

EURL-PROFICIENCY TEST-FV-17, 2015

Pesticide Residues in Broccoli Homogenate

Final Report

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CONTENTS

1. INTRODUCTION	6
2. TEST ITEMS	8
2.1 Preparation of the treated test item	8
2.2 Preparation of 'blank' test item	8
2.3 Homogeneity test	8
2.4 Stability tests	9
2.5 Distribution of test items and protocol to participants	11
3. STATISTICAL METHODS	12
3.1 False positives and negatives	12
3.2 Estimation of the assigned values	12
3.3 Fixed target standard deviations	13
3.4 z-Scores	13
3.5 Combined z-Scores	14
4. RESULTS	15
4.1 Summary of reported results	15
4.2 Assigned values and target standard deviations	17
4.3 Assessment of laboratory performance	17
5. CONCLUSIONS	24
6. SUGGESTIONS FOR FUTURE WORK	26
7. REFERENCES	27
8. ACKNOWLEDGEMENTS	28
APPENDIX 1. Homogeneity data.	29
APPENDIX 2. Histograms of residue data for each pesticide from all the laboratories.	31
APPENDIX 3. Results (mg/Kg) and z-scores for FFP RSD (25 %).	33
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).	37
APPENDIX 5. Average of the Squared z-scores (AZ^2) for laboratories in Category A.	49
APPENDIX 6. EUPT-FV-17– AZ^2 - Graphical representation for laboratories in Category A.	54
ANNEX 1. Protocols and Target list of pesticides to be sought.	55
ANNEX 2. List of laboratories that agreed to participate in EUPT-FV-17.	79

EURL-EUROPEAN UNION PROFICIENCY TEST 17
FOR THE DETERMINATION OF PESTICIDES IN FRUIT AND VEGETABLES USING
MULTIRESIDUE METHODS
2015

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 origin¹, 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 by other Member States, within the framework of the EU multi-annual coordinated control programme and national monitoring programmes.

Regulation (EC) No 882/2004² 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. European Proficiency Test 17 has been organised by the EURL in Fruit and Vegetables at the University of Almería, Spain⁴.

Participation in European Proficiency Test 17 was mandatory for all National Reference Laboratories (NRLs), as well as all other EU official laboratories, involved in the determination of pesticide residues in fruit and vegetables for the EU multi-annual coordinated control programme or for their own national monitoring programmes. Additionally, laboratories from Brazil, China, Egypt, Kenya, Peru, Saudi Arabia, Serbia, Singapore, Thailand, Turkey and Uruguay, who had been invited to take part in the previous tests, again participated. Laboratories from Costa Rica, Jamaica and Lebanon participated in this test for the first time.

DG-SANTE will have full access to all data from the EUPTs including the lab-code/lab-name key. The NRLs will also have that information for the OfLs within their network. This report may be presented to the European Union Standing Committee on Plants, Animals, Food and Feed (PAFF).

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

One hundred and eighty-five laboratories agreed to participate in European Union Proficiency Test 17.

The proficiency test was performed in 2015 using a broccoli homogenate. The broccoli plants were organically cultivated by the organisers in a greenhouse in Almería, Spain, and were treated before harvest using commercial formulations applied by spraying with conventional diffusors. Eleven pesticides were used for the treatment. Participating laboratories were also provided with a 'blank' broccoli homogenate as well as the treated broccoli test item.

The test items, 300 g of broccoli homogenate containing pesticide residues, together with 300 g of 'blank' broccoli homogenate, were shipped to participants on 16th March 2015. The deadline for results submission to the Organiser was 24th April 2015. The participants were provided with a list of one hundred and eighty-three target pesticide residues (Annex 1) and 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 to report the concentrations in mg/kg. This list of target pesticides also contained the Minimum Required Reporting Level (MRRL) for each pesticide fixed at 0.01 mg/kg, except for the following pesticides which have lower MRRLs based on Regulation (EU) No. 396/2005 and EU Directive 2006/125/EC: cadusafos (0.006 mg/kg); dimethoate and omethoate (0.003 mg/kg); ethoprophos (0.008 mg/kg); fipronil (0.004 mg/kg) along with oxydemeton-methyl and demeton-S-methylsulfone (0.006 mg/kg).

Participants were asked to analyse the blank test item and report results for any of the pesticides they found which were included in the target list. This 'blank' material was intended to be used for recovery experiments with the pesticides detected in the treated test item and, if necessary, for the preparation of matrix-matched calibration standard solutions.

The robust mean values of the analytical data submitted 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 the individual pesticides.

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 were able to detect at least 90% of the pesticides present in the test item and report no false positives have been 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 the overall accuracy of their results has not been assessed. They have been listed in order of the number of pesticides detected (of those used for the statistical evaluation) and the number of acceptable z-scores achieved.

Laboratories that did not report results have not been classified into any category and are listed in Annex 2 with the remainder of laboratories that participated in EUPT-FV-17.

2. TEST ITEMS

2.1 Preparation of the treated test item

The broccoli plants were organically cultivated by the organisers in a greenhouse in Almería, Spain, and were treated before harvest using commercial formulations applied by spraying with conventional diffusers.

Before preparation of the test item, the pesticides and target residue levels were selected, following recommendations made by the Quality Control Group (QCG), which had been appointed specifically for Proficiency Test 17. Approximately one hectare of broccoli plants was treated. All the pesticides were used as commercial pesticide formulations dissolved in water. Between seven and twenty one days after the application, a representative sample of the treated broccoli heads was taken from the plants and analysed to check if the residue levels present were close to the target levels or whether any additional spraying was necessary. When the residue levels in the broccolis were close to those recommended by the QCG, the entire production (120 kg) was harvested, frozen and processed using liquid nitrogen and a mincer. The frozen minced broccoli heads were mixed in a constantly-spinning container until a homogeneous material was obtained. 300 g portions of the well-mixed homogenate were weighed out into screw-capped polyethylene plastic bottles, sealed and stored in a freezer at about - 20 °C prior to distribution to participants.

2.2 Preparation of 'blank' test item

The broccoli plants used for the production of the blank test item were organically grown in the same greenhouse as the test item. Before the treatment of the broccoli plants, 125 kg were harvested in order to use in the preparation of the blank test items. A homogenate was prepared in the same way as the treated test item described previously.

2.3 Homogeneity test

The homogeneity and stability tests were subcontracted to the laboratory SICA AgriQ S.L., which is accredited under ISO/IEC 17025 by the Spanish accreditation body (ENAC). Ten bottles of the treated test item were randomly chosen from those stored in the freezer and analyses were performed on duplicate portions taken from each bottle. 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-MS/MS and LC-MS/MS was performed using calibration curves constructed from matrix-matched standards prepared from the 'blank' broccoli test item.

The statistical evaluation was performed according to the International Harmonized Protocol published by IUPAC, ISO and AOAC [1]. The individual residues 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 $c = F_1\sigma_{all}^2 + F_2s_{an}^2$; F_1 and F_2 being constant values of 1.88 and 1.01, respectively, from the ten samples taken, and $\sigma_{all}^2 = 0.3 \times \text{FFP RSD}(25\%) \times \text{the analytical sampling mean for all the pesticides}$. This was used to demonstrate that the between-bottle variance was not higher than the within-bottle variance.

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
Bupirimate	0.132	3.3×10^{-5}	2.2×10^{-4}	Pass
Carbendazim	0.492	2.5×10^{-4}	2.8×10^{-3}	Pass
Diazinon	0.114	0	3.1×10^{-2}	Pass
Difenoconazole	0.302	0	1.4×10^{-3}	Pass
Diflubenzuron	0.341	5.7×10^{-5}	1.3×10^{-3}	Pass
Methoxyfenozide	0.294	1.8×10^{-4}	4.3×10^{-3}	Pass
Pendimethalin	0.048	1.0×10^{-6}	3.2×10^{-5}	Pass
Permethrin	0.144	1.1×10^{-5}	2.5×10^{-4}	Pass
Spinosad (sum of spinosyn A and spinosyn D expr. as spinosad)	0.062	2.4×10^{-7}	4.3×10^{-5}	Pass
Thiabendazole	2.320	0	6.1×10^{-2}	Pass
Trifloxystrobin	0.556	0	3.4×10^{-3}	Pass

Ss : Between-Sampling Standard Deviation

As can be seen from Table 2.1, all the pesticides used to treat the broccoli matrix passed the homogeneity test.

2.4 Stability tests

The stability tests were also subcontracted to the laboratory SICA AgriQ S.L., which is accredited under ISO/IEC 17025 by the Spanish accreditation body (ENAC). The tests were performed on two occasions. On each occasion, a single bottle stored in the freezer at -20°C was chosen randomly and duplicate analyses were performed.

The two occasions were:

- Day 1: shortly before the test item shipment, which took place on March 16th 2015.
- Day 2: shortly after the deadline for reporting results, on April 24th 2015.

A pesticide is considered to be adequately stable if $|x_1 - y_i| \leq 0.3\sigma$, where x_1 is the mean value of the first stability test, y_i the mean value of the last stability test and σ the standard deviation used for proficiency assessment (typically 25% of the assigned value).

The individual results are given in Table 2.2. In general, these tests did not show any significant decrease in the pesticide concentrations with time. 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.

Moreover, regarding the stability of the sample arriving not completely frozen, a duplicate analysis of a bottle reproducing the delivery conditions that the samples experienced during 48 hours was performed. Laboratories could therefore be sufficiently confident in accepting the treated test item even if it was not completely frozen. Results for this 48 hours stability test are indicated in Table 2.3.

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

(mg/Kg)	Day 1 (Sample 041_A)	Day 1 (Sample 041_B)	Day 1 (Sample 092_A)	Day 1 (Sample 092_B)	Day 1 (Sample 119_A)	Day 1 (Sample 119_B)	Mean 1	Day 3 (Sample 021_A)	Day 3 (Sample 021_B)	Day 3 (Sample 098_A)	Day 3 (Sample 098_B)	Day 3 (Sample 160_A)	Day 3 (Sample 160_B)	Mean3	(M3 - M1)	M3-M1 ≤ 0.3*σ
Bupirimate	0.150	0.140	0.150	0.140	0.140	0.150	0.145	0.130	0.130	0.150	0.130	0.145	0.130	0.136	-0.009	Pass
Carbendazim	0.500	0.450	0.560	0.460	0.480	0.550	0.500	0.460	0.480	0.480	0.460	0.450	0.470	0.467	-0.033	Pass
Diazinon	0.059	0.058	0.060	0.052	0.053	0.059	0.057	0.051	0.051	0.057	0.053	0.049	0.051	0.052	-0.005	Pass
Difenocozone	0.320	0.300	0.310	0.380	0.370	0.400	0.347	0.310	0.320	0.350	0.360	0.330	0.330	0.338	-0.009	Pass
Diflubenzuron	0.340	0.320	0.350	0.350	0.340	0.350	0.342	0.330	0.330	0.320	0.310	0.320	0.320	0.322	-0.020	Pass
Methoxyfenozide	0.350	0.300	0.330	0.340	0.320	0.350	0.332	0.290	0.310	0.290	0.290	0.380	0.380	0.323	-0.008	Pass
Pendimethalin	0.053	0.049	0.050	0.046	0.045	0.049	0.049	0.045	0.044	0.048	0.043	0.044	0.042	0.044	-0.004	Pass
Permethrin	0.150	0.150	0.150	0.140	0.140	0.150	0.147	0.150	0.140	0.170	0.160	0.140	0.150	0.152	0.005	Pass
Spinosad	0.054	0.051	0.063	0.058	0.059	0.065	0.058	0.064	0.061	0.060	0.063	0.062	0.062	0.062	0.004	Pass
Thiabendazole	2.200	2.100	2.200	2.300	2.200	2.100	2.183	2.100	2.100	2.100	2.100	2.100	2.200	2.117	-0.067	Pass
Trifloxystrobin	0.510	0.510	0.540	0.520	0.520	0.520	0.520	0.550	0.550	0.530	0.530	0.530	0.570	0.543	0.023	Pass

Table 2.3 Statistical test for analytical precision and to demonstrate stability for the 48-hour time-elapse interval.

(mg/Kg)	Day 1 (Sample 041_A)	Day 1 (Sample 041_B)	Day 1 (Sample 092_A)	Day 1 (Sample 092_B)	Day 1 (Sample 119_A)	Day 1 (Sample 119_B)	Mean 1	Day 2 (Sample 107_A)	Day 2 (Sample 107_B)	Day 2 (Sample 154_A)	Day 2 (Sample 154_B)	Day 2 (Sample 155_A)	Day 2 (Sample 155_B)	Mean2	(M2 - M1)	M2-M1 ≤ 0.3*σ
Bupirimate	0.150	0.140	0.150	0.140	0.140	0.150	0.145	0.140	0.130	0.140	0.120	0.140	0.130	0.133	-0.012	Pass
Carbendazim	0.500	0.450	0.560	0.460	0.480	0.550	0.500	0.440	0.496	0.480	0.475	0.442	0.438	0.462	-0.038	Pass
Diazinon	0.059	0.058	0.060	0.052	0.053	0.059	0.057	0.053	0.056	0.053	0.057	0.053	0.050	0.054	-0.003	Pass
Difenocozone	0.320	0.300	0.310	0.380	0.370	0.400	0.347	0.340	0.370	0.350	0.380	0.420	0.390	0.375	0.028	Pass
Diflubenzuron	0.340	0.320	0.350	0.350	0.340	0.350	0.342	0.340	0.320	0.340	0.310	0.350	0.310	0.328	-0.013	Pass
Methoxyfenozide	0.350	0.300	0.330	0.340	0.320	0.350	0.332	0.360	0.360	0.350	0.340	0.360	0.370	0.357	0.025	Pass

(mg/Kg)	Day 1 (Sample 041_A)	Day 1 (Sample 041_B)	Day 1 (Sample 092_A)	Day 1 (Sample 092_B)	Day 1 (Sample 119_A)	Day 1 (Sample 119_B)	Mean 1	Day 2 (Sample 107_A)	Day 2 (Sample 107_B)	Day 2 (Sample 154_A)	Day 2 (Sample 154_B)	Day 2 (Sample 155_A)	Day 2 (Sample 155_B)	Mean2	(M2 - M1)	M2-M1 ≤ 0.3*σ
Pendimethalin	0.053	0.049	0.050	0.046	0.045	0.049	0.049	0.047	0.048	0.049	0.042	0.048	0.049	0.047	-0.001	Pass
Permethrin	0.150	0.150	0.150	0.140	0.140	0.150	0.147	0.140	0.142	0.120	0.130	0.140	0.130	0.134	-0.013	Pass
Spinosad	0.054	0.051	0.063	0.058	0.059	0.065	0.058	0.051	0.064	0.057	0.051	0.055	0.050	0.055	-0.004	Pass
Thiabendazole	2.200	2.100	2.200	2.300	2.200	2.100	2.183	2.100	2.100	2.000	2.100	2.200	2.000	2.083	-0.100	Pass
Trifloxystrobin	0.510	0.510	0.540	0.520	0.520	0.520	0.520	0.480	0.540	0.530	0.450	0.480	0.500	0.497	-0.023	Pass

2.5 Distribution of test items and protocol to participants

One bottle of frozen treated test items and one bottle of frozen 'blank' material were shipped to each participant in boxes containing dry ice. The test items were sent out on 16th March 2015.

Before sample shipment, the laboratories received full instructions (Annex 1) for the receipt and storage of the test items and 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 also available as an on-line form. When applying to participate in the test, each laboratory decided on their own password, which was required in order to enter the restricted zone where Forms 0-5 could be accessed on-line. This information was made available when laboratories received an e-mail from the Organiser confirming their acceptance along with their Lab Code and thus allowing them to participate. This ensured that confidentiality was maintained throughout the duration of Proficiency Test 17. The Target Pesticide List and the Minimum Required Reporting Levels (MRRLs), as established by the Organiser, were uploaded onto the EURL-FV open website to allow laboratories sufficient time to purchase standards and to validate their methods.

3. STATISTICAL METHODS

3.1 False positives and negatives

3.1.1 False positives

These are results of pesticides from the Target Pesticides List, that are reported, at or above, their respective MRRLs although they 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 pesticides. In certain instances, case-by-case decisions by the EUPT-Panel may be necessary.

Any results reported lower than the MRRL will not be considered as false positives, even though these results should not have been reported.

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 pesticides reported by the laboratories as 'analysed' but without reporting numerical values although they were: a) used by the Organiser to treat the Test Item and b) detected by the Organiser as well as the majority of the participants that had targeted these specific pesticides at or above the respective MRRLs. Results reported as '< RL' (RL= Reporting Limit of the laboratory) will be considered as not detected and will be judged as false negatives. 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 typically not be assigned. The EUPT-Panel may decide to take case-by-case decisions in this respect after considering all relevant factors such as the result distribution and the reporting limits of the affected labs. z-Scores have been calculated for all pesticides detected and reported at levels at, or above, the MRRL. They have also been calculated for 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 (x^*)

In order to minimise the influence of out-lying results on the statistical evaluation, the assigned value (= consensus concentration) was estimated using robust statistics as described in ISO 13528:2009-01 10[2], taking into account the results reported by EU and EFTA countries laboratories only. 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. In special justifiable cases, the EUPT-Panel may decide to eliminate certain results traceably associated with gross errors or to use only the results of a subgroup consisting of laboratories that have repeatedly demonstrated good performance for the specific compound in the past.

Taking into account the normative for robust analysis in ISO 13528 [2], the uncertainty was accompanying the assigned value for each pesticide was calculated according to the following equation:

$$x_i = 1.25 \frac{s^*}{\sqrt{n}}$$

Where:

- x_i is the uncertainty in mg/Kg.
- s^* is the robust standard deviation.
- n is the total number of results.

3.3 Fixed target standard deviations

Based on the experience gained from previous EU proficiency tests and recommendations from the EURL Advisory Group, a fixed relative standard deviation (FFP-RSD) of 25 % was chosen [3]. This is in line with the internationally-accepted target Measurement Uncertainty of 50 % for multiresidue analysis of pesticides [4], which is derived from, and linked to, the EUPTs. The same target RSD has been applied to all the pesticides, independent of concentration. For informative purposes the robust relative standard deviation (CVs*) is calculated according to ISO 13528:2009-01; Chapter 5.6 (Consensus value from participants) following Algorithm A in Annex C, and it can be compared to the FFP-RSD in Table 4.4.

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, or the MRRL or the reporting level (RL) (whichever one is lower) for those labs that have not 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 2.0$	Acceptable
$2.0 < z < 3.0$	Questionable
$ z \geq 3.0$	Unacceptable

- Any z-score values of $|z| > 5$ have been reported as '>5' and a value of '5' has been used to calculate combined z-scores.
- No z-score calculations have been performed for false positive results.
- 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 correctly identify and report quantitative results (that is *sought and detected*) for 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) [5].

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-FV-12. The AZ^2 is calculated as follows:

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

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'.

$$\begin{aligned} |AZ^2| \leq 2.0 & \quad \text{Good} \\ 2.0 < |AZ^2| < 3.0 & \quad \text{Satisfactory} \\ |AZ^2| \geq 3.0 & \quad \text{Unsatisfactory} \end{aligned}$$

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.

Laboratories that did not detect and quantify 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

One hundred and eighty-five laboratories agreed to participate in this proficiency test. One hundred and eighty submitted results, three did not submit results and two cancelled their participation. The results reported by all the laboratories are presented in this report. However, only results reported by laboratories from EU-countries and EFTA-countries (Iceland, Norway, and Switzerland) have been included in the statistical treatment. The results from the laboratories in Brazil, China, Costa Rica, Egypt, Jamaica, Kenya, Peru, Saudi Arabia, Serbia, Singapore, Thailand, Turkey and Uruguay and have not been included. This last group totals fifteen laboratories that reported results.

Eleven pesticides were used to treat the sample, and all of them met the necessary statistical criteria. Therefore those eleven were used in the performance evaluation in this report. A summary of the reported results for the pesticides evaluated 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 165)
Bupirimate	154	0	11	93
Carbendazim	140	0	25	85
Diazinon	160	2	3	97
Difenoconazole	150	2	13	91
Diflubenzuron	111	2	52	67
Methoxyfenozide	128	0	37	78
Pendimethalin	152	1	12	92
Permethrin	152	1	12	92
Spinosad (sum of spinosyn A and spinosyn D expressed as spinosad)	125	2	38	76
Thiabendazole	143	2	20	87
Trifloxystrobin	152	0	13	92

* The % of Reported Results comes from 165 laboratories. It does not take into account the fifteen laboratories from Brazil, China, Costa Rica, Egypt, Jamaica, Kenya, Peru, Saudi Arabia, Serbia, Singapore, Thailand, Turkey and Uruguay.

The laboratories that agreed to participate are listed in Annex 2. All results reported by the participants are given in Appendix 3, whilst the analytical methods used are given in Appendix 7 (available in the EURL-FV web page in electronic format).

4.1.1 False positives

Six laboratories from EU and/or EFTA-countries reported results for additional pesticides that had not been used to treat the test item. These pesticides and the residue levels reported are presented in Table 4.2 together with the MRRs and RLs. Where the reported concentrations of the erroneously-detected pesticide were higher than the assigned MRRL value in the Target Pesticide List (Annex 1), the result has been considered as a false positive.

Three out of those six laboratories reporting a false positive result have not been classified into Category A despite achieving sufficient scope.

Table 4.2 Laboratories that reported as quantitative results for pesticides that were not present in the treated test item

Laboratory Code	Pesticide	Concentration (mg/kg)	Determination Technique	RL (mg/kg)	MRRL (mg/kg)
Lab027	Bromopropylate	0.014	GC-MS/MS	0.01	0.01
Lab098	Bromopropylate	0.021	GC-MS	0.01	0.01
Lab051	Bromopropylate	0.039	GC-MS	0.01	0.01
Lab173	Carbaryl	0.020	GC-MS/MS	0.01	0.01
Lab158	Fenprothrin	0.020	GC-MS	0.01	0.01
Lab164	Kresoxim-methyl	0.015	GC-MS/MS	0.01	0.01

False positives from Brazil, China, Costa Rica, Egypt, Jamaica, Kenya, Peru, Saudi Arabia, Serbia, Singapore, Thailand, Turkey and Uruguay (if any) have not been included in this table.

If the concentrations reported were below the MRRs, or if the pesticides did not appear in the pesticide 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 presented as 'Not Detected' (ND).

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

Laboratory Code	Bupirimate	Carbendazim	Diazinon	Difenoconazole	Diffubenzuron	Methoxyfenozide	Pendimethalin	Permethrin	Spinosad	Thiabendazole	Trifloxystrobin
Lab019					ND			ND			
Lab054									ND		
Lab059				ND							
Lab110			ND							ND	
Lab128									ND		
Lab163										ND	
Lab164				ND							
Lab169			ND				ND				
Lab173					ND						

False negatives from from Brazil, China, Costa Rica, Egypt, Jamaica, Kenya, Peru, Saudi Arabia, Serbia, Singapore, Thailand, Turkey and Uruguay (if any) have not been included in this table.

4.1.3 Distribution of data

The distribution of the concentrations of the pesticides reported by the laboratories has been plotted as histograms with a bandwidth of $0.75 \cdot \sigma$ (σ is the target standard deviation (the FFP-RSD of 25 % multiplied by the assigned value) after removing outliers. The histograms of the eleven pesticides present in the test item are presented in Appendix 2.

4.2 Assigned values and target standard deviations

The assigned values were based on the robust mean values calculated using all the results reported by laboratories from EU and EFTA countries. The assigned values for the eleven pesticides and their uncertainties 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 (CV*) was also calculated for informative purposes, employing also this value for the calculation of the uncertainty. These RSDs can be seen in Table 4.4.

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

Pesticides	MRRL (mg/kg)	Robust mean (mg/kg)	Uncertainty (mg/kg)	FFP-RSD (%)	CV* (%)
Bupirimate	0.010	0.16	0.006	25	18.2
Carbendazim	0.010	0.51	0.023	25	21.1
Diazinon	0.010	0.071	0.003	25	18.5
Difenoconazole	0.010	0.53	0.016	25	15.1
Diflubenzuron	0.010	0.32	0.019	25	25.6
Methoxyfenozide	0.010	0.35	0.014	25	17.7
Pendimethalin	0.010	0.062	0.003	25	21.6
Permethrin	0.010	0.17	0.007	25	21.4
Spinosad (sum of spinosyn A and spinosyn D expressed as spinosad)	0.010	0.051	0.003	25	24.0
Thiabendazole	0.010	1.90	0.071	25	18.0
Trifloxystrobin	0.010	0.47	0.016	25	17.5

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 assigned values for each pesticide. The z-scores of laboratories from Brazil, China, Costa Rica, Egypt, Jamaica, Kenya, Peru, Saudi Arabia, Serbia, Singapore, Thailand, Turkey 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 (%)
Bupirimate	93.5	1.9	4.5
Carbendazim	86.4	5.7	7.9
Diazinon	92.6	4.3	3.1
Difenoconazole	97.4	1.3	1.3
Diflubenzuron	84.1	8.0	8.0
Methoxyfenozide	96.1	3.1	0.8
Pendimethalin	92.2	5.9	2.0
Permethrin	93.5	2.6	3.9
Spinosad (sum of spinosyn A and spinosyn D expressed as spinosad)	90.6	1.6	7.9
Thiabendazole	93.1	1.4	5.5
Trifloxystrobin	96.7	1.3	2.0

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 used for each particular pesticide.

4.3.2 Combined z-Scores

As previously mentioned in Section 3.5, the AZ^2 formula has only been applied to those participants categorised into Category A.

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 EU and EFTA laboratories in Category A. In this category are the laboratories that sought and detected ten 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.

One hundred thirteen of the one hundred and sixty-five EU and EFTA laboratories that submitted results have been classified into Category A (68.5 %).

From the AZ², eighty percent were classed as 'good', seven percent as 'satisfactory' and thirteen percent as 'unsatisfactory'.

Of the fifty-two laboratories in Category B, three would have been in Category A if they had not reported a false positive result.

Table 4.6 shows the laboratories in Category A, the number of pesticides reported, the AZ² values and their subclassifications. Laboratories that reported false negative results in Category A are marked with an asterisk.

Table 4.7 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 AZ² graphical representation 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.

