01	0.11	70	2	Yes	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition	MS (Q)		GC-MS (Q)	Via Standard Addition	Yes: Other	PCB 31	EI (Electron Ionisation)	Negative
054	D	0.03	0.208		2	No	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	ECD		Two columns		No			

APPENDIX 7.Methods used by participants for determining pesticides.

Cypermethrin																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
001	D	0.01	0.095	96.7	5	Yes (10ml)	Acetone	Other (Petroleum ether)	Dichloromethane	No	None	Other (Apple matrix multiple level)	MS (QQQ)		GC-MS (QQQ)	Via Standard Addition	Yes: Other	PCB 153	EI (Electron Ionisation)	Positive
002	D	0.02	0.109	96	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, citrate buffered, dSPE with PSA	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP, Tributylphosphat	EI (Electron Ionisation)	Positive
003	D	< 0,01	0.06	96		No	Acetone	Dichloromethane		No	None	Matrix matched-Multiple Level	MS (IT)		None	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
004	D	0.1	0.147	112	5	No	Ethyl acetate			No	GPC, gel permeation chromatography	Matrix matched-Multiple Level	ECD		GC-MS (Q)	Rec. from the same batch	No		CI (Chemical Ionisation)	Negative
005	D	0.02	0.124	90	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition	MS (QQQ)		None	Via Standard Addition	Yes: Other	Triphenylphosphate	EI (Electron Ionisation)	Positive
006	D	0.02	0.111	104			Acetone	Dichloromethane	Petroleum ether	No	None	Matrix matched-Multiple Level	MS (QQQ)			Rec. from the same batch	No		EI (Electron Ionisation)	
007	ND	0.01	0.01		2	4	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)		Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	Positive
008	D	0.01	0.092	73	5		Accelerated Solvent	Cyclohexane	Ethyl acetate	No	Accelerated Solvent, GPC, gel permeation chromatography Mini-Silicagel column	Pure solvent-Single Level	Other FPD and ECD		Two columns	Other untreated green tea was spiked	Yes: Other	no calculation, only to check extraction efficiency		
009	D	0.01	0.181	102	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	anthracene	EI (Electron Ionisation)	
010	NA																			
011	D	0.02	0.08	62	2	4	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
012	NA																			
013	D	0.05	0.16	100	1	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level	Other		Other					
014	NA																			
015	NA																			
016	ND	0.02	0.02		2	Yes (10mL)	Acetonitrile			No	DSPE, Graphitized Carbon Black	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	
017	D	0.01	0.08	100.5	2.5	7.5	Acetonitrile			No	DSPE + Liquid-liquid partitioning with hexane	Matrix matched-Multiple Level	MS (QQQ)		LC-MS (QQQ)	Rec. from the same batch	Yes: Other	Bromophos methyl	EI (Electron Ionisation)	

APPENDIX 7.Methods used by participants for determining pesticides.

Cypermethrin																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
018	ND	0.02	0.02																	
019	NA																			
020	D	0.02	0.046	95	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	Negative
021	D	0.02	0.114	96	2	10	Acetonitrile			No	DSPE, CaCl2 and -NH2 (instead of PSA)	Matrix matched-Single Level	MS (QQQ)		GC-MS (Q)	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
022	NA																			
023	D	0.01	0.106	105	2	4	Ethyl acetate			Yes	Other (filtration)	Standard Addition	MS (QQQ)		None	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
024	D	0.02	0.144	100	2	10	Acetonitrile	n-Hexane		No	DSPE, LLE with n-hexane	Matrix matched-Multiple Level	MS (Q)		None	Other - via procedural matrix calibration	Yes: Other		CI (Chemical Ionisation)	Negative
025	ND	0.1	0.02		2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	No		EI (Electron Ionisation)	
026	D	0.04	0.128	93.6	2.5	7.5	Acetone			No	Liquid-liquid partitioning	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
027	D	0.02	0.15	111.6	2	5	acetone/hexane/ethyl acetate			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (Q)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	
028	D	0.01	0.122	128	2	Yes (4 mL)	Acetonitrile			No	None	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
029	D	0.02	0.189	110.1	2	10	Acetonitrile	n-Hexan		No	Liquid-liquid partitioning	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	PCB 209	EI (Electron Ionisation)	Positive
030	D	0.02	0.2424	80	2	Yes 10 ml	Acetonitrile	Ethyl acetate		No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (Q)	Rec. from validation data	No		EI (Electron Ionisation)	Positive
031	ND	0.08	0.02	77	2	Yes (8 mL)	Ethyl acetate			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (Q)		GC-MS (Q)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	Positive
032	D	0.02	0.136	78	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (Q)			Rec. from the same batch	Yes: Other	TRIS	CI (Chemical Ionisation)	Negative
033	D	0.02	0.0473	385	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction, then CaCl2	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
034	D	0.02	0.105	90	3	Yes 6ml	Acetone	Dichloromethane	Petroleum ether	No	None	Matrix matched-Multiple Level	MS (QQQ) FPD ECD		None	Rec. from the same batch	No			
035	D	0.01	0.112	83	2g	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level	MS (QQQ)	MS (QQQ)		Via Standard Addition	Yes: Other	tris(2-chloro-1(chloromethyl)ethyl)phosphate	EI (Electron Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Cypermethrin																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
036	D	0.01	0.061	82	2	10	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TRISCP	EI (Electron Ionisation)	Positive
037	No Results Submited																			
038	No Results Submited																			
039	ND	0.02	0.02		2	Yes (10 mL)	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		Other (GC-ECD)	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
040	D	0.02	0.09	Blank with large amount of particular pesticide	2	Yes,10	Acetonitrile+ water(1:1)			Yes	DSPE, dispersive solid phase extraction	Standard Addition		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
041	NA																			
042	NA																			
043	ND	0.05	0.02		5	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Other	TDCPP		
044	D	0.01	0.178	94.2	1	Yes (2.5 mL)	Acetonitrile			No	SPE, solid phase extraction column(Carb/PSA)	Pure solvent-Multiple Level	MS (Q)		GC-MS (Q)	Rec. from the same batch	Yes: Isotopically labelled (target pesticide)		CI (Chemical Ionisation)	Negative
045	D	0.01	0.38	88.3	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (QQQ)		None	Rec. from the same batch	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
046	D	0.02	0.149	20	2	10	Ethyl acetate			No	GPC, gel permeation chromatography	Standard Addition	MS (QQQ)		GC-MS (QQQ)	Via Standard Addition	Yes: Other	TPP	EI (Electron Ionisation)	Positive
047	D	0.1	0.119	121	2.5	10	Ethyl acetate			No	None	Standard Addition	MS (QQQ)		None	Rec. from the same batch	Yes: Other	PCB-28	EI (Electron Ionisation)	Positive
048	NA																			
049	D	0.025	0.18	117	2	4	Acetonitrile			Yes	DSPE, dispersive solid phase extraction(PSA/CaCl2)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP		
050	D	0.01	0.128	101	2	Yes (13 mL)	Acetone	Dichloromethane	Petroleum ether (PE)	No	None	Matrix matched-Multiple Level	MS (QQQ)		LC-MS (QQQ)	Rec. from the same batch	Yes: Other	Bromophos-ethyl	CI (Chemical Ionisation)	Negative
051	D	0.01	0.026	60	2	10	Acetonitrile			No	DSPE, C18 y PSA	Matrix matched-Multiple Level	MS (QQQ)			Rec. from the same batch	No		EI (Electron Ionisation)	
052	D	0.01	0.1	89	2.5	Yes (5 mL)	Acetone	Dichloromethane	Other (PE)	No	Other (Na2SO4)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
053	ND	0.01	0.01		2	Yes	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition	MS (Q)		GC-MS (Q)	Via Standard Addition	Yes: Other	PCB 31	EI (Electron Ionisation)	Negative

APPENDIX 7.Methods used by participants for determining pesticides.

Cypermethrin																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
054	D	0.03	0.049		2	No	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level with complete sample preparation		MS (QQQ)	LC-MS (QQQ)		Yes: Isotopically labelled	Atrazin D5	ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Difenoconazole																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
001	D	0.01	0.362	93.5	5	Yes (10ml)	Acetone	Other (Petroleum ether)	Dichloromethane	No	None	Other (Apple matrix multiple level)	MS (QQQ)		LC-MS (QQQ)	Via Standard Addition	Yes: Other	PCB 153	EI (Electron Ionisation)	Positive
002	D	0.02	0.427		2	Yes (10 mL)	Acetonitrile			Yes	DSPE, citrate buffered, dSPE with PSA	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Via Standard Addition	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
003	NA																			
004	D	0.05	0.3		5	No	Ethyl acetate			No	GPC, gel permeation chromatography	Matrix matched-Multiple Level	ECD		GC-MS (Q)	Rec. from the same batch	No		CI (Chemical Ionisation)	Negative
005	D	0.02	0.432	83	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition		MS (QQQ)	None	Via Standard Addition	No		ESI (Electrospray Ionisation)	Positive
006	D	0.02	0.407	103	2	4	Acetonitrile			Yes	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
007	D	0.01	0.25	100	2	4	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)		Rec. from validation data	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
008	D	0.02	0.38	89	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)	LC-MS (QQQ)	Other untreated green tea was spiked	Yes: Other	no calculation, only to check extraction efficiency	ESI (Electrospray Ionisation)	Negative
009	D	0.01	0.45	100	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	triphenylphosphate	ESI (Electrospray Ionisation)	Positive
010	D	0.02	0.28	97	5	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from validation data	No		ESI (Electrospray Ionisation)	Positive
011	D	0.02	0.215	60	2	4	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
012	D	0.01	0.6	100	2	10	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (QQQ)	MS (QQQ)	None	Rec. from validation data	Yes: Other	TFP	ESI (Electrospray Ionisation)	Positive
013	D	0.05	0.44	80	1	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level	Other (Please Specify)		Other (Please Specify)					
014	NA																			
015	D	0.02	0.47	127	2	Yes - 10ml	Acetonitrile			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (IT)	MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP (LC) / Bromophos methyl - (GC)	ESI (Electrospray Ionisation)	Positive
016	D	0.01	0.34	112	2	Yes (10mL)	Acetonitrile			No	DSPE, Graphitized Carbon Black	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive

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Difenoconazole																			
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
017	D	0.01	0.371	82.5	2.5	7.5	Acetonitrile			No	DSPE, DSPE with graphitised carbon and PSA	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
018	D	0.02	0.25	102.5	2	10	Acetonitrile			Yes	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (QQQ)		LC-MS (QQQ)	Rec. from the same batch	Yes: Other	Triphenyl phosphate	EI (Electron Ionisation)	Positive
019	D	0.01	0.35	115	2	10	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch			EI (Electron Ionisation)	
020	D	0.02	0.315	93	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level		MS (Orbitrap)	LC-MS (Orbitrap)	Rec. from validation data	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
021	D	0.02	0.404	95	2	10	Acetonitrile			No	DSPE, CaCl2 and -NH2 (instead of PSA)	Matrix matched-Single Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
022	NA																			
023	D	0.01	0.475	88	2	4	Ethyl acetate			Yes	Other (filtration)	Standard Addition		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
024	D	0.02	0.467	100	2	10	Acetonitrile	n-Hexane		No	DSPE, LLE with n-hexane	Matrix matched-Multiple Level	MS (QQQ)		LC-MS (QQQ)	Other - via procedural matrix calibration	Yes: Other	Triphenylphosphate	EI (Electron Ionisation)	
025	D	0.05	0.28	175	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
026	D	0.04	0.412	94	2.5	7.5	Methanol	Other: ammonium acetate		No	Other: dilution by 10 and filtration	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	Oxfendazole	ESI (Electrospray Ionisation)	Positive
027	D	0.05	0.32	95	2	5	acetone/hexane/ethyl acetate			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (Q)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	
028	D	0.01	0.307	100	2	Yes (4 mL)	Acetonitrile			No	None	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
029	D	0.01	0.448	97.3	2	10	Acetonitrile	Acetonitrile		Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TRIS	ESI (Electrospray Ionisation)	Positive
030	D	0.02	0.553	92	2	Yes 10 ml	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from validation data	No		ESI (Electrospray Ionisation)	Positive
031	D	0.05	0.42	88	2	Yes (8 mL)	Ethyl acetate			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (Q)		GC-MS (Q)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	Positive
032	D	0.02	0.432	80	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Isotopically labelled)	Linuron-D6	ESI (Electrospray Ionisation)	Positive
033	D	0.02	0.312	89.3	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction, then CaCl2	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Difenoconazole

Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
034	D	0.02	0.338	70	3	Yes 6ml	Acetone	Dichloromethane	Petroleum ether	No	None	Matrix matched-Multiple Level	MS (QQQ) FPD ECD		None	Rec. from the same batch	No			
035	D	0.01	0.338	60.2	2g	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level	MS (QQQ)	MS (QQQ)		Via Standard Addition	Yes: Other	tris(2-chloro-1(chloromethyl)ethyl)phosphate	EI (Electron Ionisation)	Positive
036	D	0.01	0.32	62	2	10	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TRISCP	EI (Electron Ionisation)	Positive
037	No Results Submited																			
038	No Results Submited																			
039	D	0.02	0.28	74.1	2	Yes (10 mL)	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		LC-MS (QQQ)	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
040	D	0.005	0.48	55	2	Yes,10	Acetonitrile+ water(1:1)			Yes	DSPE, dispersive solid phase extraction	Standard Addition		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
041	D	0.01	0.38	105	2.5	Yes (10 ml)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from validation data	No		ESI (Electrospray Ionisation)	Positive
042	NA																			
043	D	0.01	0.601	85	5	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Other	TDCPP		
044	D	0.02	0.152	63.8	1	Yes (2.5 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction(PSA)	Matrix matched-Multiple Level	Other (Please Specify)	MS (Orbitrap)+MSMS(QQQ)	LC-MS (Orbitrap) +LC-MS （QQQ）	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
045	NA																			
046	D	0.02	0.336	92	2	10	Ethyl acetate			No	Filtration	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
047	D	0.02	0.472	68	0.5	10	Acetonitrile			No	DSPE, PSA	Pure solvent-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
048	NA																			
049	D	0.025	0.36	91	2	4	Acetonitrile			Yes	DSPE, dispersive solid phase extraction (PSA/CaCl2)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
050	D	0.01	0.45	85	2	Yes (13 mL)	Acetone	Dichloromethane	Petroleum ether (PE)	No	None	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
051	D	0.01	0.019	90	2	10	Acetonitrile			No	DSPE, C18 y PSA	Matrix matched-Multiple Level	MS (QQQ)			Rec. from the same batch	No		EI (Electron Ionisation)	
052	D	0.01	0.36	75	2.5	Yes (5 mL)	Acetone	Dichloromethane	Other (PE)	No	Other (Na2SO4)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
053	ND	0.01	0.01		2	Yes	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition		MS (QQQ)	LC-MS (QQQ)	Via Standard Addition	Yes: Other	tris-(1,3-dichlorisopropyle)-phosphate	ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Difenoconazole																			
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
054	ND	0.1	0.02		2	No	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (Q)		GC-MS (Q)		No		EI (Electron Ionisation)	

APPENDIX 7.Methods used by participants for determining pesticides.

