

EURL-PROFICIENCY TEST-FV-16, 2014

Pesticide Residues in Sweet Pepper 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	12
3. STATISTICAL METHODS	13
3.1 False positives and negatives	13
3.2 Estimation of the assigned values	13
3.3 Fixed target standard deviations	14
3.4 z-Scores	14
3.5 Combined z-Scores	14
4. RESULTS	16
4.1 Summary of reported results	16
4.2 Assigned values and target standard deviations	19
4.3 Assessment of laboratory performance	20
5. CONCLUSIONS	27
6. SUGGESTIONS FOR FUTURE WORK	30
7. REFERENCES	31
8. ACKNOWLEDGEMENTS	32
APPENDIX 1. Homogeneity data.	33
APPENDIX 2. Histograms of residue data for each pesticide from all the laboratories.	35
APPENDIX 3. Results (mg/Kg) and z-scores for FFP RSD (25 %).	39
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).	47
APPENDIX 5. Average of the Squared z-scores (AZ^2) for laboratories in Category A.	69
APPENDIX 6. EUPT 16 – AZ^2 - Graphical representation for laboratories in Category A.	73
ANNEX 1. Protocols and Target list of pesticides to be sought.	74
ANNEX 2. List of laboratories that agreed to participate in EUPT-FV-16.	97

EURL-EUROPEAN UNION PROFICIENCY TEST 16
FOR THE DETERMINATION OF PESTICIDES IN FRUIT AND VEGETABLES USING
MULTIRESIDUE METHODS
2014

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 16 has been organised by the EURL in Fruit and Vegetables at the University of Almería, Spain⁴.

Participation in European Proficiency Test 16 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 control programme or for their own national monitoring programmes. Additionally, laboratories from Brazil, China, Egypt, Iceland, Israel, Norway, Saudi Arabia, Serbia, Switzerland, Turkey and Uruguay, who had been invited to take part in the previous tests, again participated. Kenya, Peru, Singapore and Thailand participated in this test for the first time.

DG-SANCO 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 the Food Chain and the Animal Health.

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-three laboratories agreed to participate in European Union Proficiency Test 16.

The proficiency test was performed in 2014 using pepper homogenate. The peppers 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. Twenty-one pesticides were used for the treatment. Participating laboratories were also provided with a 'blank' pepper homogenate as well as the treated test item.

The test items, 300 g of pepper homogenate containing pesticide residues, together with 300 g of 'blank' pepper homogenate, were shipped to participants on 24th February 2014. The deadline for results submission to the Organiser was 17th March 2014. The participants were provided with a list of one hundred and seventy-five target pesticide residues (Annex 1) and informed that any of those 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. 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 median 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 agreed to participate in EUPT-FV-16.

2. TEST ITEMS

2.1 Preparation of the treated test item

The peppers 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 16. Around 1 hectare of pepper plants was treated. All the pesticides were used as commercial pesticide formulations dissolved in water. Between four and ten days after the application, a representative sample of the treated peppers 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 peppers were close to those recommended by the QCG, the entire production (200 kg) was harvested, frozen and processed using liquid nitrogen and a mincer. The frozen minced peppers 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 peppers 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 pepper 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 Labcolor, 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' pepper 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: $S_s^2 < c$, where S_s 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)	S_s^2	c	$S_s^2 < c$ Pass/Fail
Acetamiprid	0.621	4.23×10^{-3}	8.49×10^{-3}	Pass
Acrinathrin	0.264	2.03×10^{-4}	8.30×10^{-4}	Pass
Buprofezin	0.303	3.18×10^{-4}	1.68×10^{-3}	Pass
Chlorothalonil	2.077	8.66×10^{-3}	1.03×10^{-1}	Pass
Chlorpyrifos	3.038	7.01×10^{-2}	1.70×10^{-1}	Pass
Cypermethrin	0.410	5.24×10^{-4}	4.04×10^{-3}	Pass
Cyprodinil	0.387	1.03×10^{-3}	3.23×10^{-3}	Pass
Diazinon	0.065	3.44×10^{-5}	6.92×10^{-5}	Pass
Difenoconazole	0.553	1.80×10^{-3}	5.11×10^{-3}	Pass
Endosulfan α	0.776	2.26×10^{-3}	1.41×10^{-2}	Pass
Endosulfan β	0.538	2.70×10^{-3}	5.60×10^{-3}	Pass
Fenamiphos ⁺	8.243	4.99×10^{-1}	1.61×10^0	Pass
Fenamiphos Sulfone ⁺	0.102	1.44×10^{-4}	1.52×10^{-4}	Pass
Fenamiphos Sulfoxide ⁺	6.571	8.70×10^{-2}	4.62×10^{-1}	Pass
Fenhexamid	0.681	2.05×10^{-3}	1.45×10^{-2}	Pass
Fludioxonil	0.191	3.86×10^{-4}	9.44×10^{-4}	Pass
λ -Cyhalothrin	0.080	1.28×10^{-5}	1.02×10^{-4}	Pass
Methoxyfenozide	0.106	3.11×10^{-5}	2.84×10^{-4}	Pass
Pirimicarb	0.412	-6.97×10^{-4}	4.51×10^{-3}	Pass
Pyridaben	0.127	1.72×10^{-4}	3.06×10^{-4}	Pass
Spinosad	0.049	2.22×10^{-6}	8.60×10^{-5}	Pass
Tetraconazole	0.073	1.61×10^{-5}	7.07×10^{-5}	Pass

S_s : Between-Sampling Standard Deviation

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

2.4 Stability tests

The stability tests were also subcontracted to the laboratory Labcolor, 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 February 24th 2014.
- Day 2: shortly after the deadline for reporting results, on March 17th 2014.

The individual results are given in Table 2.2. In general, these tests did not show any significant decrease in the pesticide concentrations. 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. The differences between the results of these analyses were not greater than 10 %. 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

Pesticide	Concentration (mg/kg)							
	Day 1 (1 st analysis)	Day 1 (2 nd analysis)	Mean 1	Day 2 (1 st analysis)	Day 2 (2 nd analysis)	Mean 2	$\frac{(M2-M1)}{M1}$	%
Acetamiprid	0.603	0.610	0.607	0.618	0.623	0.621	0.023	2
Acrinathrin	0.271	0.278	0.275	0.264	0.266	0.265	-0.035	-3
Buprofezin	0.305	0.296	0.301	0.298	0.302	0.300	-0.002	0
Chlorothalonil	2.109	2.098	2.104	2.043	2.055	2.049	-0.026	-3
Chlorpyrifos	3.127	3.115	3.121	3.025	3.017	3.021	-0.032	-3
Cypermethrin	0.421	0.418	0.420	0.398	0.401	0.400	-0.048	-5
Cyprodinil	0.377	0.380	0.379	0.375	0.384	0.380	0.003	0
Diazinon	0.066	0.072	0.069	0.063	0.071	0.067	-0.029	-3
Difenoconazole	0.544	0.540	0.542	0.552	0.550	0.551	0.017	2
Endosulfan a	0.793	0.788	0.791	0.773	0.768	0.771	-0.025	-3
Endosulfan β	0.525	0.537	0.531	0.529	0.520	0.525	-0.012	-1
Fenamiphos ⁺	8.276	8.268	8.272	8.201	8.195	8.198	-0.009	-1
Fenamiphos Sulfone ⁺	0.108	0.115	0.112	0.109	0.101	0.105	-0.058	-6
Fenamiphos Sulfoxide ⁺	6.930	6.250	6.590	7.010	6.850	6.930	0.052	5
Fenhexamid	0.700	0.691	0.696	0.669	0.674	0.672	-0.035	-3
Fludioxonil	0.185	0.197	0.191	0.185	0.193	0.189	-0.010	-1

Pesticide	Concentration (mg/kg)							
	Day 1 (1 st analysis)	Day 1 (2 nd analysis)	Mean 1	Day 2 (1 st analysis)	Day 2 (2 nd analysis)	Mean 2	$\frac{(M2-M1)}{M1}$	%
λ-Cyhalothrin	0.074	0.072	0.073	0.083	0.079	0.081	0.110	11
Methoxyfenozide	0.115	0.112	0.114	0.101	0.099	0.100	-0.119	-12
Pirimicarb	0.428	0.426	0.427	0.410	0.418	0.414	-0.030	-3
Pyridaben	0.136	0.141	0.139	0.125	0.119	0.122	-0.119	-12
Spinosad	0.050	0.045	0.048	0.043	0.041	0.042	-0.116	-12
Tetraconazole	0.065	0.063	0.064	0.075	0.068	0.072	0.117	12

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

Pesticide	Concentration (mg/kg)							
	Day 1 (1 st analysis)	Day 1 (2 nd analysis)	Mean 1	48h (1 st analysis)	48h (2 nd analysis)	Mean 2	$\frac{(M2-M1)}{M1}$	%
Acetamiprid	0.603	0.610	0.607	0.611	0.614	0.613	0.010	1
Acrinathrin	0.271	0.278	0.275	0.264	0.270	0.267	-0.027	-3
Buprofezin	0.305	0.296	0.301	0.295	0.294	0.295	-0.020	-2
Chlorothalonil	2.109	2.098	2.104	2.041	2.032	2.037	-0.032	-3
Chlorpyrifos	3.127	3.115	3.121	3.073	3.061	3.067	-0.017	-2
Cypermethrin	0.421	0.418	0.420	0.405	0.411	0.408	-0.027	-3
Cyprodinil	0.377	0.380	0.379	0.370	0.371	0.371	-0.021	-2
Diazinon	0.066	0.072	0.069	0.064	0.060	0.062	-0.101	-10
Difenoconazole	0.544	0.540	0.542	0.571	0.567	0.569	0.050	5
Endosulfan α	0.793	0.788	0.791	0.785	0.780	0.783	-0.010	-1
Endosulfan β	0.525	0.537	0.531	0.526	0.532	0.529	-0.004	0
Fenamiphos ⁺	8.276	8.268	8.272	8.204	8.212	8.208	-0.008	-1
Fenamiphos Sulfone ⁺	0.108	0.115	0.112	0.120	0.098	0.109	-0.022	-2
Fenamiphos Sulfoxide ⁺	6.930	6.250	6.590	6.490	6.380	6.435	-0.024	-2
Fenhexamid	0.700	0.691	0.696	0.673	0.674	0.674	-0.032	-3
Fludioxonil	0.185	0.197	0.191	0.193	0.188	0.191	-0.003	0
λ-Cyhalothrin	0.074	0.072	0.073	0.073	0.069	0.071	-0.027	-3
Methoxyfenozide	0.115	0.112	0.114	0.105	0.107	0.106	-0.066	-7
Pirimicarb	0.428	0.426	0.427	0.413	0.411	0.412	-0.035	-4
Pyridaben	0.136	0.141	0.139	0.139	0.137	0.138	-0.004	0
Spinosad	0.050	0.045	0.048	0.048	0.049	0.049	0.021	2
Tetraconazole	0.065	0.063	0.064	0.068	0.070	0.069	0.078	8

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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 24th February 2014.

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 16. 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 above the MRRLs that show the apparent presence of any pesticide listed in the Target Pesticide List, but which was: (i) not detected by the Organiser, even after repeated analyses, and (ii) not detected by most of the participating laboratories (i.e. 95 % of the laboratories) that had targeted that specific pesticide.

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

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

3.1.2 False negatives

These are results for any pesticide indicated by the laboratories as "analysed" but not reported or reported without numerical values, although they were used by the Organiser to treat the test item and were detected by the Organiser and the majority of the participants that had targeted this specific pesticide, at or above the MRRL.

z-Scores have been calculated for all pesticides detected and reported at levels at, or above, the MRRL. 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

The assigned values for each pesticide are based on the median level of the results reported by EU and EFTA countries laboratories, excluding outliers. Individual results without any numerical values reported, such as detected (D), were not considered. The spread of results for each pesticide was tested for multimodality. Taking into account the normative for robust analysis in ISO 13528 [2], the uncertainty was accompanying the assigned value for each pesticide, which was calculated according to the following equation:

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

Where:

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

3.3 Fixed target standard deviations

Based on the experience gained from previous EU proficiency tests and recommendations from the 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. The FFP RSD for each pesticide can be compared to Q_n RSD for informative purposes 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:

$$|z| \leq 2 \quad \text{Acceptable}$$

$$|z| > 2 \text{ but } \leq 3 \quad \text{Questionable}$$

$$|z| > 3 \quad \text{Unacceptable}$$

- Any z-score values of $|z| > 5$ have been reported as '5'.
- 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 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'.

$$\begin{aligned} |AZ^2| \leq 2 & \quad \text{Good} \\ |AZ^2| > 2 \text{ but } \leq 3 & \quad \text{Satisfactory} \\ |AZ^2| > 3 & \quad \text{Unsatisfactory} \end{aligned}$$

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-three laboratories agreed to participate in this proficiency test and all submitted results. 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, Egypt, Israel, Kenya, Peru, Saudi Arabia, Serbia, Singapore, Thailand, Turkey and Uruguay and have not been included. This last group totals fourteen laboratories.

Twenty-one pesticides were used to treat the sample. Due to degradation processes and to the presence of impurities in the commercial formulations, twenty-seven compounds (Acetamiprid, Acrinathrin, Buprofezin, Captan, Chlorothalonil, Chlorpyrifos, Cypermethrin, Cyprodinil, Diazinon, Difenoconazole, Endosulfan α , Endosulfan β , Fenhexamid, Fenamiphos, Fludioxonil, λ -Cyhalothrin, Methoxyfenozide, Pirimicarb, Pyridaben, Spinosad, Tetraconazole plus Endosulfan sulfate, Fenamiphos Sulfone, Fenamiphos Sulfoxide, Pirimicarb-desmethyl, Propiconazole and Tebuconazole) were found by both the Organiser and the participants. From them only twenty-two met the statistical criteria. Therefore those 22 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. In the case of captan, it was introduced in this proficiency test in order to have an overview on how the laboratories analysed it by multiresidue methods. As the results showed a big dispersion, the Advisory Group decided not to use it for the evaluation of the results. For Endosulfan sulfate, Pirimicarb-Desmethyl, Propiconazole and Tebuconazole, the reason for not being used for the evaluation of the laboratories was that their assigned value was lower than a factor of 4 times the MRRL, and therefore, the Advisory Group considered that they should not be evaluated. However, for informative purposes, those compounds' histograms will be included in the Final Report.

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 169)
Acetamiprid	127	1	41	75
Acrinathrin	136	4	29	80
Buprofezin	146	1	22	86
Chlorothalonil	139	6	24	82
Chlorpyrifos	166	0	3	98
Cypermethrin	154	1	14	91
Cyprodinil	152	1	16	90

Pesticides	No. of Reported Results	No. of False Negative Results	No. of Not Analysed Results	Percentage of Reported Results* (out of 169)
Diazinon	163	1	5	96
Difenoconazole	142	0	27	84
Endosulfan α	159	1	9	94
Endosulfan β	158	1	10	93
Fenamiphos	130	2	37	77
Fenamiphos Sulfone	101	4	64	60
Fenamiphos Sulfoxide	102	3	64	60
Fenhexamid	143	0	26	85
Fludioxonil	138	3	28	82
λ -Cyhalothrin	147	6	16	87
Methoxyfenozide	119	2	48	70
Pirimicarb	154	0	15	91
Pyridaben	138	1	30	82
Spinosad	115	1	53	68
Tetraconazole	139	1	29	82

* The % of Reported Results comes from 169 laboratories. It does not take into account the fourteen laboratories from Brazil, China, Egypt, Israel, 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

Two 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 MRRLs 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.

One out of those two laboratories reporting a false positive result has 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)
Lab003	Tefluthrin	0.012	GC-ECD	0.01	0.01
Lab160	Folpet	0.240	GC-MS/MS (EI)	0.02	0.01

False positives from Brazil, China, Egypt, Israel, 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 MRRLs, 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	Acetamiprid	Azinphos methyl	Buprofezin	Chlorothalonil	Chlorpyrifos	Cypermethrin	Cyprodinil	Diazinon	Difenoconazole	Endosulfan alpha	Endosulfan beta	Fenamiphos	Fenamiphos sulfone	Fenamiphos sulfoxide	Fenhexamid	Fludioxonil	Lambda-Cyhalothrin	Methoxyfenozide	Pyrimicarb	Pyridaben	Spinosad	Tetracozonole		
003					ND				ND	ND							ND							
012													ND											
014																	ND							
028				ND																				
034																ND	ND							
035																							ND	
065	ND																							
069														ND		ND								
073				ND																				
083												ND	ND	ND					ND					
092			ND																					
095				ND									ND											
098	ND																							
103																	ND							
108																					ND			
135				ND																				
143		ND																						
146	ND	ND		ND								ND	ND	ND			ND							
148	ND																ND							
164																ND		ND						
170							ND	ND																
179																							ND	
180				ND																				

False negatives from from Brazil, China, Egypt, Israel, 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 twenty-seven pesticides present in the test item are displayed in Appendix 2.

4.2 Assigned values and target standard deviations

The assigned values were based on the median values calculated using all the results reported by laboratories from EU and EFTA countries, but excluding those values that were far from the median, i.e. outliers. The assigned values for the twenty-two pesticides and the 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 (Qn) 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 Median values, uncertainty and %RSDs for all pesticides present in the test item.

Pesticides	MRRL (mg/kg)	Median (mg/kg)	Uncertainty (mg/kg)	FFP RSD (%)	Qn RSD (%)
Acetamiprid	0.010	0.632	0.010	25	15
Acrinathrin	0.010	0.261	0.005	25	19
Buprofezin	0.010	0.467	0.007	25	14
Chlorothalonil	0.010	2.230	0.076	25	32
Chlorpyrifos	0.010	3.680	0.075	25	21
Cypermethrin	0.010	0.528	0.009	25	17
Cyprodinil	0.010	0.591	0.009	25	15
Diazinon	0.010	0.084	0.001	25	16
Difenoconazole	0.010	0.953	0.019	25	19
Endosulfan α	0.010	1.160	0.020	25	17
Endosulfan β	0.010	0.975	0.017	25	18
Fenamiphos	0.010	7.385	0.170	25	21
Fenamiphos Sulfone	0.010	0.110	0.003	25	20
Fenamiphos Sulfoxide	0.010	6.635	0.195	25	24
Fenhexamid	0.010	0.861	0.018	25	20
Fludioxonil	0.010	0.241	0.005	25	18
λ -Cyhalothrin	0.010	0.077	0.002	25	20
Methoxyfenozide	0.010	0.165	0.004	25	23
Pirimicarb	0.010	0.731	0.011	25	15
Pyridaben	0.010	0.151	0.003	25	18
Spinosad	0.010	0.094	0.003	25	24
Tetraconazole	0.010	0.104	0.002	25	17

4.3 Assessment of laboratory performance

4.3.1 z-Scores

z-Scores were calculated using the FFP RSD of 25 % for all the pesticides present.

In Appendix 3, the individual z-scores are presented for each laboratory, together with the median values for each pesticide. The z-scores of laboratories from Brazil, China, Egypt, Israel, 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 (%)
Acetamiprid	98.4	0.8	0.8
Acrinathrin	90.0	5.0	5.0
Buprofezin	96.6	1.4	2.0
Chlorothalonil	84.8	7.6	7.6
Chlorpyrifos	92.2	6.6	1.2
Cypermethrin	96.1	2.6	1.3
Cyprodinil	98.0	1.3	0.7
Diazinon	96.3	1.2	2.4
Difenoconazole	96.5	3.5	0.0
Endosulfan α	91.9	3.8	4.4
Endosulfan β	93.1	3.1	3.8
Fenamiphos	89.4	6.1	4.5
Fenamiphos Sulfone	87.6	3.8	8.6
Fenamiphos Sulfoxide	89.5	5.7	4.8
Fenhexamid	97.9	2.1	0.0
Fludioxonil	93.6	2.8	3.5
λ -Cyhalothrin	92.2	2.0	5.9
Methoxyfenozide	95.0	3.3	1.7
Pirimicarb	98.1	1.9	0.0
Pyridaben	97.1	2.2	0.7
Spinosad	85.3	3.4	11.2
Tetraconazole	95.0	1.4	3.6

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 categorise the laboratories into Category A and B.