Laboratory performance over the last three EUPTs using the AZ² formula has been summarized as follows:

- For EUPT-FV-17, out of 165 laboratories (EU and EFTA), 113 were in Category A with the following classes: 15 'unsatisfactory', 8 'satisfactory' and 90 'good'.
- For EUPT-FV-16, out of 169 laboratories (EU and EFTA), 100 were in Category A with the following classes: 2 'unsatisfactory', 5 'satisfactory' and 93 'good'.
- For EUPT-FV-15, out of 160 laboratories (EU and EFTA), 87 were in Category A with the following classes: 1 'unsatisfactory', 6 'satisfactory' and 80 'good'.
- For EUPT-FV-14, out of 151 laboratories (EU and EFTA), 83 were in Category A with the following classes: 5 'unsatisfactory', 2 'satisfactory' and 76 'good'.

Table 4.6 Performance and Classification of laboratories in Category A using the AZ² formula

Lab Code	No. of pesticides detected	AZ ²	Classification
Lab010	11	0.1	Good
Lab013	11	0.1	Good
Lab072	11	0.1	Good
Lab078	11	0.1	Good
Lab084	11	0.1	Good
Lab086	11	0.1	Good
Lab097	11	0.1	Good
Lab100	11	0.1	Good
Lab108	11	0.1	Good

Lab Code	No. of pesticides detected	AZ ²	Classification
Lab137	11	0.1	Good
Lab147	11	0.1	Good
Lab159	11	0.1	Good
Lab171	11	0.1	Good
Lab014	11	0.2	Good
Lab024	11	0.2	Good
Lab035	11	0.2	Good
Lab046	11	0.2	Good
Lab048	11	0.2	Good
Lab062	11	0.2	Good
Lab067	11	0.2	Good
Lab069	11	0.2	Good
Lab080	11	0.2	Good
Lab083	11	0.2	Good
Lab102	11	0.2	Good
Lab135	11	0.2	Good
Lab144	11	0.2	Good
Lab012	11	0.3	Good
Lab029	11	0.3	Good
Lab030	11	0.3	Good
Lab032	11	0.3	Good
Lab047	11	0.3	Good
Lab056	10	0.3	Good
Lab064	11	0.3	Good
Lab073	11	0.3	Good
Lab101	10	0.3	Good
Lab109	11	0.3	Good
Lab121	10	0.3	Good
Lab160	11	0.3	Good
Lab179	11	0.3	Good
Lab009	11	0.4	Good
Lab049	11	0.4	Good
Lab053	11	0.4	Good
Lab082	11	0.4	Good
Lab126	11	0.4	Good
Lab143	11	0.4	Good
Lab161	11	0.4	Good
Lab018	11	0.5	Good
Lab040	11	0.5	Good
Lab087	11	0.5	Good
Lab105	11	0.5	Good
Lab167	11	0.5	Good

Lab Code	No. of pesticides detected	AZ ²	Classification
Lab168	11	0.5	Good
Lab176	11	0.5	Good
Lab022	11	0.6	Good
Lab041	11	0.6	Good
Lab077	11	0.6	Good
Lab089	11	0.6	Good
Lab111	11	0.6	Good
Lab120	11	0.6	Good
Lab031	10	0.7	Good
Lab037	11	0.7	Good
Lab060	11	0.7	Good
Lab068	11	0.7	Good
Lab132	11	0.7	Good
Lab162	11	0.7	Good
Lab005	11	0.8	Good
Lab015	11	0.8	Good
Lab026	11	0.8	Good
Lab034	10	0.8	Good
Lab070	11	0.8	Good
Lab079	11	0.8	Good
Lab088	11	0.8	Good
Lab093	11	0.9	Good
Lab114	10	0.9	Good
Lab123	11	0.9	Good
Lab124	11	0.9	Good
Lab185	11	0.9	Good
Lab113	10	1.1	Good
Lab183	11	1.1	Good
Lab156	11	1.3	Good
Lab038	11	1.4	Good
Lab095	11	1.4	Good
Lab128*	11	1.4	Good
Lab061	11	1.5	Good
Lab148	10	1.5	Good
Lab044	11	1.7	Good
Lab182	11	1.7	Good
Lab057	11	1.8	Good
Lab116	11	1.9	Good
Lab181	11	2.0	Good
Lab011	11	2.1	Satisfactory
Lab017	10	2.1	Satisfactory
Lab119	10	2.2	Satisfactory

Lab Code	No. of pesticides detected	AZ ²	Classification
Lab071	10	2.3	Satisfactory
Lab058	11	2.5	Satisfactory
Lab076	11	2.5	Satisfactory
Lab007	10	2.7	Satisfactory
Lab107	10	2.7	Satisfactory
Lab025	11	3.0	Unsatisfactory
Lab050	10	3.0	Unsatisfactory
Lab091	11	3.0	Unsatisfactory
Lab103	11	3.0	Unsatisfactory
Lab085	10	3.1	Unsatisfactory
Lab042	11	3.3	Unsatisfactory
Lab094	11	3.4	Unsatisfactory
Lab074	11	3.5	Unsatisfactory
Lab036	11	3.6	Unsatisfactory
Lab136	10	3.9	Unsatisfactory
Lab021	11	4.2	Unsatisfactory
Lab054*	11	4.9	Unsatisfactory
Lab112	11	5	Unsatisfactory
Lab043	11	>5	Unsatisfactory
Lab142	11	>5	Unsatisfactory

* Laboratories reporting a false negative result.

Table 4.7 Performance of laboratories in Category B

Lab Code	% No. of pesticides detected / No. of pesticides evaluated (11)	No. of pesticides detected	No. of total z-scores	No. of acceptable z-scores (z- score ≤ 2)
Lab027+	100	11	11	11
Lab098+	100	11	11	11
Lab158+	91	10	10	1
Lab003	82	9	9	9
Lab019*	82	9	11	8
Lab045	82	9	9	7
Lab052	82	9	9	8
Lab092	82	9	9	8
Lab106	82	9	9	9
Lab133	82	9	9	9
Lab141	82	9	9	7
Lab145	82	9	9	7
Lab169*	82	9	11	9
Lab174	82	9	9	8
Lab023	73	8	8	6

Lab Code	% No. of pesticides detected / No. of pesticides evaluated (11)	No. of pesticides detected	No. of total z-scores	No. of acceptable z-scores (z- score ≤ 2)
Lab065	73	8	8	7
Lab099	73	8	8	8
Lab110*	73	8	10	7
Lab117	73	8	8	8
Lab138	73	8	8	8
Lab001	64	7	7	7
Lab016	64	7	7	5
Lab020	64	7	7	6
Lab127	64	7	7	4
Lab140	64	7	7	7
Lab151	64	7	7	6
Lab154	64	7	7	7
Lab157	64	7	7	7
Lab166	64	7	7	4
Lab002	55	6	6	6
Lab006	55	6	6	6
Lab081	55	6	6	6
Lab115	55	6	6	5
Lab134	55	6	6	6
Lab170	55	6	6	6
Lab075	45	5	5	5
Lab130	45	5	5	5
Lab131	45	5	5	5
Lab146	45	5	5	5
Lab152	45	5	5	5
Lab163*	45	5	6	4
Lab173*	45	5	6	3
Lab149	36	4	4	4
Lab164	36	4	4	3
Lab039	27	3	3	3
Lab051	27	3	3	3
Lab059*	27	3	4	2
Lab129	27	3	3	3
Lab139	27	3	3	3
Lab150	27	3	3	0
Lab008	18	2	2	2
Lab118	18	2	2	2

* Laboratories reporting a false negative result.

+ Laboratories reporting a false positive result.

5. CONCLUSIONS

One hundred and eighty-five laboratories agreed to participate in EUPT-FV-17. All but three of them submitted results for the analysis of the broccoli homogenate test item and another two cancelled their participation. Fifteen of those submitting results were not from EU or EFTA countries, so no statistical analysis was conducted on their results.

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, either gas or liquid, are shown in the z-score charts. 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.

Average of Squared z-Scores formula was used for the overall evaluation of the participant laboratories. Laboratories reporting ten or more results and no false positives 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.

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

The robust mean for each pesticide was used as the assigned value or "true" concentration, which was also used to calculate the z-scores.

Overall, the results were very good with regard to the individual z-scores for each pesticide present in the test item. Most of the pesticides had only a few unacceptable z-scores. Therefore, laboratories generally achieved accurate results for all the pesticides present in the test item – at, or above, 84 %.

Moreover, the percentage of laboratories in Category A (68.5 %) has increased slightly compared to last year's (59 %).

Participation in this year's European Proficiency Test 17 involved at least one laboratory from each Member State. Additionally, laboratories from Iceland, Norway and Switzerland participated as EFTA countries. Non-European laboratories in Brazil, China, Egypt, Kenya, Peru, Saudi Arabia, Serbia, Singapore, Thailand, Turkey and Uruguay also participated (as in previous years) although this year, they were joined by Costa Rica, Jamaica and Lebanon for the first

time. These Non-EU laboratories, however, are official laboratories in their own countries. As laid down in Article 32 of Regulation (EC) N° 882/2004, one of the EURL's duties is to collaborate with non EU laboratories that are responsible for analysing food and feed samples and to help them improve the quality of their analyses.

6. SUGGESTIONS FOR FUTURE WORK

The use of incurred pesticides in the test item will continue in next EUPT-FV-18 where ever commercial formulations are available. The number and concentrations of pesticides that will be present in the treated test item will be in line with the previous years, with the intention that they do not have an unacceptable impact on the workflow of participating laboratories.

As a result of the continuing trend in performance improvement, the stricter criteria applied to EUPT-FV-17 will be carried forward to the PT next year. The aim is that laboratories continue to increase the scope of their methods so that they are able to fully enforce EU legislation.

All the MRM amenable pesticides included in the working document SANCO/12745/2013 (Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin) will be added to the pesticide target list as voluntary pesticides, so it will be possible to have an overview about the extent of the scope of the NRLs and Ofls as regards pesticides not included in Annex I of the EU multiannual coordinated control programme [6].

For future EUPTs-FV, the ADVG will evaluate different ways of categorisation into Category A or B. Participants in Category A will be those laboratories that are able to analyse at least 90% of the pesticides in the target list, that are able to detect at least 90% of the pesticides present in the test material and that report no false positives. Within Category A, the laboratories will be sub-classified as "good", "satisfactory" or "unsatisfactory". All the other laboratories will be classified in Category B.

These changes are aimed at ensuring that, year on year, laboratories continue to strive to increase the scope of their methods, improve their performance (both in terms of correctly identifying the pesticides present in the test item, and also accurately quantify the concentrations present). It is recommended that laboratories should continue to evaluate and adopt new techniques/instrumentation that will help them to attain, or maintain, a Category A classification.

7. REFERENCES

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8. ACKNOWLEDGEMENTS

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APPENDIX 1. Homogeneity data.

Bupirimate (mg/kg)		Carbendazim (mg/kg)		Diazinon (mg/kg)		Difenoconazole (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.130	0.130	0.500	0.490	0.057	0.058	0.290	0.290
0.130	0.130	0.510	0.450	0.058	0.061	0.280	0.320
0.120	0.130	0.510	0.490	0.058	0.061	0.300	0.310
0.130	0.140	0.480	0.490	0.056	0.062	0.290	0.350
0.120	0.130	0.480	0.480	0.056	0.057	0.260	0.290
0.120	0.140	0.500	0.500	0.057	0.062	0.290	0.320
0.130	0.130	0.490	0.480	0.056	0.057	0.290	0.280
0.130	0.130	0.480	0.490	0.570	0.058	0.300	0.320
0.130	0.130	0.510	0.490	0.059	0.058	0.290	0.300
0.150	0.150	0.510	0.500	0.065	0.650	0.340	0.330

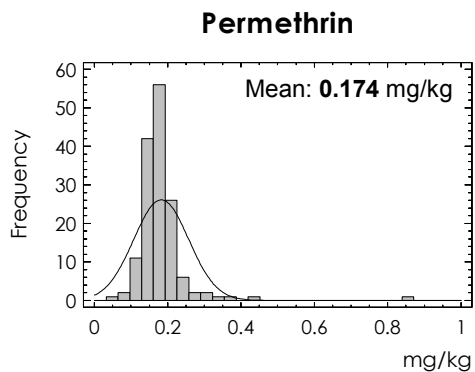
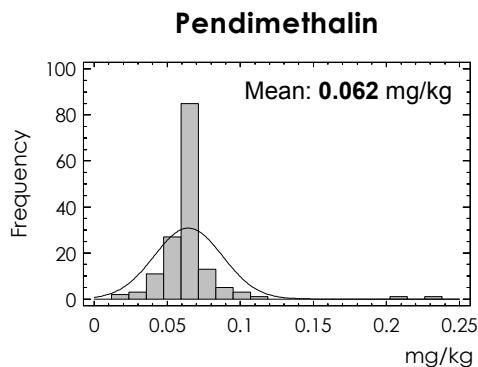
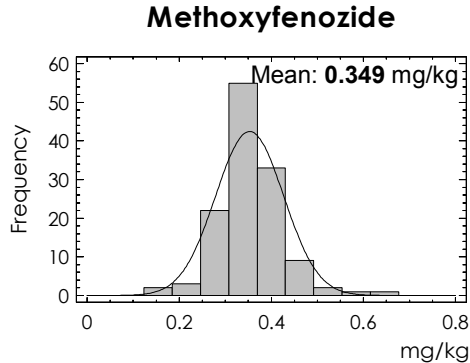
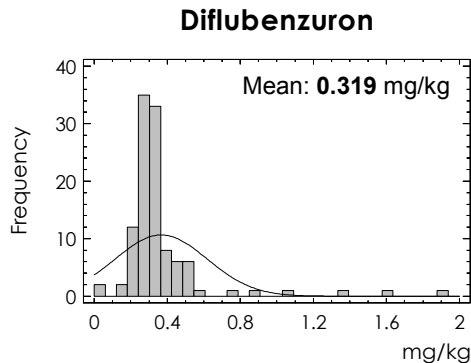
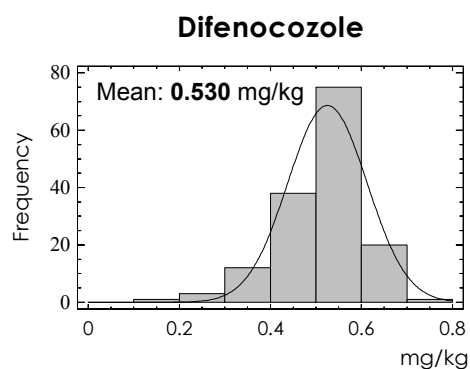
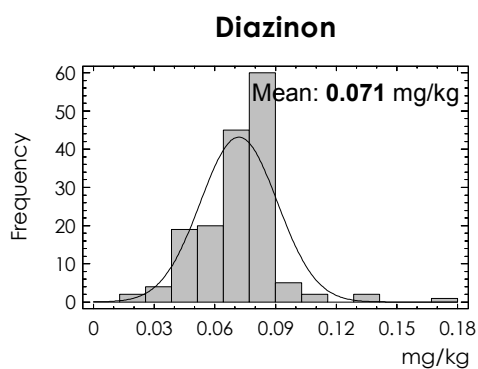
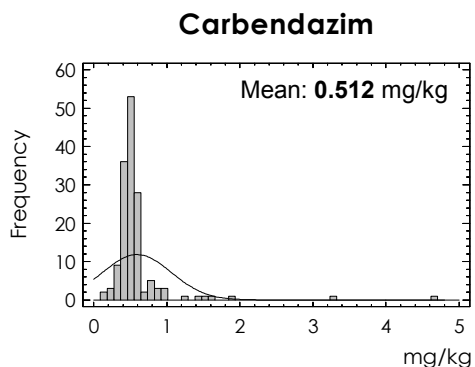
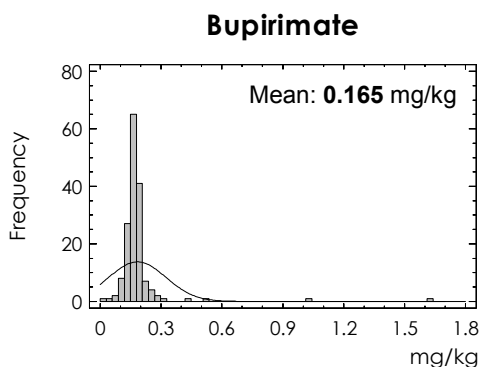
Diflubenzuron (mg/kg)		Methoxyfenozide (mg/kg)		Pendimetalin (mg/kg)		Permethrin (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.360	0.350	0.310	0.300	0.046	0.047	0.140	0.140
0.360	0.350	0.310	0.310	0.046	0.053	0.140	0.150
0.350	0.340	0.310	0.310	0.046	0.051	0.140	0.150
0.330	0.330	0.330	0.300	0.045	0.052	0.140	0.150
0.340	0.330	0.300	0.300	0.045	0.047	0.130	0.140
0.340	0.350	0.310	0.310	0.046	0.050	0.140	0.150
0.330	0.330	0.300	0.044	0.045	0.044	0.140	0.140
0.340	0.330	0.310	0.300	0.047	0.050	0.140	0.150
0.340	0.340	0.300	0.300	0.047	0.046	0.140	0.140
0.350	0.330	0.320	0.300	0.053	0.052	0.160	0.150

Spinosad (mg/kg)		Thiabendazole (mg/kg)		Trifloxystrobin (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.063	0.060	2.300	2.300	0.560	0.550
0.063	0.063	2.400	2.300	0.590	0.540
0.062	0.060	2.300	2.300	0.560	0.560
0.061	0.063	2.300	2.400	0.560	0.550
0.060	0.060	2.300	2.300	0.560	0.550
0.060	0.063	2.400	2.400	0.550	0.550
0.062	0.064	2.400	2.200	0.560	0.550
0.061	0.062	2.300	2.200	0.550	0.550
0.063	0.063	2.400	2.300	0.550	0.550
0.066	0.062	2.300	2.300	0.570	0.550

The sample numbers used for this test were: 6, 16, 35, 50, 66, 78, 97, 111, 204 and 261.

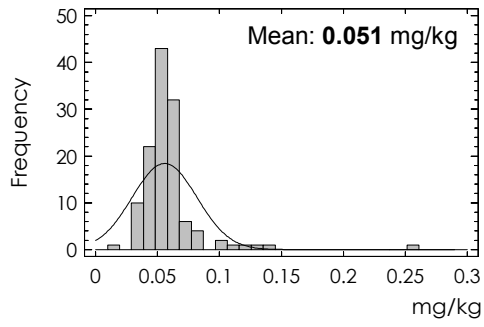
APPENDIX 2. Histograms of residue data for each pesticide from all the laboratories.

Results presented as histograms.

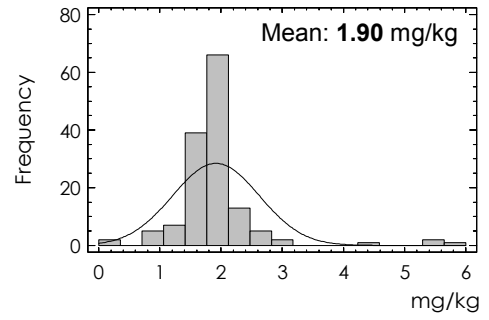


APPENDIX 2. Histograms of residue data for each pesticide from all the laboratories.

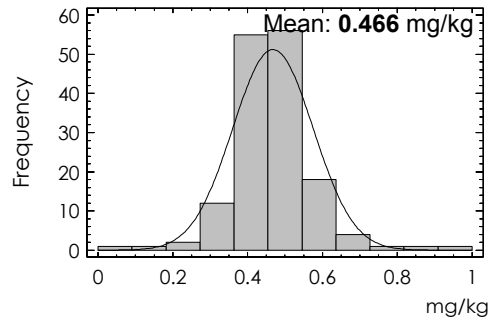
Spinosad



Thiabendazole



Trifloxystrobin



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

Results given by the laboratories for bupirimate, carbendazim, diazinon, difenoconazole, diflubenzuron, methoxyfenozide, pendimethalin, permethrin, spinosad, thiabendazole and trifloxystrobin (mg/kg) and their calculated z-score value using FFP RSD 25 %

Lab Code	Bupirimate	Carbendazim		Diazinon		Difenoconazole		Diflubenzuron		Methoxyfenozide		Pendimethalin		Permethrin		Spinosad		Thiabendazole		Trifloxystrobin		
MRRL	0.01	z-Score (FFP RSD 25%)		z-Score (FFP RSD 25%)		z-Score (FFP RSD 25%)		z-Score (FFP RSD 25%)		z-Score (FFP RSD 25%)		z-Score (FFP RSD 25%)		z-Score (FFP RSD 25%)		z-Score (FFP RSD 25%)		z-Score (FFP RSD 25%)		z-Score (FFP RSD 25%)		
Robust mean (mg/kg)	0.16	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	
		0.51	0.51	0.071	0.071	0.53	0.53	0.32	0.32	0.35	0.35	0.062	0.062	0.17	0.17	0.051	0.051	1.9	1.9	0.47	0.47	
Lab001	0.211	1.1	NA	NA	0.062	-0.5	0.4	-1	NA	NA	NA	NA	0.072	0.6	0.173	0	NA	NA	1.58	-0.7	0.412	-0.5
Lab002	0.158	-0.2	NA	NA	0.071	0	0.606	0.6	NA	NA	NA	NA	0.071	0.5	0.178	0.1	NA	NA	NA	NA	0.483	0.1
Lab003	0.22	1.3	0.36	-1.2	0.079	0.5	0.58	0.4	NA	NA	NA	NA	0.04	-1.4	0.22	1.1	0.056	0.4	1	-1.9	0.47	0
Lab004	0.15	-0.4	0.462	-0.4	0.07	-0.1	0.46	-0.5	ND	-3.9	0.354	0.1	0.05	-0.8	0.175	0	0.044	-0.6	1.24	-1.4	0.508	0.4
Lab005	0.11	-1.3	0.572	0.5	0.047	-1.4	0.422	-0.8	0.279	-0.5	0.307	-0.5	0.047	-1	0.155	-0.4	0.033	-1.4	1.94	0.1	0.4	-0.6
Lab006	0.174	0.2	0.537	0.2	0.096	1.4	0.751	1.7	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	1.953	0.1	0.615	1.3
Lab007	0.22	1.3	0.484	-0.2	0.093	1.2	0.64	0.8	NA	NA	0.429	0.9	0.083	1.3	0.358	4.2	0.053	0.1	2.061	0.3	0.636	1.5
Lab008	NA	NA	NA	NA	0.053	-1	NA	NA	NA	NA	NA	NA	NA	NA	0.104	-1.6	NA	NA	NA	NA	NA	NA
Lab009	0.137	-0.7	0.427	-0.7	0.06	-0.6	0.403	-1	0.315	-0.1	0.314	-0.4	0.054	-0.6	0.138	-0.8	0.045	-0.5	1.766	-0.3	0.38	-0.7
Lab010	0.198	0.8	0.532	0.2	0.077	0.3	0.574	0.3	0.333	0.2	0.362	0.1	0.067	0.3	0.169	-0.1	0.054	0.2	1.887	0	0.517	0.4
Lab011	0.11	-1.3	0.41	-0.8	0.03	-2.3	0.27	-2	0.36	0.5	0.34	-0.1	0.03	-2.1	0.09	-1.9	0.04	-0.9	2.18	0.6	0.3	-1.4
Lab012	0.138	-0.7	0.421	-0.7	0.063	-0.5	0.478	-0.4	0.276	-0.5	0.399	0.6	0.061	-0.1	0.151	-0.5	0.064	1	1.66	-0.5	0.406	-0.5
Lab013	0.17	0.1	0.45	-0.5	0.066	-0.3	0.53	0	0.27	-0.6	0.34	-0.1	0.063	0	0.18	0.1	0.048	-0.2	2.05	0.3	0.45	-0.1
Lab014	0.173	0.2	0.431	-0.6	0.071	0	0.529	0	0.389	0.9	0.341	-0.1	0.069	0.4	0.212	0.9	0.056	0.4	1.953	0.1	0.485	0.2
Lab015	0.173	0.2	0.334	-1.4	0.054	-1	0.507	-0.2	0.265	-0.7	0.459	1.3	0.04	-1.4	0.165	-0.2	0.041	-0.8	2.04	0.3	0.356	-0.9
Lab016	0.183	0.4	NA	NA	0.086	0.8	0.667	1	NA	NA	NA	NA	0.206	5	0.446	5	NA	NA	2.19	0.6	0.551	0.7
Lab017	0.038	-3.1	0.301	-1.6	0.024	-2.6	0.541	0.1	NA	NA	0.335	-0.2	0.062	0	0.131	-1	0.047	-0.3	1.61	-0.6	0.442	-0.2
Lab018	0.16	-0.1	0.49	-0.2	0.076	0.3	0.61	0.6	0.35	0.4	0.38	0.4	0.066	0.2	0.18	0.1	0.078	2.1	1.8	-0.2	0.49	0.2
Lab019	0.12	-1.1	0.42	-0.7	0.06	-0.6	0.41	-0.9	ND	-3.9	0.26	-1	0.05	-0.8	ND	-3.8	0.04	-0.9	5.5	5	0.26	-1.8
Lab020	1.016	5	0.533	0.2	0.079	0.5	0.576	0.3	0.259	-0.8	0.345	0	NA	NA	NA	NA	NA	NA	1.862	-0.1	NA	NA
Lab021	0.175	0.2	0.78	2.1	0.07	-0.1	0.525	0	0.29	-0.4	0.67	3.7	0.054	-0.5	0.148	-0.6	0.077	2	2.79	1.9	0.98	4.4
Lab022	0.168	0.1	0.745	1.8	0.066	-0.3	0.55	0.2	0.402	1	0.289	-0.7	0.064	0.1	0.157	-0.4	0.051	0	2.28	0.8	0.54	0.6
Lab023	0.43	5	0.53	0.1	0.08	0.5	NA	NA	NA	NA	NA	NA	0.07	0.5	0.19	0.4	0.05	-0.1	1.15	-1.6	0.23	-2
Lab024	0.156	-0.2	0.423	-0.7	0.076	0.3	0.479	-0.4	0.287	-0.4	0.326	-0.3	0.074	0.7	0.201	0.6	0.052	0.1	1.646	-0.5	0.539	0.6
Lab025	0.068	-2.4	0.399	-0.9	0.027	-2.5	0.242	-2.2	0.204	-1.4	0.267	-0.9	0.024	-2.5	0.085	-2	0.035	-1.3	1.53	-0.8	0.354	-1
Lab026	0.13	-0.8	0.47	-0.3	0.048	-1.3	0.4	-1	0.23	-1.1	0.26	-1	0.056	-0.4	0.17	-0.1	0.035	-1.3	1.53	-0.8	0.35	-1
Lab027	0.17	0.1	0.51	0	0.074	0.2	0.54	0.1	0.26	-0.7	0.35	0	0.062	0	0.18	0.1	0.061	0.8	1.61	-0.6	0.54	0.6
Lab028	No results reported																					
Lab029	0.19	0.6	0.58	0.5	0.065	-0.3	0.52	-0.1	0.37	0.6	0.34	-0.1	0.049	-0.9	0.15	-0.6	0.042	-0.7	2.16	0.5	0.44	-0.2
Lab030	0.158	-0.2	0.493	-0.1	0.082	0.6	0.597	0.5	0.332	0.2	0.413	0.7	0.047	-1	0.176	0	0.058	0.5	1.82	-0.2	0.537	0.6
Lab031	0.15	-0.4	0.41	-0.8	0.06	-0.6	0.45	-0.6	0.25	-0.9	NA	NA	0.05	-0.8	0.13	-1	0.03	-1.7	1.83	-0.1	0.43	-0.3
Lab032	0.2	0.8	0.5	-0.1	0.08	0.5	0.45	-0.6	0.27	-0.6	0.35	0	0.07	0.5	0.17	-0.1	0.06	0.7	2.1	0.4	0.55	0.7
Lab033	NA	NA	0.15	-2.8	0.06	-0.6	0.45	-0.6	NA	NA	0.3	-0.6	0.31	5	0.13	-1	ND	-3.2	0.76	-2.4	0.35	-1
Lab034	0.14	-0.6	0.41	-0.8	0.07	-0.1	0.48	-0.4	NA	NA	0.226	-1.4	0.043	-1.2	0.146	-0.6	0.033	-1.4	1.59	-0.7	0.475	0.1
Lab035	0.16	-0.1	0.61	0.8	0.069	-0.1	0.52	-0.1	0.26	-0.7	0.31	-0.4	0.063	0	0.18	0.1	0.062	0.8	1.6	-0.6	0.46	-0.1
Lab036	0.13	-0.8	0.56	0.4	0.071	0	0.474	-0.4	0.782	5	0.368	0.2	0.062	0	0.128	-1.1	0.097	3.6	1.961	0.1	0.46	-0.1
Lab037	0.14	-0.6	0.62	0.8	0.06	-0.6	0.44	-0.7	0.26	-0.7	0.34	-0.1	0.041	-1.4	0.15	-0.6	0.032	-1.5	1.7	-0.4	0.41	-0.5
Lab038	0.149	-0.4	0.298	-1.7	0.054	-1	0.541	0.1	0.538	2.7	0.318	-0.4	0.049	-0.9	0.108	-1.5	0.046	-0.4	1.81	-0.2	0.437	-0.2
Lab039	NA	NA	NA	NA	0.06	-0.6	NA	NA	NA	NA	NA	NA	0.04	-1.4	0.14	-0.8	NA	NA	NA	NA	NA	NA
Lab040	0.16	-0.1	0.48	-0.3	0.072	0.1	0.53	0	0.36	0.5	0.475	1.4	0.064	0.1	0.185	0.2	0.071	1.5	1.91	0	0.588	1
Lab041	0.178	0.3	0.588	0.6	0.064	-0.4	0.578	0.4	0.482	2	0.438	1	0.06	-0.2	0.19	0.4	0.049	-0.2	1.94	0.1	0.457	-0.1
Lab042	0.294	3.1	0.48	-0.3	0.071	0	0.58	0.4	0.309	-0.1	0.37	0.2	0.071	0.5	0.194	0.5	0.125	5	1.56	-0.7	0.538	0.6
Lab043	0.267	2.5	0.445	-0.5	0.053	-1	0.407	-0.9	1.37	5	0.321	-0.3	0.069	0.4	0.232	1.3	0.114	4.9	2.26	0.8	0.444	-0.2
Lab044	0.14	-0.6	0.45	-0.5	0.05	-1.2	0.6	0.5	0.25	-0.9	0.43	0.9	0.055	-0.5	0.19	0.4	0.1	3.8	1.8	-0.2	0.5	0.3