Endosulfan-beta																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
001	D	0.01	0.102	85.6	5	Yes (10ml)	Acetone	Petroleum ether	Dichloromethane	No	None	Other (Apple matrix multiple level)	MS (QQQ)		GC-MS (QQQ)	Via Standard Addition	Yes: Other	PCB 153	EI (Electron Ionisation)	Positive
002	D	0.02	0.13	92	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, citrate buffered, dSPE with PSA	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP, Tributylphosphat	EI (Electron Ionisation)	Positive
003	D	< 0,01	0.03	92		No	Acetone	Dichloromethane		No	None	Matrix matched-Multiple Level	MS (IT)		None	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
004	D	0.01	0.062	91	5	No	Ethyl acetate			No	GPC, gel permeation chromatography	Matrix matched-Multiple Level	ECD		GC-MS (Q)	Rec. from the same batch	No		CI (Chemical Ionisation)	Negative
005	D	0.02	0.079	79	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition	MS (QQQ)		None	Via Standard Addition	Yes: Other	Triphenylphosphate	EI (Electron Ionisation)	Positive
006	D	0.02	0.055	118			Acetone	Dichloromethane	Petroleum ether	No	None	Matrix matched-Multiple Level	MS (QQQ)			Rec. from the same batch	No		EI (Electron Ionisation)	
007	D	0.01	0.025	90	2	4	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)		Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	Positive
008	D	0.005	0.065	84	5		Accelerated Solvent	Cyclohexane	Ethyl acetate	No	Accelerated Solvent, GPC, gel permeation chromatography Mini-Silicagel column	Pure solvent-Single Level	Other FPD and ECD		Two columns	Other untreated green tea was spiked	Yes: Other	no calculation, only to check extraction efficiency		
009	D	0.01	0.111	120	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	anthracene	EI (Electron Ionisation)	
010	NA																			
011	D	0.02	0.067	70	2	4	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
012	D	0.01	0.051	82	4	No	Acetonitrile	Dichloromethane	Petroleum ether	No	Liquid-liquid partitioning	Matrix matched-Multiple Level	MS (QQQ)		None	Rec. from validation data	Yes: Other	TFP	EI (Electron Ionisation)	
013	D	0.05	0.098	78	1	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level	Other (Please Specify)		Other (Please Specify)					
014	NA																			
015	D	0.02	0.123	98	2	Yes - 10ml	Acetonitrile			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (IT)	MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP (LC) / Bromophos methyl - (GC)	EI (Electron Ionisation)	
016	ND	0.04	0.02	83	2	Yes (10mL)	Acetonitrile			No	DSPE, Graphitized Carbon Black	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	

APPENDIX 7.Methods used by participants for determining pesticides.

Endosulfan-beta																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
017	D	0.01	0.075	79.6	2.5	7.5	Acetonitrile			No	DSPE + Liquid-liquid partitioning with hexane	Matrix matched-Multiple Level	MS (QQQ)		LC-MS (QQQ)	Rec. from the same batch	Yes: Other	Bromophos methyl	EI (Electron Ionisation)	
018	D	0.02	0.11	100.7	2	10	Acetonitrile			Yes	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	ECD		GC-MS (Q)	Rec. from the same batch	Yes: Other	PCB 209	no	
019	NA																			
020	D	0.02	0.032	89	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	Negative
021	D	0.02	0.065	90	2	10	Acetonitrile			No	DSPE, CaCl2 and -NH2 (instead of PSA)	Matrix matched-Single Level	MS (QQQ)		GC-MS (Q)	Rec. from validation data	No		EI (Electron Ionisation)	Positive
022	D	0.02	0.04	80	2	Yes (4 mL)	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level	MS (IT)		None	Rec. from the same batch	Yes: Isotopically labelled	fenclorfos	EI (Electron Ionisation)	Positive
023	D	0.01	0.111	92	2	4	Ethyl acetate			Yes	Other (filtration)	Standard Addition	MS (QQQ)		None	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
024	D	0.02	0.118	100	2	10	Acetonitrile	n-Hexane		No	DSPE, LLE with n-hexane	Matrix matched-Multiple Level	MS (Q)		None	Other - via procedural matrix calibration	Yes: Other		CI (Chemical Ionisation)	Negative
025	D	0.05	0.12	132	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	No		EI (Electron Ionisation)	
026	D	0.04	0.089	101.6	2.5	7.5	Acetone			No	Liquid-liquid partitioning	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
027	D	0.01	0.082	82.8	2	5	acetone/hexane/ethyl acetate			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (Q)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	
028	D	0.01	0.101	106	2	Yes (4 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (QQQ)		None	Rec. from the same batch	Yes: Other	Aldrin	EI (Electron Ionisation)	Positive
029	D	0.02	0.0911	102.2	2	10	Acetonitrile	n-Hexan		No	Liquid-liquid partitioning	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	PCB 209	EI (Electron Ionisation)	Positive
030	D	0.02	0.145	99	2	Yes 10 ml	Acetonitrile	Ethyl acetate		No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (Q)	Rec. from validation data	No		EI (Electron Ionisation)	Positive
031	ND	0.04	0.02	86	2	Yes (8 mL)	Ethyl acetate			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (Q)		GC-MS (Q)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	Positive
032	D	0.02	0.083	76	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (Q)			Rec. from the same batch	Yes: Other	TRIS	CI (Chemical Ionisation)	Negative
033	D	0.02	0.14	106	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction, then CaCl2	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Endosulfan-beta

Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
034	D	0.02	0.083	82	3	Yes 6ml	Acetone	Dichloromethane	Petroleum ether	No	None	Matrix matched-Multiple Level	MS (QQQ) FPD ECD		None	Rec. from the same batch	No			
035	D	0.01	0.104	103.6	2g	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level	MS (QQQ)	MS (QQQ)		Via Standard Addition	Yes: Other	tris(2-chloro-1(chloromethyl)ethyl)phosphate	EI (Electron Ionisation)	Positive
036	D	0.01	0.065	87	2	10	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TRISCP	EI (Electron Ionisation)	Positive
037	No Results Submited																			
038	No Results Submited																			
039	D	0.02	0.11	108.6	2	Yes (10 mL)	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		Other (GC-ECD)	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
040	D	0.01	0.15	66.4	15	Yes, 9ml	ACETONE with 30% water	Dichloromethane	Petroleum Ether	No	Liquid-liquid partitioning	Standard Addition	XSD		GC-MS (Q)	Rec. from the same batch	No		EI (Electron Ionisation)	
041	NA																			
042	D	0.05	0.08	70	2	10 mL	Acetonitrile	isoottano		No	Liquid-liquid partitioning	Pure solvent-Single Level	ECD		GC-MS (Q)	Rec. from validation data	Yes: Other	Ethion	EI (Electron Ionisation)	Positive
043	ND	0.01	0.01		5	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Other	TDCPP		
044	D	0.01	0.095	94.3	1	Yes (2.5 mL)	Acetonitrile			No	SPE, solid phase extraction column(Carb/PSA)	Pure solvent-Multiple Level	MS (Q)		GC-MS (Q)	Rec. from the same batch	Yes: Isotopically labelled (target pesticide)		CI (Chemical Ionisation)	Negative
045	NA																			
046	D	0.02	0.108	19	2	10	Ethyl acetate			No	GPC, gel permeation chromatography	Standard Addition	MS (QQQ)		GC-MS (QQQ)	Via Standard Addition	Yes: Other	TPP	EI (Electron Ionisation)	Positive
047	ND	0.1	0.02		2.5	10	Ethyl acetate			No	None	Standard Addition	MS (QQQ)		None	Rec. from the same batch	Yes: Other	PCB-28	EI (Electron Ionisation)	Positive
048	NA																			
049	D	0.025	0.068	93	2	4	Acetonitrile			Yes	DSPE, dispersive solid phase extraction(PSA/CaCl2)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP		
050	D	0.01	0.072	95	2	Yes (13 mL)	Acetone	Dichloromethane	Petroleum ether (PE)	No	None	Matrix matched-Multiple Level	MS (QQQ)		LC-MS (QQQ)	Rec. from the same batch	Yes: Other	Bromophos-ethyl	CI (Chemical Ionisation)	Negative
051	D	0.01	0.018	60	2	10	Acetonitrile			No	DSPE, C18 y PSA	Matrix matched-Multiple Level	MS (QQQ)			Rec. from the same batch	No		EI (Electron Ionisation)	
052	D	0.01	0.066	61	2.5	Yes (5 mL)	Acetone	Dichloromethane	Other (PE)	No	Other (Na2SO4)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Endosulfan-beta																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
053	ND	0.01	0.01		2	Yes	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition	MS (Q)		GC-MS (Q)	Via Standard Addition	Yes: Other	PCB 31	EI (Electron Ionisation)	Negative
054	D	0.01	0.028		2	No	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	ECD		Two columns		No			

APPENDIX 7.Methods used by participants for determining pesticides.

Etofenprox																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
001	D	0.01	0.157	105.0	5	Yes (10ml)	Acetone	Other (Petroleum ether)	Dichloromethane	No	None	Pure solvent-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Via Standard Addition	No		ESI (Electrospray Ionisation)	Positive
002	D	0.01	0.203		2	Yes (10 mL)	Acetonitrile			Yes	DSPE, citrate buffered, dSPE with PSA	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Via Standard Addition	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
003	NA																			
004	D	0.05	0.17	72	3	Yes (15 mL)	Acetonitrile			No	Freezing out	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
005	D	0.02	0.311	77	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition		MS (QQQ)	None	Via Standard Addition	No		ESI (Electrospray Ionisation)	Positive
006	D	0.02	0.154	101			Acetone	Dichloromethane	Petroleum ether	No	None	Matrix matched-Multiple Level	MS (QQQ)			Rec. from the same batch	No		EI (Electron Ionisation)	
007	D	0.01	0.2		2	4	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)		Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	Positive
008	D	0.01	0.15	77	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)	LC-MS (QQQ)	Other untreated green tea was spiked	Yes: Other	no calculation, only to check extraction efficiency	ESI (Electrospray Ionisation)	Negative
009	D	0.01	0.238	100	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	triphenylphosphate	ESI (Electrospray Ionisation)	Positive
010	NA																			
011	D	0.01	0.169	65	2	4	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
012	D	0.01	0.229	110	4	No	Acetonitrile	Dichloromethane	Petroleum ether	No	Liquid-liquid partitioning	Matrix matched-Multiple Level	MS (QQQ)		None	Rec. from validation data	Yes: Other	TFP	EI (Electron Ionisation)	
013	NA																			
014	NA																			
015	D	0.02	0.051	51	2	Yes - 10ml	Acetonitrile			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (IT)	MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP (LC) / Bromophos methyl - (GC)	ESI (Electrospray Ionisation)	Positive
016	NA																			
017	D	0.01	0.155	92	2.5	7.5	Acetonitrile			No	DSPE, DSPE with graphitised carbon and PSA	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
018	D	0.02	0.16	79.3	2	10	Acetonitrile			Yes	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	Yes: Other	Triphenyl phosphate	ESI (Electrospray Ionisation)	Positive
019	D	0.01	0.191	104	2	10	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch			EI (Electron Ionisation)	

APPENDIX 7.Methods used by participants for determining pesticides.

Etofenprox																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
020	D	0.01	0.085	106	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level		MS (Orbitrap)	LC-MS (Orbitrap)	Rec. from validation data	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
021	D	0.02	0.152	95	2	10	Acetonitrile			No	DSPE, CaCl2 and -NH2 (instead of PSA)	Matrix matched-Single Level	MS (Q)		GC-MS (QQQ)	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
022	NA																			
023	D	0.01	0.19	78	2	4	Ethyl acetate			Yes	Other (filtration)	Standard Addition		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
024	D	0.01	0.23	100	2	10	Acetonitrile	n-Hexane		No	DSPE, LLE with n-hexane	Matrix matched-Multiple Level	MS (QQQ)		LC-MS (QQQ)	Other - via procedural matrix calibration	Yes: Other	Triphenylphosphate	EI (Electron Ionisation)	
025	D	0.05	0.072	76	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
026	D	0.02	0.188	87.1	2.5	7.5	Acetone			No	Liquid-liquid partitioning	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
027	D	0.02	0.18	102	2	5	acetone/hexane/ethyl acetate			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (Q)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	
028	D	0.01	0.117	113	2	Yes (4 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (QQQ)		None	Rec. from the same batch	Yes: Other	Aldrin	EI (Electron Ionisation)	Positive
029	D	0.02	0.205	96.8	2	10	Acetonitrile	n-Hexan		No	Liquid-liquid partitioning	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	PCB 209	EI (Electron Ionisation)	Positive
030	D	0.02	0.264	90	2	Yes 10 ml	Acetonitrile	Ethyl acetate		No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (Q)	Rec. from validation data	No		EI (Electron Ionisation)	Positive
031	NA																			
032	D	0.01	0.233	75	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Isotopically labelled	Linuron-D6	ESI (Electrospray Ionisation)	Positive
033	D	0.02	0.103	173	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction, then CaCl2	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
034	D	0.02	0.162	87	3	Yes 6ml	Acetone	Dichloromethane	Petroleum ether	No	None	Matrix matched-Multiple Level	MS (QQQ) FPD ECD		None	Rec. from the same batch	No			
035	ND	0.01	0.01		2g	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level	MS (QQQ)	MS (QQQ)		Via Standard Addition	Yes: Other	tris(2-chloro-1(chloromethyl)ethyl)phosphate	EI (Electron Ionisation)	Positive
036	D	0.01	0.12	82	2	10	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TRISCP	EI (Electron Ionisation)	Positive
037	No Results Submited																			
038	No Results Submited																			

APPENDIX 7.Methods used by participants for determining pesticides.

Etofenprox																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
039	D	0.01	0.17	85.1	2	Yes (10 mL)	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		Other (GC-ECD)	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
040	D	0.01	0.33	73.4	15	Yes, 9ml	ACETONE with 30% water	Dichloromethane	Petroleum Ether	No	Liquid-liquid partitioning	Standard Addition	MS (Q)		GC-MS (Q)	Rec. from the same batch	No		EI (Electron Ionisation)	
041	NA																			
042	NA																			
043	ND	0.01	0.01		5	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Other	TDCPP		
044	ND	0.01	0.01		1	Yes (2.5 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction(PSA)	Matrix matched-Multiple Level	Other	MS (Orbitrap)+MSMS(QQQ)	LC-MS (Orbitrap) +LC-MS（QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
045	NA																			
046	D	0.01	0.189	38	2	10	Ethyl acetate			No	GPC, gel permeation chromatography	Standard Addition	MS (QQQ)		GC-MS (QQQ)	Via Standard Addition	Yes: Other	TPP	EI (Electron Ionisation)	Positive
047	D	0.01	0.185	107	2.5	10	Ethyl acetate			No	None	Standard Addition	MS (QQQ)		None	Rec. from the same batch	Yes: Other	PCB-28	EI (Electron Ionisation)	Positive
048	D	0.05	0.09	84	2	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (IT)		GC-MS (Q)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	
049	D	0.025	0.14	94	2	4	Acetonitrile			Yes	DSPE, dispersive solid phase extraction (PSA/CaCl2)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
050	D	0.01	0.231	54	2	Yes (13 mL)	Acetone	Dichloromethane	Petroleum ether (PE)	No	None	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
051	D	0.01	0.029	60	2	10	Acetonitrile			No	DSPE, C18 y PSA	Matrix matched-Multiple Level	MS (QQQ)			Rec. from the same batch	No		EI (Electron Ionisation)	
052	D	0.01	0.15	84	2.5	Yes (5 mL)	Acetone	Dichloromethane	Other (PE)	No	Other (Na2SO4)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
053	D	0.01	0.068	70	2	Yes	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition	MS (Q)		GC-MS (Q)	Via Standard Addition	Yes: Other	PCB 31	EI (Electron Ionisation)	Negative
054	D	0.03	0.14		2	No	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (Q)		GC-MS (Q)		No		EI (Electron Ionisation)	

APPENDIX 7.Methods used by participants for determining pesticides.