The table in Appendix 5 shows the values of individual z-scores for each pesticide and the combined 'Average of the Squared z-Scores' (AZ^2) for those EU and EFTA laboratories in Category A. In this category are the laboratories that sought and detected twenty 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 of the one hundred and sixty-nine EU and EFTA laboratories that submitted results have been classified into Category A (59 %).

From the AZ^2 , ninety-three percent were classed as 'good', five percent as 'satisfactory' and two percent as 'unsatisfactory'.

Of the sixty-nine laboratories in Category B, one 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 AZ^2 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 AZ^2 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 AZ^2 formula has been summarized as follows:

- 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
Lab126	22	0.1	Good
Lab079	22	0.1	Good
Lab060	22	0.2	Good
Lab053	22	0.2	Good
Lab072	22	0.2	Good
Lab182	22	0.2	Good
Lab101	22	0.2	Good
Lab104	22	0.2	Good
Lab110	22	0.2	Good
Lab068	22	0.2	Good
Lab056	22	0.2	Good
Lab063	22	0.2	Good
Lab001	22	0.2	Good
Lab033	22	0.2	Good
Lab050	22	0.2	Good
Lab133	22	0.2	Good
Lab082	22	0.2	Good
Lab099	20	0.3	Good
Lab128	22	0.3	Good
Lab084	22	0.3	Good
Lab178	22	0.3	Good
Lab075	22	0.3	Good
Lab112	22	0.3	Good
Lab138	22	0.3	Good
Lab046	22	0.3	Good
Lab122	22	0.3	Good
Lab085	21	0.3	Good
Lab008	21	0.3	Good
Lab057	22	0.3	Good
Lab027	21	0.3	Good
Lab136	22	0.3	Good
Lab107	22	0.3	Good
Lab119	22	0.4	Good
Lab070	22	0.4	Good
Lab011	22	0.4	Good
Lab076	22	0.4	Good
Lab016	22	0.4	Good
Lab077	22	0.4	Good
Lab058	22	0.4	Good
Lab013	22	0.4	Good
Lab009	21	0.4	Good

Lab Code	No. of pesticides detected	AZ ²	Classification
Lab017	22	0.4	Good
Lab061	22	0.4	Good
Lab177	20	0.5	Good
Lab131	22	0.5	Good
Lab093	21	0.6	Good
Lab089	22	0.6	Good
Lab054	22	0.6	Good
Lab040	22	0.6	Good
Lab167	22	0.6	Good
Lab055	22	0.6	Good
Lab051	22	0.6	Good
Lab147	21	0.6	Good
Lab002	22	0.6	Good
Lab166	22	0.6	Good
Lab123	22	0.7	Good
Lab115	22	0.7	Good
Lab096	22	0.7	Good
Lab015	22	0.8	Good
Lab078	22	0.8	Good
Lab103*	21	0.8	Good
Lab153	21	0.8	Good
Lab139	22	0.8	Good
Lab155	21	0.8	Good
Lab140	22	0.8	Good
Lab173	22	0.9	Good
Lab021	22	0.9	Good
Lab145	22	0.9	Good
Lab171	22	0.9	Good
Lab030	20	0.9	Good
Lab121	21	0.9	Good
Lab031	22	0.9	Good
Lab062	22	0.9	Good
Lab097	22	0.9	Good
Lab042	22	1.0	Good
Lab049	22	1.0	Good
Lab020	22	1.0	Good
Lab091	21	1.0	Good
Lab118	22	1.0	Good
Lab100	22	1.2	Good
Lab018	20	1.3	Good
Lab066	22	1.3	Good
Lab081	22	1.3	Good

Lab Code	No. of pesticides detected	AZ ²	Classification
Lab007	22	1.3	Good
Lab151	22	1.4	Good
Lab172	22	1.4	Good
Lab028*	20	1.4	Good
Lab106	21	1.4	Good
Lab067	21	1.5	Good
Lab105	22	1.6	Good
Lab065*	21	1.7	Good
Lab095*	20	1.9	Good
Lab032	22	2.0	Good
Lab149	20	2.1	Satisfactory
Lab090	22	2.2	Satisfactory
Lab174	22	2.2	Satisfactory
Lab141	21	2.2	Satisfactory
Lab012*	20	2.3	Satisfactory
Lab157	22	3.8	Unsatisfactory
Lab164*	20	5.0	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 (22)	No. of pesticides detected	No. of total z-scores	No. of acceptable z-scores (z- score ≤ 2)
Lab160 ⁺	100	22	22	18
Lab044	86	19	19	19
Lab080	86	19	19	19
Lab120	86	19	19	19
Lab179*	86	19	20	19
Lab059	86	19	19	17
Lab034*	86	19	21	15
Lab169	86	19	19	19
Lab117	82	18	18	18
Lab130	82	18	18	18
Lab102	82	18	18	17
Lab163	82	18	18	17
Lab083*	82	18	22	16
Lab069*	82	18	20	15
Lab038	77	17	17	17
Lab064	77	17	17	17
Lab159	77	17	17	16
Lab161	77	17	17	16

Lab Code	% No. of pesticides detected / No. of pesticides evaluated (22)	No. of pesticides detected	No. of total z-scores	No. of acceptable z-scores (z- score ≤ 2)
Lab010	77	17	17	15
Lab023	73	16	16	16
Lab036	73	16	16	16
Lab087	73	16	16	16
Lab088	73	16	16	16
Lab181	73	16	16	16
Lab146*	73	16	22	13
Lab045	68	15	15	15
Lab047	68	15	15	15
Lab003**	68	15	19	14
Lab029	68	15	15	14
Lab108*	68	15	16	12
Lab048	64	14	14	14
Lab086	64	14	14	14
Lab152	64	14	14	14
Lab014*	64	14	15	13
Lab129	64	14	14	13
Lab150	64	14	14	13
Lab035*	64	14	15	12
Lab098*	64	14	15	12
Lab158	64	14	14	11
Lab073*	59	13	14	13
Lab134	59	13	13	13
Lab041	59	13	13	11
Lab127	55	12	12	9
Lab124	55	12	12	0
Lab005	50	11	11	11
Lab043	50	11	11	11
Lab074	50	11	11	11
Lab125	50	11	11	11
Lab168	50	11	11	10
Lab026	50	11	11	8
Lab148*	50	11	13	4
Lab109	45	10	10	10
Lab143*	45	10	11	10
Lab004	41	9	9	9
Lab180*	41	9	10	9
Lab039	41	9	9	8
Lab092*	36	8	9	8
Lab154	36	8	8	8
Lab113	32	7	7	7
Lab170*	32	7	9	2
Lab175	27	6	6	6

Lab Code	% No. of pesticides detected / No. of pesticides evaluated (22)	No. of pesticides detected	No. of total z-scores	No. of acceptable z-scores (z- score ≤ 2)
Lab037	27	6	6	4
Lab022	23	5	5	5
Lab094	23	5	5	5
Lab144	23	5	5	5
Lab176	23	5	5	1
Lab135*	18	4	5	1
Lab132	9	2	2	2
Lab156	9	2	2	0

* Laboratories reporting a false negative result.

+ Laboratories reporting a false positive result.

5. CONCLUSIONS

One hundred and eighty-three laboratories agreed to participate in EUPT-FV-16. All of them submitted results for the analysis of the pepper homogenate test item. Fourteen of those submitting results were not from EU or EFTA countries, so no statistical analysis was conducted on their results.

Some of the pesticide residue levels in the test item were intentionally higher than in previous EUPTs-FV. The main reason was to evaluate the capacity of the EU NRLs and OfLs for reporting values above the maximum residue levels (MRLs) and the generation of RASFF notifications. However, as the test item was prepared with incurred pesticides, sometimes it was very difficult to achieve a specific concentration value, and that was the reason for extremely high values. The high number of analytes present in the test item together with the high values of some of them increased the complexity of the exercise.

Six additional pesticides that were not used to treat the test item were reported by the laboratories and detected by the Organisers: Endosulfan sulfate, Fenamiphos Sulfone, Fenamiphos Sulfoxide, Pirimicarb-desmethyl, Propiconazole and Tebuconazole. In the cases of Endosulfan sulfate, Fenamiphos Sulfone, Fenamiphos Sulfoxide, Pirimicarb-desmethyl, they were formed from the degradation of Endosulfan, Fenamiphos and Pirimicarb, respectively. The presence of Propiconazole and Tebuconazole in the test item was probably due to contamination or impurities in the commercial formulations.

Captan, Endosulfan sulfate, Pirimicarb-desmethyl, Propiconazole and Tebuconazole were not used in the evaluation of the laboratories for statistical reasons.

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 twenty 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 twenty 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 (twenty-two). Laboratories reporting false negatives were

marked with an asterisk. Laboratories having reported a false positive have been marked with a '+'.

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

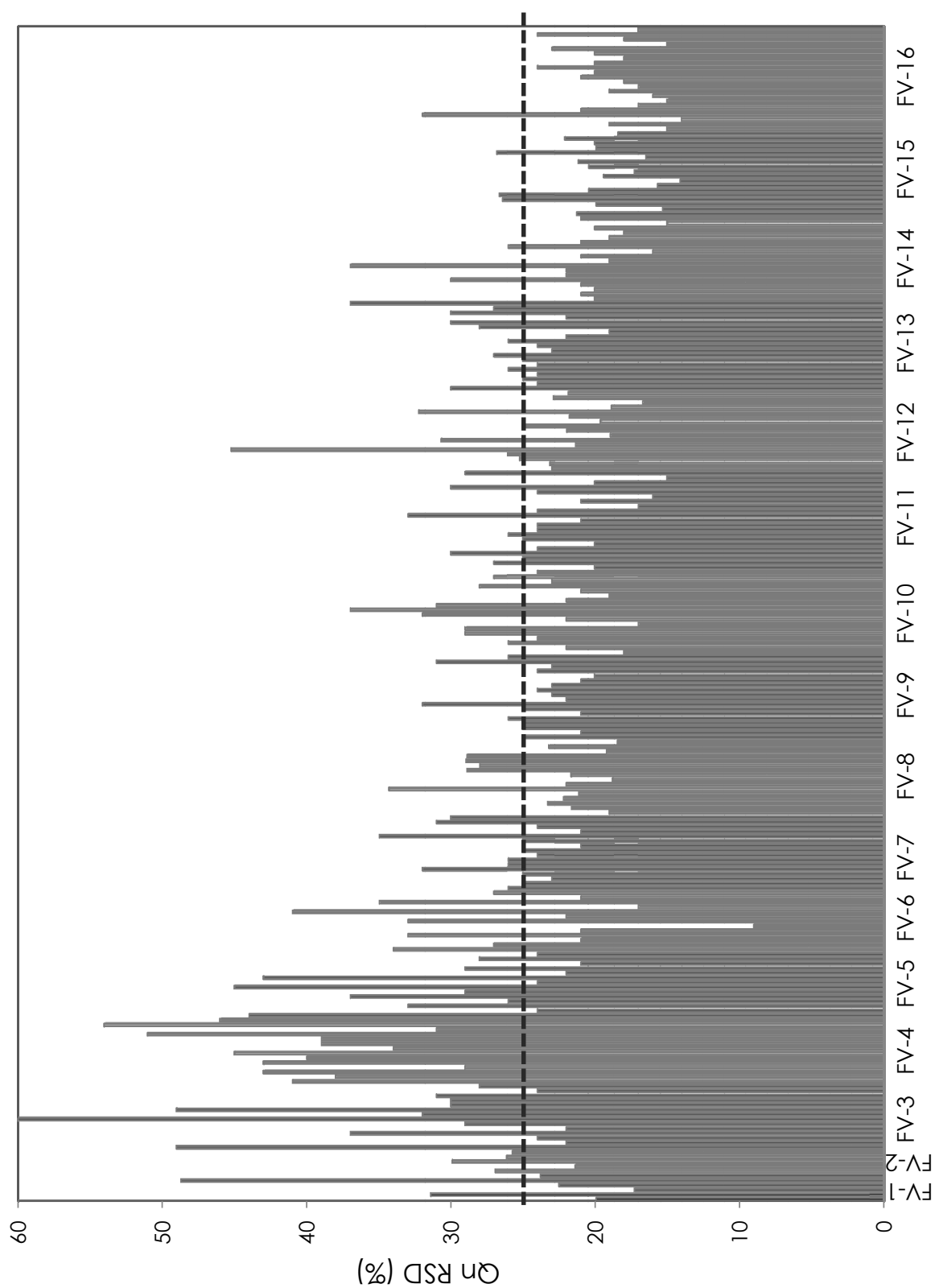
Overall, the results were very good with regard to the individual z-scores for each pesticide present in the test item. Most of the pesticides had only a few unacceptable z-scores. Therefore, laboratories generally achieved accurate results for all the pesticides present in the test item – at, or above, 85 %. As in EUPT-FV-15, chlorothalonil was the pesticide with the lowest percentage (85 %) of acceptable results in terms of z-scores.

Moreover, despite the high number of pesticides present in the test item and the wide range of concentrations, the percentage of laboratories in Category A (59 %) has increased slightly compared to last year's (55 %).

Participation in this year's European Proficiency Test 16 involved at least one laboratory from each Member State. Additionally, Iceland, Norway and Switzerland participated as EFTA countries. Non-European laboratories in Brazil, China, Egypt, Israel, Saudi Arabia, Serbia, Turkey and Uruguay also participated (as in previous years) although this year, they were joined by Kenya, Peru, Singapore and Thailand 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.

The concentration of the pesticides in the test item does not affect in a relevant way the data dispersion (Qn RSD) (See Table 4.4). Figure 1 shows the different Qn RSD values for all the pesticides present in the test items from EUPT-FV1 to EUPT-FV16. Along the years, the dispersion of the results has decreased markedly, which demonstrates how participants performances have improved with time.

Figure1. Evolution of data dispersion along the EUPT-FV history (Qn RSD, %)



6. SUGGESTIONS FOR FUTURE WORK

The use of incurred pesticides in the test item will continue in next EUPT-FV-17. The number and concentrations of pesticides that will be present in the treated test item will be chosen so that they do not have a big impact on the workflow of participating laboratories.

As a result of the continuing trend in performance improvement, the stricter criteria applied to EUPT-FV-16 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.

It is a desire of the Organisation to delete from the pesticide target list those pesticides that are clearly "single residue methods" compounds and include the new pesticides in Annex I of the EU multi-annual control programme [6].

In future PTs, the assigned value will be estimated using the robust mean, as described in ISO 13528 [2].

These changes are aimed at ensuring that, year on year, laboratories strive even more 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 quantifying 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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The Organiser wishes to give a special thank-you to the University of Almeria for the use of their facilities.

APPENDIX 1. Homogeneity data.

The analytical results marked with + are not covered by the ENAC accreditation

Acetamidrid (mg/kg)		Acrinathrin (mg/kg)		Buprofezin (mg/kg)		Chlorothalonil (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.620	0.650	0.270	0.270	0.270	0.320	2.120	2.160
0.670	0.660	0.230	0.230	0.260	0.260	1.740	1.820
0.480	0.670	0.260	0.250	0.320	0.260	2.300	1.980
0.700	0.630	0.250	0.270	0.290	0.300	1.970	2.130
0.430	0.470	0.260	0.290	0.290	0.330	1.910	2.550
0.640	0.630	0.270	0.270	0.300	0.290	2.040	1.980
0.640	0.690	0.280	0.260	0.350	0.300	2.600	1.880
0.620	0.430	0.250	0.260	0.300	0.300	1.880	1.770
0.720	0.680	0.260	0.260	0.300	0.300	2.060	1.850
0.660	0.720	0.290	0.290	0.330	0.390	2.340	2.460

Chlorpyrifos (mg/kg)		Cypermethrin (mg/kg)		Cyprodinil (mg/kg)		Diazinon (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
2.850	3.240	0.390	0.480	0.350	0.440	0.060	0.060
2.610	2.550	0.330	0.380	0.330	0.330	0.050	0.060
3.150	2.730	0.440	0.360	0.390	0.350	0.070	0.060
2.930	2.960	0.360	0.420	0.330	0.360	0.060	0.060
2.910	3.510	0.390	0.480	0.360	0.440	0.060	0.070
2.900	2.870	0.390	0.390	0.390	0.390	0.060	0.060
3.660	2.970	0.480	0.390	0.470	0.380	0.080	0.070
2.960	2.960	0.410	0.350	0.360	0.380	0.060	0.060
2.810	2.690	0.410	0.350	0.390	0.330	0.070	0.070
3.500	4.000	0.480	0.510	0.450	0.510	0.070	0.080

Difenoconazole (mg/kg)		Endosulfan α (mg/kg)		Endosulfan β (mg/kg)		Fenamiphos ⁺ (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.510	0.600	0.750	0.900	0.530	0.600	9.230	8.010
0.500	0.500	0.660	0.690	0.470	0.470	8.280	9.420
0.570	0.500	0.830	0.680	0.530	0.480	7.320	8.910
0.500	0.540	0.690	0.770	0.450	0.480	9.380	8.130
0.530	0.630	0.720	0.900	0.500	0.630	8.620	6.530
0.530	0.530	0.750	0.750	0.560	0.530	7.490	6.950
0.630	0.540	0.890	0.720	0.650	0.510	7.670	8.030
0.530	0.530	0.720	0.710	0.510	0.500	8.260	5.900
0.530	0.500	0.770	0.710	0.530	0.470	8.140	7.810
0.650	0.710	0.860	1.050	0.660	0.690	10.680	10.100

APPENDIX 1. Homogeneity data.