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

Lab Code	Bupirimate	Carbendazim		Diazinon	Difenoconazole		Diflubenzuron	Methoxyfenozide		Pendimethalin	Permethrin		Spinosad	Thiabendazole		Trifloxystrobin						
		z-Score (FFP RSD 25%)	0.01		z-Score (FFP RSD 25%)	0.01		z-Score (FFP RSD 25%)	0.01		z-Score (FFP RSD 25%)	0.01		z-Score (FFP RSD 25%)	0.01		z-Score (FFP RSD 25%)	0.01				
MRRL	0.01																					
Robust mean (mg/kg)	0.16	0.51	0.071	0.53	0.32	0.35	0.062	0.17	0.051	1.9	0.47											
Lab045	0.197	0.8	0.244	-2.1	0.068	-0.2	0.57	0.3	NA	NA	0.133	-2.5	0.06	-0.2	0.179	0.1	NA	NA	1.403	-1	0.558	0.8
Lab046	0.206	1	0.467	-0.4	0.079	0.5	0.55	0.2	0.331	0.2	0.359	0.1	0.076	0.9	0.194	0.5	0.055	0.3	1.973	0.2	0.53	0.5
Lab047	0.185	0.5	0.578	0.5	0.079	0.5	0.573	0.3	0.386	0.8	0.374	0.3	0.063	0	0.219	1	0.056	0.4	1.97	0.1	0.408	-0.5
Lab048	0.143	-0.5	0.458	-0.4	0.067	-0.2	0.44	-0.7	0.319	0	0.335	-0.2	0.065	0.2	0.144	-0.7	0.042	-0.7	1.886	0	0.393	-0.6
Lab049	0.14	-0.6	0.47	-0.3	0.063	-0.5	0.59	0.5	0.31	-0.1	0.34	-0.1	0.046	-1.1	0.13	-1	0.047	-0.3	1.5	-0.8	0.41	-0.5
Lab050	NA	NA	0.539	0.2	0.098	1.5	0.547	0.1	0.348	0.4	0.445	1.1	0.071	0.5	0.214	0.9	0.14	5	1.792	-0.2	0.558	0.8
Lab051	0.161	-0.1	NA	NA	0.071	0	NA	NA	NA	NA	NA	NA	NA	NA	0.207	0.8	NA	NA	NA	NA	NA	NA
Lab052	0.189	0.6	0.912	3.1	0.069	-0.1	0.55	0.2	NA	NA	0.397	0.5	0.066	0.2	0.101	-1.7	NA	NA	1.75	-0.3	0.49	0.2
Lab053	0.124	-1	0.502	-0.1	0.074	0.2	0.515	-0.1	0.234	-1.1	0.325	-0.3	0.059	-0.2	0.161	-0.3	0.053	0.1	1.991	0.2	0.288	-1.5
Lab054	0.13	-0.8	0.985	3.7	0.06	-0.6	0.48	-0.4	1.62	5	0.265	-1	0.039	-1.5	0.15	-0.6	ND	-3.2	1.94	0.1	0.42	-0.4
Lab055	0.08	-2.1	0.47	-0.3	0.06	-0.6	0.43	-0.8	0.13	-2.4	NA	NA	0.06	-0.2	0.16	-0.3	0.04	-0.9	0.5	-2.9	0.47	0
Lab056	0.16	-0.1	0.56	0.4	0.08	0.5	0.57	0.3	NA	NA	0.38	0.4	0.057	-0.4	0.21	0.8	0.059	0.6	1.79	-0.2	0.57	0.9
Lab057	0.177	0.3	0.701	1.5	0.098	1.5	0.568	0.3	0.55	2.9	0.396	0.5	0.088	1.7	0.193	0.4	0.072	1.6	2.27	0.8	0.474	0.1
Lab058	0.16	-0.1	0.57	0.5	0.078	0.4	0.657	1	0.525	2.6	0.41	0.7	0.066	0.2	0.254	1.8	0.054	0.2	2.85	2	0.82	3
Lab059	NA	NA	NA	NA	NA	NA	ND	-3.9	NA	NA	NA	NA	NA	NA	0.174	0	NA	NA	0.94	-2	0.582	1
Lab060	0.198	0.8	0.492	-0.2	0.108	2.1	0.494	-0.3	0.315	-0.1	0.47	1.4	0.07	0.5	0.192	0.4	0.058	0.5	1.892	0	0.537	0.6
Lab061	0.244	1.9	0.829	2.5	0.103	1.8	0.581	0.4	0.281	-0.5	0.357	0.1	0.064	0.1	0.205	0.7	0.055	0.3	2.58	1.4	0.483	0.1
Lab062	0.152	-0.3	0.619	0.8	0.059	-0.7	0.555	0.2	0.328	0.1	0.325	-0.3	0.061	-0.1	0.175	0	0.046	-0.4	1.96	0.1	0.422	-0.4
Lab063	0.148	-0.4	NA	NA	0.072	0.1	0.707	1.3	NA	NA	0.476	1.5	NA	NA	0.299	2.9	NA	NA	NA	NA	NA	NA
Lab064	0.198	0.8	0.414	-0.8	0.077	0.3	0.537	0.1	0.298	-0.3	0.375	0.3	0.083	1.3	0.194	0.5	0.058	0.5	1.87	-0.1	0.507	0.4
Lab065	0.17	0.1	0.47	-0.3	0.088	1	0.55	0.2	NA	NA	NA	NA	0.082	1.2	0.197	0.5	NA	NA	0.233	-3.5	0.55	0.7
Lab066	NA	NA	NA	NA	97.28	5	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Lab067	0.171	0.1	0.437	-0.6	0.079	0.4	0.497	-0.2	0.283	-0.5	0.335	-0.2	0.07	0.5	0.206	0.7	0.055	0.3	1.75	-0.3	0.5	0.3
Lab068	0.2	0.8	0.6	0.7	0.09	1.1	0.62	0.7	0.38	0.8	0.43	0.9	0.08	1.1	0.19	0.4	0.04	-0.9	1.7	-0.4	0.6	1.1
Lab069	0.136	-0.7	0.534	0.2	0.077	0.3	0.532	0	0.297	-0.3	0.37	0.2	0.054	-0.5	0.133	-0.9	0.046	-0.4	1.618	-0.6	0.428	-0.3
Lab070	0.18	0.4	0.49	-0.2	0.095	1.4	0.64	0.8	0.34	0.3	0.4	0.6	0.087	1.6	0.2	0.6	0.073	1.7	2.09	0.4	0.5	0.3
Lab071	0.086	-1.9	0.63	0.9	0.038	-1.9	0.3	-1.7	0.15	-2.1	0.34	-0.1	0.04	-1.4	0.12	-1.2	0.027	-1.9	1.51	-0.8	NA	NA
Lab072	0.19	0.6	0.49	-0.2	0.074	0.2	0.53	0	0.34	0.3	0.35	0	0.076	0.9	0.199	0.6	0.05	-0.1	2.01	0.2	0.473	0.1
Lab073	0.151	-0.3	0.577	0.5	0.075	0.2	0.471	-0.4	0.343	0.3	0.302	-0.5	0.047	-1	0.145	-0.7	0.054	0.2	1.64	-0.5	0.397	-0.6
Lab074	0.178	0.3	0.586	0.6	0.105	1.9	0.568	0.3	1.91	5	0.411	0.7	0.103	2.6	0.228	1.2	0.056	0.4	1.75	-0.3	0.556	0.8
Lab075	NA	NA	NA	NA	0.045	-1.5	0.574	0.3	NA	NA	NA	NA	0.044	-1.2	0.164	-0.2	NA	NA	NA	NA	0.287	-1.5
Lab076	0.17	0.1	0.468	-0.3	0.071	0	0.564	0.3	0.314	-0.1	0.439	1	0.067	0.3	0.192	0.4	0.259	5	2.233	0.7	0.504	0.3
Lab077	0.185	0.5	0.45	-0.5	0.052	-1.1	0.492	-0.3	0.462	1.8	0.383	0.4	0.049	-0.9	0.166	-0.2	0.041	-0.8	2.149	0.5	0.446	-0.2
Lab078	0.195	0.7	0.56	0.4	0.075	0.2	0.55	0.2	0.32	0	0.41	0.7	0.068	0.4	0.17	-0.1	0.046	-0.4	2.03	0.3	0.485	0.2
Lab079	0.168	0.1	0.459	-0.4	0.076	0.3	0.43	-0.8	0.208	-1.4	0.245	-1.2	0.073	0.7	0.176	0	0.029	-1.7	1.52	-0.8	0.434	-0.3
Lab080	0.168	0.1	0.598	0.7	0.081	0.6	0.563	0.3	0.271	-0.6	0.328	-0.2	0.069	0.4	0.195	0.5	0.046	-0.4	2.03	0.3	0.46	-0.1
Lab081	0.167	0	NA	NA	0.075	0.2	0.57	0.3	NA	NA	NA	NA	0.067	0.3	0.218	1	NA	NA	NA	NA	0.475	0.1
Lab082	0.167	0	0.568	0.4	0.071	0	0.529	0	0.267	-0.7	0.329	-0.2	0.064	0.1	0.194	0.5	0.028	-1.8	2.214	0.7	0.476	0.1
Lab083	0.185	0.5	0.557	0.4	0.068	-0.2	0.472	-0.4	0.331	0.2	0.29	-0.7	0.066	0.2	0.199	0.6	0.045	-0.5	1.798	-0.2	0.416	-0.4
Lab084	0.173	0.2	0.511	0	0.08	0.5	0.59	0.5	0.284	-0.4	0.36	0.1	0.061	-0.1	0.19	0.4	0.056	0.4	1.98	0.2	0.51	0.4
Lab085	0.17	0.1	1.4	5	0.086	0.8	0.5	-0.2	0.3	-0.2	0.28	-0.8	0.062	0	NA	NA	0.043	-0.6	1.2	-1.5	0.64	1.5
Lab086	0.185	0.5	0.497	-0.1	0.075	0.2	0.61	0.6	0.31	-0.1	0.38	0.4	0.068	0.4	0.186	0.3	0.053	0.1	2.173	0.6	0.52	0.5
Lab087	0.205	1	0.444	-0.5	0.07	-0.1	0.45	-0.6	0.315	-0.1	0.331	-0.2	0.084	1.4	0.201	0.6	0.039	-1	2.07	0.4	0.455	-0.1
Lab088	0.18	0.4	0.847	2.6	0.063	-0.5	0.58	0.4	0.349	0.4	0.287	-0.7	0.074	0.7	0.183	0.2	0.045	-0.5	1.86	-0.1	0.518	0.4
Lab089	0.17	0.1	0.55	0.3	0.08	0.5	0.62	0.7	0.48	2	0.41	0.7	0.07	0.5	0.21	0.8	0.061	0.8	1.88	0	0.5	0.3
Lab090	NA	NA	0.46	-0.4	0.04	-1.7	NA	NA	NA	NA	NA	NA	0.04	-1.4	0.1	-1.7	NA	NA	NA	NA	0.33	-1.2
Lab091	0.236	1.7	0.538	0.2	0.078	0.4	0.571	0.3	0.214	-1.3	0.341	-0.1	0.08	1.1	0.208	0.8	0.116	5	1.916	0	0.588	1
Lab092	0.14	-0.6	NA	NA	0.05	-1.2	0.61	0.6	0.35	0.4	0.27	-0.9	0.07	0.5	0.29	2.7	0.043	-0.6	NA	NA	0.45	-0.1
Lab093	0.098	-1.6	0.401	-0.9	0.07	-0.1	0.5	-0.2	0.332	0.2	0.32	-0.3	0.093	2	0.127	-1.1	0.044	-0.6	1.429	-1	0.421	-0.4
Lab094	0.17	0.1	0.55	0.3	0.078	0.4	0.33	-1.5	0.43	1.4	0.222	-1.5	0.06	-0.2	0.143	-0.7	0.075	1.9	4.4	5	0.3	-1.4

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

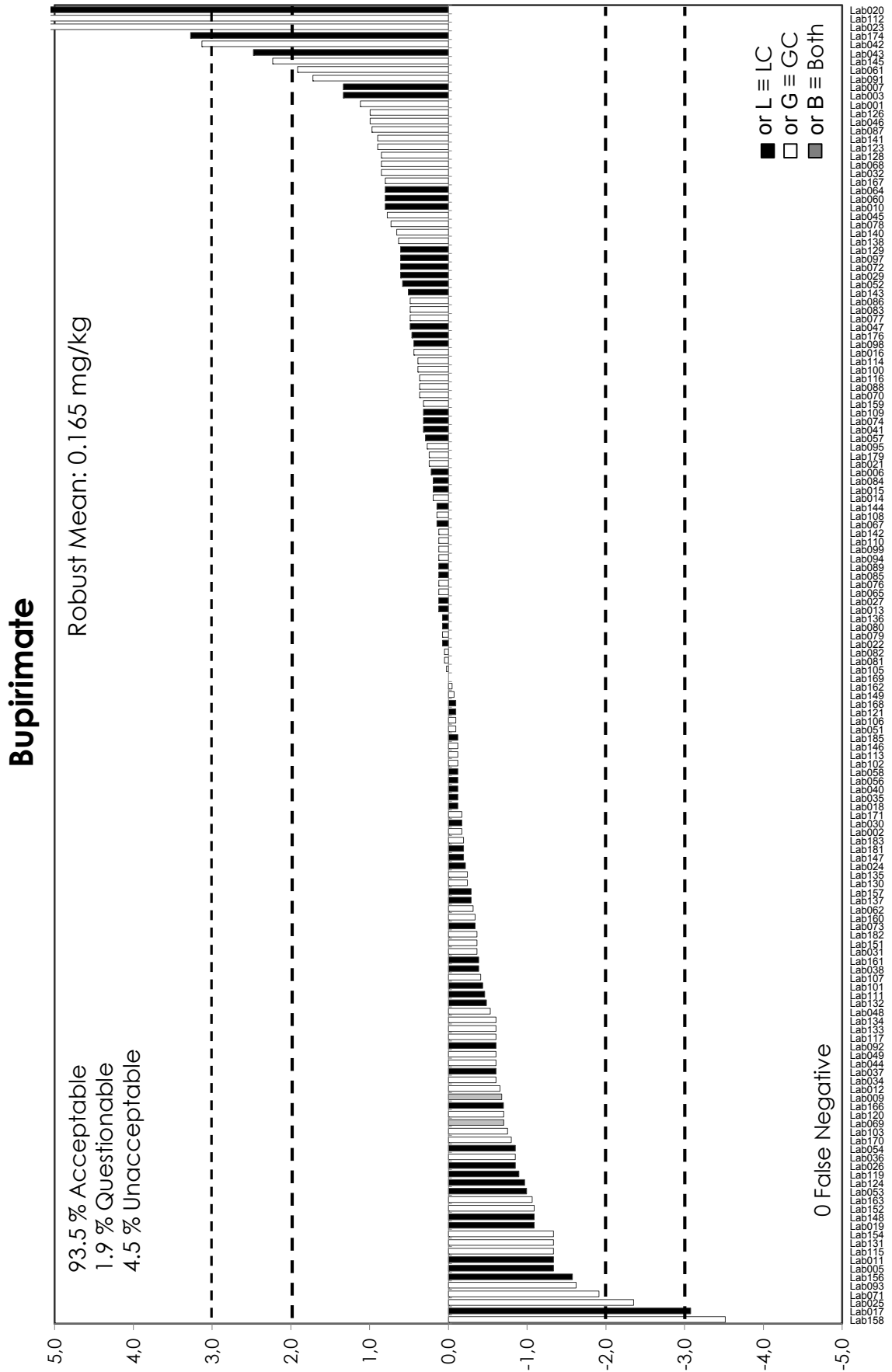
Lab Code	Bupirimate	Carbendazim		Diazinon	Difenoconazole	Diflubenzuron	Methoxyfenozide	Pendimethalin	Permethrin	Spinosad	Thiabendazole	Trifloxystrobin										
		z-Score (FFP RSD 25%)	z-Score (FFP RSD 25%)																			
MRRL	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01										
Robust mean (mg/kg)	0.16	0.51	0.071	0.53	0.32	0.35	0.062	0.17	0.051	1.9	0.47	z-Score (FFP RSD 25%)										
Lab095	0.176	0.3	0.454	-0.5	0.086	0.8	0.594	0.5	0.28	-0.5	0.328	-0.2	0.074	0.7	0.313	3.2	0.059	0.6	2.53	1.3	0.605	1.2
Lab096	No results reported																					
Lab097	0.19	0.6	0.5	-0.1	0.079	0.5	0.56	0.2	0.32	0	0.35	0	0.059	-0.2	0.17	-0.1	0.053	0.1	2	0.2	0.49	0.2
Lab098	0.183	0.4	0.548	0.3	0.077	0.3	0.588	0.4	0.333	0.2	0.368	0.2	0.075	0.8	0.184	0.2	0.057	0.5	2.11	0.4	0.394	-0.6
Lab099	0.17	0.1	0.553	0.3	0.077	0.3	0.58	0.4	NA	NA	NA	NA	0.067	0.3	0.18	0.1	NA	NA	1.709	-0.4	0.494	0.2
Lab100	0.181	0.4	0.559	0.4	0.077	0.3	0.573	0.3	0.275	-0.6	0.351	0	0.07	0.5	0.182	0.2	0.056	0.4	1.826	-0.2	0.503	0.3
Lab101	0.147	-0.4	0.385	-1	0.054	-1	0.463	-0.5	0.31	-0.1	0.335	-0.2	0.05	-0.8	NA	NA	0.046	-0.4	1.81	-0.2	0.411	-0.5
Lab102	0.16	-0.1	0.505	-0.1	0.065	-0.3	0.558	0.2	0.272	-0.6	0.426	0.9	0.058	-0.3	0.179	0.1	0.057	0.5	2.27	0.8	0.411	-0.5
Lab103	0.134	-0.8	1.89	5	0.084	0.7	0.465	-0.5	0.297	-0.3	0.244	-1.2	0.06	-0.2	0.133	-0.9	0.025	-2	1.58	-0.7	0.432	-0.3
Lab104	0.25	2.1	1.01	3.9	0.096	1.4	0.84	2.3	0.23	-1.1	0.37	0.2	0.079	1.1	0.24	1.5	0.17	5	1.65	-0.5	0.58	1
Lab105	0.166	0	0.44	-0.6	0.066	-0.3	0.503	-0.2	0.154	-2.1	0.35	0	0.059	-0.2	0.161	-0.3	0.047	-0.3	1.66	-0.5	0.424	-0.4
Lab106	0.161	-0.1	0.682	1.3	0.075	0.2	0.56	0.2	NA	NA	0.333	-0.2	0.061	-0.1	NA	NA	0.05	-0.1	1.67	-0.5	0.435	-0.3
Lab107	0.148	-0.4	0.414	-0.8	0.169	5	0.517	-0.1	NA	NA	0.299	-0.6	0.068	0.4	0.18	0.1	0.045	-0.5	1.7	-0.4	0.452	-0.1
Lab108	0.171	0.1	0.538	0.2	0.078	0.4	0.523	-0.1	0.279	-0.5	0.349	0	0.072	0.6	0.192	0.4	0.054	0.2	1.789	-0.2	0.47	0
Lab109	0.178	0.3	0.568	0.4	0.076	0.3	0.603	0.6	0.331	0.2	0.375	0.3	0.072	0.6	0.215	0.9	0.04	-0.9	1.96	0.1	0.544	0.7
Lab110	0.17	0.1	4.7	5	ND	-3.4	0.63	0.8	NA	NA	0.33	-0.2	0.07	0.5	0.18	0.1	0.046	-0.4	ND	-4	0.53	0.5
Lab111	0.146	-0.5	0.394	-0.9	0.069	-0.1	0.464	-0.5	0.348	0.4	0.273	-0.9	0.053	-0.6	0.116	-1.3	0.043	-0.6	1.64	-0.5	0.317	-1.3
Lab112	0.53	5	0.231	-2.2	0.13	3.3	0.562	0.2	0.195	-1.6	0.473	1.4	0.072	0.6	0.3	2.9	0.056	0.4	1.582	-0.7	0.489	0.2
Lab113	0.16	-0.1	0.78	2.1	0.065	-0.3	0.5	-0.2	NA	NA	0.43	0.9	0.095	2.1	0.14	-0.8	0.06	0.7	1.87	-0.1	0.38	-0.7
Lab114	0.181	0.4	0.55	0.3	0.08	0.5	0.38	-1.1	NA	NA	0.3	-0.6	0.075	0.8	0.2	0.6	0.025	-2	1.5	-0.8	0.58	1
Lab115	0.11	-1.3	NA	NA	0.07	-0.1	0.59	0.5	NA	NA	NA	NA	0.03	-2.1	0.11	-1.5	NA	NA	NA	NA	0.38	-0.7
Lab116	0.18	0.4	0.63	0.9	0.091	1.1	0.57	0.3	0.051	-3.4	0.41	0.7	0.079	1.1	0.204	0.7	0.055	0.3	0.78	-2.4	0.5	0.3
Lab117	0.14	-0.6	0.5	-0.1	0.06	-0.6	0.69	1.2	NA	NA	NA	NA	0.05	-0.8	0.18	0.1	NA	NA	1.9	0	0.44	-0.2
Lab118	NA	NA	NA	NA	0.09	1.1	NA	NA	NA	NA	NA	NA	NA	NA	0.14	-0.8	NA	NA	NA	NA	NA	NA
Lab119	0.128	-0.9	0.247	-2.1	0.07	-0.1	0.583	0.4	0.303	-0.2	0.276	-0.8	0.1	2.4	0.052	-2.8	0.037	-1.1	NA	NA	0.331	-1.2
Lab120	0.136	-0.7	0.608	0.8	0.058	-0.7	0.489	-0.3	0.244	-0.9	0.285	-0.7	0.047	-1	0.118	-1.3	0.042	-0.7	1.83	-0.1	0.418	-0.4
Lab121	0.161	-0.1	0.644	1	0.069	-0.1	0.504	-0.2	NA	NA	0.316	-0.4	0.054	-0.5	0.133	-0.9	0.047	-0.3	1.917	0	0.416	-0.4
Lab122	0.194	0.7	0.547	0.3	0.095	1.4	0.613	0.6	0.328	0.1	0.321	-0.3	0.064	0.1	0.172	-0.1	0.066	1.2	1.943	0.1	0.532	0.6
Lab123	0.202	0.9	0.52	0.1	0.085	0.8	0.614	0.6	0.412	1.2	0.41	0.7	0.089	1.7	0.242	1.6	0.042	-0.7	2	0.2	0.517	0.4
Lab124	0.125	-1	0.587	0.6	0.054	-1	0.4	-1	0.26	-0.7	0.517	1.9	0.057	-0.4	0.157	-0.4	0.04	-0.9	2.017	0.2	0.353	-1
Lab125	1.621	5	0.284	-1.8	0.059	-0.7	0.458	-0.5	0.265	-0.7	NA	NA	0.035	-1.8	0.145	-0.7	0.046	-0.4	1.441	-1	0.392	-0.6
Lab126	0.206	1	0.468	-0.3	0.069	-0.1	0.569	0.3	0.214	-1.3	0.359	0.1	0.051	-0.7	0.132	-1	0.051	0	2.104	0.4	0.484	0.2
Lab127	NA	NA	NA	NA	0.066	-0.3	NA	NA	NA	NA	0.35	0	0.047	-1	0.18	0.1	0.08	2.3	0.2	-3.6	0.052	-3.6
Lab128	0.2	0.8	0.306	-1.6	0.054	-1	0.52	-0.1	0.271	-0.6	0.328	-0.2	0.063	0	0.192	0.4	ND	-3.2	1.591	-0.7	0.444	-0.2
Lab129	0.19	0.6	NA	NA	0.083	0.7	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.67	1.7
Lab130	0.155	-0.2	NA	NA	0.075	0.2	NA	NA	NA	NA	NA	NA	0.065	0.2	0.157	-0.4	NA	NA	NA	NA	0.462	0
Lab131	0.11	-1.3	NA	NA	0.05	-1.2	0.4	-1	NA	NA	NA	NA	0.04	-1.4	0.18	0.1	NA	NA	NA	NA	NA	NA
Lab132	0.145	-0.5	0.816	2.4	0.072	0	0.464	-0.5	0.317	0	0.308	-0.5	0.051	-0.7	0.154	-0.5	0.057	0.5	1.524	-0.8	0.404	-0.5
Lab133	0.14	-0.6	0.38	-1	0.052	-1.1	0.4	-1	NA	NA	NA	NA	0.047	-1	0.15	-0.6	0.042	-0.7	1.8	-0.2	0.38	-0.7
Lab134	0.14	-0.6	0.4	-0.9	0.06	-0.6	NA	NA	NA	NA	NA	NA	0.06	-0.2	0.14	-0.8	NA	NA	NA	NA	0.4	-0.6
Lab135	0.155	-0.2	0.426	-0.7	0.076	0.3	0.494	-0.3	0.268	-0.6	0.321	-0.3	0.063	0	0.164	-0.2	0.047	-0.3	1.83	-0.1	0.366	-0.9
Lab136	0.168	0.1	0.611	0.8	0.077	0.3	0.537	0.1	1.05	5	0.544	2.2	0.105	2.7	NA	NA	0.048	-0.2	1.517	-0.8	0.506	0.3
Lab137	0.153	-0.3	0.501	-0.1	0.074	0.2	0.533	0	0.301	-0.2	0.321	-0.3	0.062	0	0.179	0.1	0.058	0.5	1.69	-0.4	0.479	0.1
Lab138	0.191	0.6	0.49	-0.2	0.076	0.3	0.545	0.1	NA	NA	NA	NA	0.081	1.2	0.197	0.5	NA	NA	1.949	0.1	0.55	0.7
Lab139	NA	NA	NA	NA	0.076	0.3	NA	NA	NA	NA	NA	NA	0.066	0.2	0.125	-1.1	NA	NA	NA	NA	NA	NA
Lab140	0.192	0.7	0.528	0.1	0.088	1	0.603	0.6	NA	NA	0.441	1.1	NA	NA	NA	NA	NA	NA	1.89	0	0.529	0.5
Lab141	0.202	0.9	NA	NA	0.135	3.6	0.66	1	NA	NA	0.484	1.5	0.089	1.7	0.264	2.1	0.059	0.6	1.97	0.1	0.584	1
Lab142	0.17	0.1	3.316	5	0.068	-0.2	0.547	0.1	0.524	2.6	0.327	-0.3	0.053	-0.6	0.167	-0.2	0.029	-1.7	5.405	5	0.45	-0.1
Lab143	0.186	0.5	0.386	-1	0.085	0.8	0.598	0.5	0.362	0.5	0.374	0.3	0.052	-0.7	0.142	-0.7	0.062	0.8	1.71	-0.4	0.438	-0.2
Lab144	0.171	0.1	0.518	0	0.061	-0.6	0.654	0.9	0.296	-0.3	0.361	0.1	0.057	-0.4	0.165	-0.2	0.045	-0.5	1.86	-0.1	0.448	-0.2