Fenpropathrin																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
001	D	0.01	0.159	104.6	5	Yes (10ml)	Acetone	Petroleum ether	Dichloromethane	No	None	Other (Apple matrix multiple level)	MS (QQQ)		GC-MS (QQQ)	Via Standard Addition	Yes: Other	PCB 153	EI (Electron Ionisation)	Positive
002	D	0.02	0.211	111	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, citrate buffered, dSPE with PSA	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP, Tributylphosphat	EI (Electron Ionisation)	Positive
003	NA																			
004	D	0.05	0.203	100	3	Yes (15 mL)	Acetonitrile			No	Liquid-liquid partitioning, isooctane with the adition of 20% NaCL	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Isotopically labelled	TPP	EI (Electron Ionisation)	
005	D	0.02	0.179	92	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition	MS (QQQ)		None	Via Standard Addition	Yes: Other	Triphenylphosphate	EI (Electron Ionisation)	Positive
006	D	0.02	0.147	95			Acetone	Dichloromethane	Petroleum ether	No	None	Matrix matched-Multiple Level	MS (QQQ)			Rec. from the same batch	No		EI (Electron Ionisation)	
007	D	0.01	0.19	102	2	4	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)		Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	Positive
008	D	0.01	0.11	80	5		Accelerated Solvent	Cyclohexane	Ethyl acetate	No	Accelerated Solvent, GPC, gel permeation chromatography Mini-Silicagel column	Pure solvent-Single Level	Other FPD and ECD		Two columns	Other untreated green tea was spiked	Yes: Other	no calculation, only to check extraction efficiency		
009	D	0.01	0.22	100	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	anthracene	EI (Electron Ionisation)	
010	NA																			
011	D	0.02	0.135	60	2	4	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
012	D	0.01	0.215	98	4	No	Acetonitrile	Dichloromethane	Petroleum ether	No	Liquid-liquid partitioning	Matrix matched-Multiple Level	MS (QQQ)		None	Rec. from validation data	Yes: Other	TFP	EI (Electron Ionisation)	
013	D	0.02	0.18	110	1	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level	Other		Other					
014	NA																			
015	D	0.02	0.15	88	2	Yes - 10ml	Acetonitrile			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (IT)	MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP (LC) / Bromophos methyl - (GC)	EI (Electron Ionisation)	
016	D	0.01	0.045	63	2	Yes (10mL)	Acetonitrile			No	DSPE, Graphitized Carbon Black	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	

APPENDIX 7.Methods used by participants for determining pesticides.

Fenpropathrin																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
017	D	0.01	0.176	96	2.5	7.5	Acetonitrile			No	DSPE + Liquid-liquid partitioning with hexane	Matrix matched-Multiple Level	MS (QQQ)		LC-MS (QQQ)	Rec. from the same batch	Yes: Other	Bromophos methyl	EI (Electron Ionisation)	
018	D	0.02	0.2	78.7	2	10	Acetonitrile			Yes	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (QQQ)		LC-MS (QQQ)	Rec. from the same batch	Yes: Other	Triphenyl phosphate	EI (Electron Ionisation)	Positive
019	D	0.01	0.182	102	2	10	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch			EI (Electron Ionisation)	
020	D	0.02	0.185	88	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level		MS (Orbitrap)	LC-MS (Orbitrap)	Rec. from validation data	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
021	D	0.02	0.175	95	2	10	Acetonitrile			No	DSPE, CaCl2 and -NH2 (instead of PSA)	Matrix matched-Single Level	MS (QQQ)		LC-MS (QQQ)	Rec. from validation data	No		EI (Electron Ionisation)	Positive
022	NA																			
023	D	0.01	0.183	86	2	4	Ethyl acetate			Yes	Other (filtration)	Standard Addition	MS (QQQ)		None	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
024	D	0.02	0.25	100	2	10	Acetonitrile	n-Hexane		No	DSPE, LLE with n-hexane	Matrix matched-Multiple Level	MS (QQQ)		LC-MS (QQQ)	Other - via procedural matrix calibration	Yes: Other	Triphenylphosphate	EI (Electron Ionisation)	
025	D	0.05	0.23	136.1	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
026	D	0.02	0.213	94	2.5	7.5	Methanol	Ammonium acetate		No	Other: dilution by 10 and filtration	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	Oxfendazole	ESI (Electrospray Ionisation)	Positive
027	D	0.02	0.19	78.5	2	5	acetone/hexane/ethyl acetate			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (Q)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	
028	D	0.01	0.18	113	2	Yes (4 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (QQQ)		None	Rec. from the same batch	Yes: Other	Aldrin	EI (Electron Ionisation)	Positive
029	D	0.02	0.242	103.7	2	10	Acetonitrile	n-Hexan		No	Liquid-liquid partitioning	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	PCB 209	EI (Electron Ionisation)	Positive
030	D	0.02	0.279	95	2	Yes 10 ml	Acetonitrile	Ethyl acetate		No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (Q)	Rec. from validation data	No		EI (Electron Ionisation)	Positive
031	NA																			
032	D	0.02	0.225	89	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (Q)	Rec. from the same batch	Yes: Isotopically labelled	Linuron-D6	ESI (Electrospray Ionisation)	Positive
033	D	0.02	0.192	116.3	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction, then CaCl2	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Fenpropathrin																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
034	ND	0.02	0.02		3	Yes 6ml	Acetone	Dichloromethane	Petroleum ether	No	None	Matrix matched-Multiple Level	MS (QQQ) FPD ECD		None	Rec. from the same batch	No			
035	D	0.01	0.228	102.8	2g	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level	MS (QQQ)	MS (QQQ)		Via Standard Addition	Yes: Other	tris(2-chloro-1(chloromethyl)ethyl)phosphate	EI (Electron Ionisation)	Positive
036	D	0.01	0.13	111	2	10	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TRISCP	EI (Electron Ionisation)	Positive
037	No Results Submited																			
038	No Results Submited																			
039	D	0.02	0.21	76.0	2	Yes (10 mL)	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		None	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
040	D	0.02	0.24	Blank with large amount of particular pesticide	15	Yes, 9ml	ACETONE with 30% water	Dichloromethane	Petroleum Ether	No	Liquid-liquid partitioning	Standard Addition	MS (Q)		GC-MS (Q)	Rec. from the same batch	No		EI (Electron Ionisation)	
041	NA																			
042	D	0.05	0.45	70	2	10 mL	Acetonitrile	isoottano		No	Liquid-liquid partitioning	Pure solvent-Single Level	ECD		GC-MS (Q)	Rec. from validation data	Yes: Other	Ethion	EI (Electron Ionisation)	Positive
043	D	0.01	0.188	99	5	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (Q)			Rec. from the same batch	Yes: Other	TDCPP		
044	D	0.01	0.214	114.5	1	Yes (2.5 mL)	Acetonitrile			No	SPE, solid phase extraction column(Carb/PSA)	Pure solvent-Multiple Level	MS (Q)		GC-MS (Q)	Rec. from the same batch	Yes: Isotopically labelled (target pesticide)		CI (Chemical Ionisation)	Negative
045	NA																			
046	D	0.02	0.189	28	2	10	Ethyl acetate			No	GPC, gel permeation chromatography	Standard Addition	MS (QQQ)		GC-MS (QQQ)	Via Standard Addition	Yes: Other	TPP	EI (Electron Ionisation)	Positive
047	D	0.02	0.17	111	2.5	10	Ethyl acetate			No	None	Standard Addition	MS (QQQ)		None	Rec. from the same batch	Yes: Other	PCB-28	EI (Electron Ionisation)	Positive
048	D	0.05	0.13	79	2	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (IT)		GC-MS (Q)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	
049	D	0.025	0.19	98	2	4	Acetonitrile			Yes	DSPE, dispersive solid phase extraction(PSA/CaCl2)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP		
050	D	0.01	0.228	102	2	Yes (13 mL)	Acetone	Dichloromethane	Petroleum ether (PE)	No	None	Matrix matched-Multiple Level	MS (QQQ)		LC-MS (QQQ)	Rec. from the same batch	Yes: Other	Bromophos-ethyl	CI (Chemical Ionisation)	Negative
051	D	0.01	0.039	60	2	10	Acetonitrile			No	DSPE, C18 y PSA	Matrix matched-Multiple Level	MS (QQQ)			Rec. from the same batch	No		EI (Electron Ionisation)	

APPENDIX 7.Methods used by participants for determining pesticides.

Fenpropathrin																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
052	D	0.01	0.18	79	2.5	Yes (5 mL)	Acetone	Dichloromethane	Other (PE)	No	Other (Na2SO4)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
053	ND	0.01	0.01		2	Yes	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition	MS (Q)		GC-MS (Q)	Via Standard Addition	Yes: Other	PCB 31	EI (Electron Ionisation)	Negative
054	D	0.1	0.115		2	No	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (Q)		GC-MS (Q)		No		EI (Electron Ionisation)	

APPENDIX 7.Methods used by participants for determining pesticides.

Imidacloprid																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
001	D	0.01	0.108	71.0	5	Yes (10ml)	Acetone	Other (Petroleum ether)	Dichloromethane	No	None	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Via Standard Addition	No		ESI (Electrospray Ionisation)	Positive
002	D	0.02	0.101		2	Yes (10 mL)	Acetonitrile			Yes	DSPE, citrate buffered, dSPE with PSA	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Via Standard Addition	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
003	ND	< 0,01	0.01		10	No	Acetone	Dichloromethane		No	None	Pure solvent-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
004	D	0.05	0.095	98	3	Yes (15 mL)	Acetonitrile			No	Freezing out	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
005	D	0.02	0.102	91	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition		MS (QQQ)	None	Via Standard Addition	No		ESI (Electrospray Ionisation)	Positive
006	D	0.02	0.054	114	2	4	Acetonitrile			Yes	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
007	D	0.01	0.035	110	2	4	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)		Rec. from validation data	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
008	D	0.01	0.083	89	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)	LC-MS (QQQ)	Other untreated green tea was spiked	Yes: Other	no calculation, only to check extraction efficiency	ESI (Electrospray Ionisation)	Negative
009	D	0.01	0.124	108	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	triphenylphosphate	ESI (Electrospray Ionisation)	Positive
010	NA																			
011	ND	0.02	0.02		2	4	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
012	D	0.01	0.05	90	2	10	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (QQQ)	MS (QQQ)	None	Rec. from validation data	Yes: Other	TFP	ESI (Electrospray Ionisation)	Positive
013	NA																			
014	D	0.01	0.048	77	2	no	Methanol	Water		no	Filter	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
015	D	0.02	0.076	58	2	Yes - 10ml	Acetonitrile			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (IT)	MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP (LC) / Bromophos methyl - (GC)	ESI (Electrospray Ionisation)	Positive
016	ND	0.01	0.01		2	Yes (10mL)	Acetonitrile			No	DSPE, Graphitized Carbon Black	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
017	D	0.01	0.096	74.4	2.5	7.5	Acetonitrile			No	DSPE, DSPE with graphitised carbon and PSA	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Imidacloprid																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
018	D	0.02	0.052		2	10	Acetonitrile			Yes	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	Yes: Other	Triphenyl phosphate	ESI (Electrospray Ionisation)	Positive
019	D	0.01	0.286	101.4	2	10	Acetonitrile			No	SPE, solid phase extraction column	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch			ESI (Electrospray Ionisation)	Positive
020	ND	0.02	0.02		2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level		MS (Orbitrap)	LC-MS (Orbitrap)	Rec. from validation data	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
021	D	0.02	0.066	79	2	10	Acetonitrile			No	DSPE, CaCl2 and -NH2 (instead of PSA)	Matrix matched-Single Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
022	NA																			
023	D	0.01	0.099	84	2	4	Ethyl acetate			Yes	Other (filtration)	Standard Addition		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
024	D	0.02	0.11	100	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	None	Other - via procedural matrix calibration	Yes: Other	Triphenylphosphate	ESI (Electrospray Ionisation)	Positive
025	D	0.05	0.05	81.8	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
026	D	0.04	0.09	108	2.5	7.5	Methanol	Other: ammonium acetate		No	Other: dilution by 10 and filtration	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	Oxfendazole	ESI (Electrospray Ionisation)	Positive
027	ND	0.01	0.01		3	5	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from validation data	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
028	D	0.01	0.084	84	2	Yes (4 mL)	Acetonitrile			No	None	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
029	D	0.02	0.112	93.2	2	10	Acetonitrile	Acetonitrile		Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TRIS	ESI (Electrospray Ionisation)	Positive
030	D	0.02	0.0949	75	2	Yes 10 ml	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from validation data	No		ESI (Electrospray Ionisation)	Positive
031	NA																			
032	D	0.02	0.091	87	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Isotopically labelled	Linuron-D6	ESI (Electrospray Ionisation)	Positive
033	D	0.02	0.0387	72.3	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction, then CaCl2	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
034	D	0.02	0.091	75	2	Yes 4mL	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Imidacloprid																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
035	D	0.01	0.078	62.2	2g	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level	MS (QQQ)	MS (QQQ)		Via Standard Addition	Yes: Other (Please Specify)	tris(2-chloro-1(chloromethyl)ethyl)phosphate	EI (Electron Ionisation)	Positive
036	NA																			
037	No Results Submited																			
038	No Results Submited																			
039	D	0.02	0.067	96.6	2	Yes (10 mL)	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
040	D	0.005	0.097	Blank with large amount of particular pesticide	2	Yes,10	Acetonitrile+water(1:1)			Yes	DSPE, dispersive solid phase extraction	Standard Addition		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
041	D	0.02	0.063	81	2.5	Yes (10 ml)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from validation data	No		ESI (Electrospray Ionisation)	Positive
042	NA																			
043	D	0.01	0.048	113	5	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Other (TDCPP)	TDCPP		
044	D	0.02	0.0 91	118.4	1	Yes (2.5 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction(PSA)	Matrix matched-Multiple Level	Other (Please Specify)	MS (Orbitrap)+MSMS(QQQ)	LC-MS (Orbitrap) +LC-MS（QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
045	NA																			
046	D	0.02	0.088	123	2	10	Ethyl acetate			No	Filtration	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
047	D	0.02	0.109	37	0.5	10	Acetonitrile			No	DSPE, PSA	Pure solvent-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
048	NA																			
049	D	0.025	0.067	101	2	4	Acetonitrile			Yes	DSPE, dispersive solid phase extraction (PSA/CaCl2)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
050	D	0.01	0.064	101	2	Yes (13 mL)	Acetone	Dichloromethane	Petroleum ether (PE)	No	None	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
051	ND	0.01	0.01		2	10	Acetonitrile			No	DSPE, C18 y PSA	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
052	D	0.01	0.07	77	2.5	Yes (5 mL)	Acetone	Dichloromethane	Other (PE)	No	Other (Na2SO4)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other (Please Specify)	TPP	ESI (Electrospray Ionisation)	Positive
053	ND	0.01	0.01		2	Yes	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition		MS (QQQ)	LC-MS (QQQ)	Via Standard Addition	Yes: Other (Please Specify)	tris-(1,3-dichlorisopropyle)phosphate	ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Imidacloprid																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
054	ND	0.03	0.02		2	No	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level with complete sample preparation		MS (QQQ)	LC-MS (QQQ)		Yes: Isotopically labelled	Atrazin D5	ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Lambda-cyhalothrin																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
001	D	0.01	0.136	95.5	5	Yes (10ml)	Acetone	Other (Petroleum ether)	Dichloromethane	No	None	Other (Apple matrix multiple level)	MS (QQQ)		GC-MS (QQQ)	Via Standard Addition	Yes: Other	PCB 153	EI (Electron Ionisation)	Positive
002	D	0.02	0.137	80	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, citrate buffered, dSPE with PSA	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP, Tributylphosphat	EI (Electron Ionisation)	Positive
003	D	< 0,02	0.08	97		No	Acetone	Dichloromethane		No	None	Matrix matched-Multiple Level	MS (IT)		None	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
004	D	0.02	0.134	102	5	No	Ethyl acetate			No	GPC, gel permeation chromatography	Matrix matched-Multiple Level	ECD		GC-MS (Q)	Rec. from the same batch	No		CI (Chemical Ionisation)	Negative
005	D	0.02	0.134	87	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition	MS (QQQ)		None	Via Standard Addition	Yes: Other	Triphenylphosphate	EI (Electron Ionisation)	Positive
006	D	0.02	0.142	101			Acetone	Dichloromethane	Petroleum ether	No	None	Matrix matched-Multiple Level	MS (QQQ)			Rec. from the same batch	No		EI (Electron Ionisation)	
007	D	0.01	0.16	94	2	4	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)		Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	Positive
008	D	0.01	0.14	81	5		Accelerated Solvent	Cyclohexane	Ethyl acetate	No	Accelerated Solvent, GPC, gel permeation chromatography Mini-Silicagel column	Pure solvent-Single Level	FPD and ECD		Two columns	Other untreated green tea was spiked	Yes: Other	no calculation, only to check extraction efficiency		
009	D	0.01	0.188	109	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	anthracene	EI (Electron Ionisation)	
010	D	0.05	0.12	84	5	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level	MS (Q)		GC-MS (Q)	Rec. from validation data	No		EI (Electron Ionisation)	Positive
011	D	0.02	0.102	96	2	4	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
012	D	0.02	0.055	119	4	No	Acetonitrile	Dichloromethane	Petroleum ether	No	Liquid-liquid partitioning	Matrix matched-Multiple Level	MS (QQQ)		None	Rec. from validation data	Yes: Other	TFP	EI (Electron Ionisation)	
013	D	0.02	0.16	100	1	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level	Other		Other (Please Specify)					
014	NA																			
015	D	0.02	0.129	132	2	Yes - 10ml	Acetonitrile			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (IT)	MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP (LC) / Bromophos methyl - (GC	EI (Electron Ionisation)	

APPENDIX 7.Methods used by participants for determining pesticides.