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Fenamiphos Sulfone ⁺ (mg/kg)		Fenamiphos Sulfoxide ⁺ (mg/kg)		Fenhexamid (mg/kg)		Fludioxonil (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.110	0.120	6.630	6.645	0.720	0.710	0.180	0.190
0.097	0.092	6.060	6.120	0.670	0.610	0.140	0.150
0.092	0.083	6.921	7.015	0.560	0.630	0.190	0.170
0.112	0.108	6.528	6.641	0.820	0.610	0.180	0.200
0.110	0.116	6.045	6.220	0.680	0.510	0.180	0.190
0.083	0.083	6.566	6.637	0.750	0.740	0.200	0.170
0.092	0.105	6.820	6.723	0.670	0.750	0.260	0.170
0.116	0.120	6.491	6.555	0.710	0.430	0.180	0.210
0.097	0.085	6.469	6.400	0.760	0.610	0.180	0.180
0.105	0.120	7.063	6.880	0.860	0.820	0.240	0.250

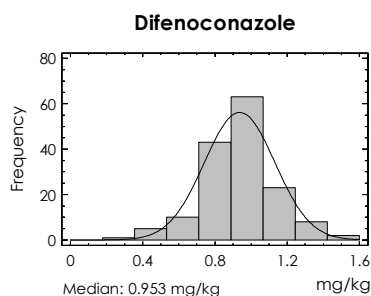
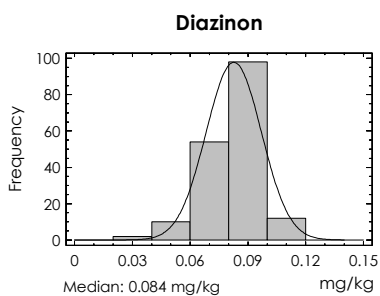
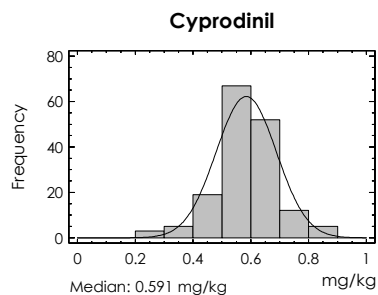
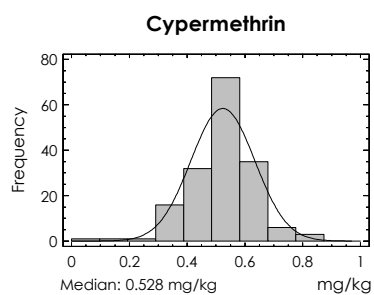
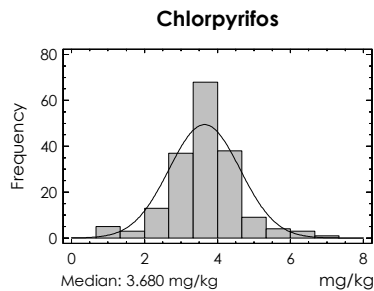
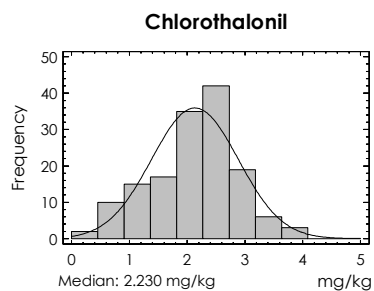
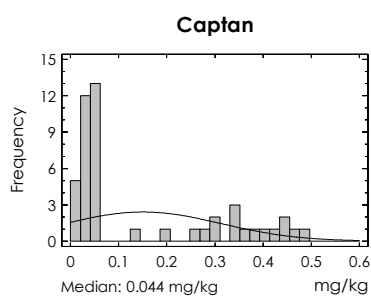
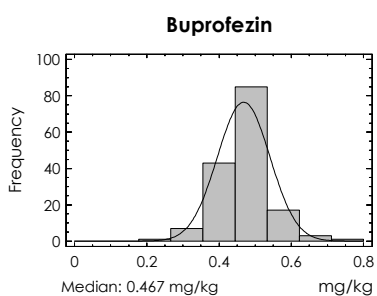
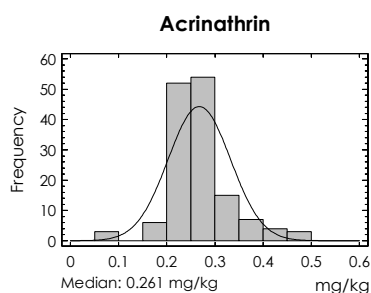
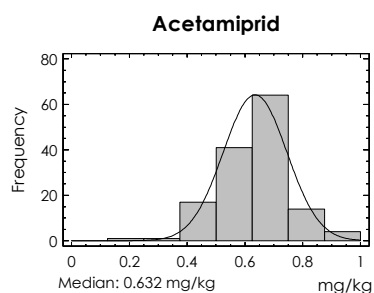
λ-Cyhalothrin (mg/kg)		Methoxyfenozide (mg/kg)		Pirimicarb (mg/kg)		Pyridaben (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.080	0.080	0.110	0.100	0.390	0.470	0.120	0.130
0.070	0.070	0.100	0.090	0.410	0.450	0.100	0.100
0.080	0.070	0.100	0.100	0.380	0.360	0.130	0.110
0.080	0.080	0.120	0.110	0.390	0.410	0.120	0.120
0.070	0.090	0.110	0.080	0.410	0.420	0.130	0.160
0.080	0.080	0.110	0.110	0.390	0.390	0.120	0.120
0.090	0.080	0.110	0.100	0.510	0.410	0.160	0.130
0.080	0.080	0.120	0.080	0.390	0.440	0.120	0.120
0.080	0.070	0.110	0.090	0.410	0.420	0.120	0.120
0.090	0.090	0.130	0.130	0.300	0.480	0.140	0.160

Spinosad (mg/kg)		Tetraconazole (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.050	0.040	0.07	0.08
0.050	0.050	0.06	0.07
0.040	0.050	0.07	0.07
0.060	0.050	0.07	0.07
0.040	0.040	0.07	0.08
0.050	0.050	0.07	0.07
0.050	0.050	0.08	0.08
0.060	0.040	0.07	0.07
0.050	0.040	0.07	0.07
0.070	0.050	0.08	0.08

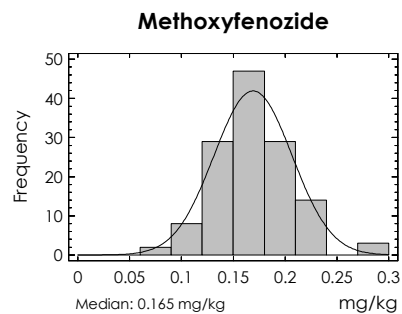
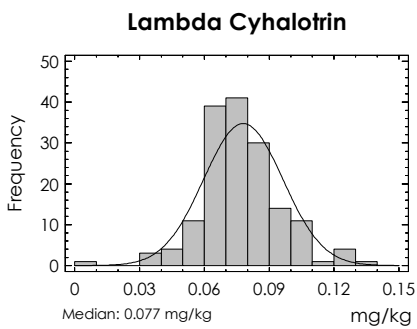
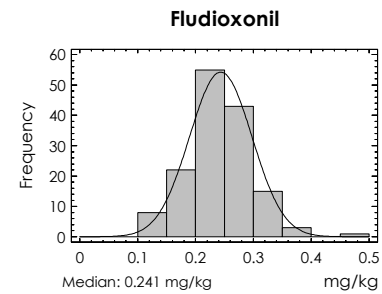
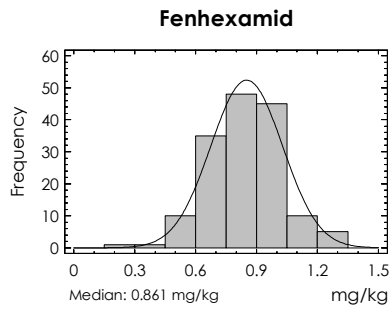
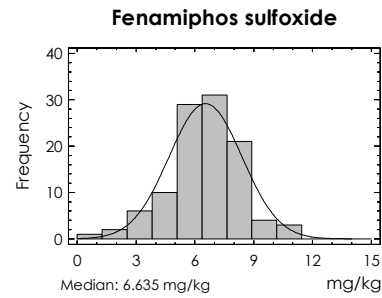
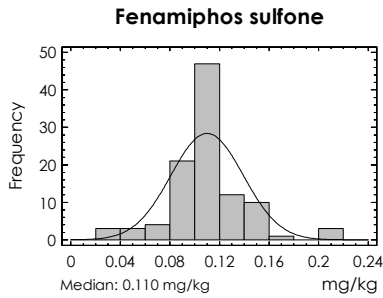
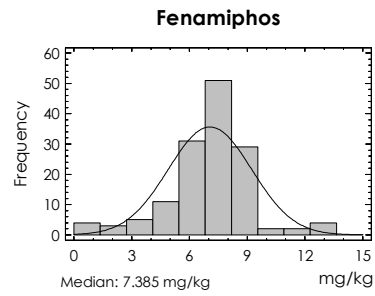
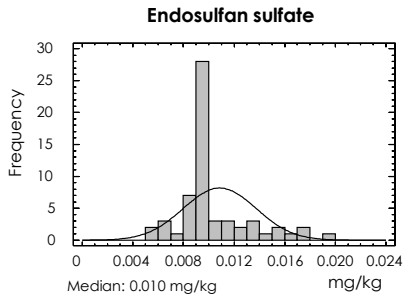
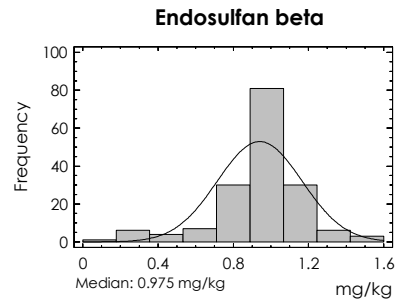
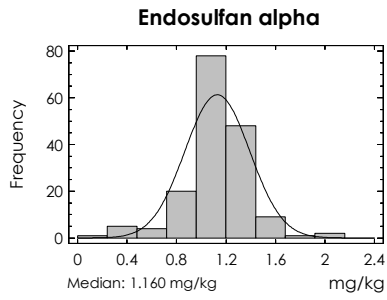
The sample numbers used for this test were: 54, 69, 86, 126, 128, 130, 147, 178, 191 and 207.

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

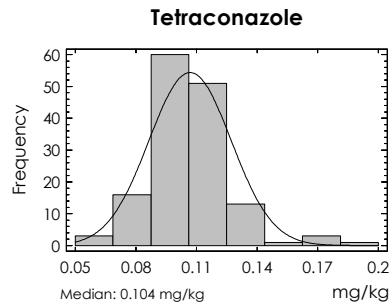
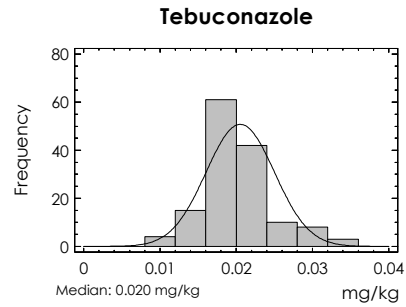
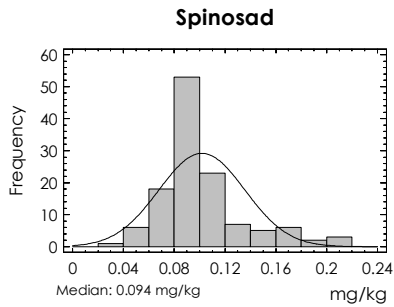
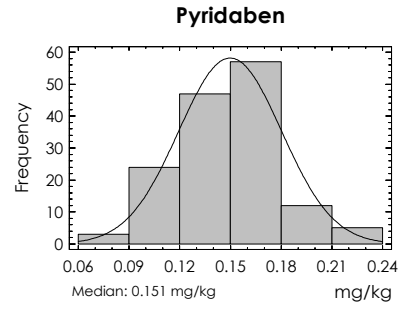
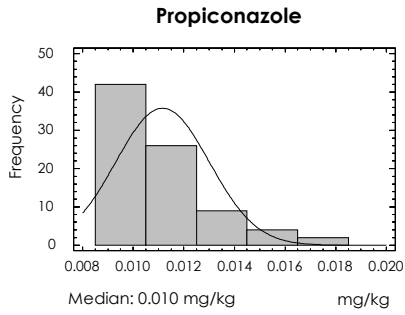
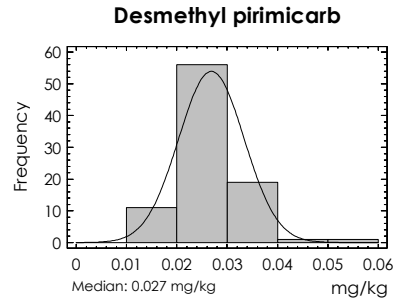
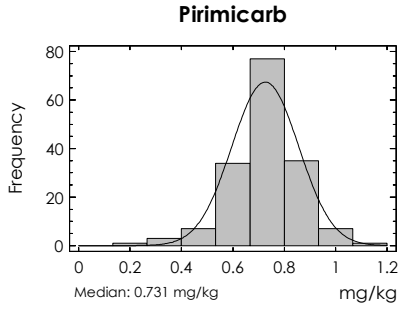
Results presented as histograms.



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



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



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

Lab Code	Acetamiprid	Acinathrin		Buprofezin	Chlorothalonil		Chlorpyrifos	Cypermethrin		Cyprodinil	Diazinon		Difenoconazole	Endosulfan alpha		Endosulfan beta	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	0.01	0.01	0.01	0.01	0.01				
Median (mg/kg)	0.632	0.261	0.467	2.230	3.680	0.528	0.591	0.084	0.953	1.160	0.975											
Lab147	0.510	-0,8	0.380	1,8	0.500	0,3	2.580	0,6	3.910	0,3	0.710	1,4	0.730	0,9	0.092	0,4	1.240	1,2	1.260	0,3	1.100	0,5
Lab148	NA		ND	-3,8	0.750	2,4	NA		5.960	2,5	2.737	5,0	0.808	1,5	0.084	0,0	1.474	2,2	1.565	1,4	1.970	4,1
Lab149	0.226	-2,6	0.415	2,4	0.484	0,1	2.262	0,1	2.057	-1,8	0.520	-0,1	0.526	-0,4	0.084	0,0	0.627	-1,4	0.997	-0,6	0.879	-0,4
Lab150	NA		0.300	0,6	0.620	1,3	NA		3.500	-0,2	0.490	-0,3	0.610	0,1	0.090	0,3	1.100	0,6	0.990	-0,6	0.820	-0,6
Lab151	0.636	0,0	0.216	-0,7	0.449	-0,2	1.160	-1,9	3.040	-0,7	0.466	-0,5	0.549	-0,3	0.075	-0,4	0.669	-1,2	1.020	-0,5	0.813	-0,7
Lab152	NA		NA		0.477	0,1	2.274	0,1	4.175	0,5	0.678	1,1	0.591	0,0	0.094	0,5	1.065	0,5	1.098	-0,2	0.968	0,0
Lab153	0.645	0,1	0.375	1,7	0.522	0,5	1.690	-1,0	3.850	0,2	0.518	-0,1	0.591	0,0	0.092	0,4	0.958	0,0	1.258	0,3	1.113	0,6
Lab154	NA		0.250	-0,2	NA		2.200	-0,1	4.000	0,3	0.550	0,2	NA		0.080	-0,2	NA		1.300	0,5	1.030	0,2
Lab155	0.610	-0,1	NA		0.420	-0,4	2.000	-0,4	4.300	0,7	0.540	0,1	0.500	-0,6	0.100	0,8	0.780	-0,7	1.100	-0,2	0.530	-1,8
Lab156	NA		NA		NA		NA		NA		NA		NA		NA		NA		0.080	-3,7	0.190	-3,2
Lab157	0.530	-0,6	0.230	-0,5	0.280	-1,6	0.380	-3,3	2.190	-1,6	0.350	-1,3	0.230	-2,4	0.090	0,3	0.600	-1,5	0.400	-2,6	0.320	-2,7
Lab158	NA		0.240	-0,3	0.440	-0,2	0.540	-3,0	1.090	-2,8	0.470	-0,4	0.520	-0,5	0.060	-1,1	NA		0.990	-0,6	0.810	-0,7
Lab159	0.500	-0,8	NA		0.460	-0,1	0.530	-3,0	2.710	-1,1	0.540	0,1	0.390	-1,4	0.086	0,1	1.050	0,4	0.880	-1,0	1.030	0,2
Lab160	0.572	-0,4	0.414	2,3	0.460	-0,1	2.150	-0,1	4.490	0,9	0.840	2,4	0.290	-2,0	0.085	0,0	0.524	-1,8	1.430	0,9	1.150	0,7
Lab161	NA		0.201	-0,9	0.453	-0,1	1.950	-0,5	3.370	-0,3	0.427	-0,8	0.530	-0,4	0.770	5,0	0.990	0,2	1.200	0,1	1.040	0,3
Lab162	0.644	0,1	0.242	-0,3	0.497	0,3	1.643	-1,1	3.359	-0,3	0.395	-1,0	0.532	-0,4	0.081	-0,1	0.907	-0,2	0.881	-1,0	0.726	-1,0
Lab163	0.499	-0,8	0.387	1,9	0.448	-0,2	3.012	1,4	3.680	0,0	0.623	0,7	0.640	0,3	0.082	-0,1	1.031	0,3	1.149	0,0	NA	
Lab164	0.548	-0,5	0.083	-2,7	0.371	-0,8	1.241	-1,8	7.318	4,0	0.450	-0,6	0.606	0,1	0.077	-0,3	0.752	-0,8	1.140	-0,1	0.974	0,0
Lab165	0.623	-0,1	0.490	3,5	0.417	-0,4	0.835	-2,5	3.684	0,0	0.535	0,0	0.424	-1,1	0.058	-1,3	0.818	-0,6	1.096	-0,2	1.020	0,2
Lab166	0.696	0,4	0.274	0,2	0.440	-0,2	1.090	-2,0	3.230	-0,5	0.474	-0,4	0.589	0,0	0.087	0,1	1.180	1,0	1.010	-0,5	0.936	-0,2
Lab167	0.539	-0,6	0.313	0,8	0.390	-0,7	3.070	1,5	2.206	-1,6	0.322	-1,6	0.450	-1,0	0.081	-0,2	0.822	-0,5	1.207	0,2	1.226	1,0
Lab168	NA		0.300	0,6	0.390	-0,7	1.100	-2,0	2.700	-1,1	0.820	2,2	0.430	-1,1	0.080	-0,2	NA		0.890	-0,9	0.950	-0,1
Lab169	0.435	-1,2	0.237	-0,4	0.389	-0,7	NA		3.270	-0,4	0.486	-0,3	0.508	-0,6	0.067	-0,8	0.694	-1,1	0.944	-0,7	1.260	1,2
Lab170	NA		NA		0.100	-3,1	3.060	1,5	0.920	-3,0	NA		ND	-3,9	ND	-3,5	NA		0.240	-3,2	0.210	-3,1
Lab171	0.600	-0,2	0.280	0,3	0.460	-0,1	2.600	0,7	4.000	0,3	0.580	0,4	0.700	0,7	0.120	1,7	1.100	0,6	1.200	0,1	0.990	0,1
Lab172	0.900	1,7	0.280	0,3	0.440	-0,2	2.800	1,0	3.900	0,2	0.540	0,1	0.580	-0,1	0.090	0,3	0.880	-0,3	1.700	1,9	0.730	-1,0
Lab173	0.670	0,2	0.300	0,6	0.490	0,2	3.120	1,6	4.430	0,8	0.600	0,5	0.610	0,1	0.090	0,3	1.080	0,5	1.280	0,4	1.060	0,3
Lab174	0.450	-1,2	0.466	3,1	0.354	-1,0	0.890	-2,4	2.310	-1,5	0.550	0,2	0.470	-0,8	0.060	-1,1	0.430	-2,2	0.840	-1,1	0.688	-1,2
Lab175	NA		NA		NA		NA		3.500	-0,2	0.570	0,3	NA		0.080	-0,2	NA		1.200	0,1	1.200	0,9
Lab176	NA		NA		NA		1.140	-2,0	5.860	2,4	NA		NA		0.185	4,8	NA		0.430	-2,5	0.138	-3,4
Lab177	0.761	0,8	0.274	0,2	0.486	0,2	2.504	0,5	3.536	-0,2	0.376	-1,2	0.618	0,2	0.091	0,3	1.043	0,4	1.122	-0,1	0.915	-0,2
Lab178	0.694	0,4	0.223	-0,6	0.444	-0,2	2.348	0,2	4.082	0,4	0.444	-0,6	0.575	-0,1	0.061	-1,1	1.077	0,5	1.120	-0,1	1.020	0,2
Lab179	0.750	0,7	0.250	-0,2	0.490	0,2	2.120	-0,2	4.300	0,7	0.600	0,5	0.580	-0,1	0.106	1,0	0.925	-0,1	1.290	0,4	0.890	-0,3
Lab180	NA		NA		NA		ND	-4,0	5.200	1,7	NA		NA		0.083	0,0	1.100	0,6	0.980	-0,6	1.300	1,3
Lab181	0.700	0,4	0.210	-0,8	0.460	-0,1	2.900	1,2	4.300	0,7	0.580	0,4	0.600	0,1	0.090	0,3	0.850	-0,4	1.200	0,1	0.910	-0,3
Lab182	0.598	-0,2	0.245	-0,2	0.422	-0,4	2.320	0,2	3.630	-0,1	0.568	0,3	0.644	0,4	0.093	0,4	0.930	-0,1	1.160	0,0	1.040	0,3
Lab183	0.660	0,2	0.313	0,8	0.517	0,4	NA		3.930	0,3	0.476	-0,4	0.457	-0,9	0.085	0,0	0.935	-0,1	NA		NA	
Lab184	NA		NA		NA		0.780	-2,6	3.270	-0,4	0.020	-3,8	NA		0.065	-0,9	NA		0.660	-1,7	0.500	-1,9

NA: Not analysed

NA*: A numerical value was not reported

ND: Not detected (False negative)