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

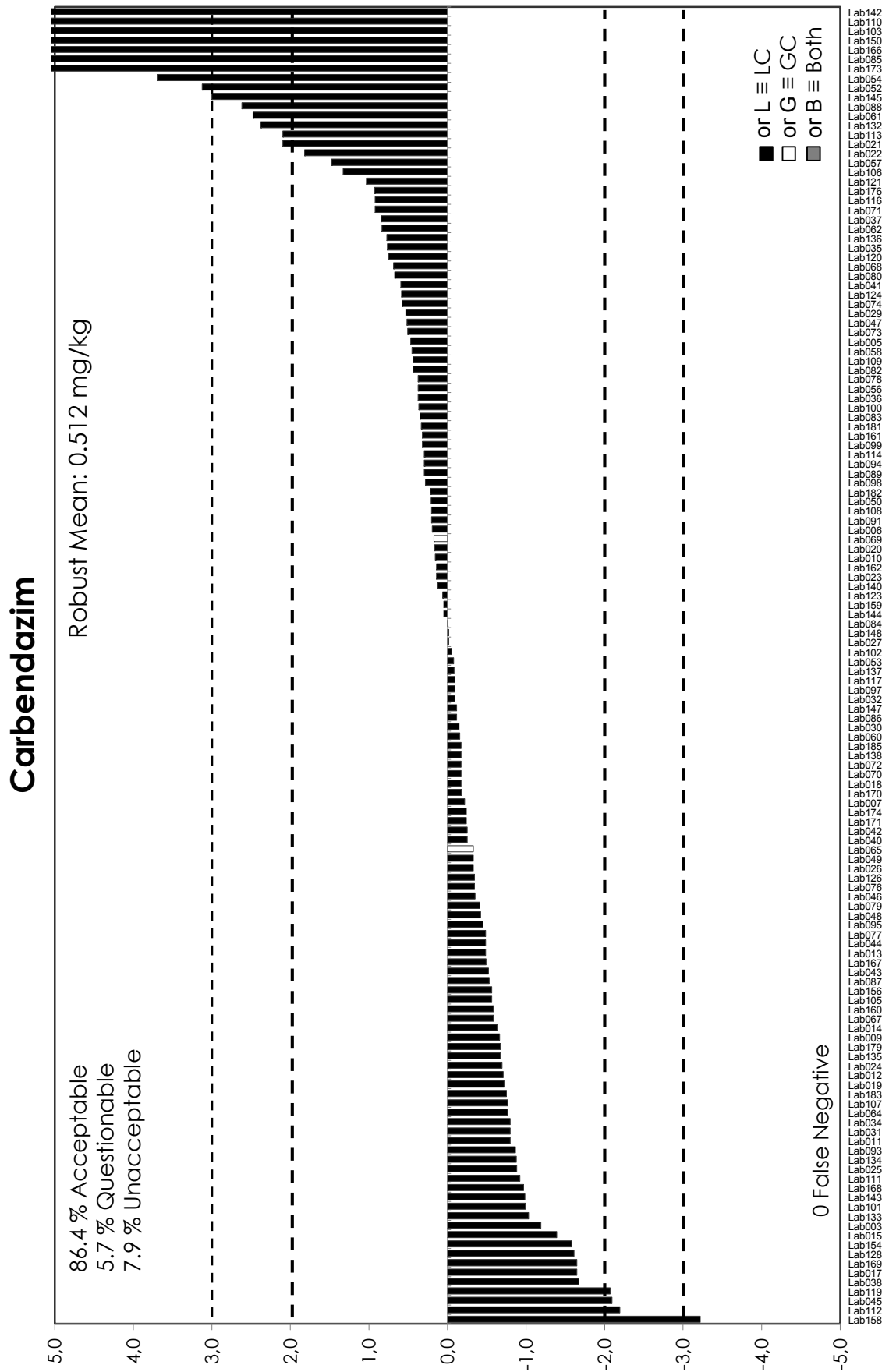
Lab Code	Bupirimate	Carbendazim		Diazinon	Difenoconazole		Diflubenzuron	Methoxyfenozide		Pendimethalin	Permethrin		Spinosad	Thiabendazole		Trifloxystrobin	z-Score (FFP RSD 25%)					
		z-Score (FFP RSD 25%)	0.01		z-Score (FFP RSD 25%)	0.01		z-Score (FFP RSD 25%)	0.01		z-Score (FFP RSD 25%)	0.01		z-Score (FFP RSD 25%)	0.01			z-Score (FFP RSD 25%)	0.01			
MRRL	0.01		0.01																			
Robust mean (mg/kg)	0.16		0.51		0.071		0.53		0.32		0.35		0.062		0.17		0.051		1.9		0.47	
Lab145	0.257	2.2	0.896	3	0.068	-0.2	0.527	0	NA	NA	0.333	-0.2	0.053	-0.6	0.12	-1.2	NA	NA	1.8	-0.2	0.407	-0.5
Lab146	0.16	-0.1	NA	NA	NA	NA	0.53	0	NA	NA	NA	NA	0.064	0.1	0.22	1.1	NA	NA	NA	NA	0.46	-0.1
Lab147	0.157	-0.2	0.497	-0.1	0.073	0.1	0.487	-0.3	0.31	-0.1	0.328	-0.2	0.053	-0.6	0.183	0.2	0.045	-0.5	1.797	-0.2	0.44	-0.2
Lab148	0.12	-1.1	0.51	0	0.04	-1.7	0.4	-1	NA	NA	0.29	-0.7	0.03	-2.1	0.12	-1.2	0.07	1.5	1.49	-0.9	0.38	-0.7
Lab149	0.162	-0.1	NA	NA	0.067	-0.2	NA	NA	NA	NA	NA	NA	0.057	-0.4	0.182	0.2	NA	NA	NA	NA	NA	NA
Lab150	NA	NA	1.65	5	0.026	-2.5	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	5.81	5	NA	NA
Lab151	0.15	-0.4	NA	NA	0.075	0.2	0.515	-0.1	NA	NA	NA	NA	0.064	0.1	0.19	0.4	NA	NA	0.95	-2	0.451	-0.1
Lab152	0.12	-1.1	NA	NA	0.06	-0.6	NA	NA	NA	NA	NA	NA	0.04	-1.4	0.14	-0.8	NA	NA	NA	NA	0.49	0.2
Lab153	NA	NA	0.28	-1.8	0.07	-0.1	0.6	0.5	0.18	-1.7	0.45	1.2	0.07	0.5	0.2	0.6	0.06	0.7	NA	NA	NA	NA
Lab154	0.11	-1.3	0.31	-1.6	0.079	0.5	0.46	-0.5	NA	NA	NA	NA	0.06	-0.2	NA	NA	NA	NA	1.24	-1.4	0.44	-0.2
Lab155	Participation cancelled																					
Lab156	0.1	-1.6	0.44	-0.6	0.08	0.5	0.64	0.8	0.425	1.3	0.364	0.2	0.067	0.3	0.182	0.2	0.071	1.6	3.1	2.5	0.53	0.5
Lab157	0.153	-0.3	NA	NA	0.069	-0.1	0.557	0.2	NA	NA	NA	NA	0.054	-0.5	0.141	-0.8	NA	NA	1.72	-0	0.42	-0
Lab158	0.02	-3.5	0.1	-3.2	0.02	-2.9	0.2	-2.5	0.01	-3.9	0.15	-2.3	0.02	-2.7	NA	NA	0.01	-3	2.45	1.2	0.17	-3
Lab159	0.178	0.3	0.518	0	0.076	0.3	0.556	0.2	0.292	-0.3	0.37	0.2	0.07	0.5	0.164	-0.2	0.061	0.8	1.96	0.1	0.51	0.4
Lab160	0.151	-0.3	0.437	-0.6	0.062	-0.5	0.458	-0.5	0.242	-1	0.272	-0.9	0.059	-0.2	0.165	-0.2	0.052	0.1	1.71	-0	0.43	-0
Lab161	0.149	-0.4	0.553	0.3	0.084	0.7	0.574	0.3	0.252	-0.8	0.283	-0.8	0.075	0.8	0.202	0.6	0.045	-1	1.73	-0	0.56	0.8
Lab162	0.163	0	0.53	0.1	0.074	0.2	0.611	0.6	0.2	-1.5	0.25	-1.1	0.065	0.2	0.17	-0.1	0.053	0.1	1.02	-2	0.52	0.5
Lab163	0.121	-1.1	NA	NA	0.054	-1	0.383	-1.1	NA	NA	NA	NA	NA	NA	0.35	4	NA	NA	ND	-4	0.29	-2
Lab164	NA	NA	NA	NA	0.08	0.5	ND	-3.9	NA	NA	NA	NA	0.074	0.7	0.156	-0.4	NA	NA	NA	NA	NA	NA
Lab165	0.212	1.1	NA	NA	0.076	0.3	0.99	3.5	NA	NA	NA	NA	0.075	0.8	0.147	-0.6	NA	NA	ND	-4	0.58	1
Lab166	0.136	-0.7	1.548	5	0.038	-1.8	0.499	-0.2	0.223	-1.2	NA	NA	0.228	5	0.865	5	NA	NA	NA	NA	NA	NA
Lab167	0.198	0.8	0.449	-0.5	0.08	0.5	0.654	0.9	0.337	0.2	0.343	-0.1	0.068	0.4	0.207	0.8	0.063	0.9	2.05	0.3	0.6	1.1
Lab168	0.161	-0.1	0.388	-1	0.06	-0.6	0.442	-0.7	0.241	-1	0.308	-0.5	0.056	-0.4	0.141	-0.8	0.044	-1	1.37	-1	0.41	-1
Lab169	0.165	0	0.301	-1.6	ND	-3.4	0.39	-1.1	0.45	1.6	0.395	0.5	ND	-3.4	0.18	0.1	0.073	1.7	2.11	0.4	0.51	0.4
Lab170	0.132	-0.8	0.489	-0.2	NA	NA	0.555	0.2	NA	NA	NA	NA	0.041	-1.4	NA	NA	NA	NA	1.77	-0	0.3	-1
Lab171	0.158	-0.2	0.481	-0.2	0.072	0.1	0.49	-0.3	0.266	-0.7	0.32	-0.3	0.063	0	0.159	-0.3	0.048	-0	2.02	0.3	0.46	0
Lab172	0.19	0.6	0.48	-0.3	0.063	-0.5	0.64	0.8	0.34	0.3	0.44	1	0.065	0.2	0.12	-1.2	0.057	0.5	1.97	0.1	0.56	0.8
Lab173	NA	NA	1.29	5	0.03	-2.3	0.37	-1.2	ND	-3.9	NA	NA	NA	NA	0.12	-1.2	NA	NA	2.45	1.2	NA	NA
Lab174	0.3	3.3	0.481	-0.2	0.066	-0.3	0.53	0	NA	NA	0.3	-0.6	NA	NA	0.183	0.2	0.046	-0	1.55	-1	0.4	-1
Lab175	NA	NA	0.495	-0.1	NA	NA	0.303	-1.7	NA	NA	NA	NA	NA	NA	0.591	5	NA	NA	8.69	5	NA	NA
Lab176	0.184	0.5	0.631	0.9	0.081	0.6	0.588	0.4	0.332	0.2	0.389	0.5	0.068	0.4	0.248	1.7	0.048	-0	1.93	0.1	0.5	0.3
Lab177	0.168	0.1	0.558	0.4	0.079	0.5	0.612	0.6	0.363	0.6	0.373	0.3	NA	NA	0.268	2.2	0.032	-2	1.01	-2	0.51	0.4
Lab178	No results reported																					
Lab179	0.175	0.2	0.426	-0.7	0.067	-0.2	0.668	1	0.386	0.8	0.422	0.8	0.059	-0.2	0.186	0.3	0.059	0.6	1.97	0.1	0.42	-0
Lab180	0.137	-0.7	1.011	3.9	0.053	-1	0.49	-0.3	0.423	1.3	0.416	0.8	0.095	2.1	0.194	0.5	0.043	-1	2.66	1.6	0.28	-2
Lab181	0.157	-0.2	0.555	0.3	0.08	0.5	0.603	0.6	0.518	2.5	0.405	0.6	0.07	0.5	0.248	1.7	0.05	-0	2.75	1.8	0.8	2.9
Lab182	0.15	-0.4	0.54	0.2	0.067	-0.2	0.655	0.9	0.403	1.1	0.599	2.9	0.06	-0.2	0.21	0.8	0.06	0.7	2.77	1.8	0.7	2
Lab183	0.157	-0.2	0.416	-0.8	0.06	-0.6	0.449	-0.6	0.524	2.6	0.322	-0.3	0.051	-0.7	0.114	-1.4	0.047	-0	1.32	-1	0.4	-1
Lab184	Participation cancelled																					
Lab185	0.16	-0.1	0.49	-0.2	0.07	-0.1	0.43	-0.8	0.51	2.4	0.28	-0.8	0.06	-0.2	0.14	-0.8	0.04	-1	1.3	-1	0.42	-0

NA: Not analysed
 ND: Not detected (False negative)

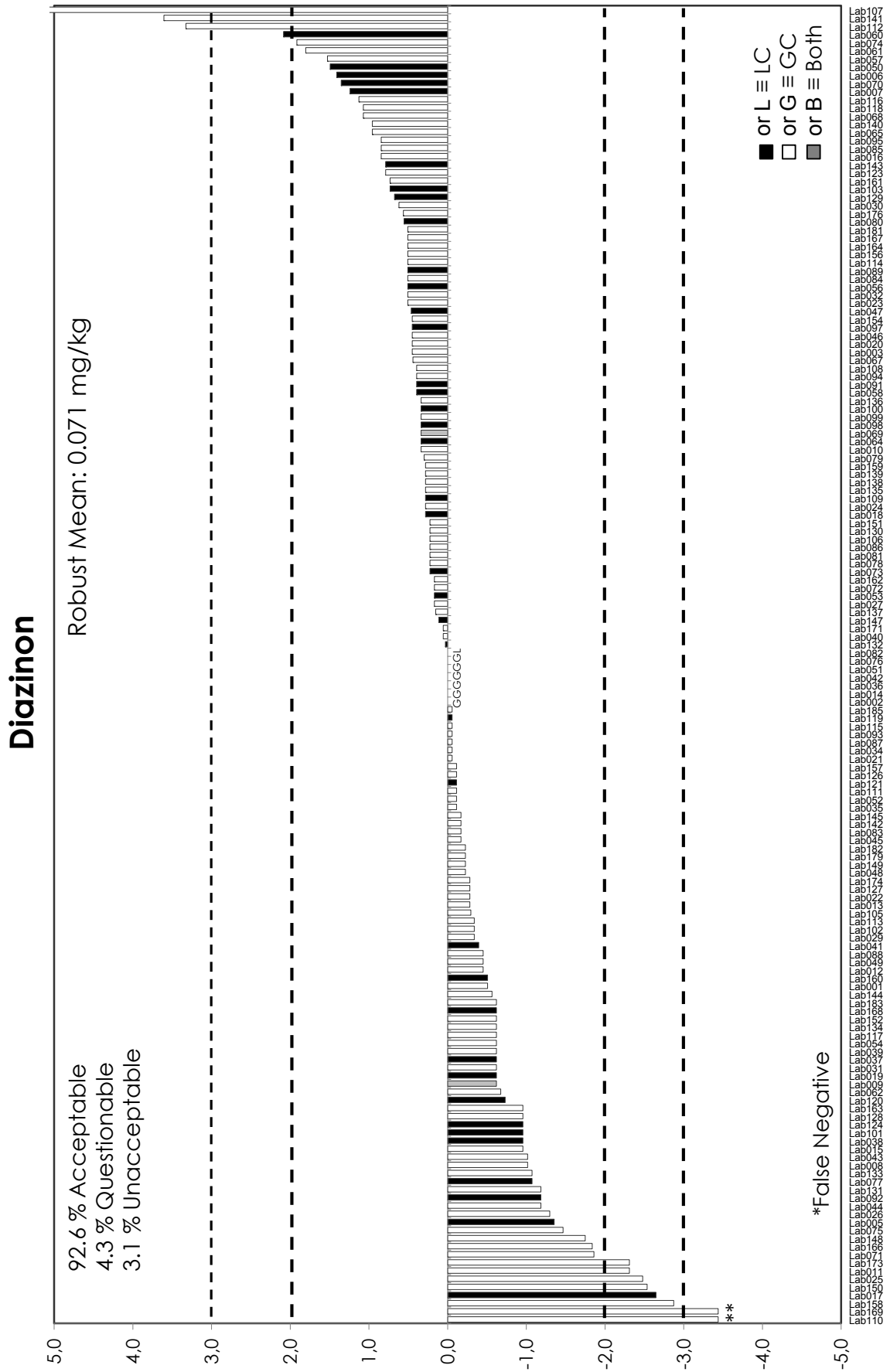
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25%).



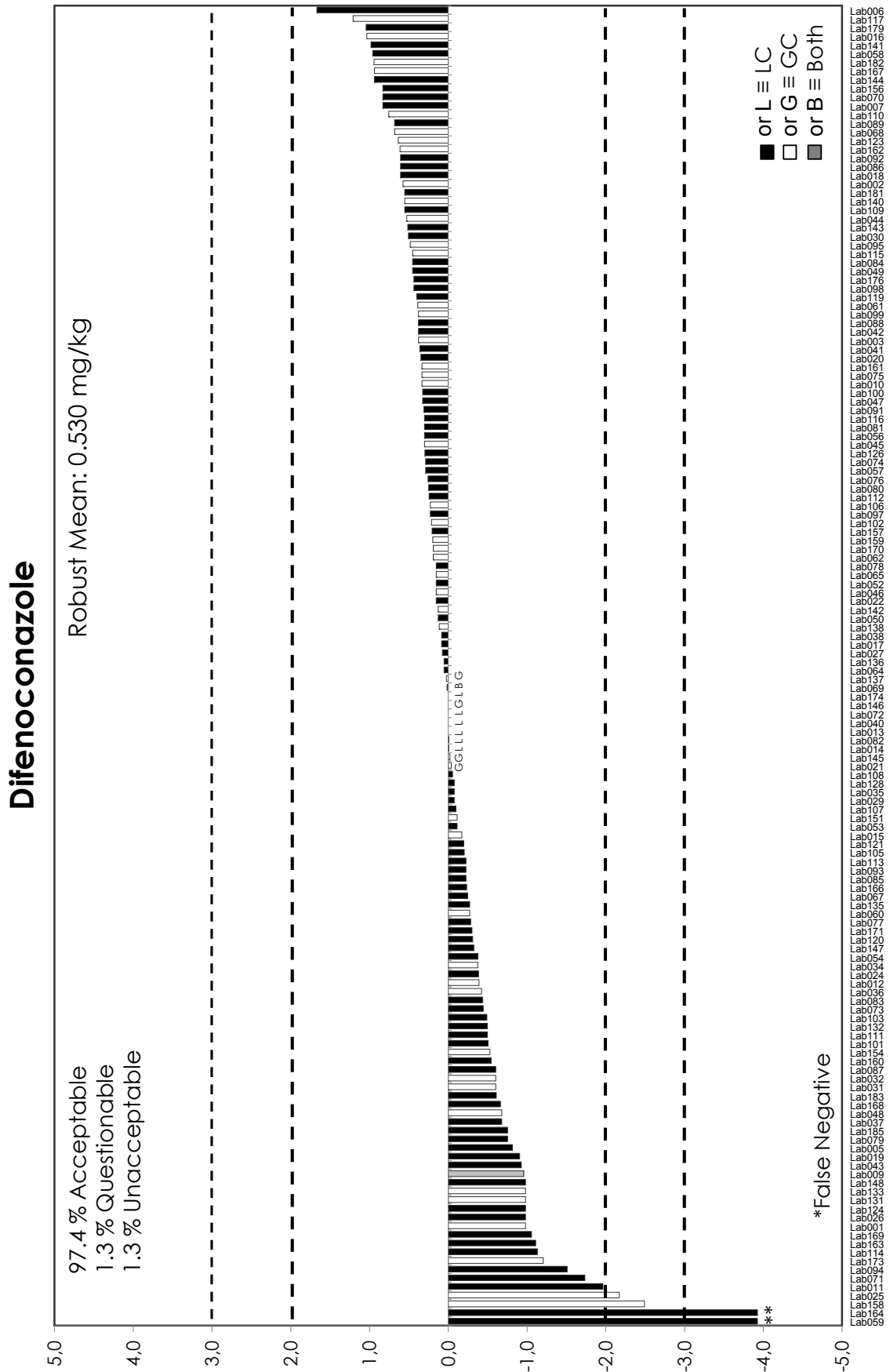
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25%).