Lambda-cyhalothrin																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
016	D	0.02	0.028	73	2	Yes (10mL)	Acetonitrile			No	DSPE, Graphitized Carbon Black	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Other (	TPP	EI (Electron Ionisation)	
017	D	0.01	0.138	88	2.5	7.5	Acetonitrile			No	DSPE + Liquid-liquid partitioning with hexane	Matrix matched-Multiple Level	MS (QQQ)		LC-MS (QQQ)	Rec. from the same batch	Yes: Other	Bromophos methyl	EI (Electron Ionisation)	
018	D	0.02	0.22	97.4	2	10	Acetonitrile			Yes	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (QQQ)		GC-ECD	Rec. from the same batch	Yes: Other	Triphenyl phosphate	EI (Electron Ionisation)	
019	D	0.01	0.162	108	2	10	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch			EI (Electron Ionisation)	
020	D	0.02	0.111	105	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	Negative
021	D	0.02	0.157	97	2	10	Acetonitrile			No	DSPE, CaCl2 and -NH2 (instead of PSA)	Matrix matched-Single Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
022	D	0.02	0.087	74	2	Yes (4 mL)	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level	MS (IT)		None	Rec. from the same batch	Yes: Isotopically labelled	fenclorfos	EI (Electron Ionisation)	Positive
023	D	0.01	0.119	97	2	4	Ethyl acetate			Yes	Other (filtration)	Standard Addition	MS (QQQ)		None	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
024	D	0.02	0.183	100	2	10	Acetonitrile	n-Hexane		No	DSPE, LLE with n-hexane	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (Q)	Other - via procedural matrix calibration	Yes: Other	Triphenylphosphate	EI (Electron Ionisation)	
025	D	0.05	0.076	99	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	No		EI (Electron Ionisation)	
026	D	0.02	0.194	89.9	2.5	7.5	Acetone			No	Liquid-liquid partitioning	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
027	D	0.01	0.16	87.4	2	5	acetone/hexane/ethyl acetate			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (Q)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	
028	D	0.01	0.117	109	2	Yes (4 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (QQQ)		None	Rec. from the same batch	Yes: Other	Aldrin	EI (Electron Ionisation)	Positive
029	D	0.02	0.183	107.9	2	10	Acetonitrile	n-Hexan		No	Liquid-liquid partitioning	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	PCB 209	EI (Electron Ionisation)	Positive
030	D	0.02	0.214	90	2	Yes 10 ml	Acetonitrile	Ethyl acetate		No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (Q)	Rec. from validation data	No		EI (Electron Ionisation)	Positive
031	D	0.05	0.15	93	2	Yes (8 mL)	Ethyl acetate			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (Q)		GC-MS (Q)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Lambda-cyhalothrin																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
032	D	0.02	0.204	93	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (Q)		GC-MS (Q)	Rec. from the same batch	Yes: Other	TRIS	CI (Chemical Ionisation)	Negative
033	D	0.02	0.142	141	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction, then CaCl2	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
034	D	0.02	0.131	94	3	Yes 6ml	Acetone	Dichloromethane	Petroleum ether	No	None	Matrix matched-Multiple Level	MS (QQQ) FPD ECD		None	Rec. from the same batch	No			
035	D	0.01	0.142	77	2g	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level	MS (QQQ)	MS (QQQ)		Via Standard Addition	Yes: Other	tris(2-chloro-1(chloromethyl)ethyl)phosphate	EI (Electron Ionisation)	Positive
036	D	0.01	0.069	91	2	10	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TRISCP	EI (Electron Ionisation)	Positive
037	No Results Submited																			
038	No Results Submited																			
039	D	0.02	0.19	83.6	2	Yes (10 mL)	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		Other (GC-ECD)	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
040	D	0.01	0.27	79.6	15	Yes, 9ml	ACETONE with 30% water	Dichloromethane	Petroleum Ether	No	Liquid-liquid partitioning	Standard Addition	MS (Q)		GC-MS (Q)	Rec. from the same batch	No		EI (Electron Ionisation)	
041	NA																			
042	D	0.05	0.1	75	2	10 mL	Acetonitrile	isoottano		No	Liquid-liquid partitioning	Pure solvent-Single Level	ECD		GC-MS (Q)	Rec. from validation data	Yes: Other	Ethion	EI (Electron Ionisation)	Positive
043	D	0.01	0.28	106	5	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Other	TDCPP		
044	D	0.01	0.209	119.4	1	Yes (2.5 mL)	Acetonitrile			No	SPE, solid phase extraction column(Carb/PSA)	Pure solvent-Multiple Level	MS (Q)		GC-MS (Q)	Rec. from the same batch	Yes: Isotopically labelled (target pesticide)		CI (Chemical Ionisation)	Negative
045	D	0.01	0.52	93.42	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (QQQ)		None	Rec. from the same batch	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
046	D	0.02	0.171	1	2	10	Ethyl acetate			No	GPC, gel permeation chromatography	Standard Addition	MS (QQQ)		GC-MS (QQQ)	Via Standard Addition	Yes: Other	TPP	EI (Electron Ionisation)	Positive
047	D	0.02	0.148	109	2.5	10	Ethyl acetate			No	None	Standard Addition	MS (QQQ)		None	Rec. from the same batch	Yes: Other	PCB-28	EI (Electron Ionisation)	Positive
048	D	0.05	0.12	92	2	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (IT)		GC-MS (Q)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	

APPENDIX 7.Methods used by participants for determining pesticides.

Lambda-cyhalothrin																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
049	D	0.025	0.18	115	2	4	Acetonitrile			Yes	DSPE, dispersive solid phase extraction (PSA/ CaCl2)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP		
050	D	0.01	0.15	86	2	Yes (13 mL)	Acetone	Dichloromethane	Petroleum ether (PE)	No	None	Matrix matched-Multiple Level	MS/MS (ITD)		GC-MS (QQQ)	Rec. from the same batch	Yes: Isotopically labelled (target pesticide)		EI (Electron Ionisation)	Positive
051	D	0.01	0.039	70	2	10	Acetonitrile			No	DSPE, C18 y PSA	Matrix matched-Multiple Level	MS (QQQ)			Rec. from the same batch	No		EI (Electron Ionisation)	
052	D	0.01	0.15	85	2.5	Yes (5 mL)	Acetone	Dichloromethane	Other (PE)	No	Other (Na2SO4)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
053	D	0.01	0.66	70	2	Yes	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition	MS (Q)		GC-MS (Q)	Via Standard Addition	Yes: Other	PCB 31	EI (Electron Ionisation)	Negative
054	D	0.1	0.121		2	No	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (Q)		GC-MS (Q)		No		EI (Electron Ionisation)	

APPENDIX 7.Methods used by participants for determining pesticides.

Methomyl

Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
001	D	0.01	0.089	76.9	5	Yes (10ml)	Acetone	Other (Petroleum ether)	Dichloromethane	No	None	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Via Standard Addition	No		ESI (Electrospray Ionisation)	Positive
002	D	0.02	0.109		2	Yes (10 mL)	Acetonitrile			Yes	DSPE, citrate buffered, dSPE with PSA	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Via Standard Addition	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
003	ND	< 0,01	0.01		10	No	Acetone	Dichloromethane		No	None	Pure solvent-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
004	D	0.05	0.066	116	3	Yes (15 mL)	Acetonitrile			No	Freezing out	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
005	D	0.02	0.089	93	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition		MS (QQQ)	None	Via Standard Addition	No		ESI (Electrospray Ionisation)	Positive
006	D	0.02	0.083	130	2	4	Acetonitrile			Yes	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
007	ND	0.01	0.01		2	4	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)		Rec. from validation data	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
008	D	0.02	0.11	117	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)	LC-MS (QQQ)	Other untreated green tea was spiked	Yes: Other	no calculation, only to check extraction efficiency	ESI (Electrospray Ionisation)	Negative
009	D	0.01	0.084	120	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	triphenylphosphate	ESI (Electrospray Ionisation)	Positive
010	NA																			
011	ND	0.02	0.02		2	4	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
012	D	0.01	0.077	80	2	10	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (QQQ)	MS (QQQ)	None	Rec. from validation data	Yes: Other	TFP	ESI (Electrospray Ionisation)	Positive
013	NA																			
014	NA																			
015	D	0.02	0.058	45	2	Yes - 10ml	Acetonitrile			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (IT)	MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP (LC) / Bromophos methyl - (GC)	ESI (Electrospray Ionisation)	Positive
016	ND	0.01	0.01		2	Yes (10mL)	Acetonitrile			No	DSPE, Graphitized Carbon Black	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
017	D	0.01	0.105	120	2.5	7.5	Acetonitrile			No	DSPE, DSPE with graphitised carbon and PSA	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Methomyl																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
018	D	0.02	0.028	92.2	2	10	Acetonitrile			Yes	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	Yes: Other	Triphenyl phosphate	ESI (Electrospray Ionisation)	Positive
019	NA																			
020	D	0.02	0.095	100	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level		MS (Orbitrap)	LC-MS (Orbitrap)	Rec. from validation data	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
021	D	0.02	0.02	94	2	10	Acetonitrile			No	DSPE, CaCl2 and -NH2 (instead of PSA)	Matrix matched-Single Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
022	NA																			
023	D	0.01	0.102	111	2	4	Ethyl acetate			Yes	Other (filtration)	Standard Addition		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
024	D	0.02	0.109	100	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	None	Other - via procedural matrix calibration	Yes: Other	Triphenylphosphate	ESI (Electrospray Ionisation)	Positive
025	ND	0.05	0.02		2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
026	ND	0.04	0.02		2.5	7.5	Methanol	Other: ammonium acetate		No	Other: dilution by 10 and filtration	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	Oxfendazole	ESI (Electrospray Ionisation)	Positive
027	ND	0.005	0.005		3	5	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from validation data	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
028	D	0.01	0.169	77	2	Yes (4 mL)	Acetonitrile			No	None	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
029	D	0.02	0.1	97.2	2	10	Acetonitrile	Acetonitrile		Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TRIS	ESI (Electrospray Ionisation)	Positive
030	D	0.02	0.164	84	2	Yes 10 ml	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from validation data	No		ESI (Electrospray Ionisation)	Positive
031	NA																			
032	D	0.02	0.116	94	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Isotopically labelled	Linuron-D6	ESI (Electrospray Ionisation)	Positive
033	D	0.02	0.0958	96.9	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction, then CaCl2	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
034	D	0.02	0.095	75	2	Yes 4mL	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
035	D	0.01	0.075	92.8	2g	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level	MS (QQQ)	MS (QQQ)		Via Standard Addition	Yes: Other	tris(2-chloro-1(chloromethyl)ethyl)phosphate	EI (Electron Ionisation)	Positive
036	NA																			

APPENDIX 7.Methods used by participants for determining pesticides.

Methomyl																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
037	No Results Submited																			
038	No Results Submited																			
039	D	0.02	0.038	96.8	2	Yes (10 mL)	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
040	ND	0.005	0.005		2	Yes,10	Acetonitrile+ water(1:1)			Yes	DSPE, dispersive solid phase extraction	Standard Addition		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
041	D	0.01	0.13	113	2.5	Yes (10 ml)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from validation data	No		ESI (Electrospray Ionisation)	Positive
042	NA																			
043	D	0.01	0.048	80	5	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Other	TDCPP		
044	ND	0.02	0.02		1	Yes (2.5 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction(PSA)	Matrix matched-Multiple Level	Other (Please Specify)	MS (Orbitrap)+MSMS(QQQ)	LC-MS (Orbitrap) +LC-MS （QQQ）	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
045	D	0.01	0.06	78.9	2	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
046	D	0.02	0.091	131	2	10	Ethyl acetate			No	Filtration	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
047	D	0.02	0.093	37	0.5	10	Acetonitrile			No	DSPE, PSA	Pure solvent-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
048	NA																			
049	D	0.025	0.06	95	2	4	Acetonitrile			Yes	DSPE, dispersive solid phase extraction (PSA/CaCl2)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
050	D	0.01	0.076	97	2	Yes (13 mL)	Acetone	Dichloromethane	Petroleum ether (PE)	No	None	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
051	ND	0.01	0.01		2	10	Acetonitrile			No	DSPE, C18 y PSA	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
052	D	0.01	0.054	75	2.5	Yes (5 mL)	Acetone	Dichloromethane	Other (PE)	No	Other (Na2SO4)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
053	ND	0.01	0.01		2	Yes	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition		MS (QQQ)	LC-MS (QQQ)	Via Standard Addition	Yes: Other	tris-(1,3-dichlorisopropyle)-phosphate	ESI (Electrospray Ionisation)	Positive
054	ND	0.03	0.02		2	No	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level with complete sample preparation		MS (QQQ)	LC-MS (QQQ)		Yes: Isotopically labelled	Atrazin D5	ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Parathion-ethyl																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
001	D	0.01	0.397	90.08	5	Yes (10ml)	Acetone	Other (Petroleum ether)	Dichloromethane	No	None	Other (Apple matrix multiple level)	MS (QQQ)		GC-MS (QQQ)	Via Standard Addition	Yes: Other	PCB 153	EI (Electron Ionisation)	Positive
002	D	0.02	0.358	93	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, citrate buffered, dSPE with PSA	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP, Tributylphosphat	EI (Electron Ionisation)	Positive
003	D	< 0,01	0.15	91		No	Acetone	Dichloromethane		No	None	Matrix matched-Multiple Level	MS (IT)		None	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
004	D	0.05	0.238	102	5	No	Ethyl acetate			No	GPC, gel permeation chromatography	Matrix matched-Multiple Level	ECD		GC-MS (Q)	Rec. from the same batch	No		CI (Chemical Ionisation)	Negative
005	D	0.02	0.328	85	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition	MS (QQQ)		None	Via Standard Addition	Yes: Other	Triphenylphosphate	EI (Electron Ionisation)	Positive
006	D	0.02	0.235	107			Acetone	Dichloromethane	Petroleum ether	No	None	Matrix matched-Multiple Level	MS (QQQ)			Rec. from the same batch	No		EI (Electron Ionisation)	
007	D	0.01	0.36		2	4	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)		Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	Positive
008	D	0.02	0.4	88	5		Accelerated Solvent	Cyclohexane	Ethyl acetate	No	Accelerated Solvent, GPC, gel permeation chromatography Mini-Silicagel column	Pure solvent-Single Level	FPD and ECD		Two columns	Other untreated green tea was spiked	Yes: Other	no calculation, only to check extraction efficiency		
009	D	0.01	0.45	100	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	anthracene	EI (Electron Ionisation)	
010	NA																			
011	D	0.02	0.296	60	2	4	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
012	NA																			
013	D	0.05	0.55	108	1	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level	Other (Please Specify)		Other (Please Specify)					
014	NA																			
015	D	0.02	0.522	95	2	Yes - 10ml	Acetonitrile			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (IT)	MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP (LC) / Bromophos methyl - (GC)	EI (Electron Ionisation)	
016	D	0.05	0.16	57	2	Yes (10mL)	Acetonitrile			No	DSPE, Graphitized Carbon Black	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	

APPENDIX 7.Methods used by participants for determining pesticides.