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

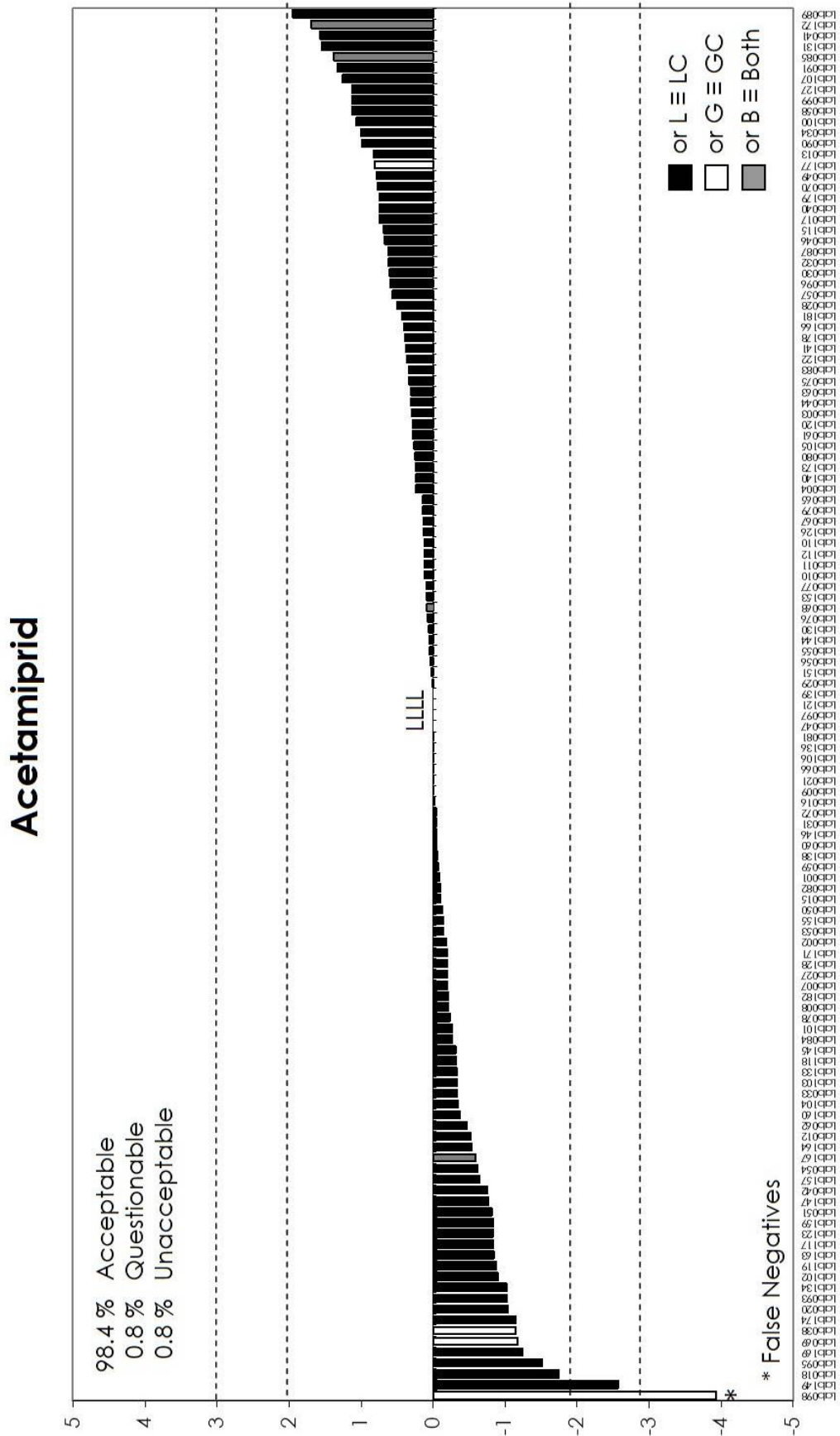
Lab Code	Fenamiphos	Fenamiphos sulfone		Fenamiphos sulfoxide	Fenhexamid		Fludioxonil	Lambda-Cyhalothrin		Methoxyfenozide	Pirimicarb		Pyridaben	Spinosad		Tetraconazole						
		z-Score (FFP RSD 25%)	0.01		z-Score (FFP RSD 25%)	0.01		z-Score (FFP RSD 25%)	0.01		z-Score (FFP RSD 25%)	0.01		z-Score (FFP RSD 25%)	0.01		z-Score (FFP RSD 25%)	0.01				
MRRL	0.01	0.110		6.635	0.861		0.241	0.077		0.165	0.731		0.151	0.094		0.104						
Median (mg/kg)	7.385	0.110		6.635	0.861		0.241	0.077		0.165	0.731		0.151	0.094		0.104						
Lab147	7.200	-0,1	0.140	1,1	8.000	0,8	0.920	0,3	0.250	0,1	0.080	0,2	0.160	-0,1	0.910	1,0	0.140	-0,3	NA	0.130	1,0	
Lab148	NA		NA		NA		1.250	1,8	NA		ND	-3,5	NA		0.815	0,5	NA		NA	0.277	5,0	
Lab149	2.101	-2,9	NA		NA		0.506	-1,6	0.171	-1,2	0.073	-0,2	0.240	1,8	0.301	-2,4	0.104	-1,2	0.073	-0,9	0.095	-0,3
Lab150	NA		NA		NA		1.200	1,6	0.400	2,6	0.090	0,7	NA		0.740	0,0	NA		NA	0.090	-0,5	
Lab151	3.230	-2,3	0.113	0,1	1.520	-3,1	1.310	2,1	0.232	-0,1	0.056	-1,1	0.142	-0,6	0.894	0,9	0.154	0,1	0.121	1,1	0.094	-0,4
Lab152	NA		NA		NA		0.951	0,4	0.208	-0,5	0.086	0,5	NA		NA		0.155	0,1	NA	0.095	-0,3	
Lab153	5.520	-1,0	0.120	0,4	3.420	-1,9	0.749	-0,5	0.198	-0,7	0.101	1,2	0.235	1,7	0.710	-0,1	0.160	0,2	NA	0.139	1,3	
Lab154	NA		NA		NA		NA		NA		0.080	0,2	NA		NA		NA		NA	NA	NA	
Lab155	6.150	-0,7	0.085	-0,9	6.650	0,0	0.720	-0,7	0.300	1,0	0.110	1,7	0.230	1,6	0.720	-0,1	0.180	0,8	0.063	-1,3	0.130	1,0
Lab156	NA		NA		NA		NA		NA		NA		NA		NA		NA		NA	NA	NA	
Lab157	4.100	-1,8	0.090	-0,7	8.630	1,2	0.400	-2,1	0.220	-0,3	0.060	-0,9	0.070	-2,3	0.720	-0,1	0.200	1,3	0.070	-1,0	0.360	5,0
Lab158	12.500	2,8	NA		NA		NA		0.260	0,3	0.070	-0,4	NA		0.830	0,5	0.140	-0,3	NA	NA	NA	
Lab159	6.850	-0,3	NA		NA		0.600	-1,2	NA		NA		0.214	1,2	0.870	0,8	0.225	2,0	0.099	0,2	0.123	0,7
Lab160	10.970	1,9	0.130	0,7	10.13	2,1	0.735	-0,6	0.243	0,0	0.140	3,3	0.106	-1,4	0.612	-0,7	0.220	1,8	0.139	1,9	0.101	-0,1
Lab161	6.920	-0,3	NA		NA		0.850	-0,1	0.240	0,0	0.073	-0,2	NA		0.750	0,1	0.160	0,2	NA	0.100	-0,2	
Lab162	8.285	0,5	0.163	1,9	ND	-4,0	0.824	-0,2	0.318	1,3	0.101	1,2	0.161	-0,1	0.789	0,3	0.164	0,3	0.091	-0,1	0.104	0,0
Lab163	NA		NA		NA		0.937	0,4	0.270	0,5	0.061	-0,8	0.240	1,8	0.602	-0,7	0.163	0,3	0.194	4,3	0.137	1,3
Lab164	7.004	-0,2	0.301	5,0	15.63	5,0	0.666	-0,9	ND	-3,8	0.081	0,2	ND	-3,8	0.655	-0,4	0.142	-0,2	0.252	5,0	0.100	-0,2
Lab165	4.770	-1,4	NA		NA		0.792	-0,3	0.207	-0,6	0.057	-1,0	0.152	-0,3	0.823	0,5	0.126	-0,7	0.074	-0,8	0.094	-0,4
Lab166	5.620	-1,0	0.040	-2,5	7.210	0,3	0.854	0,0	0.261	0,3	0.075	-0,1	0.159	-0,1	0.709	-0,1	0.171	0,5	0.082	-0,5	0.103	0,0
Lab167	6.239	-0,6	0.125	0,6	6.512	-0,1	0.660	-0,9	0.228	-0,2	0.085	0,4	0.147	-0,4	0.655	-0,4	0.136	-0,4	0.090	-0,2	0.093	-0,4
Lab168	NA		NA		NA		NA		NA		0.110	1,7	NA		0.470	-1,4	NA		NA	NA	NA	
Lab169	6.170	-0,7	NA		NA		0.663	-0,9	0.193	-0,8	0.067	-0,5	0.132	-0,8	0.682	-0,3	0.145	-0,2	0.072	-0,9	0.092	-0,5
Lab170	4.030	-1,8	NA		2.490	-2,5	NA		NA		NA		NA		NA		NA		NA	NA	NA	
Lab171	7.900	0,3	0.110	0,0	5.300	-0,8	0.760	-0,5	0.340	1,6	0.066	-0,6	0.210	1,1	0.660	-0,4	0.190	1,0	0.130	1,5	0.170	2,5
Lab172	13.400	3,3	0.110	0,0	9.100	1,5	1.000	0,6	0.310	1,1	0.074	-0,2	0.130	-0,8	1.100	2,0	0.100	-1,4	0.110	0,7	0.110	0,2
Lab173	5.620	-1,0	0.100	-0,4	7.280	0,4	0.970	0,5	0.260	0,3	0.080	0,2	0.210	1,1	0.860	0,7	0.170	0,5	0.170	3,2	0.120	0,6
Lab174	6.219	-0,6	0.157	1,7	3.229	-2,1	0.677	-0,9	0.160	-1,3	0.058	-1,0	0.138	-0,7	0.533	-1,1	0.086	-1,7	0.071	-1,0	0.070	-1,3
Lab175	NA		NA		NA		NA		NA		NA		NA		0.820	0,5	NA		NA	NA	NA	
Lab176	NA		NA		NA		NA		NA		NA		NA		NA		NA		NA	NA	NA	
Lab177	6.744	-0,3	NA		NA		1.126	1,2	0.271	0,5	0.073	-0,2	0.206	1,0	0.771	0,2	0.176	0,7	0.100	0,3	0.060	-1,7
Lab178	7.627	0,1	0.115	0,2	7.662	0,6	0.863	0,0	0.210	-0,5	0.054	-1,2	0.137	-0,7	0.785	0,3	0.124	-0,7	0.091	-0,1	0.095	-0,3
Lab179	5.100	-1,2	NA		NA		0.900	0,2	0.310	1,1	0.078	0,1	0.213	1,2	0.750	0,1	0.162	0,3	ND	-3,6	0.081	-0,9
Lab180	NA		NA		NA		0.640	-1,0	NA		NA		0.150	-0,4	0.720	-0,1	0.160	0,2	NA	NA	NA	
Lab181	NA		NA		NA		0.880	0,1	0.260	0,3	0.066	-0,6	NA		0.870	0,8	NA		NA	0.092	-0,5	
Lab182	6.140	-0,7	0.125	0,5	7.380	0,4	0.693	-0,8	0.263	0,4	0.088	0,6	0.175	0,2	0.784	0,3	0.142	-0,2	0.093	0,0	0.124	0,8
Lab183	9.507	1,1	NA		NA		1.030	0,8	0.300	1,0	0.072	-0,3	0.194	0,7	0.795	0,4	0.185	0,9	0.092	-0,1	0.176	2,8
Lab184	3.227	-2,3	NA		NA		NA		NA		0.050	-1,4	NA		NA		NA		NA	NA	NA	

NA: Not analysed

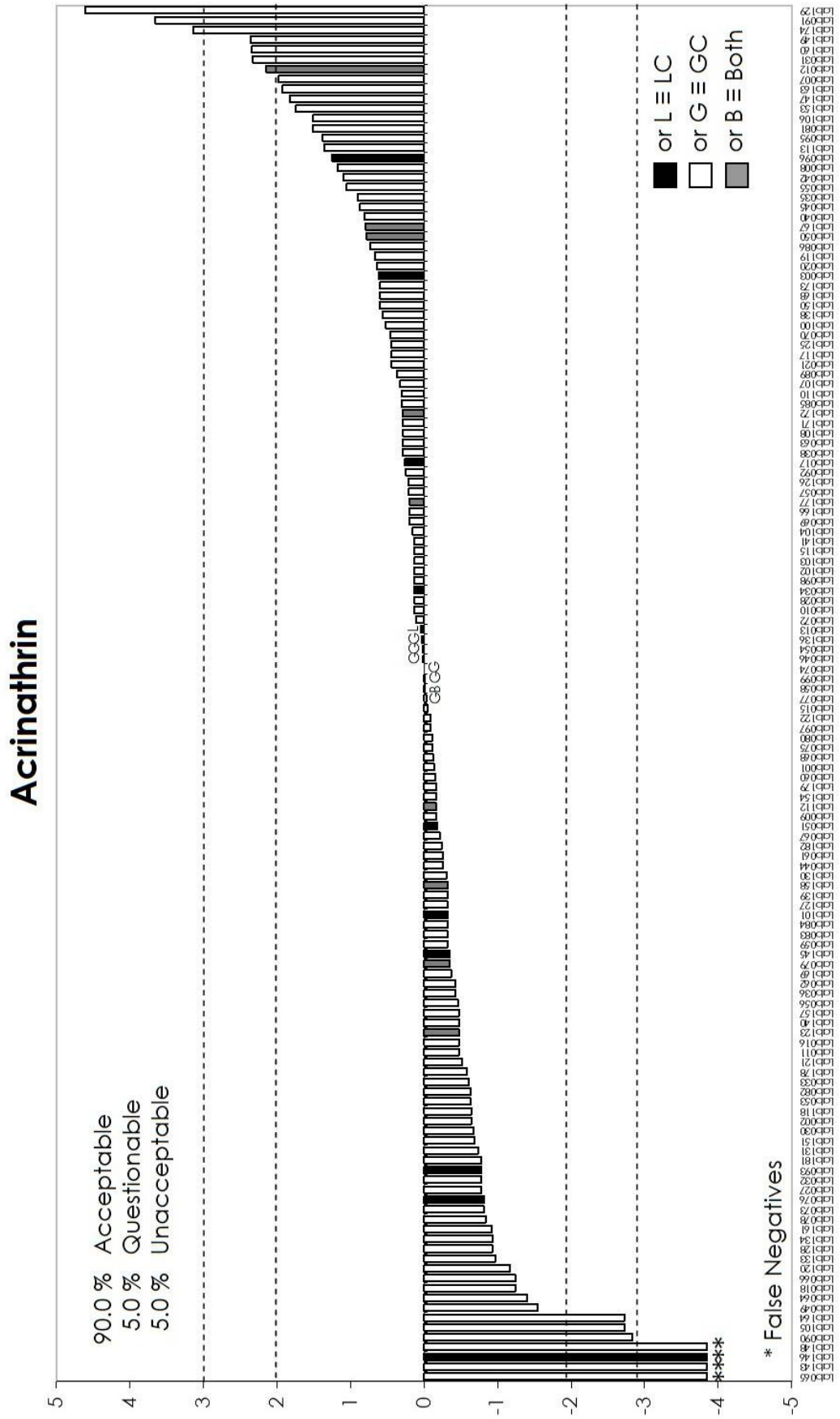
NA*: A numerical value was not reported

ND: Not detected (False negative)

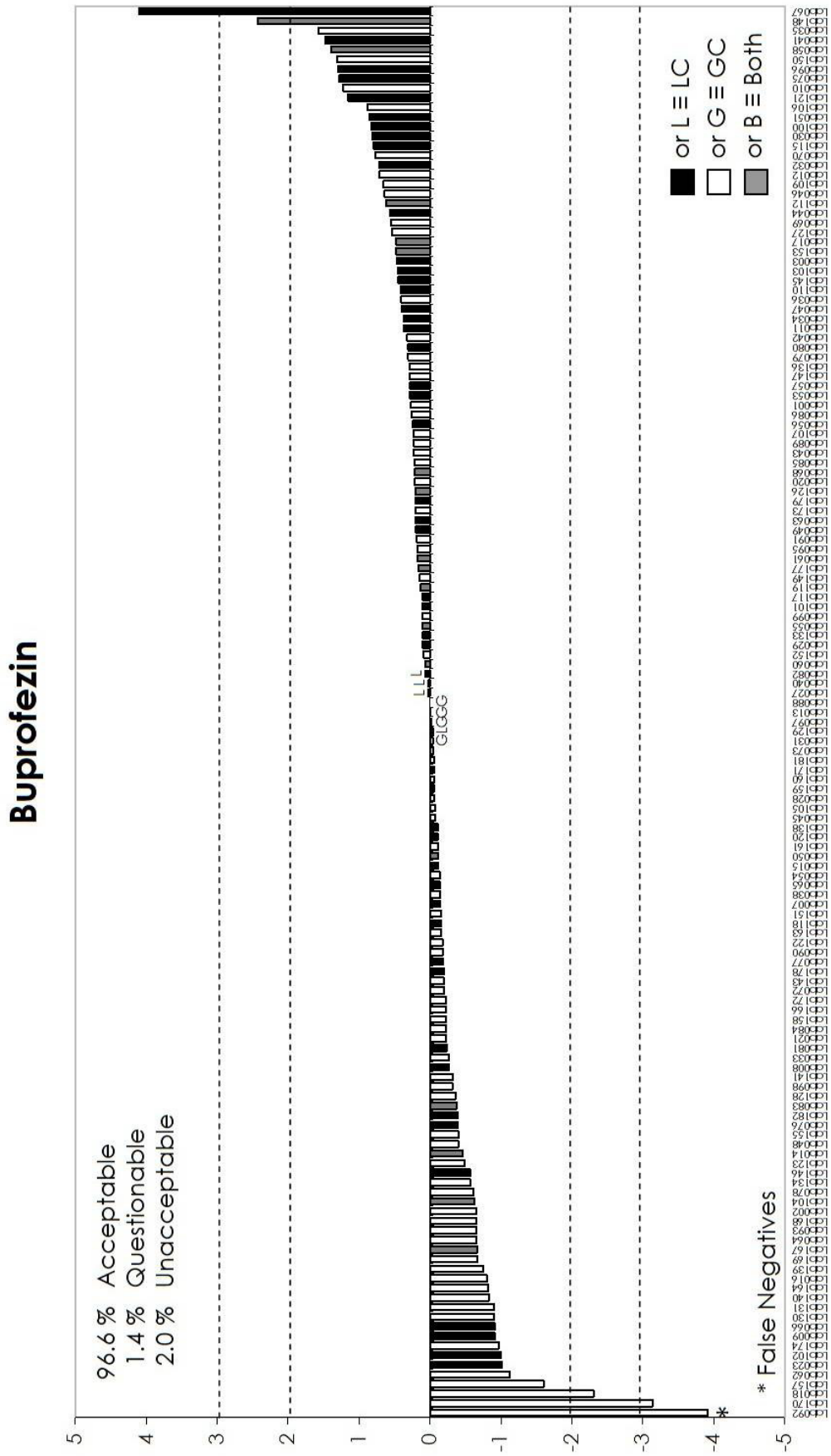
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



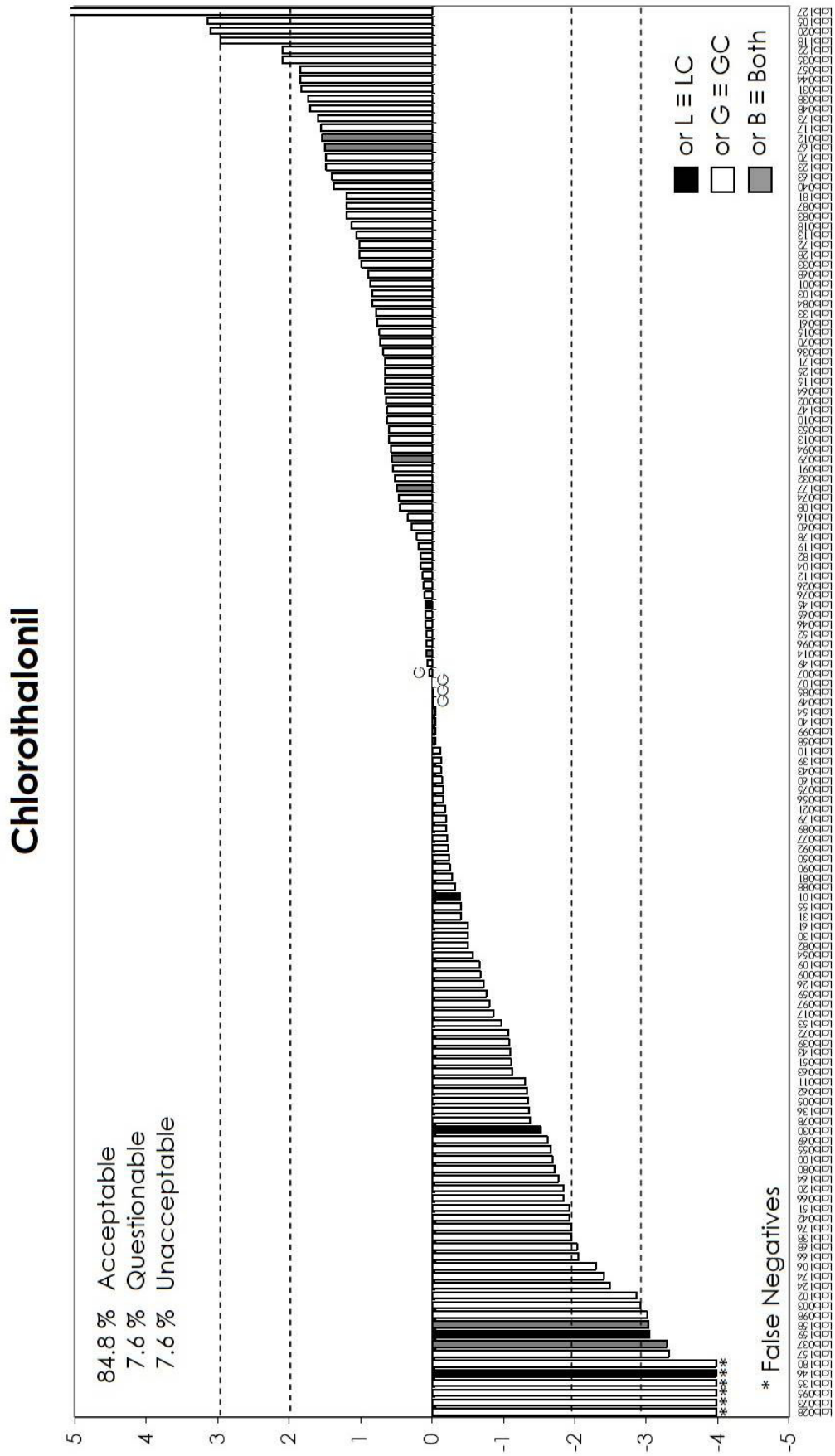
APPENDIX 4. Graphical Representation of z-scores for FFP RSD (25 %).