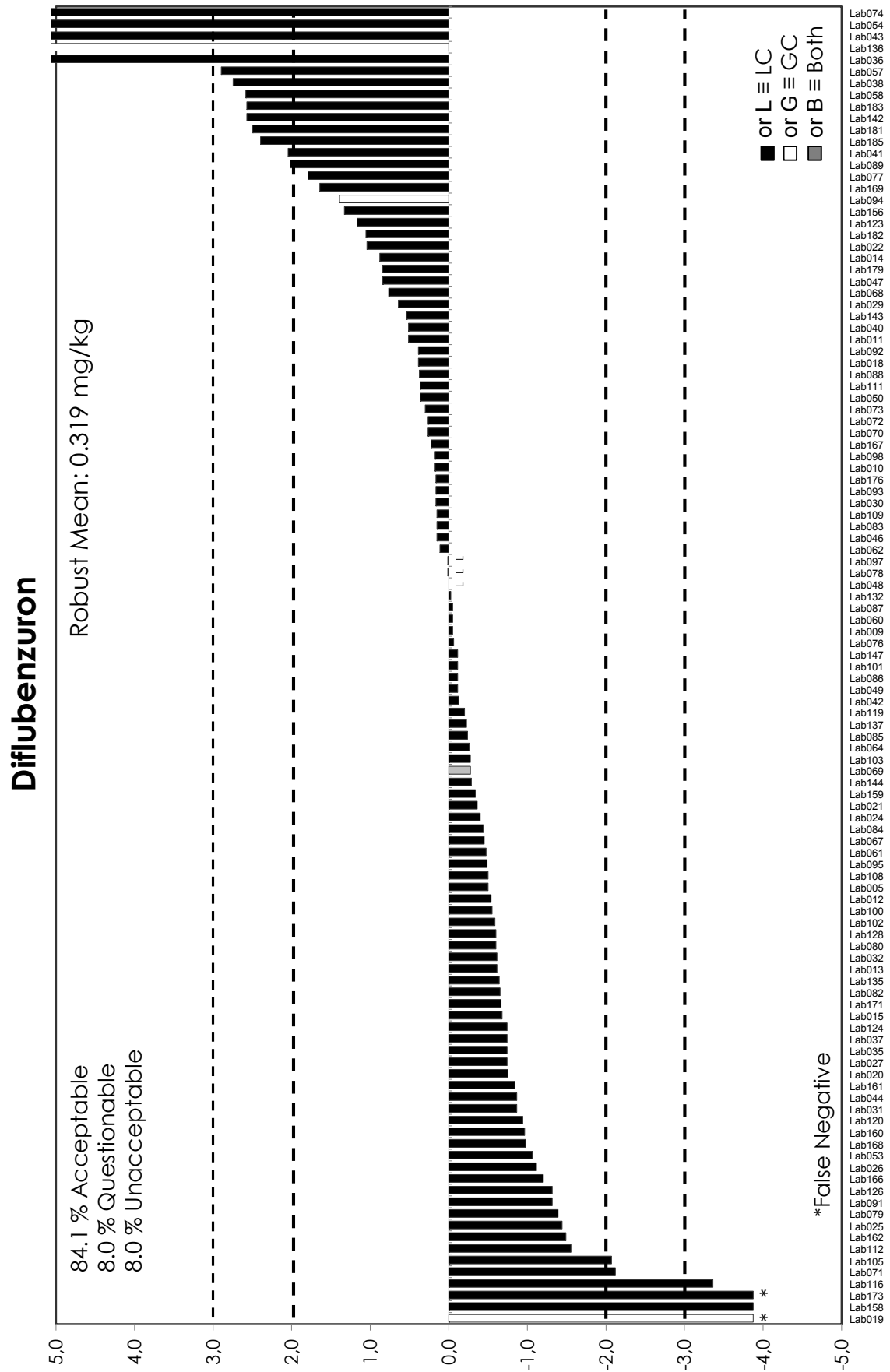
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25%).



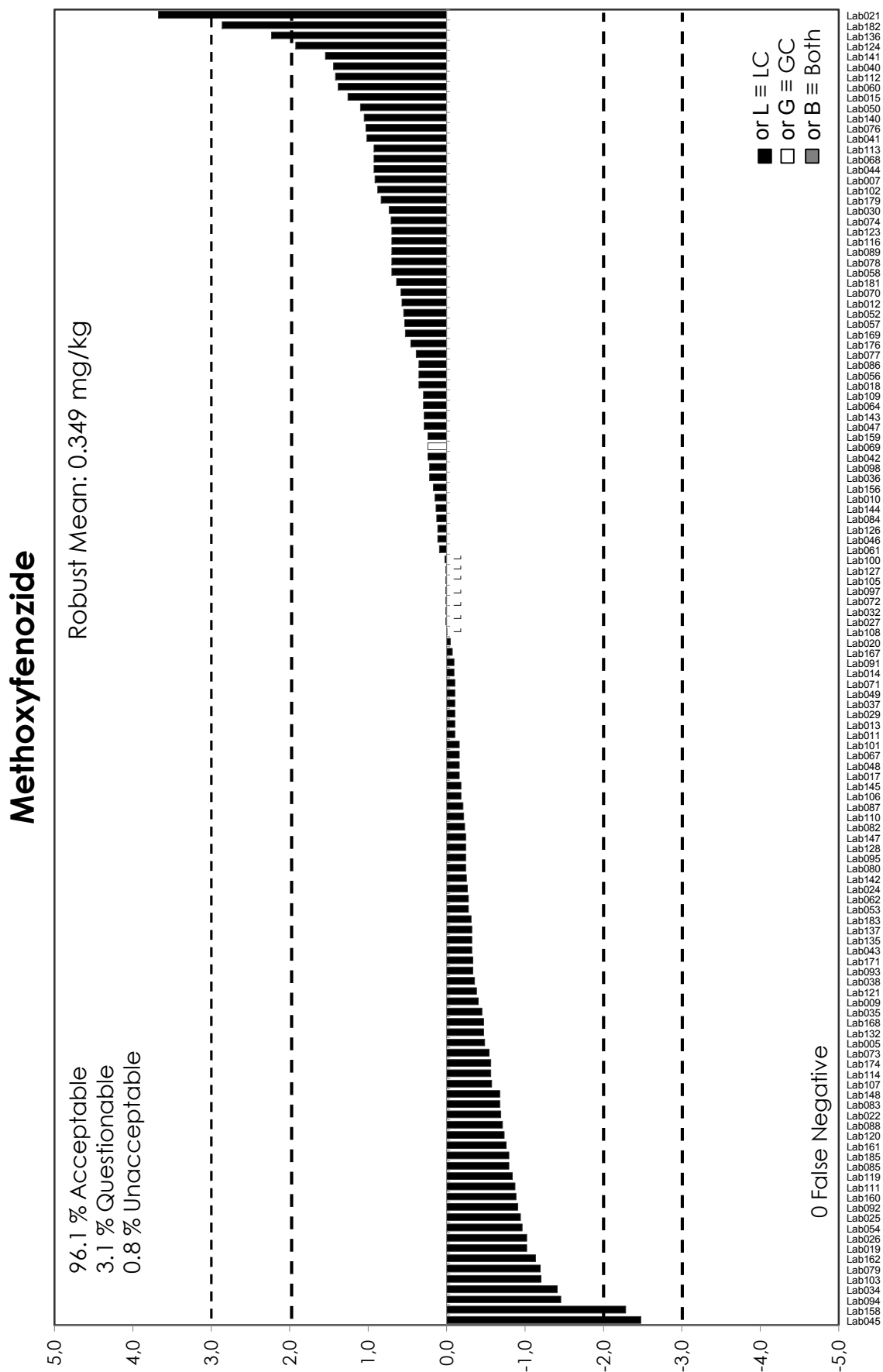
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25%).



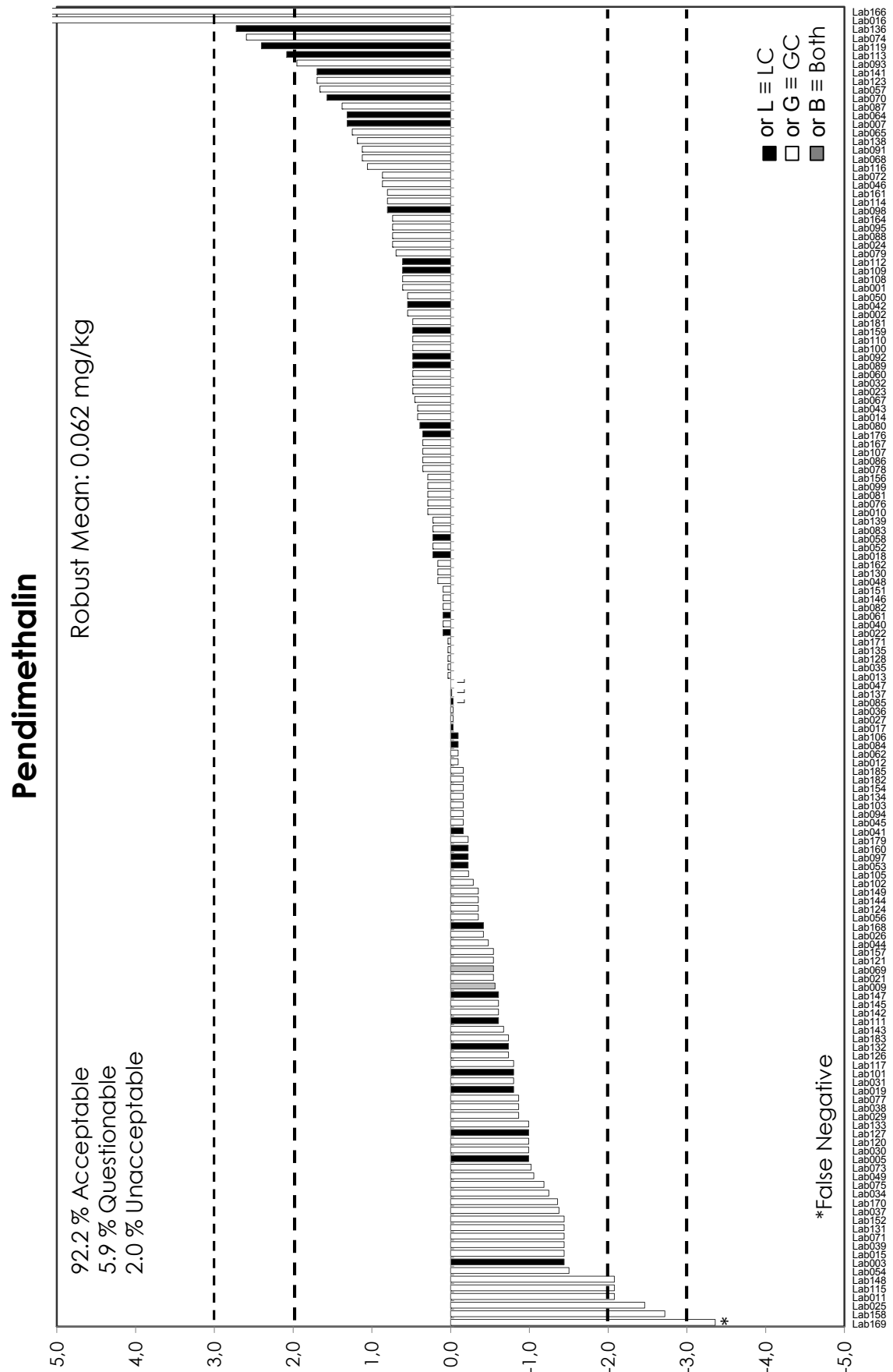
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25%).



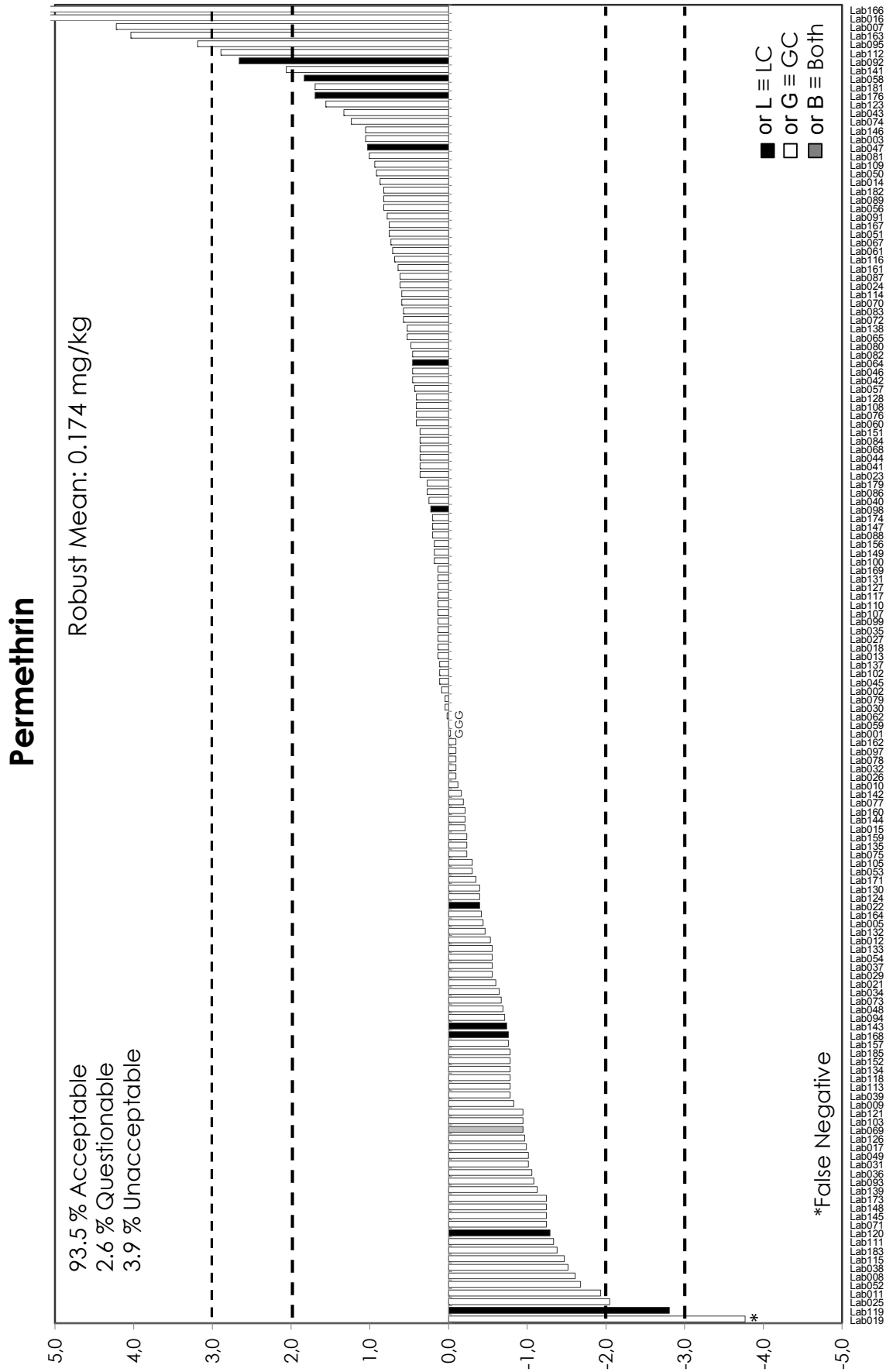
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25%).



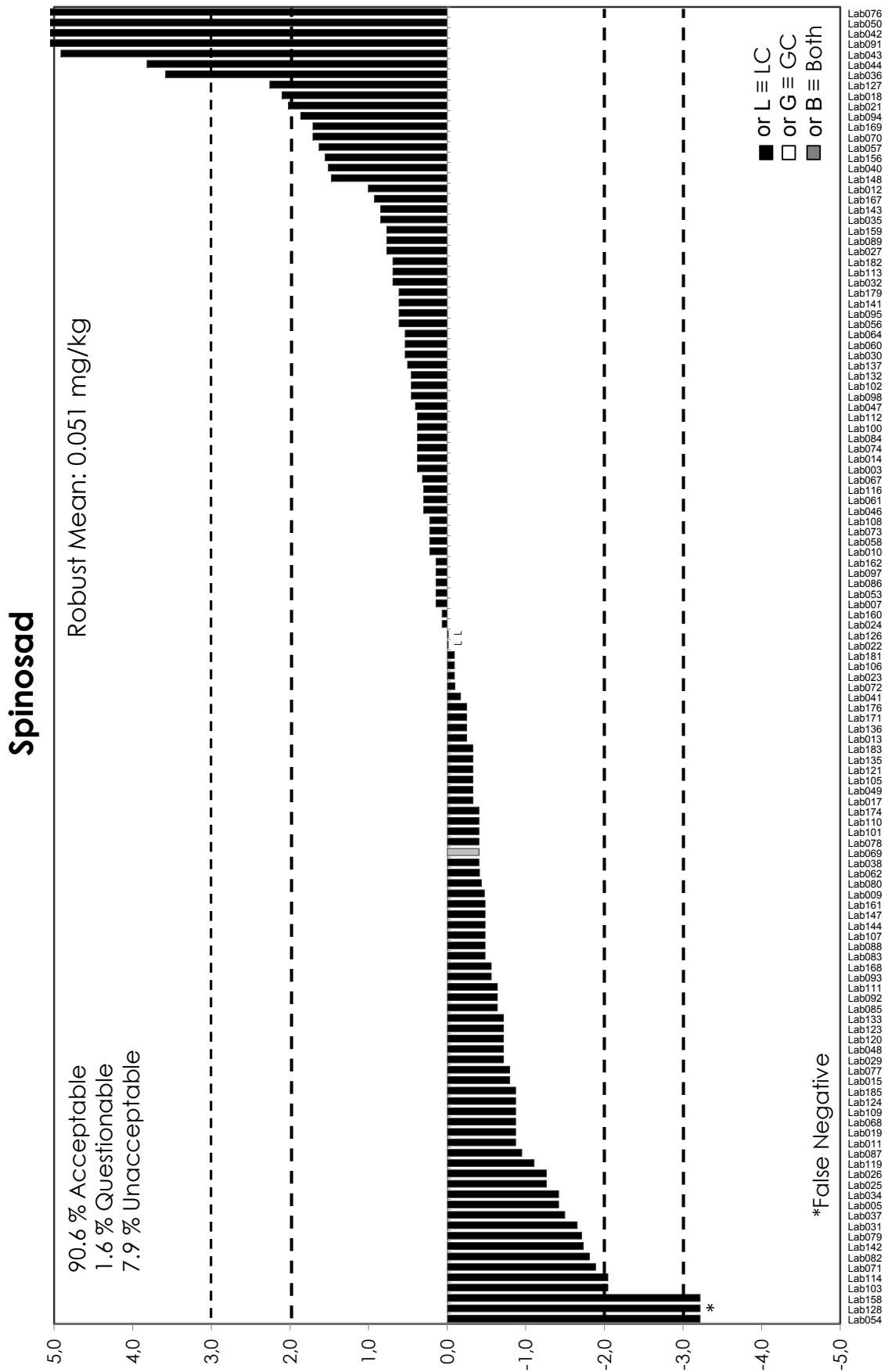
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25%).



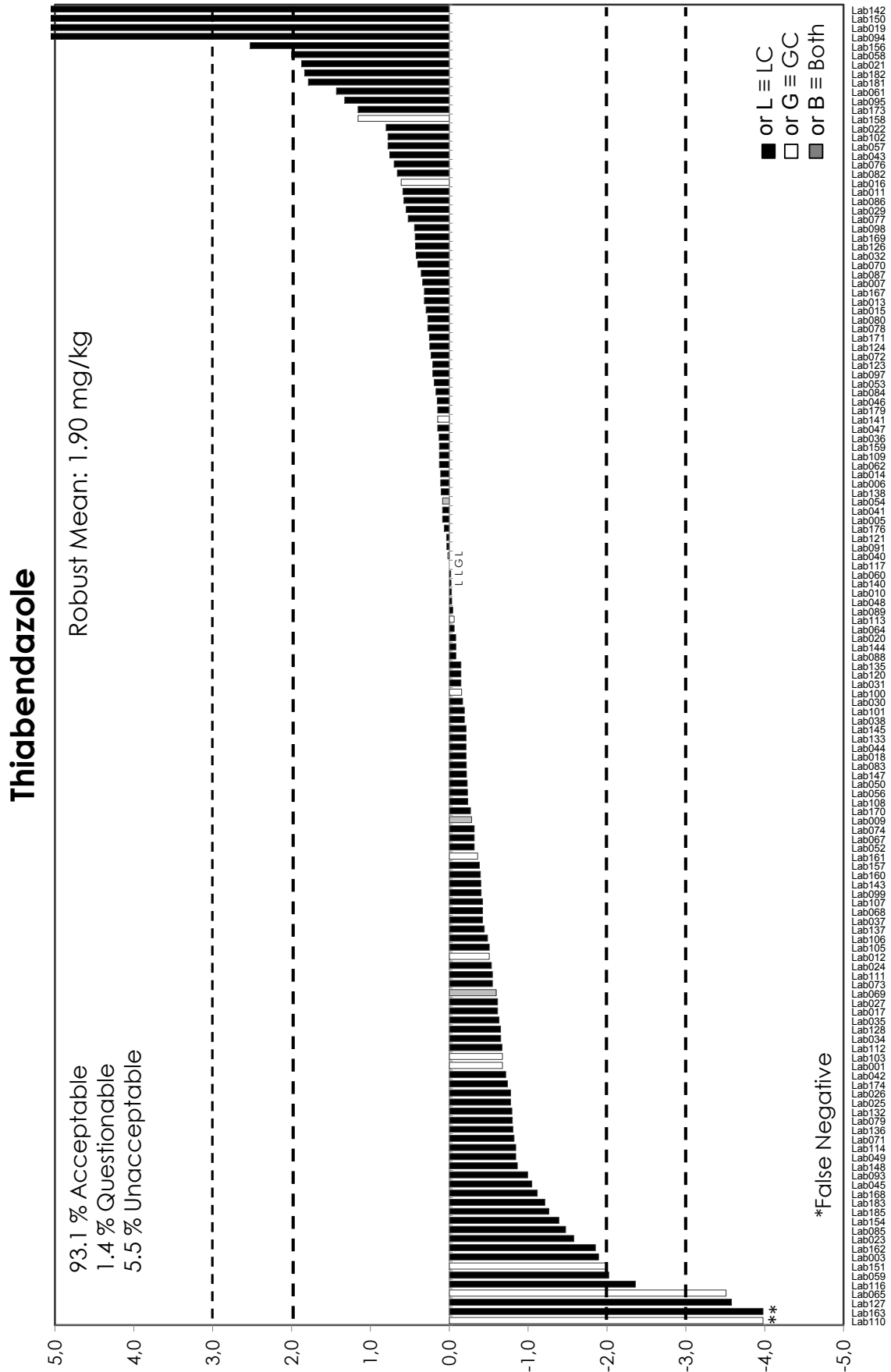
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25%).



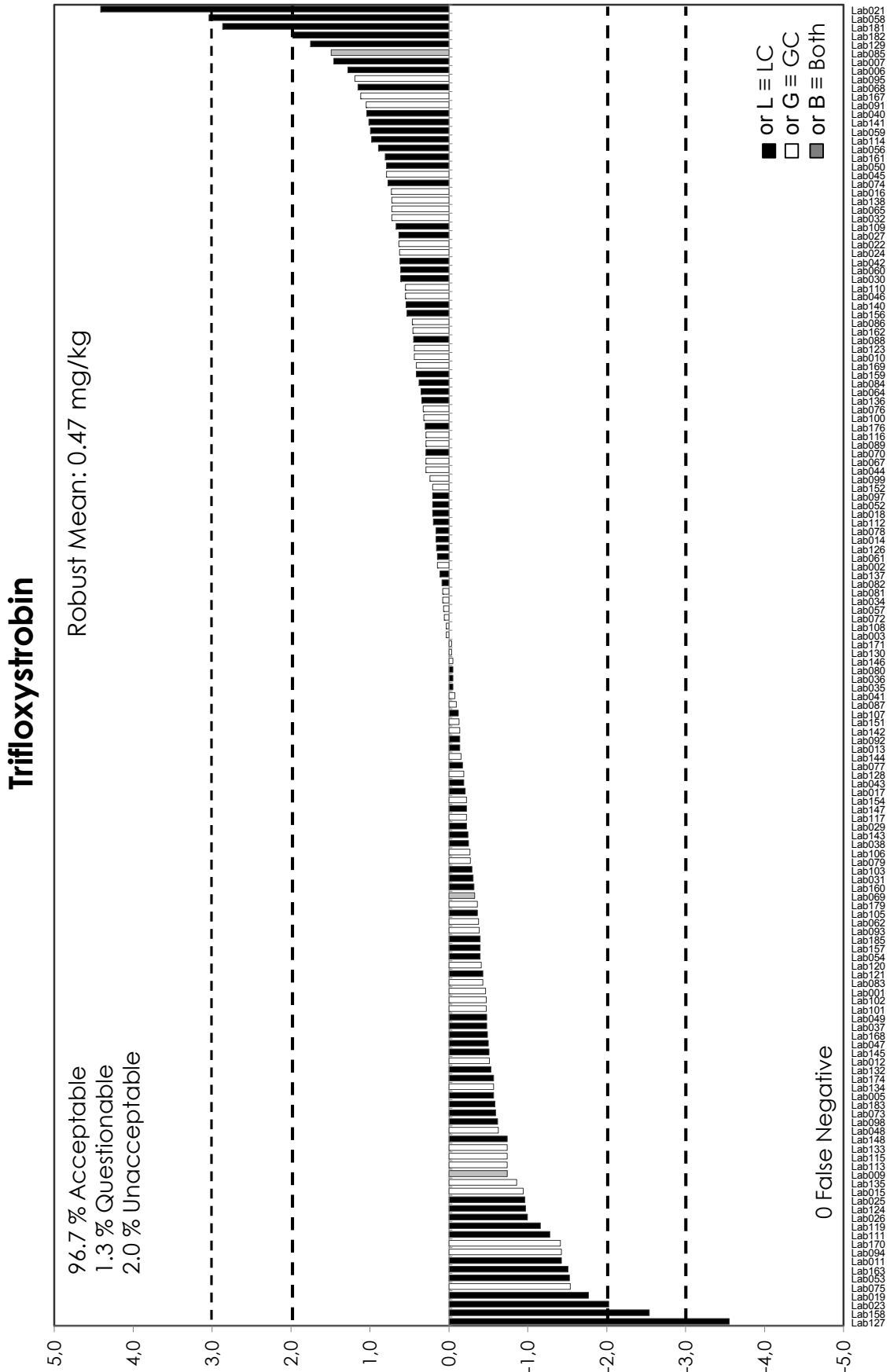
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25%).



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 of the Squared z-scores (AZ²) for laboratories in Category A.