Parathion-ethyl																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
017	D	0.01	0.324	98	2.5	7.5	Acetonitrile			No	DSPE + Liquid-liquid partitioning with hexane	Matrix matched-Multiple Level	MS (QQQ)		LC-MS (QQQ)	Rec. from the same batch	Yes: Other	Bromophos methyl	EI (Electron Ionisation)	
018	D	0.02	0.4	91	2	10	Acetonitrile			Yes	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	FPD		GC-MS (Q)	Rec. from the same batch	Yes: Other	Triphenyl phosphate	no	
019	D	0.01	0.424	101	2	10	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch			EI (Electron Ionisation)	
020	D	0.02	0.361	91	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	Negative
021	D	0.02	0.414	85	2	10	Acetonitrile			No	DSPE, CaCl2 and -NH2 (instead of PSA)	Matrix matched-Single Level	MS (QQQ)		GC-MS (Q)	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
022	ND	0.02	0.02		2	Yes (4 mL)	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level	MS (IT)		None	Rec. from the same batch	Yes: Isotopically labelled	fenclorfos	EI (Electron Ionisation)	Positive
023	D	0.01	0.363	80	2	4	Ethyl acetate			Yes	Other (filtration)	Standard Addition	MS (QQQ)		None	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
024	D	0.02	0.419	100	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Other - via procedural matrix calibration	Yes: Other	Triphenylphosphate	ESI (Electrospray Ionisation)	Positive
025	D	0.05	0.4	NONE	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	No		EI (Electron Ionisation)	
026	D	0.02	0.492	92.8	2.5	7.5	Acetone			No	Liquid-liquid partitioning	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
027	ND	0.01	0.01		2	5	acetone/hexane/ethyl acetate			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (Q)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	
028	D	0.01	0.4	72	2	Yes (4 mL)	Acetonitrile			No	None	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
029	D	0.02	0.45	124.9	2	10	Acetonitrile	n-Hexan		No	Liquid-liquid partitioning	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	PCB 209	EI (Electron Ionisation)	Positive
030	D	0.02	0.324	92	2	Yes 10 ml	Acetonitrile	Ethyl acetate		No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (Q)	Rec. from validation data	No		EI (Electron Ionisation)	Positive
031	NA																			
032	D	0.02	0.439	104	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (Q)			Rec. from the same batch	Yes: Other	Triphenylmethan	EI (Electron Ionisation)	Positive
033	D	0.02	0.168	67	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction, then CaCl2	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive

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Parathion-ethyl																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
034	D	0.02	0.402	90	3	Yes 6ml	Acetone	Dichloromethane	Petroleum ether	No	None	Matrix matched-Multiple Level	MS (QQQ) FPD ECD		None	Rec. from the same batch	No			
035	D	0.01	0.334	92.9	2g	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level	MS (QQQ)	MS (QQQ)		Via Standard Addition	Yes: Other	tris(2-chloro-1(chloromethyl)ethyl)phosphate	EI (Electron Ionisation)	Positive
036	D	0.01	0.38	63	2	10	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TRISCP	EI (Electron Ionisation)	Positive
037	No Results Submited																			
038	No Results Submited																			
039	D	0.02	0.33	74.2	2	Yes (10 mL)	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		Other (GC-ECD)	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
040	ND				15	Yes, 9ml	ACETONE with 30% water	Dichloromethane	Petroleum Ether	No	Liquid-liquid partitioning	Standard Addition	FPD		GC-MS (Q)	Rec. from the same batch	No		EI (Electron Ionisation)	
041	NA																			
042	D	0.05	0.51	70	2	10 mL	Acetonitrile	isoottano		No	Liquid-liquid partitioning	Pure solvent-Single Level	NPD		GC-MS (Q)	Rec. from validation data				
043	D	0.01	0.492	74	5	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Other	TDCPP		
044	D	0.02	0.51	79.4	1	Yes (2.5 mL)	Acetonitrile			No	SPE, solid phase extraction column(Carb/PSA)+onlineGPC	Matrix matched-Single Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
045	D	0.01	0.41	89.7	2	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
046	D	0.02	0.307	29	2	10	Ethyl acetate			No	GPC, gel permeation chromatography	Standard Addition	MS (QQQ)		GC-MS (QQQ)	Via Standard Addition	Yes: Other	TPP	EI (Electron Ionisation)	Positive
047	D	0.02	0.418	108	2.5	10	Ethyl acetate			No	None	Standard Addition	MS (QQQ)		None	Rec. from the same batch	Yes: Other	PCB-28	EI (Electron Ionisation)	Positive
048	D	0.05	0.65	88	2	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (IT)		GC-MS (Q)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	
049	D	0.025	0.28	65	2	4	Acetonitrile			Yes	DSPE, dispersive solid phase extraction(PSA/CaCl2)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP		
050	D	0.01	0.34	99	2	Yes (13 mL)	Acetone	Dichloromethane	Petroleum ether (PE)	No	None	Matrix matched-Multiple Level	MS (QQQ)		LC-MS (QQQ)	Rec. from the same batch	Yes: Other	Bromophos-ethyl	CI (Chemical Ionisation)	Negative
051	D	0.01	0.13	90	2	10	Acetonitrile			No	DSPE, C18 y PSA	Matrix matched-Multiple Level	MS (QQQ)			Rec. from the same batch	No		EI (Electron Ionisation)	

APPENDIX 7.Methods used by participants for determining pesticides.

Parathion-ethyl																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
052	D	0.01	0.38	80	2.5	Yes (5 mL)	Acetone	Dichloromethane	Other (PE)	No	Other (Na2SO4)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
053	ND	0.01	0.01		2	Yes	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition	MS (Q)		GC-MS (Q)	Via Standard Addition	Yes: Other	PCB 31	EI (Electron Ionisation)	Negative
054	D	0.03	0.135		2	No	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (Q)		GC-MS (Q)		No		EI (Electron Ionisation)	

APPENDIX 7.Methods used by participants for determining pesticides.

Pyridaben																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
001	D	0.01	0.259	100.1	5	Yes (10ml)	Acetone	Other (Petroleum ether)	Dichloromethane	No	None	Other (Apple matrix multiple level)	MS (QQQ)		LC-MS (QQQ)	Via Standard Addition	Yes: Other	PCB 153	EI (Electron Ionisation)	Positive
002	D	0.02	0.306	119	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, citrate buffered, dSPE with PSA	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP, Tributylphosphat	EI (Electron Ionisation)	Positive
003	D	< 0,01	0.12	97		No	Acetone	Dichloromethane		No	None	Matrix matched-Multiple Level	MS (IT)		None	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
004	D	0.05	0.246	85	3	Yes (15 mL)	Acetonitrile			No	Liquid-liquid partitioning, isooctane with the adition of 20% NaCL	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Isotopically labelled	TPP	EI (Electron Ionisation)	
005	D	0.02	0.388	82	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition		MS (QQQ)	None	Via Standard Addition	No		ESI (Electrospray Ionisation)	Positive
006	D	0.02	0.175	96			Acetone	Dichloromethane	Petroleum ether	No	None	Matrix matched-Multiple Level	MS (QQQ)			Rec. from the same batch	No		EI (Electron Ionisation)	
007	NA																			
008	D	0.02	0.27	82	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)	LC-MS (QQQ)	Other untreated green tea was spiked	Yes: Other	no calculation, only to check extraction efficiency	ESI (Electrospray Ionisation)	Negative
009	D	0.01	0.333	81	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	triphenylphosphate	ESI (Electrospray Ionisation)	Positive
010	D	0.02	0.15	100	5	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from validation data	No		ESI (Electrospray Ionisation)	Positive
011	D	0.02	0.227	62	2	4	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
012	D	0.01	0.304	113	4	No	Acetonitrile	Dichloromethane	Petroleum ether	No	Liquid-liquid partitioning	Matrix matched-Multiple Level	MS (QQQ)		None	Rec. from validation data	Yes: Other	TFP	EI (Electron Ionisation)	
013	D	0.05	0.46	78	1	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level	Other		Other (Please Specify)					
014	D	0.01	0.268	94	2	no	Methanol	Water		no	Filter	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
015	D	0.02	0.297	85	2	Yes - 10ml	Acetonitrile			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (IT)	MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP (LC) / Bromophos methyl - (GC)	EI (Electron Ionisation)	
016	D	0.02	0.058	70	2	Yes (10mL)	Acetonitrile			No	DSPE, Graphitized Carbon Black	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	

APPENDIX 7.Methods used by participants for determining pesticides.

Pyridaben																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
017	D	0.01	0.265	88.3	2.5	7.5	Acetonitrile			No	DSPE, DSPE with graphitised carbon and PSA	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
018	D	0.02	0.26	75.5	2	10	Acetonitrile			Yes	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Rec. from the same batch	Yes: Other	Triphenyl phosphate	ESI (Electrospray Ionisation)	Positive
019	D	0.01	0.294	99	2	10	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch			EI (Electron Ionisation)	
020	D	0.02	0.193	78	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level		MS (Orbitrap)	LC-MS (Orbitrap)	Rec. from validation data	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
021	D	0.02	0.316	95	2	10	Acetonitrile			No	DSPE, CaCl2 and -NH2 (instead of PSA)	Matrix matched-Single Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
022	ND	0.02	0.02		2	Yes (4 mL)	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level	MS (IT)		None	Rec. from the same batch	Yes: Isotopically labelled	fenclorfos	EI (Electron Ionisation)	Positive
023	D	0.01	0.283	95	2	4	Ethyl acetate			Yes	Other (filtration)	Standard Addition	MS (QQQ)		None	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
024	D	0.02	0.397	100	2	10	Acetonitrile	n-Hexane		No	DSPE, LLE with n-hexane	Matrix matched-Multiple Level	MS (QQQ)		LC-MS (QQQ)	Other - via procedural matrix calibration	Yes: Other	Triphenylphosphate	EI (Electron Ionisation)	
025	D	0.05	0.36	100.9	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	No		EI (Electron Ionisation)	
026	D	0.04	0.307	103	2.5	7.5	Methanol	Other: ammonium acetate		No	Other: dilution by 10 and filtration	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	Oxfendazole	ESI (Electrospray Ionisation)	Positive
027	D	0.02	0.3	82.2	2	5	acetone/hexane/ethyl acetate			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (Q)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	
028	D	0.01	0.275	117	2	Yes (4 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (QQQ)		None	Rec. from the same batch	Yes: Other	Aldrin	EI (Electron Ionisation)	Positive
029	D	0.02	0.361	114	2	10	Acetonitrile	n-Hexan		No	Liquid-liquid partitioning	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	PCB 209	EI (Electron Ionisation)	Positive
030	D	0.02	0.316	98	2	Yes 10 ml	Acetonitrile	Ethyl acetate		No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (Q)	Rec. from validation data	No		EI (Electron Ionisation)	Positive
031	NA																			
032	D	0.02	0.345	80	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Isotopically labelled	Linuron-D6	ESI (Electrospray Ionisation)	Positive
033	D	0.02	0.207	96.1	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction, then CaCl2	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Pyridaben																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
034	D	0.02	0.301	90	3	Yes 6ml	Acetone	Dichloromethane	Petroleum ether	No	None	Matrix matched-Multiple Level	MS (QQQ) FPD ECD		None	Rec. from the same batch	No			
035	D	0.01	0.181	69.3	2g	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level	MS (QQQ)	MS (QQQ)		Via Standard Addition	Yes: Other	tris(2-chloro-1(chloromethyl)ethyl)phosphate	EI (Electron Ionisation)	Positive
036	D	0.01	0.16	78	2	10	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TRISCP	EI (Electron Ionisation)	Positive
037	No Results Submited																			
038	No Results Submited																			
039	D	0.02	0.25	103.8	2	Yes (10 mL)	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		LC-MS (QQQ)	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
040	D	0.01	0.59	78.1	15	Yes, 9ml	ACETONE with 30% water	Dichloromethane	Petroleum Ether	No	Liquid-liquid partitioning	Standard Addition	MS (Q)		GC-MS (Q)	Rec. from the same batch	No		EI (Electron Ionisation)	
041	D	0.01	0.26	75	2.5	Yes (10 ml)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from validation data	No		ESI (Electrospray Ionisation)	Positive
042	D	0.05	0.86	70	2	10 mL	Acetonitrile	isoottano		No	Liquid-liquid partitioning	Pure solvent-Single Level	ECD		GC-MS (Q)	Rec. from validation data	Yes: Other	Ethion	EI (Electron Ionisation)	Positive
043	D	0.01	0.359	66	5	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Other	TDCPP		
044	D	0.02	0.362	96.4																
045	D	0.01	0.27	95	2	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
046	D	0.02	0.257	113	2	10	Ethyl acetate			No	Filtration	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
047	D	0.02	0.376	60	0.5	10	Acetonitrile			No	DSPE, PSA	Pure solvent-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
048	D	0.05	0.12	90	2	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (IT)		GC-MS (Q)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	
049	D	0.025	0.32	92	2	4	Acetonitrile			Yes	DSPE, dispersive solid phase extraction (PSA/CaCl2)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
050	D	0.01	0.268	72	2	Yes (13 mL)	Acetone	Dichloromethane	Petroleum ether (PE)	No	None	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
051	D	0.01	0.039	60	2	10	Acetonitrile			No	DSPE, C18 y PSA	Matrix matched-Multiple Level	MS (QQQ)			Rec. from the same batch	No		EI (Electron Ionisation)	

APPENDIX 7.Methods used by participants for determining pesticides.

Pyridaben																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
052	D	0.01	0.23	65	2.5	Yes (5 mL)	Acetone	Dichloromethane	Other (PE)	No	Other (Na2SO4)	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
053	D	0.01	0.128	70	2	Yes	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition	MS (Q)		GC-MS (Q)	Via Standard Addition	Yes: Other	PCB 31	EI (Electron Ionisation)	Negative
054	D	0.03	0.168		2	No	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (Q)		GC-MS (Q)		No		EI (Electron Ionisation)	

APPENDIX 7.Methods used by participants for determining pesticides.

Tebuconazole																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
001	D	0.01	0.364	92.4	5	Yes (10ml)	Acetone	Other (Petroleum ether)	Dichloromethane	No	None	Pure solvent-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Via Standard Addition	No		ESI (Electrospray Ionisation)	Positive
002	D	0.02	0.4	103	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, citrate buffered, dSPE with PSA	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP, Tributylphosphat	EI (Electron Ionisation)	Positive
003	NA																			
004	D	0.05	0.28	65	3	Yes (15 mL)	Acetonitrile			No	Liquid-liquid partitioning, isooctane with the adition of 20% NaCL	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Isotopically labelled	TPP	EI (Electron Ionisation)	
005	D	0.02	0.419	85	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition		MS (QQQ)	None	Via Standard Addition	No		ESI (Electrospray Ionisation)	Positive
006	D	0.02	0.184	111			Acetone	Dichloromethane	Petroleum ether	No	None	Matrix matched-Multiple Level	MS (QQQ)			Rec. from the same batch	No		EI (Electron Ionisation)	
007	D	0.01	0.3		2	4	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)		Rec. from validation data	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
008	D	0.02	0.35	89	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)	LC-MS (QQQ)	Other untreated green tea was spiked	Yes: Other	no calculation, only to check extraction efficiency	ESI (Electrospray Ionisation)	Negative
009	D	0.01	0.43	91	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	triphenylphosphate	ESI (Electrospray Ionisation)	Positive
010	D	0.02	0.32	105	5	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from validation data	No		ESI (Electrospray Ionisation)	Positive
011	D	0.02	0.195	60	2	4	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
012	D	0.01	0.203	100	4	No	Acetonitrile	Dichloromethane	Petroleum ether	No	Liquid-liquid partitioning	Matrix matched-Multiple Level	MS (QQQ)		None	Rec. from validation data	Yes: Other	TFP	EI (Electron Ionisation)	
013	D	0.05	0.42	70	1	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level	Other		Other (Please Specify)					
014	NA																			
015	D	0.02	0.34	91	2	Yes - 10ml	Acetonitrile			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (IT)	MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP (LC) / Bromophos methyl - (GC)	ESI (Electrospray Ionisation)	Positive
016	D	0.01	0.32		2	Yes (10mL)	Acetonitrile			No	DSPE, Graphitized Carbon Black	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Tebuconazole																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
017	D	0.01	0.36	80	2.5	7.5	Acetonitrile			No	DSPE, DSPE with graphitised carbon and PSA	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
018	D	0.02	0.29	82.6	2	10	Acetonitrile			Yes	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (QQQ)		LC-MS (QQQ)	Rec. from the same batch	Yes: Other	Triphenyl phosphate	EI (Electron Ionisation)	Positive
019	D	0.01	0.374	97	2	10	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch			EI (Electron Ionisation)	
020	D	0.02	0.395	108	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	Negative
021	D	0.02	0.331	89	2	10	Acetonitrile			No	DSPE, CaCl2 and -NH2 (instead of PSA)	Matrix matched-Single Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
022	ND	0.02	0.02		2	Yes (4 mL)	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level	MS (IT)		None	Rec. from the same batch	Yes: Isotopically labelled)	fenclorfos	EI (Electron Ionisation)	Positive
023	D	0.01	0.4	75	2	4	Ethyl acetate			Yes	Other (filtration)	Standard Addition		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
024	D	0.02	0.353	100	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Other - via procedural matrix calibration	Yes: Other	Triphenylphosphate	ESI (Electrospray Ionisation)	Positive
025	D	0.05	0.25	62.2	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
026	D	0.04	0.313	78	2.5	7.5	Methanol	Ammonium acetate		No	Other: dilution by 10 and filtration	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	Oxfendazole	ESI (Electrospray Ionisation)	Positive
027	D	0.01	0.285	87.6	3	5	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from validation data	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
028	D	0.01	0.441	101	2	Yes (4 mL)	Acetonitrile			No	None	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
029	D	0.02	0.491	95.8	2	10	Acetonitrile	Acetonitrile		Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TRIS	ESI (Electrospray Ionisation)	Positive
030	D	0.02	0.447	104	2	Yes 10 ml	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from validation data	No		ESI (Electrospray Ionisation)	Positive
031	D	0.10	0.29	78	2	Yes (8 mL)	Ethyl acetate			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (Q)		GC-MS (Q)	Rec. from validation data	Yes: Other	TPP	EI (Electron Ionisation)	Positive
032	D	0.02	0.408	87	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Isotopically labelled	Linuron-D6	ESI (Electrospray Ionisation)	Positive