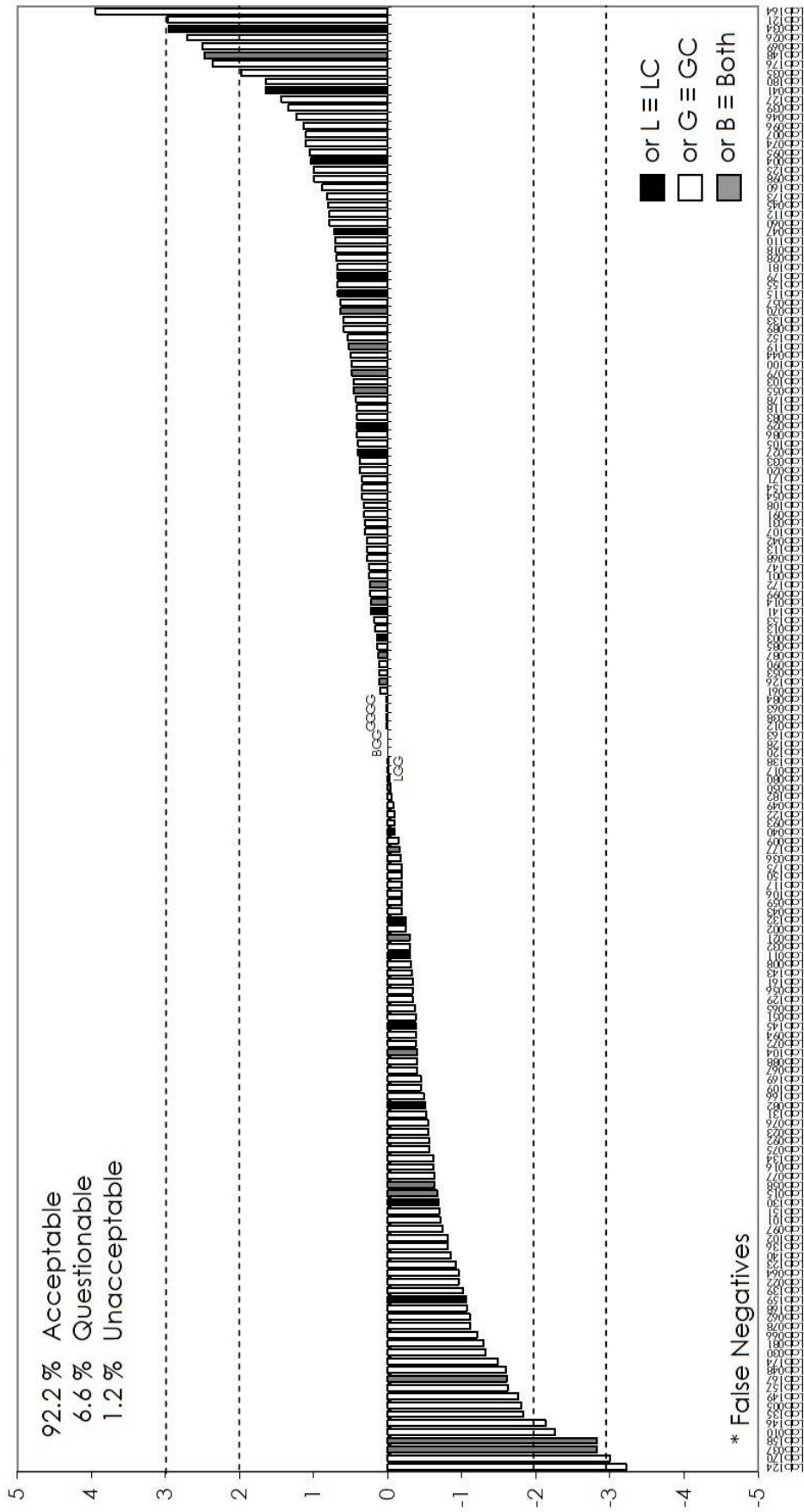
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



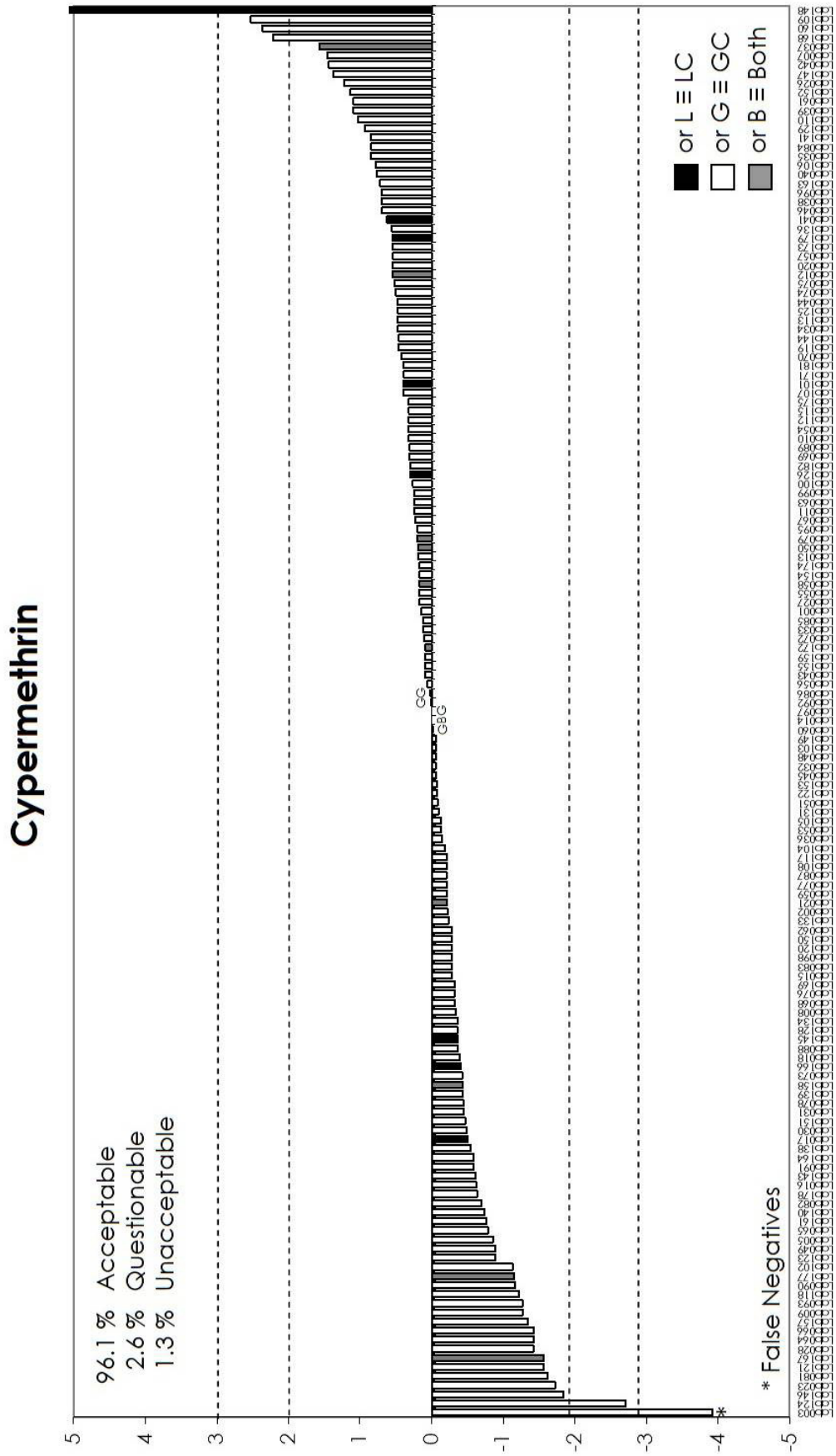
APPENDIX 4. Graphical Representation of z-scores for FFP RSD (25 %).



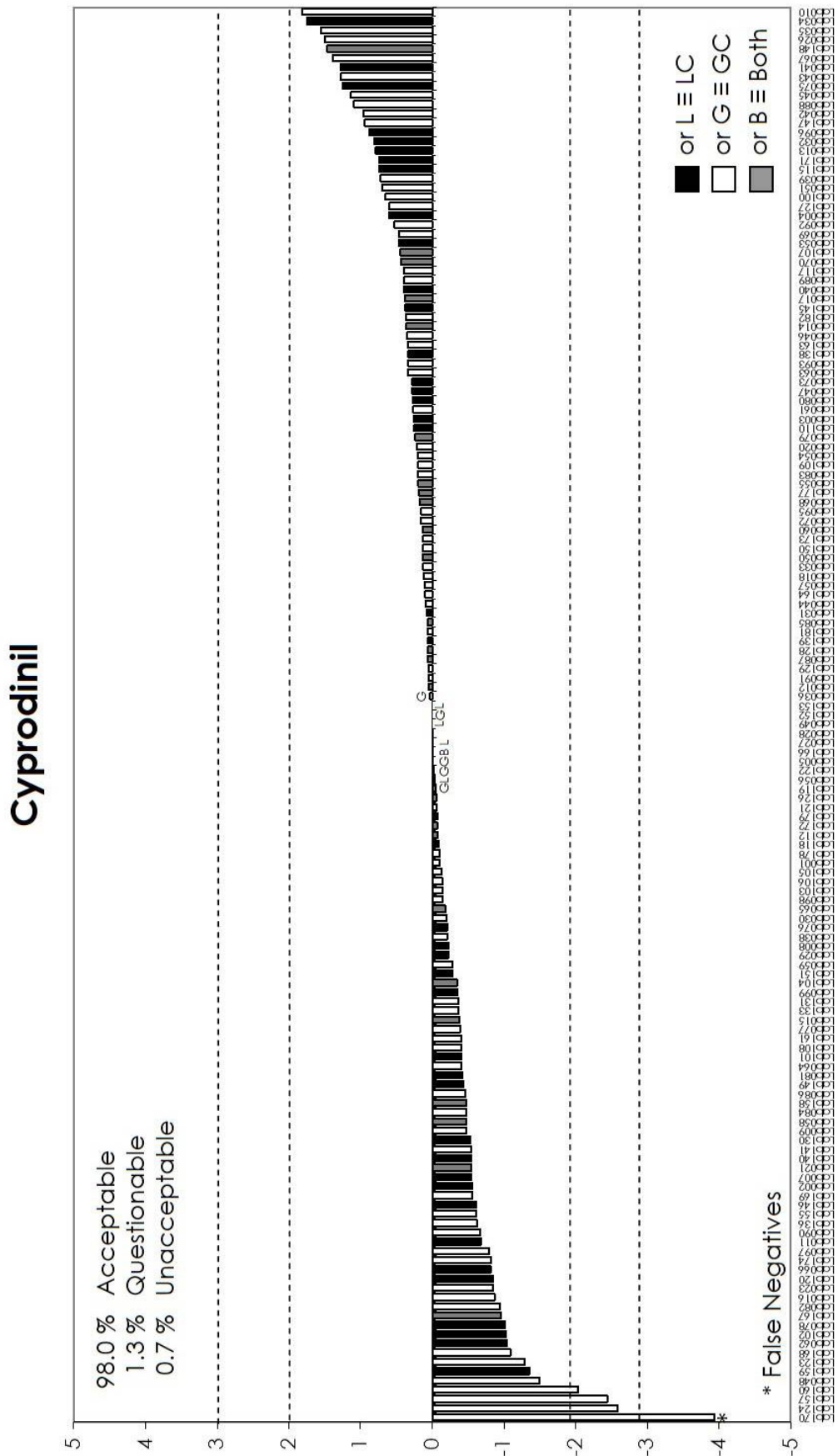
Chlorpyrifos



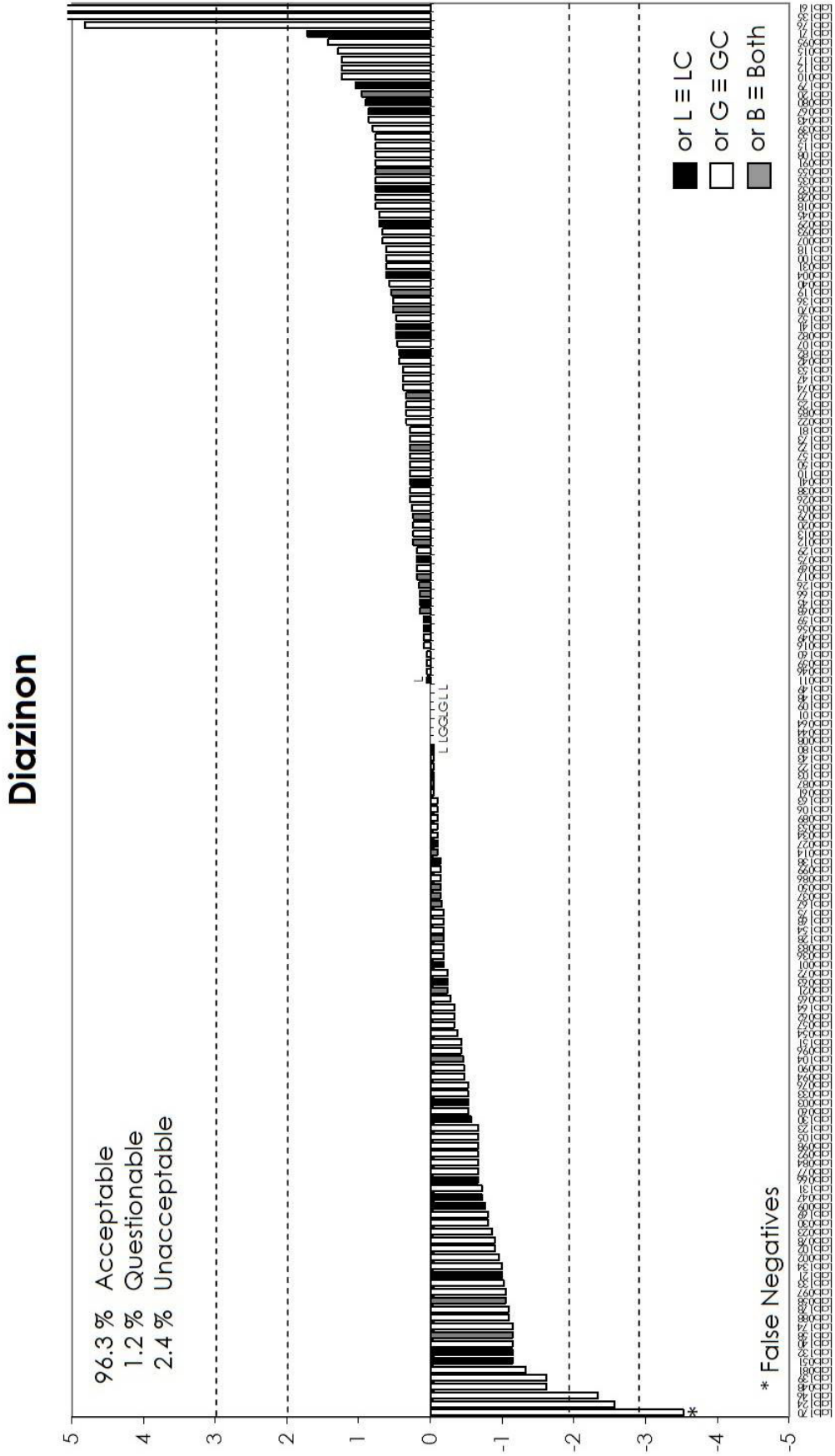
APPENDIX 4. Graphical Representation of z-scores for FFP RSD (25 %).



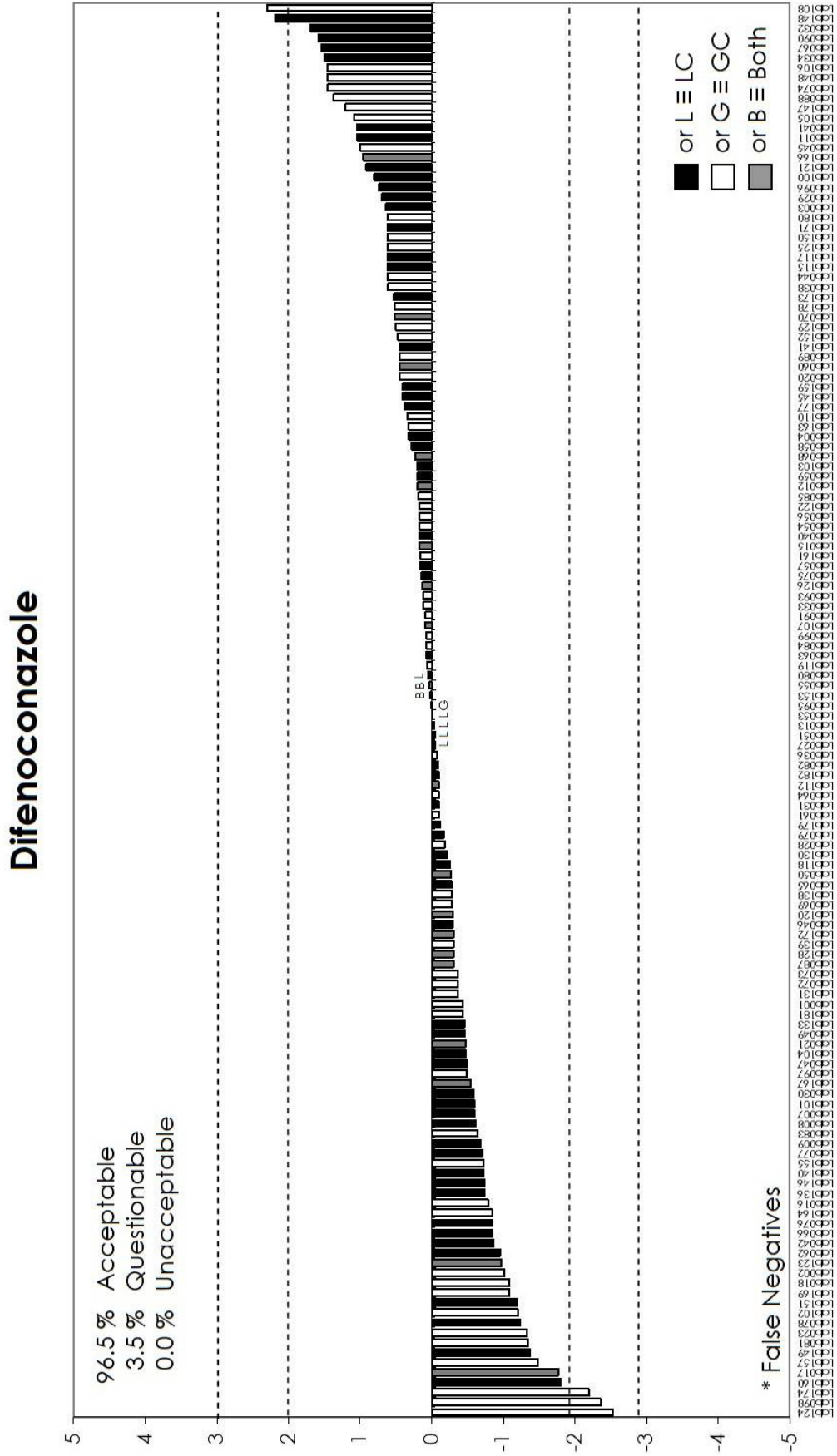
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).