Lab Code	Bupirimate	Carbendazim	Diazinon	Difenoconazole	Diflubenzuron	Methoxyfenozide	Pendimethalin	Permethrin	Spinosad	Thiabendazole	Trifloxystrobin	No. of z-scores	AZ ²
	z-scores												
Lab010	0.8	0.2	0.3	0.3	0.2	0.1	0.3	-0.1	0.2	0	0.4	11	0.1
Lab013	0.1	-0.5	-0.3	0	-0.6	-0.1	0	0.1	-0.2	0.3	-0.1	11	0.1
Lab072	0.6	-0.2	0.2	0	0.3	0	0.9	0.6	-0.1	0.2	0.1	11	0.1
Lab078	0.7	0.4	0.2	0.2	0	0.7	0.4	-0.1	-0.4	0.3	0.2	11	0.1
Lab084	0.2	0	0.5	0.5	-0.4	0.1	-0.1	0.4	0.4	0.2	0.4	11	0.1
Lab086	0.5	-0.1	0.2	0.6	-0.1	0.4	0.4	0.3	0.1	0.6	0.5	11	0.1
Lab097	0.6	-0.1	0.5	0.2	0	0	-0.2	-0.1	0.1	0.2	0.2	11	0.1
Lab100	0.4	0.4	0.3	0.3	-0.6	0	0.5	0.2	0.4	-0.2	0.3	11	0.1
Lab108	0.1	0.2	0.4	-0.1	-0.5	0	0.6	0.4	0.2	-0.2	0	11	0.1
Lab137	-0.3	-0.1	0.2	0	-0.2	-0.3	0	0.1	0.5	-0.4	0.1	11	0.1
Lab147	-0.2	-0.1	0.1	-0.3	-0.1	-0.2	-0.6	0.2	-0.5	-0.2	-0.2	11	0.1
Lab159	0.3	0	0.3	0.2	-0.3	0.2	0.5	-0.2	0.8	0.1	0.4	11	0.1
Lab171	-0.2	-0.2	0.1	-0.3	-0.7	-0.3	0	-0.3	-0.2	0.3	0	11	0.1
Lab014	0.2	-0.6	0	0	0.9	-0.1	0.4	0.9	0.4	0.1	0.2	11	0.2
Lab024	-0.2	-0.7	0.3	-0.4	-0.4	-0.3	0.7	0.6	0.1	-0.5	0.6	11	0.2
Lab035	-0.1	0.8	-0.1	-0.1	-0.7	-0.4	0	0.1	0.8	-0.6	-0.1	11	0.2
Lab046	1	-0.4	0.5	0.2	0.2	0.1	0.9	0.5	0.3	0.2	0.5	11	0.2
Lab048	-0.5	-0.4	-0.2	-0.7	0	-0.2	0.2	-0.7	-0.7	0	-0.6	11	0.2
Lab062	-0.3	0.8	-0.7	0.2	0.1	-0.3	-0.1	0	-0.4	0.1	-0.4	11	0.2
Lab067	0.1	-0.6	0.4	-0.2	-0.5	-0.2	0.5	0.7	0.3	-0.3	0.3	11	0.2
Lab069	-0.7	0.2	0.3	0	-0.3	0.2	-0.5	-0.9	-0.4	-0.6	-0.3	11	0.2
Lab080	0.1	0.7	0.6	0.3	-0.6	-0.2	0.4	0.5	-0.4	0.3	-0.1	11	0.2

APPENDIX 5. Average of the Squared z-scores (AZ²) for laboratories in Category A.

Lab Code	Bupirimate	Carbendazim	Diazinon	Difenoconazole	Diflubenzuron	Methoxyfenozide	Pendimethalin	Permethrin	Spinosad	Thiabendazole	Trifloxystrobin	No. of z-scores	AZ ²
	z-scores												
Lab083	0.5	0.4	-0.2	-0.4	0.2	-0.7	0.2	0.6	-0.5	-0.2	-0.4	11	0.2
Lab102	-0.1	-0.1	-0.3	0.2	-0.6	0.9	-0.3	0.1	0.5	0.8	-0.5	11	0.2
Lab135	-0.2	-0.7	0.3	-0.3	-0.6	-0.3	0	-0.2	-0.3	-0.1	-0.9	11	0.2
Lab144	0.1	0	-0.6	0.9	-0.3	0.1	-0.4	-0.2	-0.5	-0.1	-0.2	11	0.2
Lab012	-0.7	-0.7	-0.5	-0.4	-0.5	0.6	-0.1	-0.5	1	-0.5	-0.5	11	0.3
Lab029	0.6	0.5	-0.3	-0.1	0.6	-0.1	-0.9	-0.6	-0.7	0.5	-0.2	11	0.3
Lab030	-0.2	-0.1	0.6	0.5	0.2	0.7	-1	0	0.5	-0.2	0.6	11	0.3
Lab032	0.8	-0.1	0.5	-0.6	-0.6	0	0.5	-0.1	0.7	0.4	0.7	11	0.3
Lab047	0.5	0.5	0.5	0.3	0.8	0.3	0	1	0.4	0.1	-0.5	11	0.3
Lab056	-0.1	0.4	0.5	0.3	NA	0.4	-0.4	0.8	0.6	-0.2	0.9	10	0.3
Lab064	0.8	-0.8	0.3	0.1	-0.3	0.3	1.3	0.5	0.5	-0.1	0.4	11	0.3
Lab073	-0.3	0.5	0.2	-0.4	0.3	-0.5	-1	-0.7	0.2	-0.5	-0.6	11	0.3
Lab101	-0.4	-1	-1	-0.5	-0.1	-0.2	-0.8	NA	-0.4	-0.2	-0.5	10	0.3
Lab109	0.3	0.4	0.3	0.6	0.2	0.3	0.6	0.9	-0.9	0.1	0.7	11	0.3
Lab121	-0.1	1	-0.1	-0.2	NA	-0.4	-0.5	-0.9	-0.3	0	-0.4	10	0.3
Lab160	-0.3	-0.6	-0.5	-0.5	-1	-0.9	-0.2	-0.2	0.1	-0.4	-0.3	11	0.3
Lab179	0.2	-0.7	-0.2	1	0.8	0.8	-0.2	0.3	0.6	0.1	-0.4	11	0.3
Lab009	-0.7	-0.7	-0.6	-1	-0.1	-0.4	-0.6	-0.8	-0.5	-0.3	-0.7	11	0.4
Lab049	-0.6	-0.3	-0.5	0.5	-0.1	-0.1	-1.1	-1	-0.3	-0.8	-0.5	11	0.4
Lab053	-1	-0.1	0.2	-0.1	-1.1	-0.3	-0.2	-0.3	0.1	0.2	-1.5	11	0.4
Lab082	0	0.4	0	0	-0.7	-0.2	0.1	0.5	-1.8	0.7	0.1	11	0.4
Lab126	1	-0.3	-0.1	0.3	-1.3	0.1	-0.7	-1	0	0.4	0.2	11	0.4
Lab143	0.5	-1	0.8	0.5	0.5	0.3	-0.7	-0.7	0.8	-0.4	-0.2	11	0.4

APPENDIX 5. Average of the Squared z-scores (AZ²) for laboratories in Category A.

Lab Code	Bupirimate	Carbendazim	Diazinon	Difenoconazole	Diflubenzuron	Methoxyfenozide	Pendimethalin	Permethrin	Spinosad	Thiabendazole	Trifloxystrobin	No. of z-scores	AZ ²
	z-scores												
Lab161	-0.4	0.3	0.7	0.3	-0.8	-0.8	0.8	0.6	-0.5	-0.4	0.8	11	0.4
Lab018	-0.1	-0.2	0.3	0.6	0.4	0.4	0.2	0.1	2.1	-0.2	0.2	11	0.5
Lab040	-0.1	-0.3	0.1	0	0.5	1.4	0.1	0.2	1.5	0	1	11	0.5
Lab087	1	-0.5	-0.1	-0.6	-0.1	-0.2	1.4	0.6	-1	0.4	-0.1	11	0.5
Lab105	0	-0.6	-0.3	-0.2	-2.1	0	-0.2	-0.3	-0.3	-0.5	-0.4	11	0.5
Lab167	0.8	-0.5	0.5	0.9	0.2	-0.1	0.4	0.8	0.9	0.3	1.1	11	0.5
Lab168	-0.1	-1	-0.6	-0.7	-1	-0.5	-0.4	-0.8	-0.6	-1.1	-0.5	11	0.5
Lab176	0.5	0.9	0.6	0.4	0.2	0.5	0.4	1.7	-0.2	0.1	0.3	11	0.5
Lab022	0.1	1.8	-0.3	0.2	1	-0.7	0.1	-0.4	0	0.8	0.6	11	0.6
Lab041	0.3	0.6	-0.4	0.4	2	1	-0.2	0.4	-0.2	0.1	-0.1	11	0.6
Lab077	0.5	-0.5	-1.1	-0.3	1.8	0.4	-0.9	-0.2	-0.8	0.5	-0.2	11	0.6
Lab089	0.1	0.3	0.5	0.7	2	0.7	0.5	0.8	0.8	0	0.3	11	0.6
Lab111	-0.5	-0.9	-0.1	-0.5	0.4	-0.9	-0.6	-1.3	-0.6	-0.5	-1.3	11	0.6
Lab120	-0.7	0.8	-0.7	-0.3	-0.9	-0.7	-1	-1.3	-0.7	-0.1	-0.4	11	0.6
Lab031	-0.4	-0.8	-0.6	-0.6	-0.9	NA	-0.8	-1	-1.7	-0.1	-0.3	10	0.7
Lab037	-0.6	0.8	-0.6	-0.7	-0.7	-0.1	-1.4	-0.6	-1.5	-0.4	-0.5	11	0.7
Lab060	0.8	-0.2	2.1	-0.3	-0.1	1.4	0.5	0.4	0.5	0	0.6	11	0.7
Lab068	0.8	0.7	1.1	0.7	0.8	0.9	1.1	0.4	-0.9	-0.4	1.1	11	0.7
Lab132	-0.5	2.4	0	-0.5	0	-0.5	-0.7	-0.5	0.5	-0.8	-0.5	11	0.7
Lab162	0	0.1	0.2	0.6	-1.5	-1.1	0.2	-0.1	0.1	-1.9	0.5	11	0.7
Lab005	-1.3	0.5	-1.4	-0.8	-0.5	-0.5	-1	-0.4	-1.4	0.1	-0.6	11	0.8
Lab015	0.2	-1.4	-1	-0.2	-0.7	1.3	-1.4	-0.2	-0.8	0.3	-0.9	11	0.8
Lab026	-0.8	-0.3	-1.3	-1	-1.1	-1	-0.4	-0.1	-1.3	-0.8	-1	11	0.8

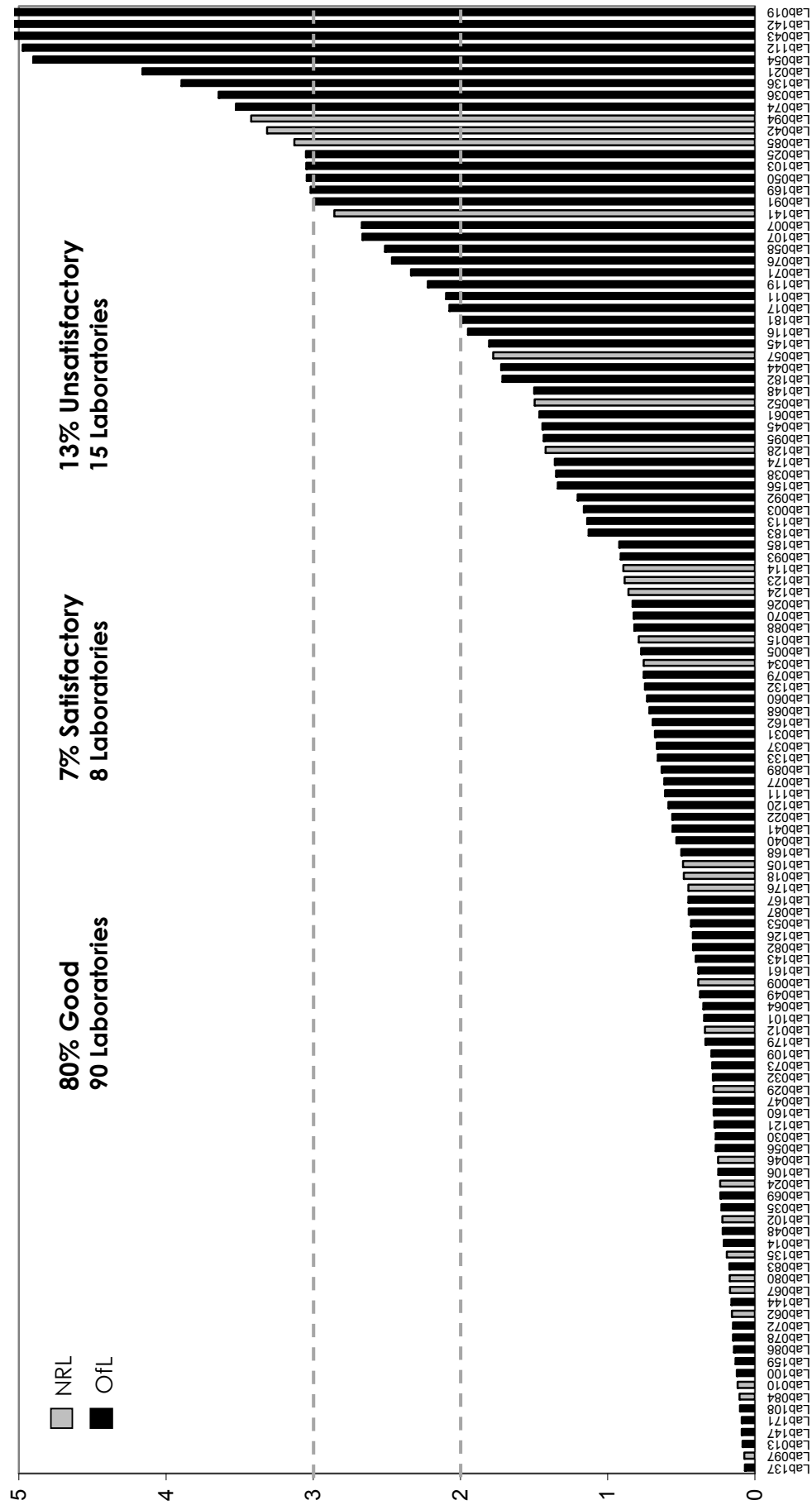
APPENDIX 5. Average of the Squared z-scores (AZ²) for laboratories in Category A.

Lab Code	Bupirimate	Carbendazim	Diazinon	Difenoconazole	Diflubenzuron	Methoxyfenozide	Pendimethalin	Permethrin	Spinosad	Thiabendazole	Trifloxystrobin	No. of z-scores	AZ ²
	z-scores												
Lab034	-0.6	-0.8	-0.1	-0.4	NA	-1.4	-1.2	-0.6	-1.4	-0.7	0.1	10	0.8
Lab070	0.4	-0.2	1.4	0.8	0.3	0.6	1.6	0.6	1.7	0.4	0.3	11	0.8
Lab079	0.1	-0.4	0.3	-0.8	-1.4	-1.2	0.7	0	-1.7	-0.8	-0.3	11	0.8
Lab088	0.4	2.6	-0.5	0.4	0.4	-0.7	0.7	0.2	-0.5	-0.1	0.4	11	0.8
Lab093	-1.6	-0.9	-0.1	-0.2	0.2	-0.3	2	-1.1	-0.6	-1	-0.4	11	0.9
Lab114	0.4	0.3	0.5	-1.1	NA	-0.6	0.8	0.6	-2	-0.8	1	10	0.9
Lab123	0.9	0.1	0.8	0.6	1.2	0.7	1.7	1.6	-0.7	0.2	0.4	11	0.9
Lab124	-1	0.6	-1	-1	-0.7	1.9	-0.4	-0.4	-0.9	0.2	-1	11	0.9
Lab185	-0.1	-0.2	-0.1	-0.8	2.4	-0.8	-0.2	-0.8	-0.9	-1.3	-0.4	11	0.9
Lab113	-0.1	2.1	-0.3	-0.2	NA	0.9	2.1	-0.8	0.7	-0.1	-0.7	10	1.1
Lab183	-0.2	-0.8	-0.6	-0.6	2.6	-0.3	-0.7	-1.4	-0.3	-1.2	-0.6	11	1.1
Lab156	-1.6	-0.6	0.5	0.8	1.3	0.2	0.3	0.2	1.6	2.5	0.5	11	1.3
Lab038	-0.4	-1.7	-1	0.1	2.7	-0.4	-0.9	-1.5	-0.4	-0.2	-0.2	11	1.4
Lab095	0.3	-0.5	0.8	0.5	-0.5	-0.2	0.7	3.2	0.6	1.3	1.2	11	1.4
Lab128	0.8	-1.6	-1	-0.1	-0.6	-0.2	0	0.4	-3.2	-0.7	-0.2	10	1.4
Lab061	1.9	2.5	1.8	0.4	-0.5	0.1	0.1	0.7	0.3	1.4	0.1	11	1.5
Lab148	-1.1	0	-1.7	-1	NA	-0.7	-2.1	-1.2	1.5	-0.9	-0.7	10	1.5
Lab044	-0.6	-0.5	-1.2	0.5	-0.9	0.9	-0.5	0.4	3.8	-0.2	0.3	11	1.7
Lab182	-0.4	0.2	-0.2	0.9	1.1	2.9	-0.2	0.8	0.7	1.8	2	11	1.7
Lab057	0.3	1.5	1.5	0.3	2.9	0.5	1.7	0.4	1.6	0.8	0.1	11	1.8
Lab116	0.4	0.9	1.1	0.3	-3.4	0.7	1.1	0.7	0.3	-2.4	0.3	11	1.9
Lab181	-0.2	0.3	0.5	0.6	2.5	0.6	0.5	1.7	-0.1	1.8	2.9	11	2
Lab011	-1.3	-0.8	-2.3	-2	0.5	-0.1	-2.1	-1.9	-0.9	0.6	-1.4	11	2.1

APPENDIX 5. Average of the Squared z-scores (AZ²) for laboratories in Category A.

Lab Code	Bupirimate	Carbendazim	Diazinon	Difenoconazole	Diflubenzuron	Methoxyfenozide	Pendimethalin	Permethrin	Spinosad	Thiabendazole	Trifloxystrobin	No. of z-scores	AZ ²
	z-scores												
Lab017	-3.1	-1.6	-2.6	0.1	NA	-0.2	0	-1	-0.3	-0.6	-0.2	10	2.1
Lab119	-0.9	-2.1	-0.1	0.4	-0.2	-0.8	2.4	-2.8	-1.1	NA	-1.2	10	2.2
Lab071	-1.9	0.9	-1.9	-1.7	-2.1	-0.1	-1.4	-1.2	-1.9	-0.8	NA	10	2.3
Lab058	-0.1	0.5	0.4	1	2.6	0.7	0.2	1.8	0.2	2	3	11	2.5
Lab076	0.1	-0.3	0	0.3	-0.1	1	0.3	0.4	5	0.7	0.3	11	2.5
Lab007	1.3	-0.2	1.2	0.8	NA	0.9	1.3	4.2	0.1	0.3	1.5	10	2.7
Lab107	-0.4	-0.8	5	-0.1	NA	-0.6	0.4	0.1	-1	-0	-0	10	2.7
Lab025	-2.4	-0.9	-2.5	-2.2	-1.4	-0.9	-2.5	-2	-1	-1	-1	11	3
Lab050	NA	0.2	1.5	0.1	0.4	1.1	0.5	0.9	5	-0	0.8	10	3
Lab091	1.7	0.2	0.4	0.3	-1.3	-0.1	1.1	0.8	5	0	1	11	3
Lab103	-0.8	5	0.7	-0.5	-0.3	-1.2	-0.2	-0.9	-2	-1	-0	11	3
Lab085	0.1	5	0.8	-0.2	-0.2	-0.8	0	NA	-1	-2	1.5	10	3.1
Lab042	3.1	-0.3	0	0.4	-0.1	0.2	0.5	0.5	5	-1	0.6	11	3.3
Lab094	0.1	0.3	0.4	-1.5	1.4	-1.5	-0.2	-0.7	1.9	5	-1	11	3.4
Lab074	0.3	0.6	1.9	0.3	5	0.7	2.6	1.2	0.4	-0	0.8	11	3.5
Lab036	-0.8	0.4	0	-0.4	5	0.2	0	-1.1	3.6	0.1	-0	11	3.6
Lab136	0.1	0.8	0.3	0.1	5	2.2	2.7	NA	-0	-1	0.3	10	3.9
Lab021	0.2	2.1	-0.1	0	-0.4	3.7	-0.5	-0.6	2	1.9	4.4	11	4.2
Lab054	-0.8	3.7	-0.6	-0.4	5	-1	-1.5	-0.6	-3	0.1	-0	10	4.9
Lab112	5	-2.2	3.3	0.2	-1.6	1.4	0.6	2.9	0.4	-1	0.2	11	5
Lab043	2.5	-0.5	-1	-0.9	5	-0.3	0.4	1.3	4.9	0.8	-0	11	5.5
Lab142	0.1	5	-0.2	0.1	2.6	-0.3	-0.6	-0.2	-2	5	-0	11	5.5

EUPT-FV17 AZ² – Graphical Representation for Laboratories in Category A



GENERAL PROTOCOL

for EU Proficiency Tests for 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-SANTE⁵ by the four European Union Reference Laboratories (EURLs) responsible for pesticide residues in food and feed. These EUPTs are directed at laboratories belonging to the Network⁶ of National Reference Laboratories (NRLs) and Official Laboratories (OfLs) of the EU Member States. OfLs from EFTA countries and EU-Candidate countries are also welcome to participate in the EUPTs. OfLs from Third countries may be permitted to participate on a case-by-case basis.

The following four EURLs for pesticide residues were appointed by DG-SANTE based on regulation 882/2004/EC⁷:

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

The aim of these EUPTs is to obtain information regarding the quality, accuracy and comparability of pesticide residue data in food and feed reported to the European Union within the framework of the national control programmes and the EU multiannual co-ordinated control programme⁸. Participating laboratories will be provided with an assessment of their analytical performance that they can use to demonstrate their analytical performance and compare themselves with other participating laboratories.

⁵ DG-SANTE = European Commission, Directorate-General for Health and Food Safety

⁶ For more information about the EURL/NRL/OfL-Network please refer to the EURL-Web-portal under: <http://www.eurl-pesticides.eu>

⁷ 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

⁸ European Commission Proficiency Tests for Pesticide Residues in Fruits and Vegetables, Trends in Analytical Chemistry, 2010, 29 (1), 70 – 83.

ANNEX 1. Protocols and Target list of pesticides to be sought.

EUPT-Organizers and Scientific Committee

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

An **Organising Team** is appointed by the EURL(s) in charge. This team is responsible for all administrative and technical matters concerning the organisation of the PT, e.g. the PT-announcement, production of Test Item and Blank Material, the undertaking of homogeneity and stability tests, packing and shipment of the Test Item and Blank Material, handling and evaluation of the results and method information submitted by the participants and the drafting of the preliminary and final reports.

To complement the internal expertise of the EURLs, a group of external consultants that form the **EUPT-Scientific Committee** (EUPT-SC)⁹ has been established and approved by DG-SANTE. The EUPT-SC consists of expert scientists with many years of experience in PTs and/or pesticide residue analysis. The actual composition of the EUPT-SC, the affiliation of each member is shown on the EURL-Website. The members of the EUPT-SC will also be listed in the Specific Protocol and the Final Report of each EUPT.

The EUPT-SC is made up of the following two subgroups:

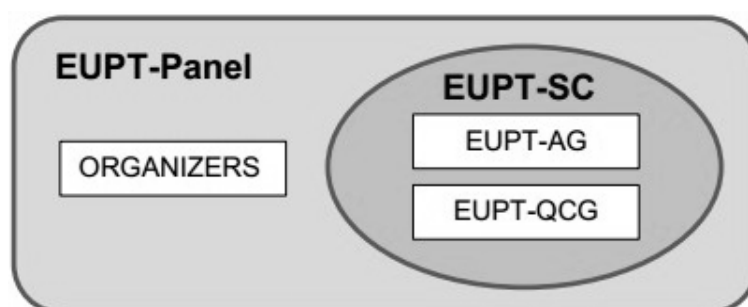
- a) An independent **Quality Control Group** (EUPT-QCG) and
- b) An **Advisory Group** (EUPT-AG) ,

The EUPT-SC's role is to help the organisers make decisions regarding the EUPT design: the selection of the commodity; 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 (in anonymous form); and the drafting of documents such as the General and Specific PT Protocols and the Final EUPT-Reports.

The EUPT-QCG has the additional function of supervising the quality of EUPTs and of assisting the EURLs in confidential aspects such as the choice of the pesticides to be present in the Test Item and the concentrations at which they should be present.

The EUPT-SC typically meets once a year, after the EUPTs of all four pesticide EURLs have been conducted, to discuss the evaluation of the EUPT-results and consult the EURLs in their decision making. Upcoming EUPTs are also planned during these meetings.

The EUPT-Organising Team and the EUPT-SC together form the **EUPT-Panel**.



⁹ Link to the List of current members of the EUPT Scientific Committee:
<http://www.eurl-pesticides.eu/library/docs/allcrl/EUPT-SC.pdf>

ANNEX 1. Protocols and Target list of pesticides to be sought.

The decisions of the EUPT-Panel will be documented.

The present EUPT General Protocol was jointly drafted by the EUPT- SC and the EURLs and was approved by DG-SANTE.

EUPT Participants

Within the European Union all NRLs operating in the same area as the organising EURL, as well as all OfLs whose scope overlaps with that of the EUPT, are legally obliged to participate in EUPTs. The legal obligation of NRLs and OfLs to participate in EUPTs arises from:

- Art. 28 of Reg. 396/2005/EC¹⁰ (for all OfLs analyzing for pesticide residues within the framework of official controls¹¹ in food or feed)
- Art. 33 of Reg. 882/2004/EC (for all NRLs)

The four EURLs will annually issue and distribute, via the EURL-website, a joint list of all OfLs that must participate in each of the EUPTs to be conducted within a given year. The list of obliged labs will be updated every year to take account of any changes in the lab profiles. Interim updates will be issued to eliminate any possible errors.

NRLs are responsible for checking whether all relevant OfLs within their network are included in the list of obligated laboratories and whether the contact information and commodity-scopes are correct.