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Tebuconazole																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
033	D	0.02	0.279	91.4	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction, then CaCl2	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
034	D	0.02	0.336	69	2	Yes 4mL	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
035	D	0.01	0.313	59.5	2g	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level	MS (QQQ)	MS (QQQ)		Via Standard Addition	Yes: Other	tris(2-chloro-1(chloromethyl)ethyl)phosphate	EI (Electron Ionisation)	Positive
036	D	0.01	0.19	86	2	10	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	Yes: Other	TRISCP	EI (Electron Ionisation)	Positive
037	No Results Submited																			
038	No Results Submited																			
039	D	0.02	0.33	98.0	2	Yes (10 mL)	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level		MS (QQQ)	GC-MS (IT)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
040	D	0.01	0.44	68.8	15	Yes, 9ml	ACETONE with 30% water	Dichloromethane	Petroleum Ether	No	Liquid-liquid partitioning	Standard Addition	MS (Q)		GC-MS (Q)	Rec. from the same batch	No		EI (Electron Ionisation)	
041	D	0.01	0.32	85	2.5	Yes (10 ml)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from validation data	No		ESI (Electrospray Ionisation)	Positive
042	D	0.05	0.65	70	2	10 mL	Acetonitrile	isoottano		No	Liquid-liquid partitioning	Pure solvent-Single Level	NPD		GC-MS (Q)	Rec. from validation data				
043	D	0.01	0.605	108	5	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Other	TDCPP		
044	D	0.02	0.482	98.3	1	Yes (2.5 mL)	Acetonitrile			No	SPE, solid phase extraction column(Carb/PSA)+onlineGPC	Matrix matched-Single Level	MS (QQQ)		GC-MS (QQQ)	Rec. from the same batch	No		EI (Electron Ionisation)	Positive
045	D	0.01	0.39	96.4	2	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
046	D	0.02	0.331	98	2	10	Ethyl acetate			No	Filtration	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
047	D	0.02	0.438	64	0.5	10	Acetonitrile			No	DSPE, PSA	Pure solvent-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
048	NA																			
049	D	0.025	0.38	90	2	4	Acetonitrile			Yes	DSPE, dispersive solid phase extraction (PSA/CaCl2)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
050	D	0.01	0.4	80	2	Yes (13 mL)	Acetone	Dichloromethane	Petroleum ether (PE)	No	None	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Tebuconazole																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
051	D	0.01	0.018	70	2	10	Acetonitrile			No	DSPE, C18 y PSA	Matrix matched-Multiple Level	MS (QQQ)			Rec. from the same batch	No		EI (Electron Ionisation)	
052	D	0.01	0.36	89	2.5	Yes (5 mL)	Acetone	Dichloromethane	Other (PE)	No	Other (Na2SO4)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
053	ND	0.01	0.01		2	Yes	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition	MS (Q)		GC-MS (Q)	Via Standard Addition	Yes: Other	PCB 31	EI (Electron Ionisation)	Negative
054	D	0.03	0.124		2	No	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level with complete sample preparation		MS (QQQ)	LC-MS (QQQ)		Yes: Isotopically labelled	Atrazin D5	ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Thiophanate-methyl																			
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
001	D	0.01	0.178	73.92	5	Yes (10ml)	Acetone	Other (Petroleum ether)	Dichloromethane	No	None	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Via Standard Addition	No		ESI (Electrospray Ionisation)	Positive
002	D	0.02	0.229		2	Yes (10 mL)	Acetonitrile			Yes	DSPE, citrate buffered, dSPE with PSA	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Via Standard Addition	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
003	NA																			
004	D	0.05	0.132	95	3	Yes (15 mL)	Acetonitrile			No	Freezing out	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
005	D	0.02	0.377	-	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition		MS (QQQ)	None	Via Standard Addition	No		ESI (Electrospray Ionisation)	Positive
006	D	0.02	0.238	92	2	4	Acetonitrile			Yes	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
007	ND	0.01	0.01		2	4	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)		Rec. from validation data	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
008	D	0.02	0.19	75	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Single Level		MS (QQQ)	LC-MS (QQQ)	Other untreated green tea was spiked	Yes: Other	no calculation, only to check extraction efficiency	ESI (Electrospray Ionisation)	Negative
009	D	0.01	0.26	111	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	triphenylphosphate	ESI (Electrospray Ionisation)	Positive
010	NA																			
011	D	0.02	0.168	80	2	4	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (IT)		GC-MS (IT)	Rec. from the same batch	Yes: Other	TPP	EI (Electron Ionisation)	Positive
012	D	0.02	0.45	85	2	10	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level	MS (QQQ)	MS (QQQ)	None	Rec. from validation data	Yes: Other	TFP	ESI (Electrospray Ionisation)	Positive
013	NA																			
014	D	0.01	0.175	59	2	no	Methanol	Water		no	Filter	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
015	D	0.02	0.66	47	2	Yes - 10ml	Acetonitrile			No	SPE, solid phase extraction column	Matrix matched-Multiple Level	MS (IT)	MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP (LC) / Bromophos methyl - (GC)	ESI (Electrospray Ionisation)	Positive
016	D	0.01	0.11	107	2	Yes (10mL)	Acetonitrile			No	DSPE, Graphitized Carbon Black	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
017	D	0.01	0.193	62.8	2.5	7.5	Acetonitrile			No	DSPE, DSPE with graphitised carbon and PSA	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Thiophanate-methyl																				
Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
018	D	0.02	0.17		2	10	Acetonitrile			Yes	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	Yes: Other	Triphenyl phosphate	ESI (Electrospray Ionisation)	Positive
019	D	0.01	0.356		2	10	Acetonitrile			No	SPE, solid phase extraction column	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch			ESI (Electrospray Ionisation)	Positive
020	D	0.02	0.175	104	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level		MS (Orbitrap)	LC-MS (Orbitrap)	Rec. from validation data	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
021	D	0.02	0.174	96	2	10	Acetonitrile			No	DSPE, CaCl2 and -NH2 (instead of PSA)	Matrix matched-Single Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
022	NA																			
023	D	0.01	0.262	NA	2	4	Ethyl acetate			Yes	Other (filtration)	Standard Addition		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
024	D	0.02	0.086	100	2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	None	Other - via procedural matrix calibration	Yes: Other	Triphenylphosphate	ESI (Electrospray Ionisation)	Positive
025	D	0.05	0.15	35.5	2	4	Acetonitrile				DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
026	D	0.04	0.218	100	2.5	7.5	Methanol	Other: ammonium acetate		No	Other: dilution by 10 and filtration	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	Oxfendazole	ESI (Electrospray Ionisation)	Positive
027	D	0.01	0.02	86.8	3	5	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from validation data	Yes: Other	TPP	ESI (Electrospray Ionisation)	Positive
028	D	0.01	0.194	67	2	Yes (4 mL)	Acetonitrile			No	None	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
029	D	0.02	0.089	78.3	2	10	Acetonitrile	Acetonitrile		Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	Yes: Other	TRIS	ESI (Electrospray Ionisation)	Positive
030	D	0.02	0.62	105	2	Yes 10 ml	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from validation data	No		ESI (Electrospray Ionisation)	Positive
031	NA																			
032	D	0.02	0.231	89	2	Yes (10 mL)	Acetonitrile			Yes	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Isotopically labelled	Linuron-D6	ESI (Electrospray Ionisation)	Positive
033	D	0.02	0.0388		2	10	Acetonitrile			No	DSPE, dispersive solid phase extraction, then CaCl2	Extracted standard from spiked tea		MS (QQQ)	LC-MS (QQQ)		No		ESI (Electrospray Ionisation)	Positive
034	D	0.02	0.482	70	2	Yes 4mL	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive

APPENDIX 7.Methods used by participants for determining pesticides.

Thiophanate-methyl

Lab. Code	Scope of Method	Reporting Level (mg/kg)	Official Concentration (mg/kg)	Recovery %	Sample Weight (g)	Water addition? (mL)	Solvent 1	Solvent 2	Solvent 3	pH Adjustment	Clean Up	Calibration	GC Detector	LC Detector	Confirmation Method	Recovery Approach	ISTD Used	ISTD Details	Ionisation mode:	Polarity
035	D	0.01	0.189	62.7	2	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Pure solvent-Multiple Level	MS (QQQ)	MS (QQQ)		Via Standard Addition	Yes: Other	tris(2-chloro-1(chloromethyl)ethyl)phosphate	EI (Electron Ionisation)	Positive
036	NA																			
037	No Results Submited																			
038	No Results Submited																			
039	D	0.02	0.18	72.0	2	Yes (10 mL)	Acetonitrile			No	DSPE, CaCl2 (instead of MgSO4)	Pure solvent-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
040	D	0.005	0.18	76	2	Yes,10	Acetonitrile+ water(1:1)			Yes	DSPE, dispersive solid phase extraction	Standard Addition		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
041	NA																			
042	NA																			
043	D	0.01	6.2	98	5	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)		Rec. from the same batch	Yes: Other	TDCPP		
044	ND	0.02	0.02		1	Yes (2.5 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction(PSA)	Matrix matched-Multiple Level	Other (Please Specify)	MS (Orbitrap)+MSMS(QQQ)	LC-MS (Orbitrap) +LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
045	D	0.01	0.21	81.3	2	Yes (10 mL)	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
046	D	0.02	0.214	282	2	10	Ethyl acetate			No	Filtration	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
047	D	0.02	0.38	53	0.5	10	Acetonitrile			No	DSPE, PSA	Pure solvent-Multiple Level		MS (QQQ)	None	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
048	NA																			
049	D	0.025	0.19	73	2	4	Acetonitrile			Yes	DSPE, dispersive solid phase extraction (PSA/CaCl2)	Matrix matched-Multiple Level		MS (QQQ)	LC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
050	ND	0.01	0.01		2	Yes (13 mL)	Acetone	Dichloromethane	Petroleum ether (PE)	No	None	Matrix matched-Multiple Level		MS (QQQ)	GC-MS (QQQ)	Rec. from the same batch	No		ESI (Electrospray Ionisation)	Positive
051	NA																			
052	D	0.05	0.25	100	2.5	Yes (5 mL)	Acetone	Dichloromethane	Other (PE)	No	Other (Na2SO4)	Standard Addition		MS (QQQ)	LC-MS (QQQ)	Via Standard Addition	No		ESI (Electrospray Ionisation)	Positive
053	ND	0.01	0.01		2	Yes	Acetonitrile			No	DSPE, dispersive solid phase extraction	Standard Addition		MS (QQQ)	LC-MS (QQQ)	Via Standard Addition	Yes: Other	tris-(1,3-dichlorisopropyle)-phosphate	ESI (Electrospray Ionisation)	Positive
054	D	0.03	0.043		2	No	Acetonitrile			No	DSPE, dispersive solid phase extraction	Matrix matched-Multiple Level with complete sample preparation		MS (QQQ)	LC-MS (QQQ)		Yes: Isotopically labelled	Atrazin D5	ESI (Electrospray Ionisation)	Positive

3rd Edition
Approved: January 2012

GENERAL PROTOCOL

for EU Proficiency Tests on Pesticide Residues in Food and Feed

Introduction

This protocol contains general procedures valid for all European Union Proficiency Tests (EUPTs) organised on behalf of the European Commission, DG-SANCO[1] by the four European Union Reference Laboratories (EURLs) for pesticide residues in food and feed. These EUPTs are directed at all National Reference Laboratories (NRLs) and Official Laboratories (OfLs) within the EU Member States. Laboratories outside of this EURL/NRL/OfL-Network[2] may be permitted to participate on a case-by-case basis after consultation with DG-SANCO.

The following four EURLs for pesticide residues were appointed by DG-SANCO based on regulation 882/2004/EC[3]:

- EURL for Fruits and Vegetables (EURL-FV)
- EURL for Cereals and Feedingstuff (EURL-CF)
- EURL for Food of Animal Origin and Commodities with High Fat Content (EURL-AO) and
- EURL for Single Residue Methods (EURL-SRM)

NRLs are appointed by Member State based on the provisions of Regulation 882/2004/EC, whereas OfLs are laboratories that are actively involved in official controls following Article 26 of Regulation 396/2004/EC (e.g. by conducting pesticide residue analyses within the framework of national and/or EU-controlled programmes).

According to Article 28 (3) of Regulation 396/2005/EC[4], all laboratories analysing samples for the official control of pesticide residues shall participate in the European Union Proficiency Test(s) organised by the European Union. The aim of these EUPTs is to obtain information regarding the quality, accuracy and comparability of the pesticide residue data in food and feed sent to the European Union within the framework of the national control programmes and the co-ordinated

[1] DG-SANCO = European Commission, Health and Consumer Protection Directorate-General
[2] For more information about the EURL/NRL/OfL-Network please refer to the EURL-Web-portal under: http://www.eurl-pesticides.eu
[3] Regulation (EC) No 882/2004 of the European Parliament and of the Council on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules. Published at OJ of the EU L191 of 28.05.2004
[4] Regulation (EC) No 396/2005, published at OJ of the EU L70 of 16.03.2005, as last amended by Regulation 839/2008 published at OJ of the EU L234 of 30.08.2008.

multiannual community control programme[5]. Participating laboratories will be provided with an assessment of their analytical performance and the reliability of their data – compared to the other participating laboratories.

EUPT-Panel

EUPTs are organised by individual EURLs or by more than one EURL in joint cooperation.

An **Organising Team** is appointed from the EURL(s) in charge. This team is responsible for all administrative and technical matters concerning the organisation of the PT, e.g. PT-announcement; Test Item production; undertaking the homogeneity and stability tests; packing and shipment of Test Item, as well as the handling and first assessment of participants' results.

Approved by DG SANCO, expert scientists with long-term experience in pesticide residue analysis will be chosen as members of a joint **EUPT-Scientific Committee** (SC). This Committee is made up of the following two subgroups:

a) An independent **Quality Control Group** (QCG) and

b) An **Advisory Group** (AG)

The SC's role is to help the organisers make decisions regarding the EUPT design: the selection of pesticides to be included in the Target Pesticide List (see below); the establishment of the Minimum Required Reporting Levels (MRRLs); the evaluation and statistical treatment of the results and the drafting of the protocol and final report. The QCG has the additional function of supervising the quality of the EUPT and to assist the EURL in confidential aspects such as the choice of the pesticides to be present in the Test Item and the concentration levels at which they should be present in the Test Item.