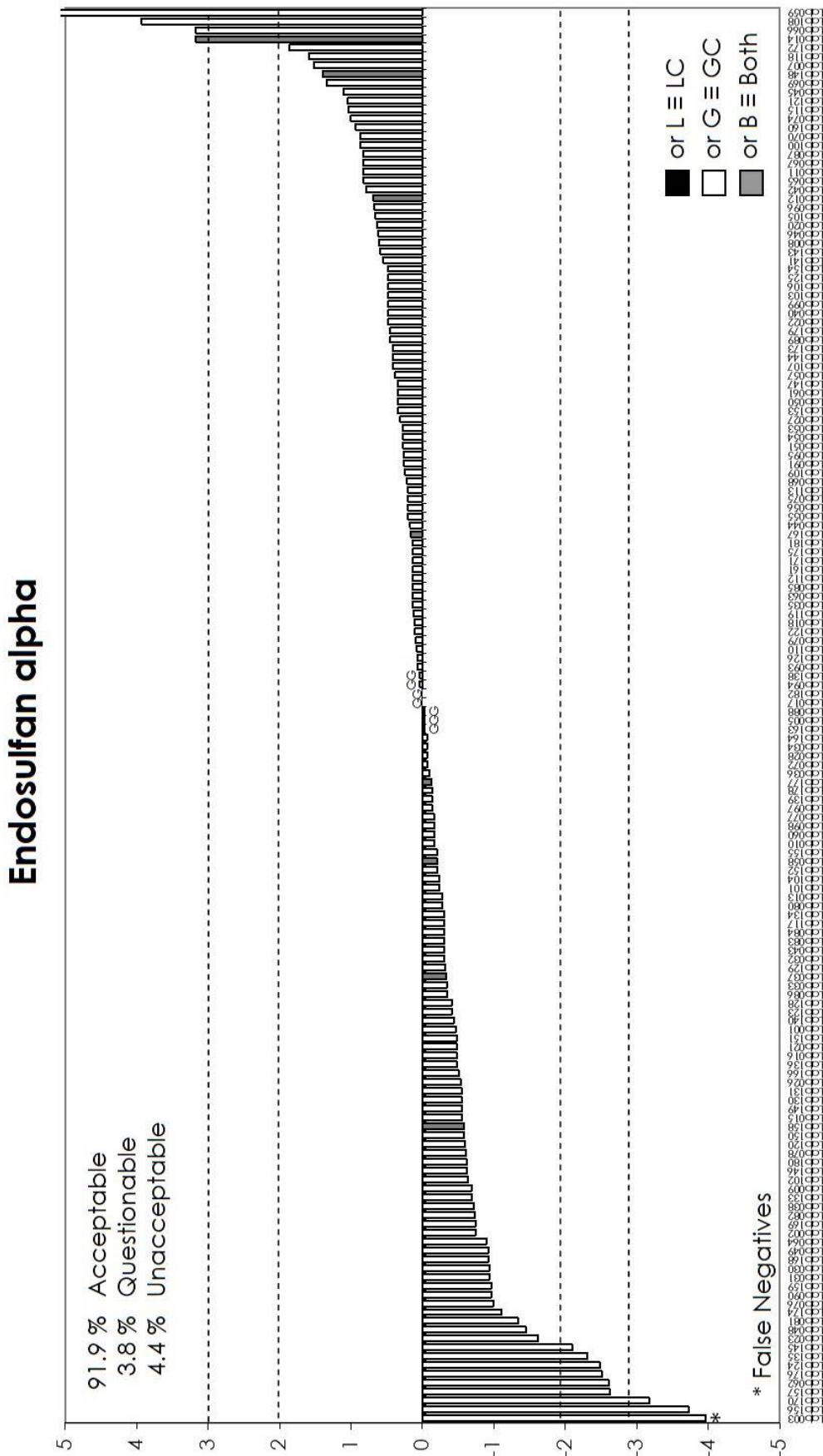


APPENDIX 4. Graphical Representation of z-scores for FFP RSD (25 %).

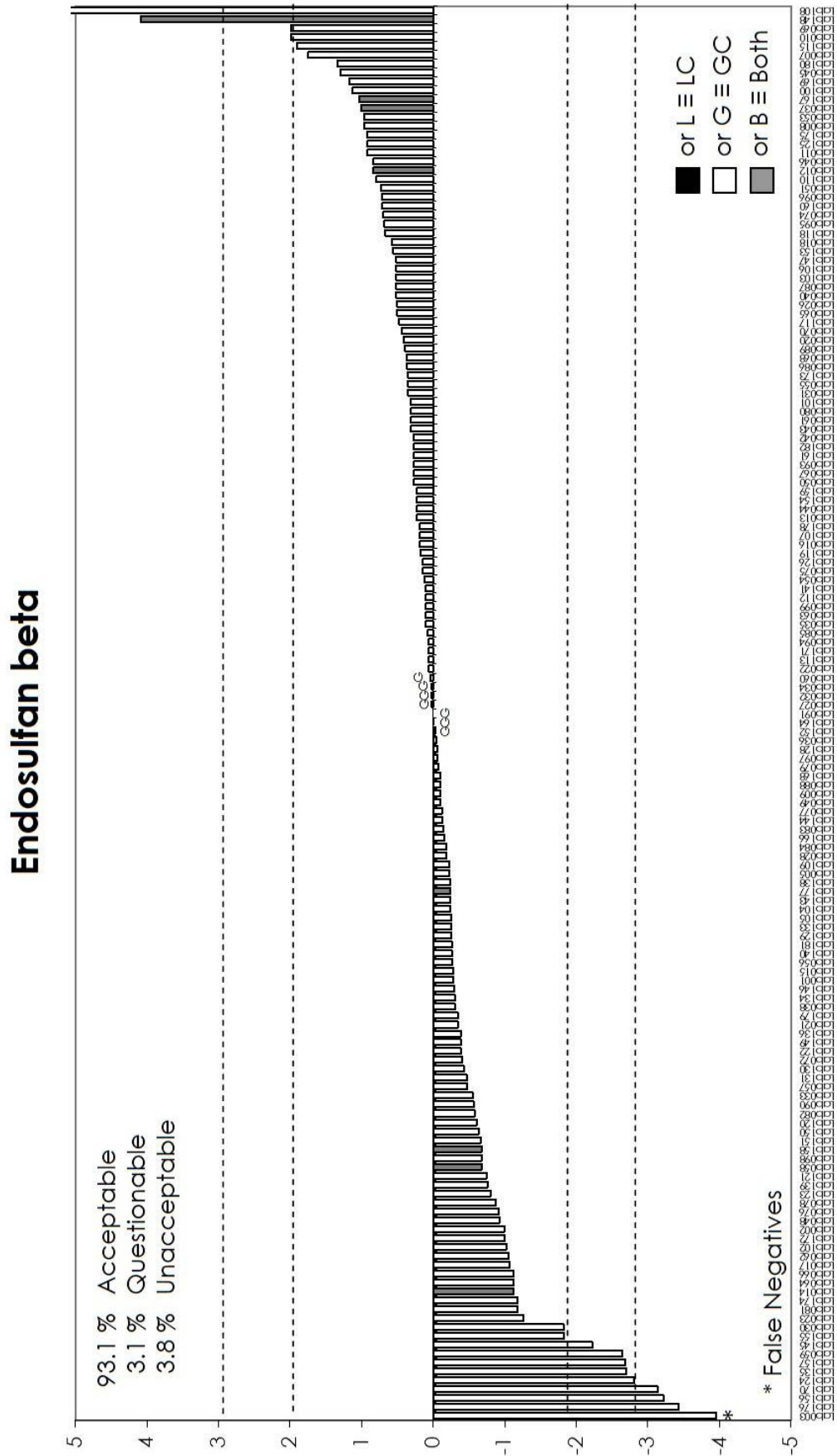


APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).

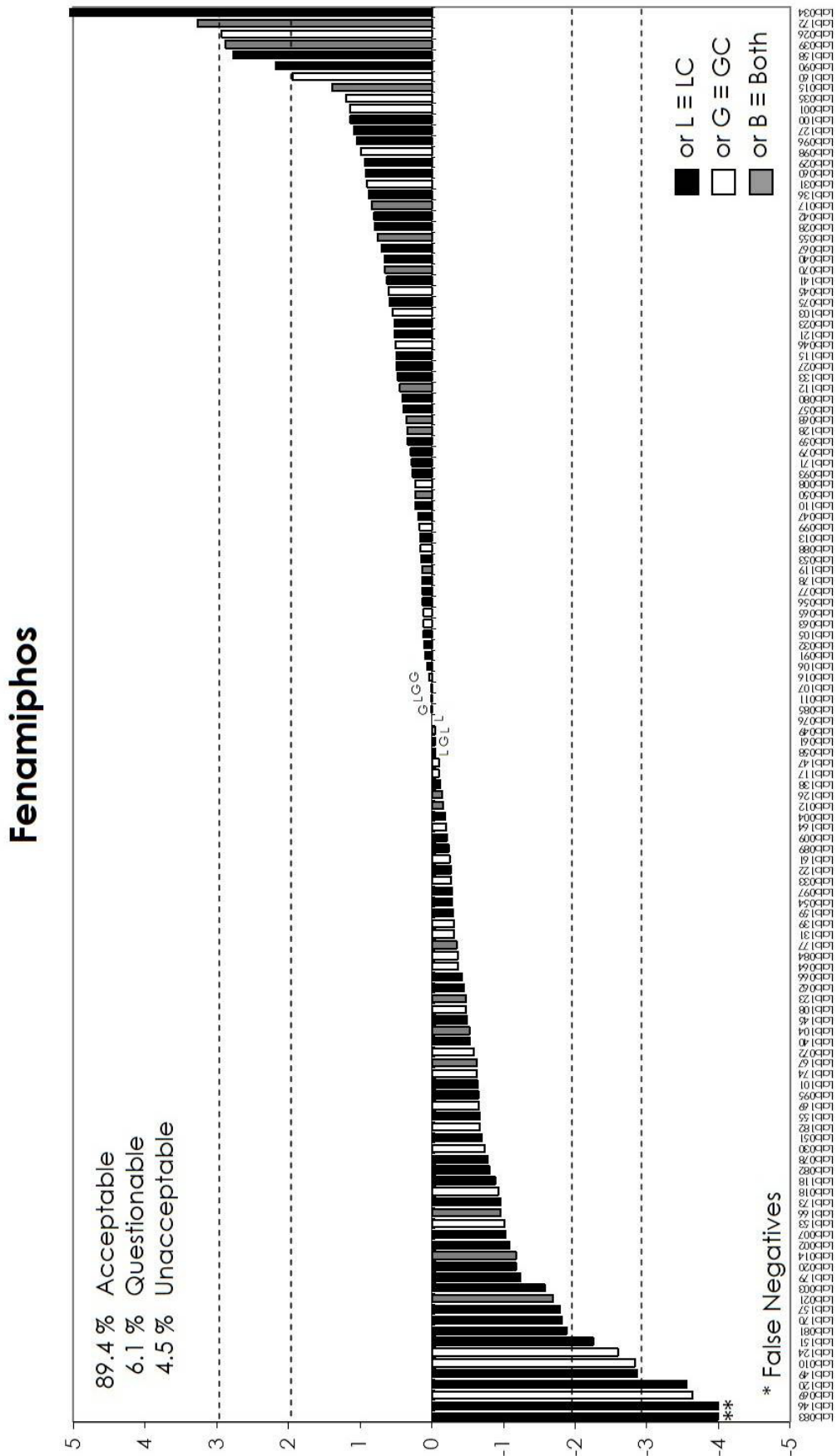




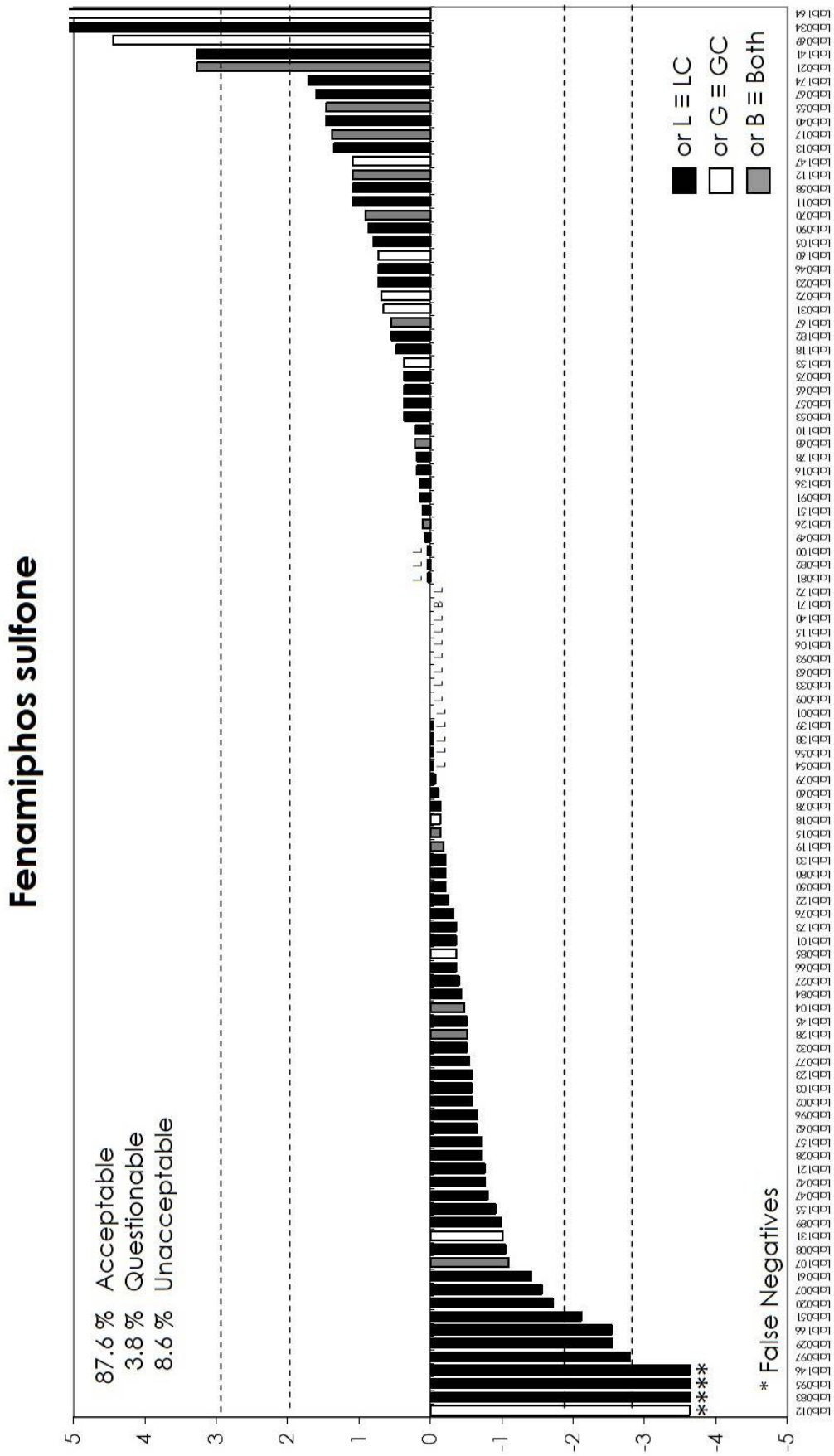
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



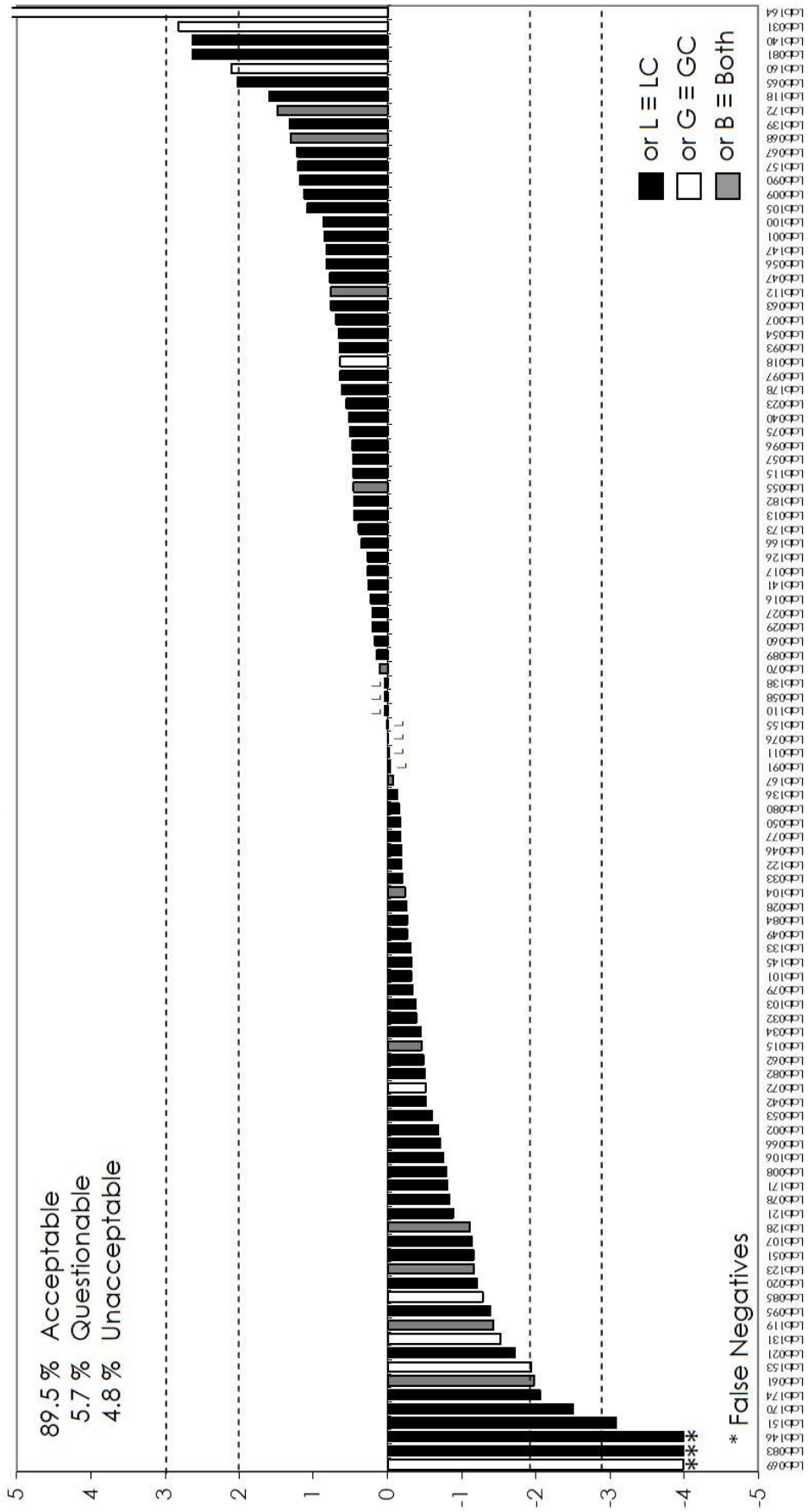
APPENDIX 4. Graphical Representation of z-scores for FFP RSD (25 %).