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

Labs that are obliged to participate in a given EUPT, and that are not able to participate, must provide the reasons for their non-participation without prejudice of any legal action taken against them for not participating. This also applies to any participating laboratories that then fail to report results.

Confidentiality and Communication

The proprietor of all EUPT data is DG-SANTE and as such has access to all information.

For each EUPT, the laboratories are given a unique code (lab code), initially only known to themselves and the Organisers. In the final EUPT-Report, the names of participating laboratories will not be linked to their laboratory codes. It should be noted, however, that the Organisers, at the request by DG-SANTE, may present the EUPT-results on a country-by-country basis. It may therefore be possible that a link between codes and laboratories could be made, especially for those countries where only one laboratory has participated. Furthermore, the EURLs reserve the

¹⁰ 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.

¹¹ Official controls in the sense of Reg. 882/2004/EC This includes labs involved in controls within the framework of national and/or EU-controlled programmes as well as labs involved in import controls according to Regulation 669/2009/EC.

ANNEX 1. Protocols and Target list of pesticides to be sought.

right to share EUPT results and codes amongst themselves: for example, for the purpose of evaluating overall lab or country performance as requested by DG-SANTE.

As laid down in Regulation 882/2004, NRLs are responsible for evaluating and improving their own OfL-Network. On request from the NRLs, the EURLs will provide them with the PT-codes of the participating OfLs belonging to their OfL-Network. This will allow NRLs to follow the participation and performance of the laboratories within their network.

Communication between participating laboratories during the test on matters concerning a PT exercise is not permitted from the start of the PT exercise until the distribution of the preliminary report.

For each EUPT the organising EURL prepares a specific EUPT-Website where all relevant documents in their latest version are linked.

The official language used in all EUPTs is English.

Announcement / Invitation Letter

At least 3 months before the Test Item of a given EUPT is distributed to the laboratories the EURLs will publish an Announcement/Invitation letter on the EURL-web-portal and distribute it via e-mail to the NRL/OfL mailing list available to the EURLs. This letter will inform about the commodity to be used as Test Item, as well as links to the tentative EUPT-Target Pesticide List and the tentative EUPT-Calendar.

Target Pesticide List

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

Labs must express their results as stated in the Target Pesticides List.

Specific Protocol

For each EUPT the organizing EURL will publish a Specific Protocol at least 2 weeks before the Test Item is distributed to the participating laboratories. The Specific Protocol will contain all the information previously included in the Invitation Letter but in its final version, information on payment and delivery, instructions on how to handle the Test Item upon receipt and on how to submit results, as well as any other relevant information.

Homogeneity of the Test Item

The Test Item will be tested for homogeneity typically before distribution to participants. The homogeneity tests involve the analysis of two replicate analytical portions, taken from at least ten

ANNEX 1. Protocols and Target list of pesticides to be sought.

randomly chosen units of treated Test Item. Both, sample preparation and measurements should be conducted in random order.

The homogeneity test data are statistically evaluated according to the International Harmonized Protocols published by ISO and IUPAC. The acceptance criterion for the Test Items to be sufficiently homogenous for the Proficiency Test is that s_{sam}^2 is less than c with s_{sam} being the between-bottle sampling standard deviation and $c = F_1 \times \sigma_{all}^2 + F_2 \times s_{an}^2$. F_1 and F_2 are constants, with values of 1.88 and 1.01, respectively, if 10 samples are used. $\sigma_{all}^2 = 0.3 \times \text{FFP-RSD}^{12}$ (25 %) \times the analytical sampling mean for all pesticides, and s_{an} is the estimate of the analytical standard deviation.

The results of all homogeneity tests are presented to the EUPT-SC. In special cases where the above homogeneity test criteria are not met, the EUPT-SC considering all relevant aspects (e.g. the homogeneity results of other pesticides spiked at the same time, the overall distribution the participants' results, the analytical difficulties faced during the test, knowledge of the analytical behaviour of the pesticide question) may decide to overrule the test. The reasons of this overruling have to be transparently explained in the Final EUPT-Report.

Stability of the analytes contained in the Test Item

The Test Items will also be tested for stability - according to ISO 13528, Annex B. The time delay between the first and the last stability test must exceed the period of the EUPT-exercise. Typically the first analysis is carried out shortly before the shipment of the Test Items and the last one shortly after the deadline for submission of results. To better recognise trends and gain additional certainty one or more additional tests may be conducted by the Organisers. At least 6 sub-samples (analytical portions) should be analysed on each test day (e.g. 2 analytical portions withdrawn from three randomly chosen containers OR 6 portions withdrawn from a single container). In principle all pesticides contained in the Test Item should be checked for stability. However, in individual cases, where sufficient knowledge exists that the stability of a certain analyte is very unlikely to be significantly affected during storage (e.g. based on experience from past stability tests or knowledge of its physicochemical properties), the Organisers, after consultation with the EUPT-QCG, may decide to omit a specific stability test. The EUPT-SC will finally decide whether analytes for which the stability test was not undertaken will be included in the final report, considering all relevant aspects such as the distribution of the participant's results (CVs*).

A pesticide is considered to be adequately stable if $|x_1 - y_1| \leq 0.3 \times \sigma$, where x_1 is the mean value of the first stability test, y_1 the mean value of the last stability test and σ the standard deviation used for proficiency assessment (typically 25% of the assigned value).

The results of all stability tests are presented to the EUPT-SC. In special cases where the above stability test criteria are not met, the EUPT-SC considering all relevant aspects (e.g. the past experience with the stability of the compound, the overall distribution the participants' results, the

¹² FFP-RSD = fit for purpose relative standard deviation.

ANNEX 1. Protocols and Target list of pesticides to be sought.

analytical difficulties faced during the test, knowledge about the analytical behavior of the pesticide question) may decide to overrule the test. The reasons of this overruling will be transparently explained in the Final EUPT-Report.

The Organizers may also decide to conduct additional stability tests at different storage conditions than those recommended to the participants e.g. at ambient temperature.

Considering knowledge about the expected susceptibility of pesticides in the Test Item to possible losses, the organizers will chose the shipment conditions to be such that pesticide losses are minimized (e.g. shipment of frozen samples, addition of dry ice). As shipment time can differ between labs/countries it is recommended that the organizers conduct additional stability tests at conditions simulating shipment. Should critical losses be detected for certain pesticides EUPT-SC should be informed (or the EUPT-QCG before or during the test). Case-by-case decisions may be taken considering all relevant aspects including the shipment time of the samples to each laboratory.

Methodologies to be used by the participants

Participating laboratories are instructed to use the analytical procedure(s) that they would routinely employ in official control activities (monitoring etc.). Where an analytical procedure has not yet been established routinely this should be stated.

General procedures for reporting results

Participating laboratories are responsible for reporting their own quantitative results to the Organiser within the stipulated deadline. Any pesticide that was targeted by a participating laboratory should be reported as "analysed". Each laboratory will be able to report only one result for each analyte detected in the Test Item. The concentrations of the pesticides detected should be expressed in 'mg/ kg' unless indicated otherwise in the specific protocol.

The Test Item is intentionally treated with pesticides whereas the Blank Material is analysed to ensure that it does not contain any of the pesticides in the Target Pesticides List, at or above, the specified MRRs. Both the Test Item and Blank Material have to be analysed by the participating laboratories and any pesticide detected in them must be reported.

Correction of results for recovery

According to the Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed¹³, 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, but also exhibiting good precision). Other approaches for recovery correction explicitly allowed in the SANCO document are the use of stable isotope labelled analogues of the target analytes used as Internal Standards (ISTDs), the

¹³ Document N° SANCO/12571/2013; Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed

ANNEX 1. Protocols and Target list of pesticides to be sought.

'procedural calibration' approach as well as the approach of 'standard addition' with additions of analyte(s) being made to analytical portions. Where reported residue data have been automatically adjusted for recovery by the method, or have subsequently been adjusted using a recovery factor, this must be indicated on the specific field of the 'Result Submission Form'. Results may be corrected for recovery only in cases where this correction is applied in routine practice (including cases of MRL-violations). Laboratories are required to report whether their results were adjusted for recovery and, if a recovery factor was used, the recovery (in percentage) must also be reported. No recovery data are required where correction for recovery is automatic by using the 'standard addition 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 actual approach that was followed.

Methodology information

All laboratories are requested to provide information on the analytical method(s) they have used. A compilation of the methodology information submitted by all participants is presented in an Annex of the final report. Where necessary the methods are evaluated and discussed, especially in those cases where the result distribution is not unimodal or very broad (e.g. CVs*>35%). 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 results

These are results of pesticides from the Target Pesticides List, that are reported, at or above, their respective MRRL although they 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 pesticides. In certain instances, case-by-case decisions by the EUPT-Panel may be necessary.

Any results reported lower than the MRRL will not be considered as false positives, even though these results should not have been reported.

– False Negatives results

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

ANNEX 1. Protocols and Target list of pesticides to be sought.

In cases of the assigned value being less than a factor of 4 times the MRRL, false negatives will typically not be assigned. The EUPT-Panel may decide to take case-by-case decisions in this respect after considering all relevant factors such as the result distribution and the reporting limits of the affected labs.

– Estimation of the assigned value (x^*)

In order to minimise the influence of out-lying results on the statistical evaluation, the assigned value (= consensus concentration) will typically be estimated using robust statistics as described in ISO 13528:2009-01¹⁴. In special justifiable cases, the EUPT-Panel may decide to eliminate certain results traceably associated with gross errors (see "Omission or Exclusion of results" below) or to use only the results of a subgroup consisting of laboratories that have repeatedly demonstrated good performance for the specific compound in the past.

– Omission or Exclusion of results

Before estimating the assigned value results associated with obvious mistakes have to be examined to decide whether they should be removed from the population. Such gross errors may include incorrect recording (e.g. due to transcription errors by the participant, decimal point faults or transposed digits, incorrect unit), calculation errors (e.g. missing factors), analysis of a wrong sample/extract (e.g. a spiked blank), use of wrong concentrations of standard solutions, incorrect data processing (e.g. integration of wrong peak), major deviations from the analytical procedure, inappropriate storage or transport conditions (in case of susceptible compounds), and the use of inappropriate procedures that demonstrably lead to significantly biased results (e.g. due to degradation or incomplete extraction). Where the Organisers (e.g. after the publication of the preliminary report) receive information of such gross errors, having a significant impact on a generated result, the affected results will be examined on a case-by-case basis to decide whether, or not, they should be excluded from the population used for robust statistics. Even results that cannot be specifically identified as outliers might be excluded. All decisions to omit/exclude results will be discussed with the EUPT-SC and the reasoning for the omission of each result clearly stated in the final EUPT-Report. However, z-scores will be calculated for all results irrespective of the fact that they were omitted from the calculation of the assigned value.

Omitted results might be interesting as they might give indications about possible source(s) of errors. The Organisers will thus ask the relevant lab(s) to provide feedback on possible sources of errors (see also "follow-up activities").

Any exclusion of results from the population is to be discussed within the EUPT-SC and the reasoning behind is to be revealed in the EUPT-final report.

¹⁴ DIN ISO 13528:2009-01, Statistical methods for use in proficiency testing by interlaboratory comparisons, International Organization for Standardization. Therein a specific robust method for determination of the consensus mean and standard deviation without the need for removal of deviating results is described (Algorithm A in Annex C).

ANNEX 1. Protocols and Target list of pesticides to be sought.

Uncertainty of the assigned value

The uncertainty of the assigned values x_i is calculated according to ISO 13528:2009-01 as:

$$x_i = 1.25 \frac{s^*}{\sqrt{n}}$$

Where s^* is the robust standard deviation and n is the number of results.

In certain cases and considering all relevant factors (e.g. the result distribution, multimodality), the number of submitted results, information regarding analyte homogeneity/stability, information regarding the use of methodologies that might produce a bias that were used by the participants), the EUPT-Panel may consider the assigned value of a specific analyte to be too uncertain and decide that the results should not be evaluated, or only evaluated for informative purposes. The provisions of ISO 13528:2009-01 concerning the uncertainty of the assigned value will be taken into account.

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

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

$$\text{FFP-}s_i = b \cdot x_i^* \quad \text{with } b = 0.25 \text{ (25 \% FFP-RSD)}$$

The percentage FFP-RSD is set at 25% based on experience from results of previous EUPTs¹⁵. 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.

For informative purposes the robust relative standard deviation (CVs^{*}) is calculated according to ISO 13528:2009-01; Chapter 5.6 (Consensus value from participants) following Algorithm A in Annex C.

– **z-scores**

This parameter is calculated using the following formula:

$$z_i = (x_i - x_i^*) / \text{FFP-}s_i$$

Where: x_i is the value reported by the laboratory, x_i^* the assigned value, and **FFP- s_i** the standard deviation for each pesticide (i). Z-scores will be rounded to one decimal place. For the calculation of combined z-scores (see below) the original z-scores will be used and rounded to one decimal place after calculation.

Any z-scores > 5 will be typically reported as '> 5' and a value of '5' will be used to calculate combined z-scores (see below).

Z-scores will be interpreted in the following way, as is set in the ISO 17043:2010¹⁶:

¹⁵Comparative 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 Target list of pesticides to be sought.

$ z \leq 2$	Acceptable
$2 < z < 3$	Questionable
$ z \geq 3$	Unacceptable

For results considered as 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 decide whether, or not, these values should appear in the z-score histograms.

– **Category A and B classification**

The EUPT-Panel will decide how to classify the laboratories into two categories - A or B. Currently, laboratories that have detected and quantified 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 can therefore be classified into Category A. The 90 % criterion will be applied following Table 1.

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	N - 2
14	12.6	13	
15	13.5	13	
16	14.4	14	
17	15.3	15	
18	16.2	16	
19	17.1	17	
20	18.0	18	
21	18.9	19	N - 3
22	19.8	20	
23	20.7	21	
24	21.6	22	
25	22.5	22	
26	23.4	23	

¹⁶ ISO/IEC 17043:2010. Conformity assessment -- General requirements for proficiency testing

ANNEX 1. Protocols and Target list of pesticides to be sought.

– Overall performance of laboratories – combined z-scores

For evaluation of the overall performance of laboratories within Category A, the Average of the Squared z-Score (AZ²)^{17,18} (see below) will be used. The AZ² is calculated as follows:

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

Where n is the number of z-scores to be considered in the calculation. In the calculation of the AZ², z-scores higher than 5 will be classified as 5. Based on the AZ² achieved, the laboratories are classified as follows:

AZ ² ≤ 2	Good
2 < AZ ² < 3	Satisfactory
AZ ² ≥ 3	Unsatisfactory

Combined z-scores are considered to be of lesser importance than the individual z-scores. The EUPT-Panel retains the right not to calculate AZ² if it is considered as not being useful or if the number of results reported by any participant is considered to be too low.

In the case of EUPT-SRMs, where only a few results per lab may be available, the Average of the Absolute z-scores (AAZ) may be calculated for informative purposes, but only for labs that have reported enough results to obtain 5 or more z-scores. For the calculation of the AAZ, z-scores higher than 5 will also be classified as 5.

Laboratories within Category B will be ranked according to the total number of pesticides that they correctly reported to be present in the Test Item. The number of acceptable z-scores achieved will be presented, too. The EURL-Panel retains the right to calculate combined z-scores (see above) also for labs within Category B, e.g. for informative purposes, provided that a minimum number of results (z-scores) are have been reported.

Publication of results

The EURLs will publish a preliminary report, containing tentative assigned and z-score values for all pesticides present in the Test Item, within 2 months of the deadline for result submission.

The Final EUPT 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 (typically in late summer or autumn) to discuss the results of all EUPTs organised annually by the EURLs in the following year, the final report may be published up to 10 months after the deadline for results submission.

¹⁷ Formerly named "Sum of squared z-scores (SZ²)"

¹⁸ 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.

ANNEX 1. Protocols and Target list of pesticides to be sought.

Certificates of participation

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

Feedback

At any time before, during or after the PT participants have the possibility to contact the Organizers and make suggestions or indicate errors. After the distribution of the Final EUPT-Report, participating laboratories will be given the opportunity to give their feedback to the Organisers and make suggestions for future improvements.

Correction of errors

Should errors be discovered in any of the documents issued prior to the EUPT (Calendar, Target Pesticides List, Specific Protocol) the corrected documents will be uploaded onto the website and in the case of substantial errors the participants will be informed. **Before starting the exercise participants should make sure to download the latest version of these documents.**

If substantial errors are discovered in the Preliminary EUPT-Report the Organizers will distribute a new corrected version, where it will be stated that the previous version is not valid. The existence of a new updated version will also appear on the EUPT-website.

Where substantial errors are discovered in the Final EUPT-Report the EUPT-Panel will decide whether a corrigendum will be issued and how this should look. The online version of the final report will be replaced by the new one and all affected labs will be contacted.

Where errors are discovered in EUPT-Certificates the relevant laboratories will be sent new corrected ones. Where necessary the laboratories will be asked to return the old ones.

Follow-up activities

Laboratories are expected to undertake follow-up activities to trace back the sources of erroneous or strongly deviating results (typically those with $|z| > 2.0$) - including all false positives and false negatives. Even results within $|z| \leq 2.0$ may have to be checked if there is indications of a significant positive or negative bias.

Upon request, the laboratory's corresponding NRL and EURL are to be informed of the outcome of any investigative activities for false positives, false negatives and for results with $|z| \geq 3.0$. Concerning z-scores between 2.0 and 3.0 the communication of the outcome of traceability activities is optional but highly encouraged where the source of deviation could be identified and could be of interest to other labs.

ANNEX 1. Protocols and Target list of pesticides to be sought.

According to instructions from DG-SANTE, 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" is to be followed.

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.



EUPT-FV-17 SPECIFIC PROTOCOL

European Union Proficiency Test for Pesticide Residues in Fruits and Vegetables (2015)

Introduction

This protocol is complementary to the General Protocol of EU Proficiency Tests (EUP) for Pesticide Residues in Food and Feed (5th Edition, Approved: 3rd March, 2015). This Proficiency Test is organised by the EURL for Pesticide Residues in Fruits and Vegetables covering Multiresidue Methods (MRM) of analysis.

According to Article 28 of Regulation 396/2005/EC (23rd February, 2005) of the European Parliament and of the Council, all laboratories analysing samples for the official control of pesticide residues shall participate in the European Union Proficiency Tests (EUPs) for pesticide residues organised by the European Union.

These proficiency tests are carried out in order to improve the quality, accuracy and comparability of the residue data and to evaluate the laboratory capacity to report results that covers the entire range of maximum residue limits (0.005 - 15 mg/kg) in all groups of fruit and vegetable matrices (high water, acid and fat content). Bearing that, a wide concentration range should be covered with the different analytes present in the test item.

Test Item

This proficiency test is based on the analysis of incurred pesticide residues in broccoli. The broccolis were grown in a greenhouse located in the University of Almería facilities. The pesticide treatments carried out were pre-harvest using commercial formulations. The test item was frozen (using liquid nitrogen), chopped, homogenised and sub-sampled into polyethylene bottles that had previously been coded.

Ten of these bottles containing the test item were chosen randomly, and analysed to check for homogeneity.

The test item was stored frozen (-20°C) prior to shipment to participants.

Six bottles, again chosen randomly, will be analysed over a period of time to confirm the stability of the pesticides in the test item (three when the test items are shipped, then other three bottles a few days after the deadline for submitting results). There will be one further analysis during this

ANNEX 1. Protocols and Target list of pesticides to be sought.

period reproducing the sample shipment to see if there is any degradation of any of the pesticides present in the test item.

Subcontracting

All analytical determinations concerning the test item treatment analysis will be performed in a laboratory which is ISO 17025 accredited.

Assigned value and robust relative standard deviation

In order to minimise the influence of out-lying results on the statistical evaluation, the assigned value will be estimated using the robust statistics as described in ANNEX A of ISO 13258:2009-01, where the robust average (x^*) according algorithm A is defined.

Also, the robust relative standard deviation (CVs^*) will be calculated for each analyte.

Steps to follow

This Proficiency Test will be made up of the following 9 essential steps:

1. To participate, each laboratory must complete the Application Form on-line, available on the EURL-FV Web page, before the deadline stipulated on the Calendar. It is recommended that laboratories download the Target Pesticide List from this web site. Laboratories should carefully read the Target Pesticide List, where important information about the reporting of the results, as well as the Minimum Required Reporting Limits (MRRLs), is given. The MRRLs do not always correspond with the EU MRLs set for broccoli.

2. When the registration period is closed, laboratories will receive an e-mail confirming their participation in this exercise, and assigning them each a Laboratory Code. Laboratories with this code will be able to access the restricted area containing the forms using their login information - consisting of their **USER NAME**, which is the Laboratory Code expressed as **Labxxx** (three digits with no spaces between them) and their **PASSWORD**, as chosen on the application form.

3. The sample delivery will cost **200 Euros** for EU and EFTA laboratories and **250 Euros** for any other participants. The laboratories will receive an invoice and after that they can start the payment procedure. An e-mail showing the bank transfer confirmation, or similar, may be requested at any time by the Organiser. **Payments without a Laboratory Code or Invoice Number identifying them will not be considered as paid.**

4. Any communication with the Organisation should be made using a **Contact Form** placed in the restricted area.

5. **Form 0 - Laboratory Scope** will be placed in the restricted area and will be open to participants from the 23rd February -6th March 2015, prior to test item shipment. The aim is that laboratories provide information regarding their scope of analysis before receipt of the test item and detailed information regarding which pesticide is within the accredited scope of the lab and which is not. After the deadline it will not be possible to change the scope.

6. When the participant laboratories receive the test item (and not before), they must enter the restricted area again and submit **Form 1 - Test Item Receipt** to inform the Organiser that they

ANNEX 1. Protocols and Target list of pesticides to be sought.

have accepted the test item. This Form has a deadline: 20th March 2015, which must be met. If no test item has been received by this deadline, the laboratories should contact the Organiser using the Contact Form of the restricted area.

7. The participant laboratories must respect the deadline for submitting their results - 13th April 2015 - using **Form 2 – Detected pesticides; Form 3 - Results** and **Form 4 - Methods** on-line.

8. One final form, **Form 5 - Additional Information Requested** can be filled in after the deadline has passed. This Form will be available from 20th – 24th April 2015. Not all laboratories may need to fill this in. It will depend upon information reported on previous Forms.

9. The Organiser will evaluate the results at the end of the proficiency test, once the deadline for receipt of results has passed. The Organiser will upload an electronic version on the EURL-FV web site and afterwards send a hard 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.

Form 0 - Laboratory Scope

Before the participant laboratories receive the sample, the restricted area will be open so that their laboratory scopes can be recorded. Form 0 will need to be filled in to ascertain which of the pesticides in the Target Pesticide List were actually sought. After the deadline it will not be possible to change the scope.

This form will also request information on which of the pesticides sought by the laboratory is within the laboratory's accredited scope.

Amount of Test Item

Participants will receive:

- Approximately 300 g of broccoli test item treated with pesticides.
- Approximately 300 g of 'blank' broccoli test item.

Shipment of Test Item

All Test Items will be frozen and packed in polystyrene boxes surrounded in dry ice and packed into cardboard boxes.

The shipment of the test items will be carried out over a one-week period from the 16th March 2015. The Organiser will try to ensure that all the packages arrive on the same day to 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

ANNEX 1. Protocols and Target list of pesticides to be sought.

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 deeply frozen (-18°C or less) 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.

Form 1 - Test Item Receipt

Once the laboratory has received the test item, its arrival must be reported to the Organiser using Form 1 in the restricted area; filling in the date of receipt, the condition of the test item, and its acceptance. The deadline for acceptance (or nonacceptance) is 20th March 2015. 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 20th March, they must inform the Organiser **immediately** using the Contact Form of the restricted area.

Submission of results:

Once the laboratory has analysed the test item and is ready to submit their data, they must enter their results at various steps on 3 forms by accessing the restricted area in the EURL –FV web site: <http://www.eurl-pesticides.eu>

Detected Pesticides – Form 2

In Form 2, the information entered in Form 0 – Laboratory Scope, will be made available again.

For each pesticide included in the laboratory scope, the Limit of Quantification (LOQ) will be requested. The MRRL and the participant's own LOQ will be used to help identify false negative results.

Before this, a question will be requested as to which approach was used for the relative expanded uncertainty estimation in multiresidue methods for fruits and vegetables. The laboratories will be also asked to report any pesticide that may have been detected in the blank test item.

ANNEX 1. Protocols and Target list of pesticides to be sought.

This form can be filled in at various stages - so once entered, the data will be saved, and the laboratories can add further data at a later date.

Results – Form 3

In this step, the laboratory should report the measured concentrations for each determination. All concentrations must be expressed in mg/kg together with the recovery as a percentage.

The number of significant figures should be based on the procedures provided in SANCO/12571/2013. Additional significant figures may be recorded for the purpose of statistical analysis.

Results should not be reported where a pesticide was not detected or was detected below the laboratory LOQ. In both cases, this should be recorded as 'ND' 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.

Methods – Form 4

In this step, the laboratory must report the details of the analytical methods they used. A list including all the pesticides detected in the sample will be shown along with a pesticide reference number. Laboratories may describe a method for the first pesticide and use this pesticide reference number to refer to other pesticides determined using the same method.

Again in this form, information must always be saved so that laboratories can go back to it and continue at any time before the final reporting deadline - which for all forms is 13th March 2015. Any results reported after this deadline will not be included in the statistical treatment, nor in the final report.

It should **not** be assumed that only pesticides registered for use on broccolis are present in the test item.

False Negatives or Further Information – Form 5

This Form will be available only for those laboratories reporting that they sought a pesticide present in the test item but for which no method was reported in Form 4. If a laboratory accesses this Form and it is empty, this will mean that there is no need to enter further information. This Form will be available after the deadline is over - from 20th – 24th April 2015.

ANNEX 1. Protocols and Target list of pesticides to be sought.

Calendar

ACTIVITY	DATE
Publishing the Target Pesticide List, Calendar and Matrix on the Web page.	15 th December 2014
Receiving Application Form from invited laboratories.	12 th Jan-13 th Feb 2015
Specific Protocol published on the Web site.	February 2015
Deadline for receiving Laboratory scope: Form 0	23 rd Feb - 6 th March 2015
Sample distribution.	16 th March 2015
Deadline for receiving sample acceptance: Form 1	20 th March 2015
Deadline for receiving results: Form 2, Form 3 and Form 4	13 th April 2015
Filling in Form 5	20 th -24 th April 2015
Preliminary Report: only results, no statistical treatment	May 2015
Final Report distributed to the Laboratories	December 2015

Cost of test item shipment.