The EUPT-Organising Team and the EUPT-Scientific Committee (the AG and the QCG) together form the **EUPT-Panel**.

The present EUPT General Protocol was drafted by the EUPT-Panel and was approved by DG-SANCO.

EUPT Participants

All NRLs operating in the same area as the organising EURL are legally obliged to participate in EUPTs - as well as all OfLs whose scope overlaps with that of the EUPT. The four EURLs will be annually issuing and distributing via the EURL website, a joint list of all OfLs that shall participate in all EUPTs to be conducted within a given year. The "list of obliged labs" is to be considered as tentative as it will be only based on information submitted by OfLs concerning their commodity scope and status. The legal obligation of NRLs and OfLs to participate in EUPTs arises from:

- Art. 28 of Reg. 396/2005/EC (for all OfLs analyzing for pesticide residues within the framework of official controls in food or feed)

[5] European Commission Proficiency Tests for Pesticide Residues in Fruits and Vegetables, Trends in Analytical Chemistry, 2010, 29 (1), 70-83.

- Art. 33 of Reg. 882/2004/EC (for all NRLs)

If necessary the "list of obliged labs" will be updated within the same year to take account of any changes in the lab profiles.

NRLs are responsible for checking whether all relevant OfLs within their network are included in the list of obliged laboratories and whether the contact information is correct.

The NRLs should further make arrangements to urge all relevant OfLs within their network to participate in all EUPT relevant to them.

OfLs are urged to keep their own profiles within the EURL-DataPool up-to-date, especially their commodity and pesticide scopes and their contact information.

Any OfL not intending to participate in a given EUPT will have to explain to the EURL its reasons for non-participation without prejudice of any legal action taken against it for not participating. This also applies to initially participating laboratories that do not deliver results.

Official labs from EFTA countries and EU-candidate countries are also welcome to participate in the EUPTs. In special cases, the Organisers, upon consultation with DG-SANCO, will also allow laboratories outside of the EURL/NRL/OfL-Network to participate in EUPTs.

Confidentiality

The proprietor of all EUPT data is DG-SANCO and thus has access to all information.

In each EUPT, the laboratories are given a unique code, initially only known to themselves and the Organisers. In the final EUPT-Report, the list of participating laboratories will not be linked to their laboratory codes. It should be noted that the organisers, at the request of DG-SANCO, may present the EUPT-results to the Standing Committee on the Food Chain and Animal Health on a country-by-country basis. It is therefore possible that a link between codes and laboratories could be made, especially for those countries where only one laboratory has participated.

As laid down in Regulation 882/2004, NRLs are responsible for evaluating and improving their own OfL network. For this reason, the EURLs will provide the OfL laboratory codes to their NRLs together with the final report. This will allow NRLs to correlate the laboratories within their network and their performance. Furthermore, the EURLs reserve the right to share EUPT results and codes among themselves: for example, for the purpose of evaluating overall lab performance as requested by DG-SANCO.

Communication

The official language used in all EUPTs is English.
Communication between participating laboratories during the test on matters concerning this PT exercise is not permitted.

ANNEX 1. Protocols and instructions. Target List of pesticides to be sought.

Announcement / Invitation Letter

The announcement of the individual EUPT will be issued at least 3 months before the Test Item is distributed to the laboratories. The announcement will be published on the EURL portal and additionally distributed via e-mail to the NRL/OfL mailing list available to the EURLs. The announcement will contain an invitation letter, details on how to register and where to find additionally-related documents, as well as some preliminary information on the specific protocol such as the tentative calendar, the name of the commodity expected to be used, and the tentative Target Pesticide List.

Target Pesticide List

This list contains all analytes (pesticides and metabolites) to be tested, along with the Minimum Required Reporting Levels (MRRLs) valid for the specific EUPT. The MRRLs are based upon the lowest MRLs found either in Regulation 396/2005/EC or Commission Directive 2006/125/EC (Baby Food Directive).

In some cases, that will be clearly marked, results calculated according to the pesticide residue definition may be requested with those residue definitions differing from the legal ones in certain cases.

Specific Protocol

For each EUPT a Specific Protocol will be published at least 2 weeks before the Test Item is distributed to the laboratories. This protocol will contain all the information previously included in the Invitation Letter but in its final version, in addition to information on payment for delivery service and/or participation. It will furthermore include instructions on how to handle the Test Item upon receipt, on how to submit results, and any other relevant information.

General procedures for reporting results

Laboratories are responsible for reporting their results to the Organiser within the stipulated deadlines. Any pesticide that was targeted by a participating laboratory should be reported as "analysed". Each laboratory must report only one result for each of the analytes detected in the Test Items, using the analytical procedure(s) that they would routinely use for each compound for monitoring purposes. The residue levels of the pesticides detected should be expressed in mg/kg and in some cases for products of animal origin in µg/kg fat.

One Test Item is intentionally treated with pesticides and one is not. Both Test Items have to be analysed by the laboratories and any pesticide detected in them shall be reported.

Correction of results for recovery

According to the Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed, (Document SANCO), it is common practice that pesticide analysis results are not corrected for recovery, but may be corrected if the average recovery is

significantly different from 100% (typically if outside the 70-120% range with good precision), therefore, if residue data are adjusted for recovery, then this must be indicated on the specific field of the 'reporting result form'. Laboratories are required to report whether their results were adjusted for recovery and, if this was the case, the recovery (as percentage) used should be also reported. No recovery data are required where correction for recovery results automatically from using the 'standard addition(s)' approach, or isotopically-labelled internal standards (in both cases with spiking of the Test Item at the beginning of the extraction procedures). In these cases, the laboratories should report the calculation technique used for the results instead of the recovery data.

Methodology information

All laboratories are requested to provide information on the analytical method(s) they have used. If no sufficient information on the methodology used is provided, the Organiser reserves the right not to accept the analytical results reported by the participants concerned.

Results evaluation

The procedures used for the treatment and assessment of results are described below.

- ***False Positives***

These are results reported above the MRRLs that suggest the presence of pesticides that were listed in the Target Pesticide List, but which were: (i) not detected by the Organiser, even after repeated analyses, and/or (ii) not detected by the overwhelming majority (e.g. 95%) of the participating laboratories that had targeted the specific pesticide. However, in certain instances, case-by-case decisions by the EUPT-Panel may be necessary.
Any results reported that are lower than the MRRL will not be considered as false positives, even though these results should not have been reported.

- ***False Negatives***

These are results for pesticides reported by the laboratories as "analysed" but without reporting numerical values although they were used by the Organiser to treat the Test Item and were detected by the Organiser and the majority of the participants that had targeted these specific pesticides, at or above the MRRL. Results reported as <RL (RL= Reporting Limit of the laboratory) will be considered as not detected and will be judged as false negatives. However, in certain instances, case-by-case decisions by the EUPT-Panel may be necessary.

In cases of the assigned value being less than a factor of 4 times the MRRL, false negatives will not be assigned as this is not statistically justifiable.

- ***Estimation of the true concentration (μ)***

The "true" concentration (assigned value) will be typically estimated using the median of all the results. In special justifiable cases, the EUPT-Panel may decide to use only part of the population of results to establish the median (e.g. only results with z-scores ≤ 5.0, or by excluding results

generated by a method that demonstrably generates significantly biased results, e.g. due to incomplete extraction).

- ***Standard deviation of the assigned value (target standard deviation)***

The target standard deviation (δ) of the assigned value will be calculated using a Fit-For-Purpose Relative Standard Deviation (FFP-RSD) approach, as follows:

$$\delta = b_i * \mu_i \quad \text{with } b_i = 0.25 \text{ (25\% FFP-RSD)}$$

The percentage FFP-RSD is set at 25% based on experience from previous EUPTs[6]. The EUPT-Panel reserves the right to also employ other approaches on a case-by-case basis considering analytical difficulties and experience gained from previous proficiency tests.

- ***z-scores***

This parameter is calculated using the following formula:

$$z_i = (x_i - \mu_i) / \delta_i$$

Where: x_i is the value reported by the laboratory, μ_i the assigned value, and δ_i the standard deviation at that level for each pesticide (i).

Any z-scores of > 5 will be reported as >5 and where combined z-scores are calculated a value of "5" will be used.

z-Scores will be interpreted in the following way:

$\|z\| \leq 2$	Acceptable
$2 < \|z\| \leq 3$	Questionable
$\|z\| > 3$	Unacceptable

For results that are considered to be false negatives, z-scores will be calculated using the MRRL or RL (the laboratory's Reporting Limit) if the RL < MRRL.

The EUPT-Panel will consider whether, or not, these values should appear in the z-score histograms.

z-Scores will not be calculated for any false positive result.

- ***Category A and B classification***

The EUPT-Panel will decide whether to classify the laboratories into two groups - A or B. Laboratories that detect a sufficiently high percentage of the pesticides present in the Test Item (e.g. at least 90%) and reported no false positives will have demonstrated 'sufficient scope' and will therefore be classified into Category A. The 90% criterion will be applied following Table 1.

[6] Comparative Study of the Main Top-down Approaches for the Estimation of Measurement Uncertainty in Multiresidue Analysis of Pesticides in Fruits and Vegetables. J. Agric. Food Chem., 2011, 59(14), 7609-7619.

ANNEX 1. Protocols and instructions. Target List of pesticides to be sought.

Table 1. No. of pesticides needed to be detected to have sufficient scope.

No. of Pesticides Present in the Sample (N)	90%	No. of Pesticides needed to be detected to have sufficient scope (n)	n
3	2.7	3	N
4	3.6	4	
5	4.5	4	N - 1
6	5.4	5	
7	6.3	6	
8	7.2	7	
9	8.1	8	
10	9.0	9	
11	9.9	10	
12	10.8	11	
13	11.7	12	
14	12.6	13	
15	13.5	13	N - 2
16	14.4	14	
17	15.3	15	
18	16.2	16	
19	17.1	17	
20	18.0	18	
21	18.9	19	
22	19.8	20	
23	20.7	21	
24	21.6	22	
25	22.5	22	N - 3
26	23.4	23	

For evaluation of the overall performance of laboratories within Category A, the Average of the Squared z-Score (AZ^2)[7,8] will be used.

Laboratories within Category B will be ranked according to the total number of pesticides present in the sample. The number of acceptable z-scores achieved will be presented too. The EURL-Panel retains the right to calculate combined z-scores (see below) also for Category B labs, e.g. for informative purposes, provided that a minimum number of results (z-scores) is available.

[7] Formerly named "Sum of squared z-scores (SZ^2)"
[8] Laboratory assessment by combined z-score values in proficiency tests: experience gained through the EUPT for pesticide residues in fruits and vegetables. Anal. Bioanal. Chem., 2010, 397, 3061–3070.

- ***Combined z-scores***

For evaluation of the overall performance, the Average of the Squared z-Score (AZ^2) will be used. The AZ^2 is calculated as follows:

$$AZ^2 = \frac{\sum_{i=1}^{n} |Z_i| |Z_i|}{n}$$

This formula multiplies each z-score by itself and not by an arbitrary number. Based on the AZ^2 achieved, the laboratories are classified as follows:

Formula	Good	Satisfactory	Unsatisfactory
AZ^2	≤ 2	$2 < AZ^2 \leq 3$	$AZ^2 > 3$

Combined z-scores are considered to be of lesser importance than the individual z-scores. The EUPT-Panel retains the right not to calculate AZ^2 if it is considered as not being useful. In the case of EUPT-SRMs, where only few results per lab are available, the Average of the Absolute z-scores (AAZ) will be calculated for informative purposes, but only for labs within Category A and as long as 5 or more z-scores are available.

Publication of results

The EURLs will publish a preliminary report, containing tentative medians and z-score values for all pesticides present in the test sample, within 2 months from the deadline for result submission.

The Final Report will be published after the EUPT-Panel has discussed the results. Taking into account that the EUPT-Panel meets normally only once a year to discuss the results of all EUPTs organised annually by the EURLs in the running year, the final report may be published up to 8 months after the deadline for results submission.

Certificates of participation

Along with the Final Report, the EURL Organiser will deliver a Certificate of Participation to each participating laboratory with the z-score achieved for each pesticide and the combined z-scores calculated (if any) together with the classification into Category A and B.

Feedback

After the distribution of the final report of an EUPT, participating laboratories will be given the opportunity to give their feedback to the Organiser and make suggestions for future improvements.

Follow-up activities

Laboratories are expected to undertake follow-up activities to trace back to the source of any erroneous or (strongly) deviating results - including all false positives and false negatives, along with results with $|z|>2$.

Upon request, the laboratory's corresponding NRL, or EURL, are to be informed of the outcome of these traceability activities.

> According to instructions by DG-SANCO, the "Protocol for management of underperformance in comparative testing and/or lack of collaboration of National Reference Laboratories (NRLs) with EU Reference Laboratories (EURLs) activities" will be followed for NRLs.

Disclaimer

The EUPT-Panel retains the right to change any parts of this EUPT – General Protocol based on new scientific or technical information. Any changes will be communicated in due course.

Laboratory Rights

After the Final Report has been sent, the laboratories will have the right to communicate the nonconformity of their result evaluation in written form. Any detected errors in the preliminary report should also be reported to the Organiser. The Organiser, assisted by the Scientific Committee, will decide upon any re-evaluation and will give a corresponding explanation.

EUPT-T01 SPECIFIC PROTOCOL

European Union Proficiency Test for

Pesticide Residues in tea

(2013)

Introduction

This protocol is complementary to the General Protocol of EU Proficiency Tests (EUPTs) for Pesticide Residues in Food and Feed. This Proficiency Test is organised by the EURL for Pesticide Residues in Fruit and Vegetables covering Multiresidue Methods (MRM) of analysis.

Test item

This proficiency test is based on the analysis of tea samples from China containing incurred residues of pesticides. The samples were purchased in a specialised shop for Chinese products, in Almería, Spain.

The test item (dried green tea containing incurred pesticide residues) was ground, homogenised and sub-sampled into self-seal bags that had previously been coded.

Ten of those bags containing the test item have been chosen randomly, and analysed to check for homogeneity.

The test item is stored at 4ºC prior to shipment to participants.

Two bags, again chosen randomly, will be analysed by the Organiser over a period of time to confirm the stability of the pesticides in the test item (firstly, when the test items are shipped, then a few days after the receipt deadline for participants´ results).

Steps to follow

This Proficiency Test will be made up of the following steps:

1. To participate, each laboratory must complete and return the Application Form, sent to the participants by e-mail, before the deadline stipulated on the Calendar. The participants will also receive the Target Pesticide List, containing the Minimum Required
Reporting Limits (MRRLs). Those MRRLs do not always correspond with the EU MRLs set for tea. Participation in this proficiency test remains on a voluntary basis.

2. Laboratories will then receive an e-mail confirming their participation in this exercise, and assigning them each a Laboratory Code.

3. The sample delivery will be provided free of charge for those laboratories that have participated in EUPT-T01.

4. The sample will be delivered to the participant laboratories on June 10th 2013. At the same time they will receive by e-mail an Excel file where they will be able to report the results.

5. The deadline for submitting the results of this proficiency test is 28th June 2013.

6. The Organiser will evaluate the results at the end of the proficiency test, once the deadline for the receipt of results has passed. The Organiser will upload an electronic version onto the EURL-FV website and will send the electronic copy of the Final Report to each participant laboratory. This report will include information regarding the design of the test, the homogeneity and stability results, a statistical evaluation of the participant's results as well as graphical displays of the results and any conclusions. Further relevant information considered to be of value may also be included.

Amount of Test Item

Participants will receive:

• Approximately 15 g of incurred commercial tea.

Shipment of Test item

The test item will be packed in self-seal bags and into cardboard boxes protected with foam in the interior.

The shipment of the test item will be carried out over a one-week period from the 10th June 2013. The Organiser will try to ensure that all the packages arrive on the same day at each laboratory. An information message will be sent out by e-mail before shipment. Laboratories must make their own arrangements for the receipt of the package. They must inform the Organiser of any public holidays in their country/city during the delivery period given in the calendar, as well as making the necessary arrangements for receiving the shipment, even if the laboratory is closed.