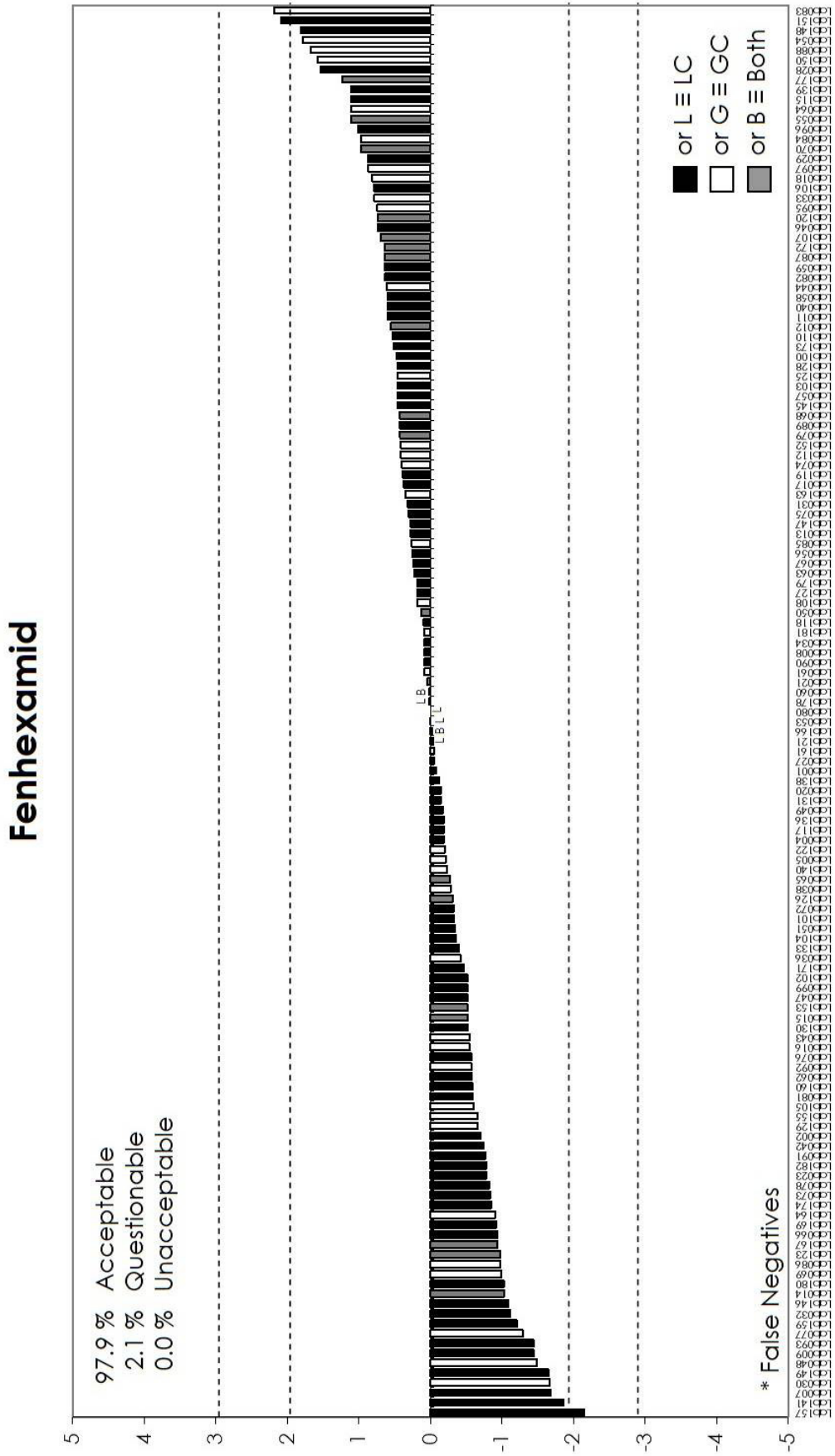
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



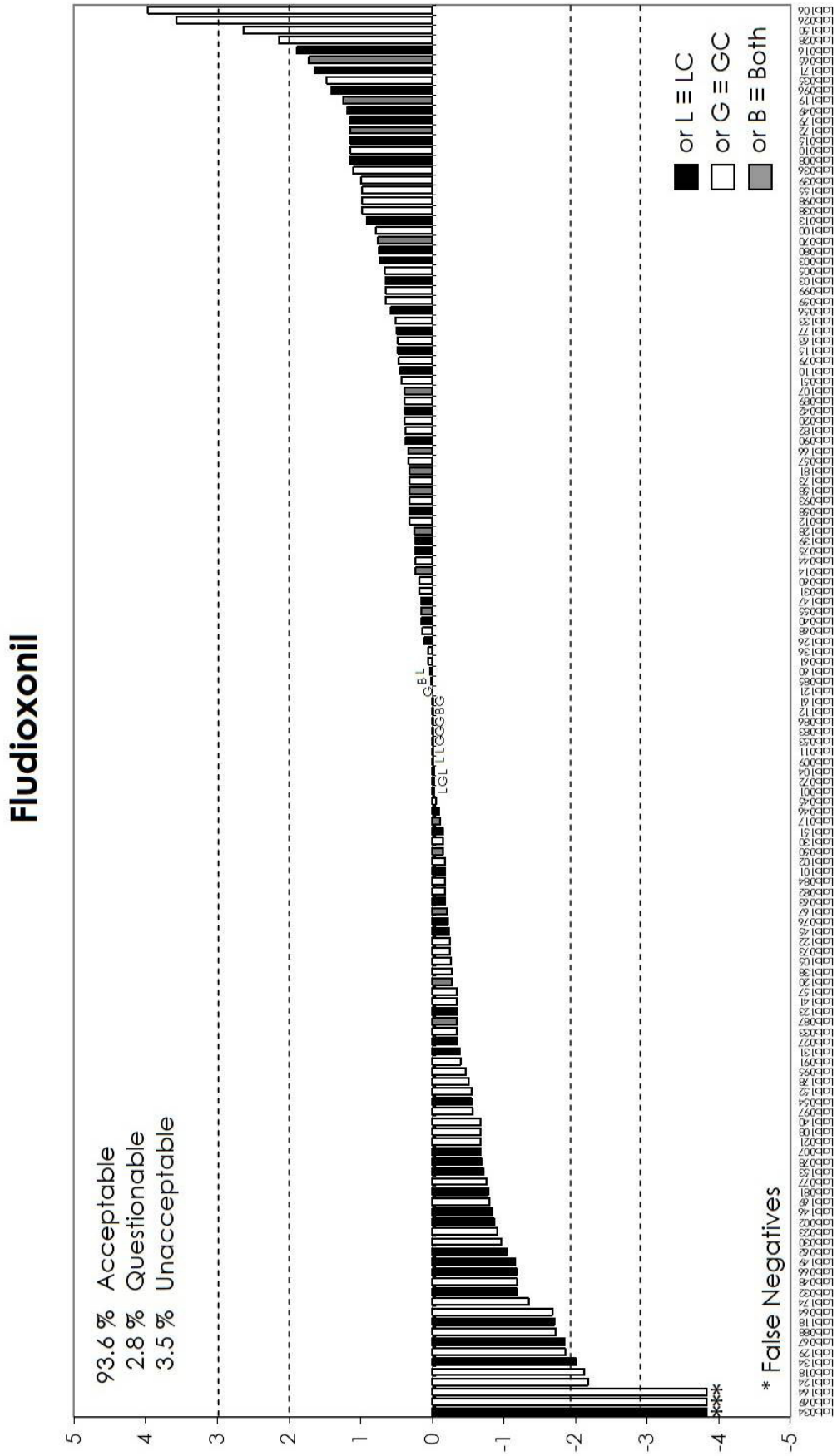
Fenamiphos sulfoxide



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

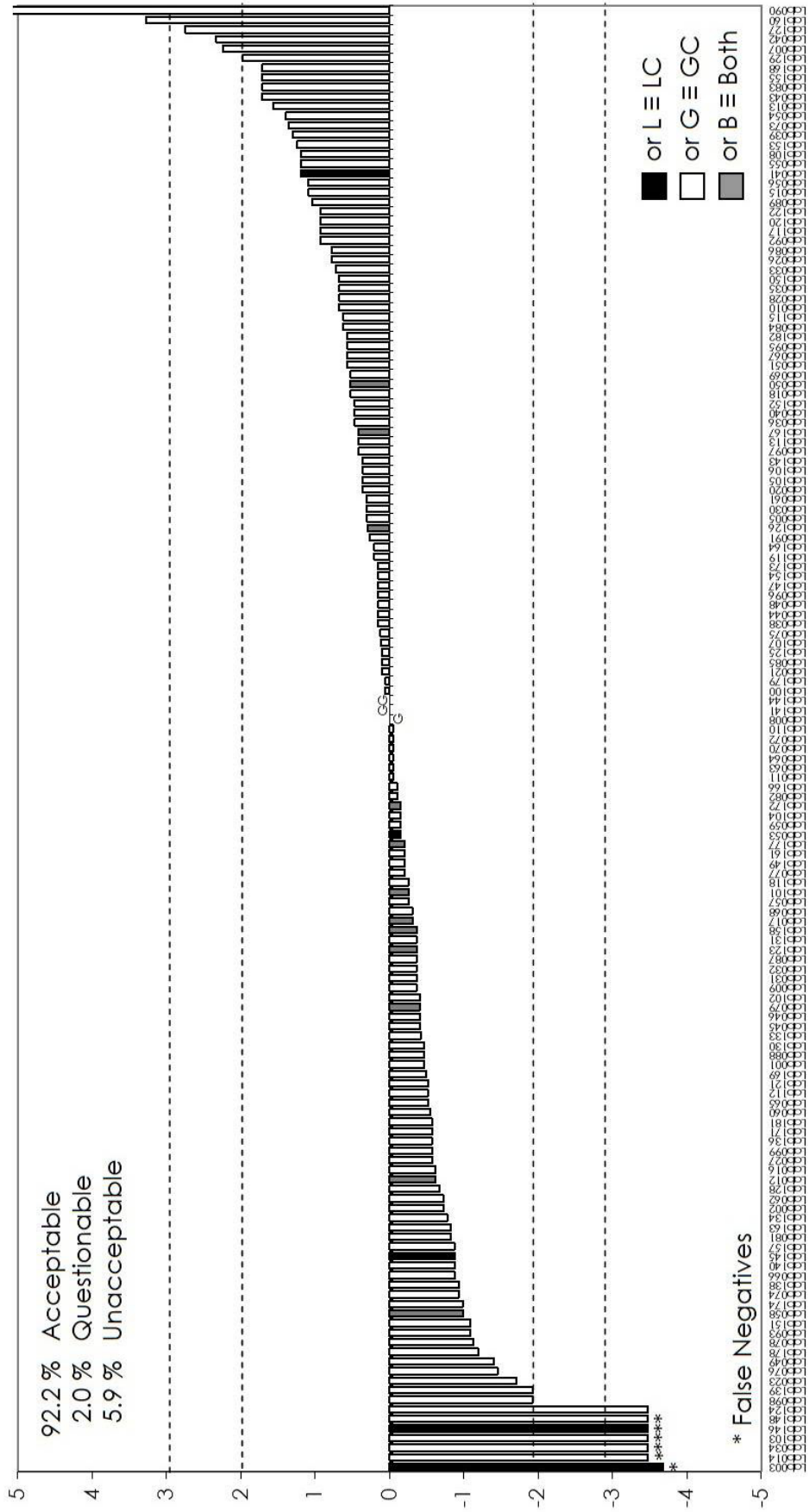


APPENDIX 4. Graphical Representation of z-scores for FFP RSD (25 %).

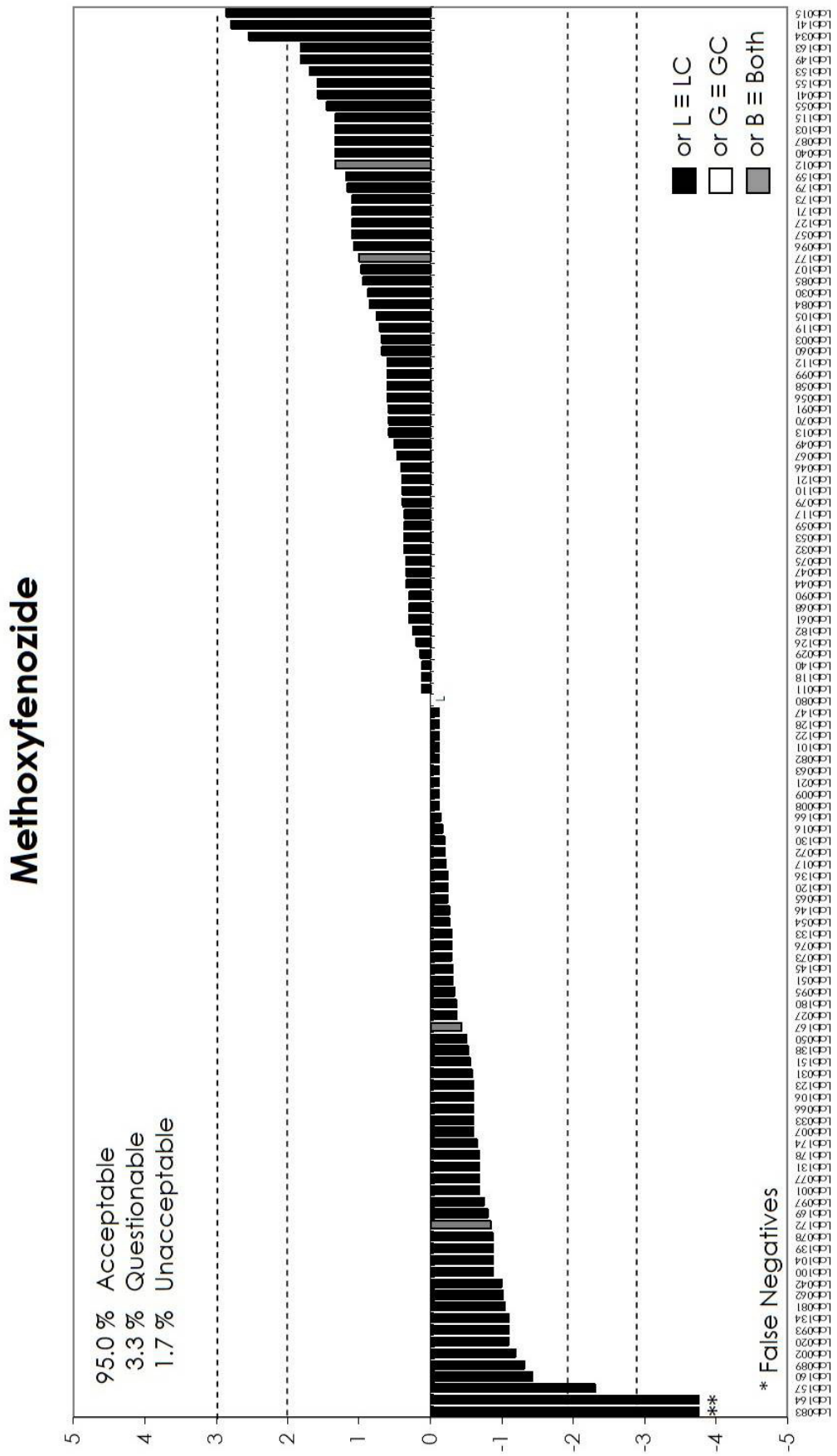


APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).