EU and EFTA laboratories will be charged **200€** for the shipment cost. Other laboratories will be charged **250 €**. Regarding payment procedures - each laboratory can specify their details and invoice requests when applying for the test.

Please, do not pay for this EUPT until we send you the invoice.

Remember to include your Laboratory Code in the subject of the bank transfer.

Payment details are as follows:

BANK NAME: CAJAMAR - Caja Rural Sociedad Corporativa de Crédito

BANK ACCOUNT OWNER: Universidad de Almería

BANK ADDRESS: Office Number 990. Universidad de Almería. Spain

ACCOUNT NUMBER: 30580130172731005000

IBAN: ES0730580130172731005000

SWIFT: CCRIES2A

CONCEPT: Invoice No. or Lab Code

ANNEX 1. Protocols and Target list of pesticides to be sought.

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 950015008

Organising team (e-mail and phone no.):

Dr. Amadeo R. Fernández-Alba	EURL-FV amadeo@ual.es +34 950015034
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Quality Control Group

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

Statistical Group

Dr. Carmelo Rodríguez, Senior Mathematician, University of Almería, Spain

Advisory Group

Dr. Michelangelo Anastasiades, Senior Chemist, CVUA, Stuttgart, Germany.
Dr. Miguel Gamón, Senior Chemist, Laboratorio Agroalimentario, Valencia, Spain.
Dr. Philippe Gros, Senior Chemist, Laboratoire du SCL, Montpellier, France.
Dr. Magnus Jezussek, Senior Chemist, Erlangen, Germany.
Dr. André de Kok, Senior Chemist, NVWA, Wageningen, The Netherlands.
Mr. Ralf Lippold, Senior Chemist, CVUA, Freiburg, Germany.
Dr. Sonja Masselter, Senior Chemist, AGES, Innsbruck, Austria.
Mr. Finbarr O'Regan, Senior Chemist, Pesticide Control Laboratory, Celbridge, Ireland.
Dr. Tuija Pihlström, Senior Chemist NFA, Uppsala, Sweden.
Dr. Mette Erecius Poulsen, Senior Chemist, DTU, Copenhagen, Denmark.
Dr. Darinka Stajnbaher, Senior Chemist, Maribor, Slovenia.

ANNEX 1. Protocols and Target list of pesticides to be sought.

TARGET PESTICIDE LIST FOR THE EUPT-FV-17

Pesticide	MRRL (mg/Kg)
3-hydroxy-carbofuran	0.01
Acephate	0.01
Acetamiprid	0.01
Acrinathrin	0.01
Aldicarb	0.01
Aldicarb Sulfone	0.01
Aldicarb Sulfoxide	0.01
Azinphos-methyl	0.01
Azoxystrobin	0.01
Benfuracarb	0.01
Benomyl	0.01
Bifenthrin	0.01
Bitertanol	0.01
Boscalid	0.01
Bromopropylate	0.01
Bromuconazole	0.01
Bupirimate	0.01
Buprofezin	0.01
Cadusafos	0.006
Carbaryl	0.01
Carbendazim	0.01
Carbofuran	0.01
Carbosulfan	0.01
Chlorfenapyr	0.01
Chlorfenvinphos	0.01
Chlorobenzilate	0.01
Chlorothalonil	0.01
Chlorpropham (only parent compound)	0.01
Chlorpyrifos	0.01
Chlorpyrifos-methyl	0.01
Clofentezine (only parent compound)	0.01
Clothianidin	0.01
Cyfluthrin (cyfluthrin incl. other mixtures of constituent isomers (sum of isomers))	0.01
Cypermethrin (cypermethrin incl. other mixtures of constituent isomers (sum of isomers))	0.01
Cyproconazole	0.01
Cyprodinil	0.01
Deltamethrin	0.01
Demeton-S-methylsulfone	0.006
Desmethyl-pirimicarb	0.01
Diazinon	0.01
Dichlofluanid (only parent compound)	0.01
Dichlorvos	0.01
Dicloran	0.01
Dicofol	0.01
Diethofencarb	0.01
Difenoconazole	0.01
Diflubenzuron	0.01
Dimethoate	0.003
Dimethomorph	0.01
Dimethylaminosulfotoluidide (DMST)	0.01
Diniconazole	0.01
Diphenylamine	0.01
Endosulfan alpha	0.01
Endosulfan beta	0.01
Endosulfan sulfate	0.01

ANNEX 1. Protocols and Target list of pesticides to be sought.

Pesticide	MRRL (mg/Kg)
EPN	0.01
Epoxiconazole	0.01
Ethion	0.01
Ethirimol	0.01
Ethoprophos	0.008
Etofenprox	0.01
Fenamidone	0.01
Fenamiphos	0.01
Fenamiphos sulfone	0.01
Fenamiphos sulfoxide	0.01
Fenarimol	0.01
Fenazaquin	0.01
Fenbuconazole	0.01
Fenhexamid	0.01
Fenitrothion	0.01
Fenoxycarb	0.01
Fenpropathrin	0.01
Fenpropimorph	0.01
Fenpyroximate	0.01
Fenthion	0.01
Fenthion oxon	0.01
Fenthion oxon sulfone	0.01
Fenthion oxon sulfoxide	0.01
Fenthion sulfone	0.01
Fenthion sulfoxide	0.01
Fenvalerate and Esfenvalerate	0.01
Fipronil (only parent compound)	0.004
Fludioxonil	0.01
Flufenoxuron	0.01
Fluopicolide	0.01
Fluopyram	0.01
Fluquinconazole	0.01
Flusilazole	0.01
Flutolanil	0.01
Flutriafol	0.01
Fosthiazate	0.01
Hexaconazole	0.01
Hexythiazox	0.01
Imazalil	0.01
Imidacloprid	0.01
Indoxacarb (Indoxacarb as sum of the isomers S and R)	0.01
Iprodione	0.01
Iprovalicarb	0.01
Isocarbophos	0.01
Isofenphos-methyl	0.01
Isoprothiolane	0.01
Kresoxim-methyl	0.01
Lambda-Cyhalothrin	0.01
Linuron	0.01
Lufenuron	0.01
Malaoxon	0.01
Malathion	0.01
Mandipropamid	0.01
Mepanipyrim (only parent compound)	0.01
Metaflumizone	0.01
Metalaxyl and metalaxyl-M	0.01
Metconazole	0.01
Methamidophos	0.01

ANNEX 1. Protocols and Target list of pesticides to be sought.

Pesticide	MRRL (mg/Kg)
Methidathion	0.01
Methiocarb	0.01
Methiocarb sulfone	0.01
Methiocarb sulfoxide	0.01
Methomyl	0.01
Methoxyfenozide	0.01
Monocrotophos	0.01
Myclobutanil	0.01
Omethoate	0.003
Orthophenylphenol	0.01
Oxadixyl	0.01
Oxamyl	0.01
Oxydemeton-methyl	0.006
Paclbutrazole	0.01
Paraoxon-methyl	0.01
Parathion-ethyl	0.01
Parathion-methyl	0.01
Penconazole	0.01
Pencycuron	0.01
Pendimethalin	0.01
Permethrin	0.01
Phenthoate	0.01
Phosalone	0.01
Phosmet	0.01
Phosmet oxon	0.01
Phoxim	0.01
Pirimicarb	0.01
Pirimiphos-methyl	0.01
Prochloraz (only parent compound)	0.01
Procymidone	0.01
Profenofos	0.01
Propargite	0.01
Propiconazole	0.01
Propyzamide	0.01
Prothioconazole (Prothioconazole-desthio)	0.01
Prothiofos	0.01
Pyraclostrobin	0.01
Pyridaben	0.01
Pyrimethanil	0.01
Pyriproxyfen	0.01
Quinoxifen	0.01
Spinosad (sum of spinosyn A and spinosyn D, expr. as spinosad)	0.01
Spirodiclofen	0.01
Spiroxamine	0.01
Tau-Fluvalinate	0.01
Tebuconazole	0.01
Tebufenozide	0.01
Tebufenpyrad	0.01
Teflubenzuron	0.01
Tefluthrin	0.01
Terbutylazine	0.01
Tetraconazole	0.01
Tetradifon	0.01
Thiabendazole	0.01
Thiacloprid	0.01
Thiamethoxam	0.01
Thiodicarb	0.01
Thiophanate-methyl	0.01

ANNEX 1. Protocols and Target list of pesticides to be sought.

Pesticide	MRRL (mg/Kg)
Tolclofos-methyl	0.01
Tolyfluanid	0.01
Triadimefon	0.01
Triadimenol	0.01
Triazophos	0.01
Trichlorfon (only parent compound)	0.01
Trifloxystrobin	0.01
Triflumuron	0.01
Trifluralin	0.01
Trificonazole	0.01
Vinclozolin (only parent compound)	0.01
Zoxamide	0.01
Triazophos	0.01

New pesticides this year

This list is based on Commission Regulation (EU) No 400/2012
The MRRLs are based in Regulation (EC) No. 396/2005 and Commission Directive 2006/125/EC.

ANNEX 2. List of laboratories that agreed to participate in EUPT-FV-17.

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
Austria	Austrian Agency For Health And Food Safety (AGES GmbH)	Innsbruck	YES
Austria	MA 38 - Lebensmitteluntersuchungsanstalt Der Stadt Wien	Wien	YES
Belgium	Scientific Institute Of Public Health (WIV-ISP)	Brussels	YES
Belgium	Fytolab Belgium CVBA	Zwijnaarde	YES
Belgium	LOVAP NV	Geel	YES
Brazil	Center Of Research And Analysis Of Residues And Contaminants	Santa Maria	YES
Brazil	Pesticide Residue Analysis Laboratory - ITEP/Labtox	Recife	YES
Bulgaria	Central Laboratory For Chemical Testing And Control	Sofia	YES
Bulgaria	Euro Lab	Svilengrad	YES
China	Shanghai Municipal Center For Disease Control And Prevention	Shanghai	YES
Costa Rica	Centro De Investigación En Contaminación Ambiental (CICA), Universidad De Costa Rica	San Pedro De Montes De Oca, San José	YES
Croatia	Euroinspekt-Croatiakontrola	Zagreb	YES
Croatia	Teaching Institute Of Public Health Dr Andrija Štampar	Zagreb	YES
Croatia	Faculty Of Food Technology And Biotechnology, Food Control Center, Zagreb, Croatia	Zagreb	YES
Croatia	Institute Of Public Health Split	Split	YES
Croatia	Croatian National Institute Of Public Health	Zagreb	YES
Croatia	Teaching Institute Of Public Health Of PGZ	Rijeka	YES
Cyprus	Pesticides Residues Laboratory Of The State General Laboratory Of Ministry Of Health	Nicosia	YES
Czech Republic	University Of Chemistry And Technology, Department Of Food Analysis And Nutrition	Prague	YES
Czech Republic	Czech Agriculture And Food Inspection Authority	Brno	YES
Czech Republic	Central Institute For Supervising And Testing In Agriculture	Brno	YES
Denmark	Danish Veterinary And Food Administration	Ringsted	YES
Denmark	National Food Institute, Technical University Of Denmark	Lyngby	YES
Egypt	Central Lab Of Residue Analysis Of Pesticides And Heavy Metals In Foods	Giza	YES
Estonia	Laboratory For Residues And Contaminants, Agricultural Research Centre	Saku	YES
Estonia	Tartu Laboratory Of Health Board	Tartu	YES
Finland	Metropolilab Ltd	Helsinki	YES
Finland	Finnish Customs Laboratory	Espoo	YES
France	Capinov - Triskalia	Landerneau Cedex	YES

ANNEX 2. List of laboratories that agreed to participate in EUPT-FV-17.

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
France	Centre Analyse Mediterranée Pyrénées	Perpignan	YES
France	Inovalys Le Mans	Le Mans	YES
France	Cereco Sud	Lieu Saint Amand	YES
France	Laboratoire Du SCL De Montpellier	Montpellier	YES
France	SCL - Laboratoire De Massy	Massy Cedex	YES
France	Fredon Pays De La Loire/Girpa	Beaucouze	YES
Germany	LUFA-ITL Gmbh	Kiel	YES
Germany	Federal Office Of Consumer Protection And Food Safety (BVL)	Berlin	YES
Germany	Labor Dr. Mang	Frankfurt	YES
Germany	Landesamt Für Verbraucherschutz	Berlin	YES
Germany	Chemical And Veterinary Analytical Institute Rhine-Ruhr-Wupper	Berlin	YES
Germany	Nds. Landesamt Für Verbraucherschutz Und Lebensmittelsicherheit, LVI OL	Berlin	YES
Germany	Landeslabor Berlin-Brandenburg (LLBB)	Berlin	YES
Germany	Bayerisches Landesamt Für Gesundheit Und Lebensmittelsicherheit	Berlin	YES
Germany	Landesuntersuchungsamt Für Chemie, Hygiene Und Veterinärmedizin	Berlin	YES
Germany	LUA Sachsen	Berlin	YES
Germany	CVUA-Westfalen	Bochum	YES
Germany	Thueringer Landesamt Fuer Verbraucherschutz	Berlin	YES
Germany	Landesamt Für Landwirtschaft Lebensmittelsicherheit Und Fischerei	Berlin	YES
Germany	Landesamt Für Verbraucherschutz Sachsen-Anhalt	Berlin	YES
Germany	State Laboratory Schleswig-Holstein	Berlin	YES
Germany	Chemisches Und Veterinäruntersuchungsamt Stuttgart	Berlin	YES
Germany	LTZ Augustenberg	Berlin	YES
Germany	CVUA Rheinland	Berlin	YES
Germany	Landesuntersuchungsamt Institut Für Lebensmittelchemie Speyer	Berlin	YES
Germany	Chemisches Und Veterinäruntersuchungsamt Ostwestfalen-Lippe (CVUA-OWL)	Berlin	YES
Germany	CVUA-MEL	Berlin	YES
Germany	Department For Consumer Protection Duesseldorf	Berlin	YES
Germany	Institut Fuer Hygiene Und Umwelt	Berlin	YES
Germany	Zentrales Institut Des Sanitätsdienstes Der Bundeswehr KIEL (Zinsanbw Kiel)	Kronshagen	YES

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COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
Germany	Labor Friedle Gmbh	Tegernheim	YES
Germany	GALAB Laboratories Gmbh	Hamburg	YES
Germany	ILAU Gmbh	Anzing	YES
Germany	Labor Dr. Lippert Gmbh	Sinzig	YES
Germany	Eurofins Dr. Specht Laboratorien Gmbh	Düsseldorf	YES
Greece	Benaki Phytopathological Institute, Pesticide Residues Laboratory	Kiphissia (Athens)	YES
Greece	Regional Center Of Plant Protection & Quality Control Of Magnesia, Volos	Volos	YES
Greece	Regional Center Of Plant Protection And Quality Control Of Iraklion, Laboratory Of Pesticide Residue	Iraklion, Crete	YES
Greece	Laboratory Of Pesticide Residue Analysis, Regional Centre Of Crop Protection & Quality Control Of Ioannina	Ioannina	YES
Greece	Pesticide Residues Laboratory Of The Regional Centre Of Plant Protection And Quality Control Of Thessaloniki	Thermi	YES
Greece	General Chemical State Laboratory	Athens	YES
Greece	Pesticides Residue Laboratory Of Regional Centre Of Piraeus	Piraeus	YES
Greece	Regional Center Of Plant Protection And Quality Control Of Achaia	Patra	YES
Greece	Periferal Center Of Plant Protection And Quality Control Kavala	Kavala	YES
Hungary	National Food Chain Safety Office, DPPSCA, Pesticide Residue Analytical Laboratory, Miskolc	Budapest	YES
Hungary	National Food Chain Office Directorate Of Plant Protection, Soil Conservation And Agri-Environment Pesticide Residue Analytical Laboratory, Szolnok	Budapest	YES
Hungary	National Food Chain Safety Office, Pesticide Residue Analytical Laboratory Of Hodmezovasarhely	Budapest	YES
Hungary	NFCISO Pesticide Analytical Laboratory, Velenca	Budapest	YES
Iceland	Matís OHF	Reykjavík	YES
Ireland	The Pesticide Control Laboratory	Celbridge	YES
Italy	ASL Milano - Laboratorio Di Prevenzione	Milano	YES
Italy	Istituto Zooprofilattico Sperimentale Della Sardegna	Sassari	YES
Italy	Istituto Zooprofilattico Sperimentale Del Lazio E Della Toscana	Roma	YES
Italy	ARPAL	Genova	YES
Italy	ARPALazio Sezione Di Latina	Rieti	YES
Italy	Servizio Laboratorio Chimico ARPACAL	Catanzaro	YES
Italy	Istituto Zooprofilattico Dell'abruzzo E Del Molise G.Caporale Teramo	Teramo	YES
Italy	ARPA Puglia - Polo Di Specializzazione Alimenti-Bari	Bari	YES
Italy	Laboratorio Contaminanti Ambientali - IZSUM	Perugia	YES

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Italy	Landesagentur Für Umwelt-Labor Für Wasseranalysen Und Chromatographie	Bolzano	YES
Italy	Istituto Zooprofilattico Sperimentale Lombardia Emilia Romagna (Izslr) - Laboratorio Pesticidi	Brescia	YES
Italy	Arpa Valle D'aosta	Saint Christophe	YES
Italy	Laboratorio Di Sanita' Pubblica Azienda Sanitaria Di Firenze	Firenze	YES
Italy	Laboratorio Tematico Fitofarmaci ARPA Emilia Romagna - Sezione Provinciale Di Ferrara	Ferrara	YES
Italy	ARPA FVG Laboratorio Unico Multisito - Sede Di Pordenone	Palmanova	YES
Italy	Laboratorio Di Sanita' Pubblica Asl Provincia Di Bergamo	Bergamo	YES
Italy	Istituto Superiore Di Sanita' - Reparto Antiparassitari	Rome	YES
Italy	ARPA Trento	Trento	YES
Italy	ARPA Piemonte Polo Alimenti	Torino	YES
Italy	ARPA Marche- Dip. Macerata	Ancona	YES
Italy	Istituto Zooprofilattico Sperimentale Della Sicilia "A.Mirri"	Palermo	YES
Italy	Arpa Veneto-SI Verona	Padova	YES
Italy	Arpacampania - Laboratorio Regionale Micotossine E Fitofarmaci	Napoli	YES
Jamaica	Residue And Biochemical Laboratory, Veterinary Services Division	Kingston	YES
Kenya	SGS Kenya Limited Mombasa Laboratory	Mombasa	YES
Kenya	Kenya Plant Health Inspectorate Service (KEPHIS) Analytical Chemistry Laboratory	Nairobi	YES
Latvia	Institute Of Food Safety, Animal Health And Environment BIOR	Riga	YES
Lebanon	Phytopharmacy Laboratory Agriculture Ministry Of Lebanon	Mount Lebanon	YES
Lithuania	National Food And Veterinary Risk Assessment Institute	Vilnius	YES
Luxembourg	Laboratoire National De Santé - Alimentaire	Dudelange	YES
Nederland	Eurofins Lab Zeeuws-Vlaanderen (ZVL) Bv	Oosterhout	YES
Netherlands	Groen Agrocontrol	Delft	YES
Netherlands	Lab Dr A Verwey Agrogroep	Rotterdam	YES
Netherlands	NVWA - Netherlands Food And Consumer Product Safety Authority	Utrecht	YES
Norway	Bioforsk, Plant Health And Plant Protection, Pesticide Chemistry Section	Aas	YES
Peru	Unidad Del Centro Del Control De Insumos Y Residuos Toxicos	Lima	YES
Poland	Laboratory Of Pesticide Residue Analysis, Institute Of Plant Protection – National Research Institute, Rzeszow	Poznan	YES
Poland	Plant Protection Institute-National Research Institute, Pesticide Laboratory In Bialystok	Poznan	YES

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Poland	Department Of Pesticide Residues, Institute Of Plant Protection - National Research Institute	Poznan	YES
Poland	Wojewodzka Stacja Sanitarno-Epidemiologiczna We Wroclawiu - Dzial Laboratoryjny	Wroclaw	YES
Poland	Institute Of Plant Protection-National Research Institute, Laboratory Of Pesticide Residue Analyses	Sosnicowice	YES
Poland	Laboratory Of Voivodship Sanitary-Epidemiological Station In Warsaw	Warsaw	YES
Poland	Main Inspectorate Of Plant Health And Seed Inspection , Central Laboratory	Warszawa	YES
Poland	Wojewodzka Stacja Sanitarno-Epidemiologiczna W Opolu	Opole	YES
Poland	Wojewodzka Stacja Sanitarno-Epidemiologiczna W Lodzi	Lodz	YES
Poland	Sgs Polska Sp. Z O.O. Laboratorium Srodowiskowe	Warszawa	YES
Poland	Institute Of Horticulture, Food Safety Laboratory	Skierniewice	YES
Portugal	Regional Laboratory Of Veterinary An Food Safety	Funchal	YES
Portugal	INIAV - UEISTSA - Laboratório De Resíduos De Pesticidas - Oeiras	Oeiras	YES
Romania	Laboratory For Pesticides Residues Control In Plants And Vegetables	Voluntari	YES
Romania	Sanitary Veterinary And Food Safety Directorate	Bucharest	YES
Romania	Regional Laboratory For Determination Of Pesticide Residues In Plant And Plant Products Mures	Targu Mures	YES
Romania	Sanitary Veterinary And Food Safety Laboratory Iasi	Iasi	YES
Saudi Arabia	National Center For Monitoring Food Contaminants	Riyadh	YES
Serbia	Sp Laboratorija	Becej	YES
Serbia	Center For Food Analysis	Belgrade	YES
Singapore	Agri-Food And Veterinary Authority Of Singapore	Singapore	YES
Slovakia	National Reference Centre For Pesticide Residues, Public Health Authority Of The Slovak Republic	Bratislava	YES
Slovakia	Veterinary And Food Institute In Bratislava	Bratislava	YES
Slovenia	National Laboratory Of Helath, Environment And Food	Maribor	YES
Slovenia	National Laboratory For Health, Environment And Food (Dep. For Chem. Anal. Maribor)	Maribor	YES
Slovenia	Kmetijski Inštitut Slovenije (Agricultural Institute Of Slovenia)	Ljubljana	YES
Spain	Laboratorio Químico Microbiológico, S.A	Murcia	YES
Spain	Laboratorio Kudam,S.L	Torre De La Horadada	YES
Spain	Laboratorio Agrario Regional. Dirección General De Producción Agropecuaria Y Desarrollo Rural. Junta De Castilla Y León.	Burgos	YES
Spain	Servicios Perifericos De La Consejería De Sanidad Y Asuntos Sociales, Laboratorio De Salud Publica	Cuenca	YES
Spain	Laboratorio Producción Y Sanidad Vegetal	Mengibar (Jaén)	YES

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Spain	Laboratorio De Producción Y Sanidad Vegetal De Huelva	Huelva	YES
Spain	CNTA	San Adrian (Navarra)	YES
Spain	Laboratorio Agroalimentario De Granada	Atarfe (Granada)	YES
Spain	Laboratorio De Residuos-Instituto Tecnológico De Canarias, S. A.-Departamento De Análisis Ambiental	Las Palmas De Gran Canaria	YES
Spain	AINIA	Paterna	YES
Spain	Analytica Allimentaria Gmbh, Sucursal En España	Almeria	YES
Spain	Laboratorios Ecosur S.A.	Lorqui - Murcia	YES
Spain	Laboratorio De Salud Publica- Madrid Salud	Madrid	YES
Spain	Laboratorio Agroalimentario Y De Sanidad Animal	El Palmar (Murcia)	YES
Spain	Laboratorio Regional De La CCAA De La Rioja	Logroño	YES
Spain	Agricultural And Phytopathological Laboratory Of Galicia	Abegondo. A Coruña	YES
Spain	NASERTIC	Villava	YES
Spain	Laboratorio De Salud Pública De Badajoz	Badajoz	YES
Spain	Laboratorio De Salud Publica De Almeria	Almeria	YES
Spain	Laboratory Of Barcelona Public Health Agency	Barcelona	YES
Spain	Laboratorio Agroalimentario Valencia	Burjassot. Valencia	YES
Spain	CNA (Aecosan)	Madrid	YES
Spain	Laboratorio Agroambiental De Zaragoza (Gobierno De Aragón)	Zaragoza	YES
Spain	Laboratorio Del SOIVRE. Dirección Provincial De Comercio De Almería	Almería	YES
Spain	Laboratorio Agroalimentario De Extremadura	Cáceres	YES
Spain	Laboratori Agroalimentari-DAAM	Vilassar De Mar	YES
Spain	Laboratorio Salud Pública De Palma	Palma De Mallorca	YES
Spain	Laboratorio Arbitral Agroalimentario	Madrid	YES
Spain	Laboratorio De Producción Y Sanidad Vegetal De Almería	La Mojonera	YES
Spain	Labs&Technological Services AGQ, S.L.	Burguillos	YES
Spain	Laboratorio Agroalimentario Y Ambiental De Castilla La Mancha	Toledo	YES
Spain	Direccion Territorial De Comercio De Valencia (SOIVRE)	Valencia	YES
Sweden	Eurofins Food & Feed Testing Sweden AB	Fagersta	YES
Sweden	Swedish National Food Agency, Science Department, Chemistry Division	Strömsund	YES

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Switzerland	Kantonales Labor Zürich	Zürich	YES
Switzerland	Amt Für Verbraucherschutz Aargau (Cantonal Office Of Consumer Protection Aargau)	Aarau	YES
Thailand	Central Laboratory (Thailand) Co., Ltd. Bangkok Branch	Bangkok	YES
Turkey	Ozel MSM Gida Kontrol Laboratuvari Ve Dan. Hiz. Tic. A.S.	Mersin	YES
Uk	Science And Advice For Scottish Agriculture (Sasa)	Edinburgh	YES
Uk	Food And Environment Research Agency	York	YES
Uk	LGC	Teddington	YES
Uk	Eurofins Food Testing Uk Ltd	Wolverhampton	YES
Uruguay	Pharmacognosy-Gact(Grupo De Analisis De Compuestos Traza)	Montevideo	YES