Advice on Test item Handling

Once received, the test item should be stored at 4°C prior to analysis thus avoiding any possible deterioration/spoilage. The test item should be mixed thoroughly before taking the analytical portion(s).

All participants should use their own routine standard operating procedures for extraction, clean-up and analytical measurement and their own reference standards for identification and quantification.

Test item Receipt

Once the laboratory has received the test item, its arrival must be reported to the Organiser by e-mail. The deadline for acceptance (or non-acceptance) is 14th June 2013. If the laboratory does

not respond by this date, the Organiser will assume that the test item has been received and accepted.

If any laboratory has not received the test item by 14th June, they must inform the Organiser **immediately** by e-mail (cferrer@ual.es or analozano@ual.es)

Submission of results:

Once the laboratory has analysed the test item and is ready to submit their data, they must enter their results in the Excel file provided by the Organisers and send it to the following e-mail address: cferrer@ual.es.

All analyte concentrations must be expressed in mg/kg together with the associated recovery expressed as a percentage.

The number of significant figures should be based on the guidelines provided in SANCO/12495/2011. Additional significant figures may be recorded for the purpose of statistical analysis. Please bear this in mind when reporting data:

- Residue levels < 0.010 mg/kg should be rounded to one significant figure
- Residue levels ≥ 0.010 mg/kg and < 10 mg/kg should be rounded to two significant figures
- Residue levels ≥ 10 mg/kg may be rounded to three significant figures or to a whole number.

Results should not be reported where a pesticide was not detected or was detected below the laboratory's LOQ. In both cases, this should be recorded as 'ND' (Not Detected) or <LOQ. If a pesticide was not sought, it should be recorded as 'NA' (Not Analysed). The actual results/residue levels measured must be reported as numbers.

Further instructions on how to fill in the Excel file will be provided in the same file.

False Negatives

After the receipt of results, participant laboratories that have reported that they sought a pesticide present in the test item but did not find it (false negative) will be asked via e-mail about the analytical method used to determine that specific pesticide.

Calendar

ACTIVITY	DATE
Sending Application Form to laboratories	9th May 2013
Sending calendar and pesticides target list to participant laboratories.	14th May 2013
Deadline for receiving Application Form from laboratories.	17th May 2013
Sample distribution.	10th June 2013
Deadline for receiving results	28th June 2013
Preliminary Report: only results, no statistical treatment.	July 2013
Final Report	September 2013

ANNEX 1. Protocols and instructions. Target List of pesticides to be sought.

Cost of test item shipment.

The sample delivery will be free of charge for those laboratories that have participated in EUPT-T01. Other laboratories will be charged **175 €**. Regarding payment procedures, each laboratory can specify their details and invoice requests when applying for the test. Payment details are as follows:

BANK NAME: CAJAMAR - Caja Rural Sociedad Corporativa de Crédito
BANK ACCOUNT HOLDER: Universidad de Almeria
BANK ADDRESS: Office Number 990. Universidad de Almeria. Spain
ACCOUNT NUMBER: 30580130172731005000
IBAN: ES0730580130172731005000
SWIFT: CCRIES2A
REFERENCE GIVEN: Invoice No. or Lab Code

Contact information

The official organising group details are as follows:

Universidad de Almería. Edificio Químicas CITE I
Ctra. Sacramento s/n
04120 Almería - Spain
Fax No.: +34 950015483

Organising team (e-mails and phone no.s) EURL-FV:

Dr. Amadeo R. Fernández-Alba	amadeo@ual.es	+34 950015034
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Ms. Ana Lozano	analozano@ual.es	+34 950015645
Mr. Łukasz Rajski	154303@edu.p.lodz.pl	+34 950015645

Quality Control Group

Dr. Antonio Valverde	University of Almería, Spain
Mr. Stewart Reynolds, Senior Chemist	FERA, York, United Kingdom

Statistical Group

Dr. Carmelo Rodriguez, Senior Mathematician, University of Almeria, Spain

Advisory Group

Dr. André de Kok, Senior Chemist	NVWA, Wageningen, The Netherlands.
Dr. Tuija Pihlström, Senior Chemist	NFA, Uppsala, Sweden.
Dr. Sonja Masselter, Senior Chemist	AGES, Innsbruck, Austria.
Dr. Darinka Stajnbaher, Senior Chemist	Maribor, Slovenia.
Dr. Magnus Jezussek, Senior Chemist	Erlangen, Germany.

ANNEX 1. Protocols and instructions. Target List of pesticides to be sought.

Dr. Miguel Gamón, Senior Chemist	Lab. Agroalimentario, Valencia, Spain.
Dr. Mette Erecius Poulsen, Senior Chemist	NFI, Copenhagen, Denmark.
Mr. Ralf Lippold, Senior Chemist	CVUA, Freiburg, Germany.
Dr. Michelangelo Anastassiades, Senior Chemist	CVUA, Stuttgart, Germany.

TARGET PESTICIDE LIST FOR THE EUPT-T01

Pesticide	MRRL (mg/Kg)
3-hydroxy-carbofuran	0.02
Acephate	0.02
Acetamiprid	0.02
Acrinathrin	0.02
Aldicarb	0.02
Aldicarb Sulfone	0.02
Aldicarb Sulfoxide	0.02
Amitraz	0.02
Azinphos-methyl	0.02
Azoxystrobin	0.02
Benfuracarb	0.02
Bifenthrin	0.02
Bitertanol	0.02
Boscalid	0.02
Bromopropylate	0.02
Bromuconazole	0.02
Bupirimate	0.02
Buprofezin	0.02
Cadusafos	0.01
Captan	0.02
Carbaryl	0.02
Carbendazim (sum of benomyl and carbendazim expressed as carbendazim)	0.02
Carbofuran	0.02
Carbosulfan	0.02
Chlorfenapyr	0.02
Chlorfenvinphos	0.02
Chlorobenzilate	0.02
Chlorothalonil	0.02
Chlorpropham (only parent compound)	0.02
Chlorpyrifos	0.02
Chlorpyrifos-methyl	0.02
Clofentezine (only parent compound)	0.02
Clothianidin	0.02
Cyfluthrin (cyfluthrin incl. other mixtures of constituent isomers (sum of isomers))	0.02
Cypermethrin (cypermethrin incl. other mixtures of constituent isomers (sum of isomers))	0.02
Cyproconazole	0.02
Cyprodinil	0.02
Deltamethrin	0.02
Demeton-S-methylsulfone	0.02
Desmethyl-pirimicarb	0.02
Diazinon	0.02
Dichlofluanid (only parent compound)	0.01
Dichlorvos	0.02
Dicloran	0.01
Dicofol	0.02
Difenoconazole	0.02
Diflubenzuron	0.02
Dimethoate	0.02
Dimethomorph	0.02
Dimethylaminosulfotoluidide (DMST)	0.02
Diphenylamine	0.02
DMF (2,4-Dimethylformanilide)	0.02
DMPF (N-2,4-Dimethylphenyl-N-Methyl-formamidine)	0.02
Endosulfan alpha	0.02
Endosulfan beta	0.02
Endosulfan sulfate	0.02

ANNEX 1. Protocols and instructions. Target List of pesticides to be sought.

Pesticide	MRRL (mg/Kg)
EPN	0.01
Epoxiconazole	0.02
Ethion	0.02
Ethoprophos	0.02
Etofenprox	0.01
Fenamiphos	0.02
Fenamiphos sulfone	0.02
Fenamiphos sulfoxide	0.02
Fenarimol	0.02
Fenazaquin	0.02
Fenbuconazole	0.02
Fenhexamid	0.02
Fenitrothion	0.02
Fenoxycarb	0.02
Fenpropathrin	0.02
Fenpropimorph	0.02
Fenthion	0.02
Fenthion oxon	0.02
Fenthion oxon sulfone	0.02
Fenthion oxon sulfoxide	0.02
Fenthion sulfone	0.02
Fenthion sulfoxide	0.02
Fipronil (only parent compound)	0.005
Fludioxonil	0.02
Flufenoxuron	0.02
Fluopicolide	0.02
Fluquinconazole	0.02
Flusilazole	0.02
Flutolanil	0.02
Flutriafol	0.02
Folpet	0.02
Fosthiazate	0.02
Hexaconazole	0.02
Hexythiazox	0.02
Imazalil	0.02
Imidacloprid	0.02
Indoxacarb (Indoxacarb as sum of the isomers S and R)	0.02
Iprodione	0.02
Iprovalicarb	0.02
Isofenphos-methyl	0.01
Kresoxim-methyl	0.02
Lambda-Cyhalothrin	0.02
Linuron	0.02
Lufenuron	0.02
Malaoxon	0.02
Malathion	0.02
Mepanipyrim (only parent compound)	0.02
Metaflumizone	0.02
Metalaxyl and metalaxyl-M	0.02
Metconazole	0.02
Methamidophos	0.02
Methidathion	0.02
Methiocarb	0.02
Methiocarb sulfone	0.02
Methiocarb sulfoxide	0.02
Methomyl	0.02
Methoxyfenozide	0.02
Monocrotophos	0.02

ANNEX 1. Protocols and instructions. Target List of pesticides to be sought.

Pesticide	MRRL (mg/Kg)
Myclobutanil	0.02
Omethoate	0.02
Orthophenylphenol	0.02
Oxadixyl	0.02
Oxamyl	0.02
Oxydemeton-methyl	0.02
Paclobutrazole	0.02
Paraoxon-methyl	0.02
Parathion-ethyl	0.02
Parathion-methyl	0.02
Penconazole	0.02
Pencycuron	0.02
Pendimethalin	0.02
Phenthoate	0.01
Phosalone	0.02
Phosmet	0.02
Phosmet oxon	0.02
Phoxim	0.02
Pirimicarb	0.02
Pirimiphos-methyl	0.02
Prochloraz (only parent compound)	0.02
Procymidone	0.02
Profenofos	0.02
Propargite	0.02
Propiconazole	0.02
Propyzamide	0.02
Prothioconazole (Prothioconazole-desthio)	0.02
Prothiofos	0.01
Pyraclostrobin	0.02
Pyridaben	0.02
Pyrimethanil	0.02
Pyriproxyfen	0.02
Quinoxyfen	0.02
Spinosad (sum of spinosyn A and spinosyn D, expr. as spinosad)	0.02
Spirodiclofen	0.02
Spiroxamine	0.02
Tau-Fluvalinate	0.01
Tebuconazole	0.02
Tebufenozide	0.02
Tebufenpyrad	0.02
Teflubenzuron	0.02
Tefluthrin	0.02
Tetraconazole	0.02
Tetradifon	0.02
Thiabendazole	0.02
Thiacloprid	0.02
Thiamethoxam	0.02
Thiodicarb	0.02
Thiophanate-methyl	0.02
Tolclofos-methyl	0.02
Tolylfluanid	0.02
Triadimefon	0.02
Triadimenol	0.02
Triazophos	0.02
Trichlorfon (only parent compound)	0.02
Trifloxystrobin	0.02
Triflumuron	0.02
Trifluralin	0.02

ANNEX 1. Protocols and instructions. Target List of pesticides to be sought.

Pesticide	**MRRL** (mg/Kg)
Triticonazole	0.02
Vinclozolin (only parent compound)	0.02
Zoxamide	0.02

This list is based on Commission Regulation (EU) No 788/2012.

ANNEX 2. List of laboratories that agreed to participate in EUPT-T01.

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
Austria	Austrian Agency for Health and Food Safety (AGES), Institute for Food Safety, Department for Pesticide and Food Analytics (PLMA)	Innsbruck	YES
Belgium	Groen Agrocontrol	Delfgauw	YES
Belgium	WIV-ISP (IPH)	Bruxelles	YES
Belgium	Agro-Analyses	Metz	YES
Belgium	Fytolab	Zwijnaarde	YES
China	Key Laboratory of Food Safety Risk Assessment of Ministry of Health, China National Center for Food Safety Risk Assessment	Beijing	YES
Denmark	Danish Veterinary and Food Administration, Ringsted	Ringsted	YES
Egypt	Central Lab of Residue Analysis of Pesticides and Heavy Metals in Foods	Dokki, Giza	YES
Finland	Finnish Customs Laboratory	Espoo	YES
France	SCL-Laboratoire SCI Massy	Massy Cedex	YES
France	SCL Laboratoire de Montpellier	Montpellier	YES
France	CERECO SUD	Garons	YES
Germany	Chemical and Veterinary Analytical Institute Rhine-Ruhr-Wupper	Krefeld	YES
Germany	Institute for Hygiene and Environment	Hamburg	YES
Germany	Eurofins Dr. Specht Laboratorien GmbH	Hamburg	YES
Germany	CVUA Rheinland	Bonn	YES
Germany	Federal Insitute of Food Safety and Consumer Protection (BVL)	Berlin	YES
Germany	Landesuntersuchungsamt für Chemie, Hygiene und Veterinärmedizin Bremen	Bremen	YES
Germany	Bayerisches Landesamt fuer Gesundheit und Lebensmittelsicherheit	Erlangen	YES
Germany	Amt für Verbraucherschutz Düsseldorf - 39/2 Chemische und Lebensmitteluntersuchung	Düsseldorf	YES
Hungary	National Food Chain Safety Office DPPSCA Pesticide Residue Analytical Laboratory, Miskolc	Miskolc	YES
Hungary	National Food Chain Safety Office, DPPSCA Pesticide Analytical Laboratory, Velence	Velence	YES
Ireland	The Pesticide Control Laboratory	Celbridge	YES
Israel	Pesticide Residues Laboratory	Bet-Dagan	YES
Italy	Istituto Superiore Di Sanita' - Dip. Ampp - Reparto Antiparassitari	Rome	YES
Italy	ARPA Emilia Romagma, Area Fitofarmaci	Ferrara	YES
Italy	ARPA Puglia -Polo di specializzazione "Alimenti" - Bari	Bari	YES
Italy	ARPA Piemonte - Polo Alimenti	La Loggia (TO)	YES

ANNEX 2. List of laboratories that agreed to participate in EUPT-T01.

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
Italy	Laboratorio di Prevenzione ASL Milano	Milano	YES
Italy	ARPAL - Dipartimento La Spezia	La Spezia	YES
Italy	ARPA Trento	Trento	YES
Italy	ARPA Marche - Dip. Macerata	Macerata	YES
Norway	Bioforsk, Plant Health and Plant Protection, Department of Pesticide Chemistry	Aas	YES
Poland	Voivodship Sanitary-Epidemiological Station in Warsaw	Warsaw	YES
Romania	Sanitary Veterinary and Food Safety Directorate	Bucharest	YES
Saudi Arabia	National Center for Monitoring Food Contaminants	Riyadh	YES
Serbia	SP Laboratorija	Becej	YES
Slovakia	State Veterinary and Food Institiute	Bratislava	YES
Slovenia	IPH Maribor	Maribor	NO
Spain	Laboratorio Agroalimentario De Granada	Atarfe, Granada	YES
Spain	Laboratorio Agroalimentario de la Generalitat Valenciana	Burjassot	YES
Spain	Laboratorio De Produccion y Sanidad Vegetal Jaen. AGAPA	Mengibar, Jaén	YES
Spain	Laboratorio Agrario Regional de la Junta de Castilla y León	Burgos	YES
Spain	Laboratorio Agroambiental	Zaragoza	YES
Spain	Laboratorio Regional de la CC.AA. de La Rioja	Logroño	YES
Spain	Laboratorio Arbitral Agroalimentario	Madrid	YES
Spain	Laboratorio Agroalimentario de Extremadura	Cáceres	NO
Spain	CNTA	San Adrián	YES
Sweden	National Food Agency	Uppsala	YES
Switzerland	Kantonales Labor Zürich	Zürich	YES
Switzerland	Amt für Verbraucherschutz Aargau (Cantonal Office of Consumer Protection Aarau)	Aarau	YES
The Netherland	Laboratorium Zeeuws-Vlaanderen bv	Graauw	YES
The Netherland	NVWA	Wageningen	YES
Uruguay	Pharmacognosy & Natural Products	Montevideo	YES