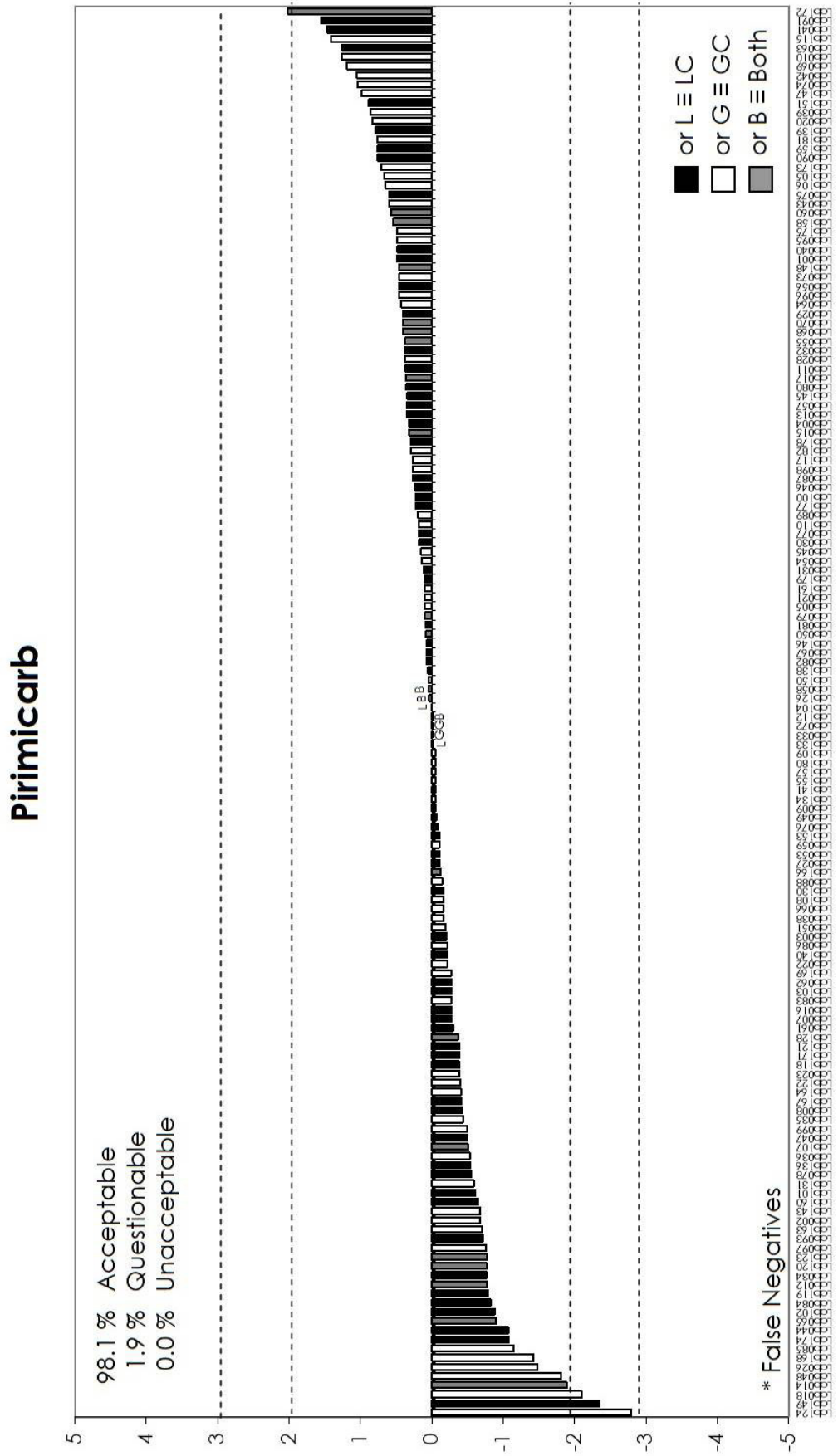
Lambda Cyhalothrin



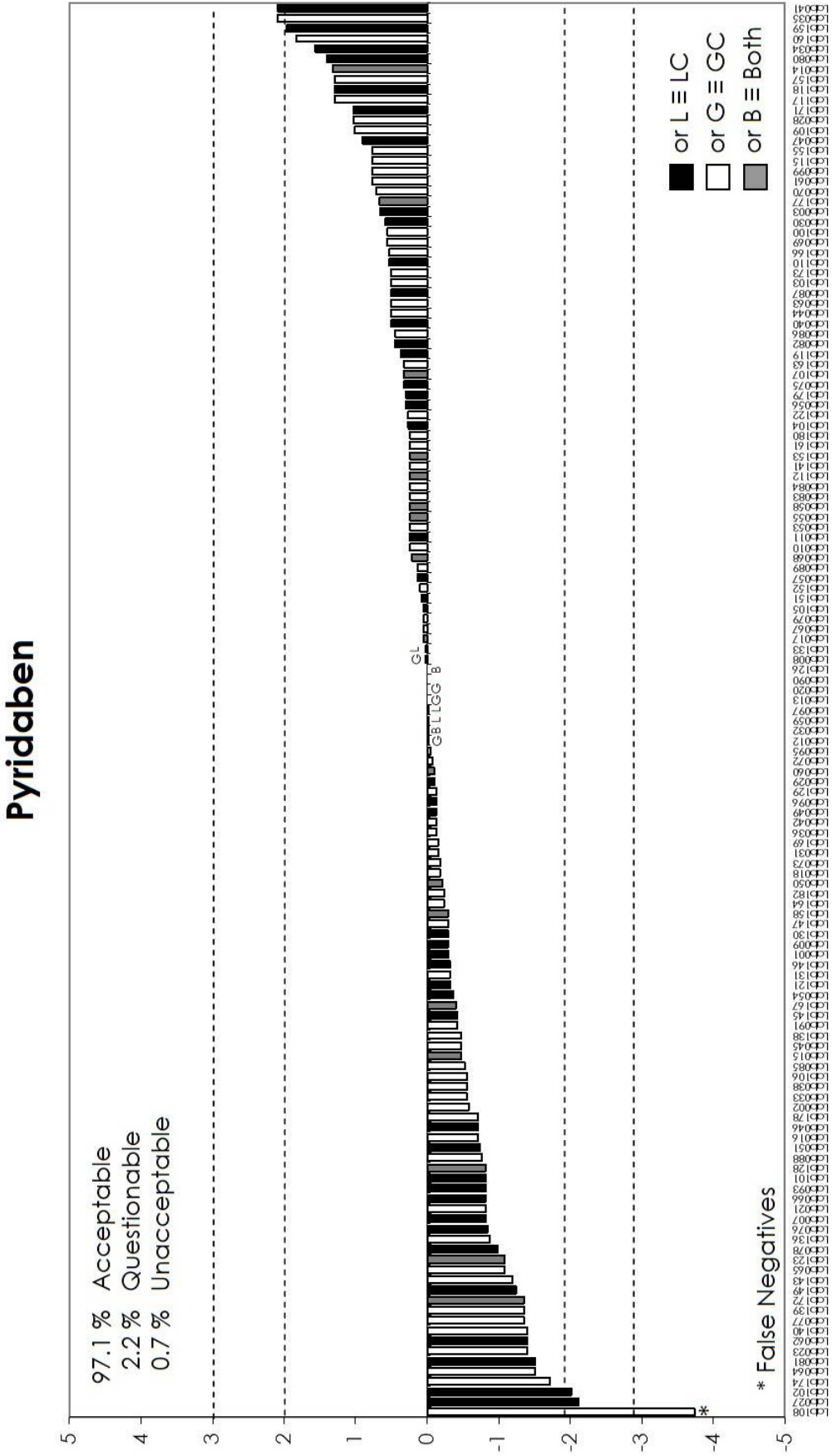
APPENDIX 4. Graphical Representation of z-scores for FFP RSD (25 %).



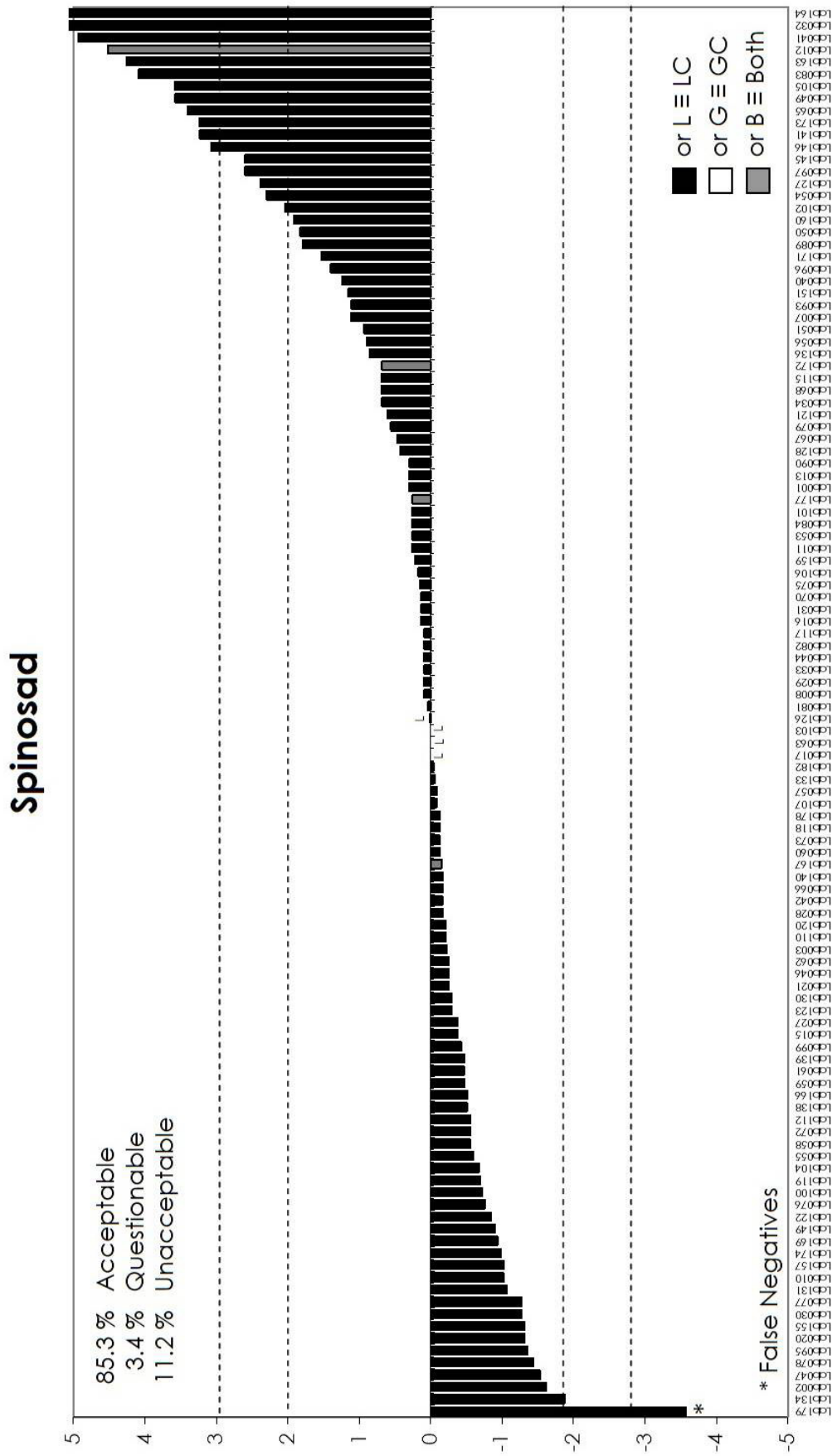
APPENDIX 4. Graphical representation of z-scores for FFP RSD (25 %).



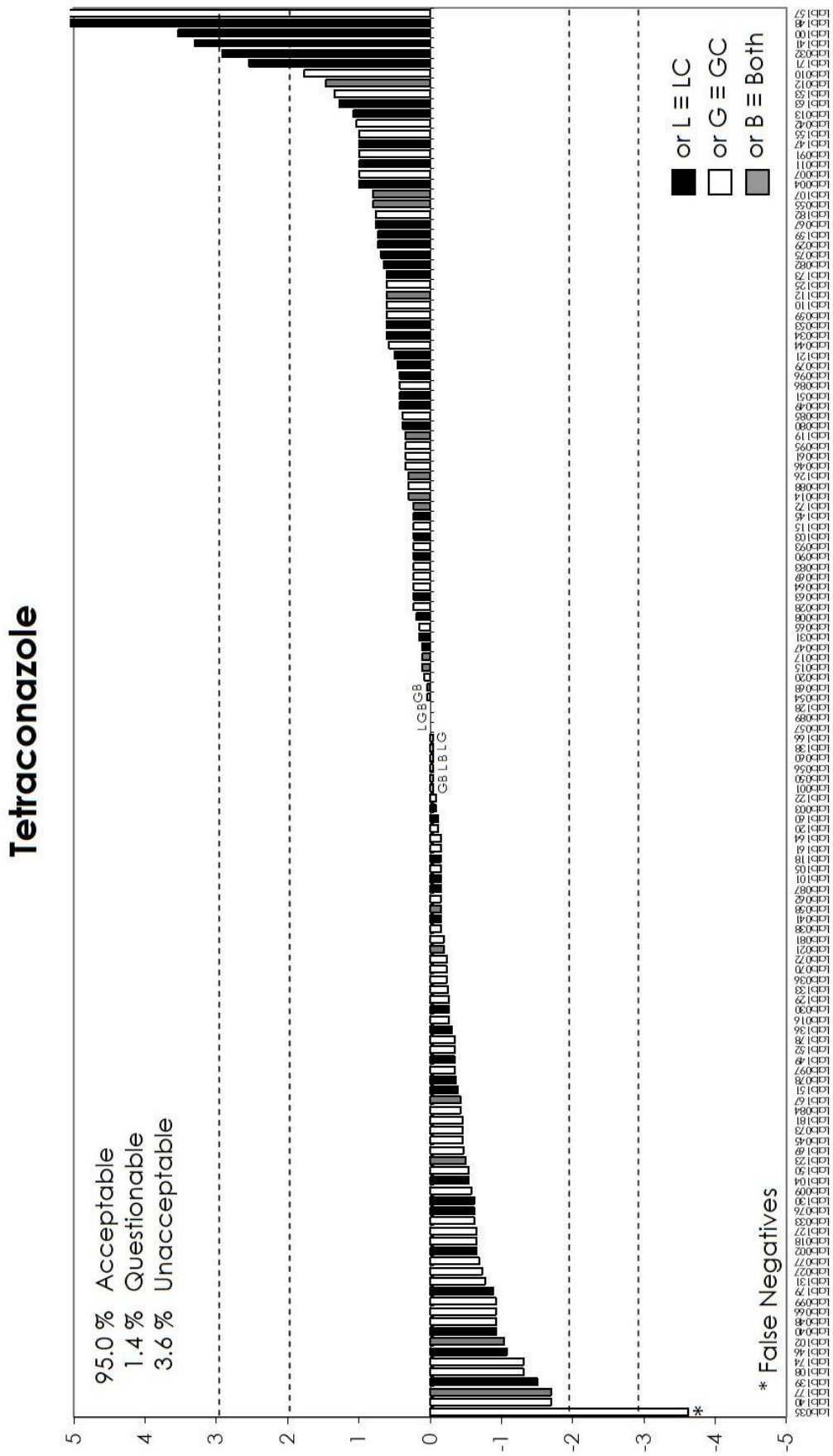
APPENDIX 4. Graphical Representation of z-scores for FFP RSD (25 %).



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-Score (AZ²) for laboratories in Category A.

Lab Code	Aceclamidrid	Acridinathrin	Buprofezin	Chlorothalonil	Chlorpyrifos	Cypermethrin	Cyprodinil	Diazinon	Difenoconazole	Endosulfan alpha	Endosulfan beta	Fenamiphos	Fenamiphos sulfone	Fenamiphos sulfoxide	Fenhexamid	Fludioxonil	Lambda-Cyhalothrin	Methoxyfenozide	Pirimicarb	Pyridaben	Spinosad	Tetraconazole	No. of z-scores	AZ ²
	z-scores																							
1	-0.1	-0.1	0.3	0.9	0.2	0.2	-0.1	-0.2	-0.4	-0.5	-0.3	1.1	0.0	0.9	-0.1	0.0	-0.5	-0.7	0.5	-0.3	0.3	0.0	22	0.2
2	-0.2	-0.6	-0.7	0.6	-0.3	-0.2	-0.6	-1.0	-1.0	-0.7	-1.0	-1.1	-0.6	-0.7	-0.7	-0.9	-0.7	-1.2	-0.7	-0.6	-1.6	-0.7	22	0.6
7	-0.2	2.0	-0.1	0.0	1.1	1.5	-0.5	0.7	-0.6	1.5	1.7	-1.0	-1.6	0.7	-1.7	-0.7	2.2	-0.6	-0.3	-0.8	1.1	1.0	22	1.3
8*	-0.2	1.2	-0.3		-0.3	-0.3	-0.2	0.0	-0.6	0.6	1.0	0.2	-1.1	-0.8	0.1	1.1	0.0	-0.1	-0.4	0.0	0.1	0.2	21	0.3
9	0.0	-0.2	-0.9	-0.7	-0.1	-1.3	-0.5	-0.8	-0.7	-0.7	-0.1	-0.2	0.0	1.1	-1.4	0.0	-0.4	-0.1	-0.1	-0.3		-0.6	21	0.4
11*	0.1	-0.5	0.4	-1.3	-0.3	0.2	-0.7	0.0	1.0	0.8	0.9	0.0	1.1	0.0	0.6	0.0	-0.1	0.1	0.4	0.2	0.3	1.0	22	0.4
12*	-0.5	2.1	0.7	1.5	0.0	0.5	0.1	0.2	0.2	0.7	0.8	-0.2	-3.6		0.6	0.3	-0.6	1.3	-0.8	0.0	4.5	1.5	21	2.3
13*	0.8	0.0	0.0	0.6	0.2	0.2	0.8	0.2	0.0	-0.3	0.2	0.2	1.3	0.4	0.3	0.9	1.6	0.6	0.4	0.0	0.3	1.1	22	0.4
15*	-0.1	0.0	-0.1	0.7	-0.7	-0.3	-0.4	1.3	0.2	-0.6	-0.3	1.4	-0.1	-0.5	-0.5	1.1	1.1	2.9	0.3	-0.5	-0.4	0.1	22	0.8
16	0.0	-0.5	-0.8	0.3	-0.6	-0.6	-0.9	0.1	-0.8	-0.5	0.2	0.0	0.2	0.2	-0.5	1.9	-0.6	-0.2	-0.3	-0.7	0.1	-0.3	22	0.4
17	0.7	0.3	0.5	-0.9	0.0	-0.5	0.4	0.2	-1.8	0.0	-1.1	0.8	1.4	0.3	0.4	-0.1	-0.3	-0.2	0.4	0.1	0.0	0.1	22	0.4
18	-1.7	-1.2	-2.3	1.1	0.7	-0.4	0.1	0.8	-1.1	0.1	0.6	-0.9	-0.1	0.6	0.8	-2.1	0.5		-2.1	-0.2		-0.7	20	1.3
20	-1.0	0.6	0.2	3.1	0.4	0.5	0.2	0.2	0.4	0.6	0.4	-1.2	-1.7	-1.2	-0.1	0.4	0.4	-1.1	0.8	0.0	-1.3	0.1	22	1.0
21*	0.0	0.4	-0.2	-0.2	-0.3	-0.2	-0.5	-0.2	-0.5	-0.5	-0.3	-1.7	3.3	-1.7	0.0	-0.7	0.1	-0.1	0.1	-0.8	-0.3	-0.2	22	0.9
27	-0.2	-0.8	0.0		0.4	0.2	0.0	-0.1	-0.1	0.3	0.0	0.5	-0.4	0.2	-0.1	-0.3	-0.6	-0.4	-0.1	-2.1	-0.4	-0.7	21	0.3
28	0.5	0.1	-0.1	-4.0	0.7	-1.4	0.0	0.8	-0.2	-0.1	-0.2	0.8	-0.7	-0.3	1.5	2.1	0.7		0.4	1.0	-0.2	0.2	21	1.4
30	0.6	-0.7	0.8	-1.5	-1.3	-0.5	-0.2	-0.8	-0.6	-0.9	-1.8	-0.7			-1.7	-1.0	0.3	0.9	0.2	0.6	-1.3	-0.3	20	0.9
31	0.0	2.3	0.0	1.8	0.3	-0.5	0.1	0.6	-0.1	-0.9	0.3	0.9	0.7	2.8	0.3	0.2	-0.4	-0.6	0.1	-0.2	0.1	0.2	22	0.9
32*	0.6	-0.8	0.7	0.5	-0.3	-0.1	0.8	0.8	1.7	-0.3	0.0	0.1	-0.5	-0.4	-1.1	-1.2	-0.4	0.4	0.4	0.0	5.0	2.9	22	2.0
33*	-0.3	-0.6	-0.3	1.0	0.4	0.1	0.1	-0.5	0.1	-0.3	-0.6	-0.3	0.0	-0.2	0.8	-0.3	0.7	-0.6	0.0	-0.6	0.1	-0.6	22	0.2
40*	0.7	0.8	0.0	1.4	-0.1	0.8	0.4	0.6	0.2	0.5	0.5	0.7	1.5	0.5	0.6	0.1	0.5	1.3	0.5	0.5	1.2	-0.9	22	0.6
42	-0.7	1.1	0.3	-1.9	0.3	1.4	1.0	0.4	-0.9	0.8	0.3	0.8	-0.8	-0.5	-0.7	0.4	2.3	-1.0	1.0	-0.1	-0.2	1.0	22	1.0
46	0.7	0.0	0.6	0.1	1.2	0.7	0.4	0.0	-0.3	0.6	0.8	0.5	0.7	-0.2	0.7	-0.1	-0.4	0.4	0.2	-0.7	-0.3	0.3	22	0.3
49*	0.8	-1.5	0.2	0.0	-0.1	-0.9	0.0	0.1	-0.5	-0.9	-0.1	0.0	0.1	-0.3	-0.2	1.2	-1.4	0.5	-0.1	-0.1	3.6	0.4	22	1.0
50	-0.1	0.8	-0.1	-0.2	0.0	0.2	0.1	-0.1	-0.3	0.3	0.3	0.2	-0.2	-0.2	0.1	-0.1	0.5	-0.5	0.1	-0.2	1.8	0.0	22	0.2
51	-0.8	-0.2	0.9	-1.1	-0.4	-0.1	0.7	-1.1	0.0	0.3	0.7	-0.7	-2.1	-1.2	-0.3	0.4	0.6	-0.3	-0.2	-0.7	0.9	0.4	22	0.6
53	-0.1	-0.6	0.3	0.6	0.1	-0.1	0.5	-0.1	0.0	0.3	1.0	0.1	0.4	-0.6	0.0	0.0	-0.2	0.4	-0.1	0.2	0.3	0.6	22	0.2
54*	-0.6	0.0	-0.1	-0.6	0.3	0.3	0.2	-0.4	0.2	0.3	0.1	-0.3	0.0	0.7	1.8	-0.5	1.4	-0.3	0.1	-0.4	2.3	0.0	22	0.6
55	0.1	1.1	0.1	-1.7	0.5	0.2	0.2	0.8	0.0	0.2	0.3	0.8	1.5	0.5	1.1	0.1	1.2	1.5	0.4	0.2	-0.6	0.8	22	0.6
56	0.0	-0.5	0.2	-0.2	-0.3	0.1	0.0	0.1	0.2	0.2	-0.3	0.1	0.0	0.8	0.3	0.6	1.1	0.6	0.5	0.3	0.9	0.0	22	0.2
57	0.6	0.2	0.3	1.8	0.6	0.5	0.1	-0.3	0.2	0.4	-0.5	0.4	0.4	0.5	0.5	0.3	-0.3	1.1	0.4	0.1	-0.1	0.0	22	0.3

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

Lab Code	Acetaminiprid	Acinathrin	Buprofezin	Chlorothaloniil	Chlorpyrifos	Cypermethrin	Cyprodinil	Diazinon	Difencanazole	Endosulfan alpha	Endosulfan beta	Fenamiphos	Fenamiphos sulfone	Fenamiphos sulfoxide	Fenhexamid	Fludoxonil	Lambda-Cyhalothrin	Methoxyfenozide	Plitmicarb	Pyridaben	Spinosad	Tetraconazole	No. of z-scores	AZ ²
	z-scores																							
58	1.1	0.0	1.4	-0.1	-0.6	0.2	-0.5	-1.0	0.3	-0.2	-0.7	0.0	1.1	0.0	0.6	0.3	-1.0	0.6	0.0	0.2	-0.6	-0.2	22	0.4
60	-0.1	-0.2	0.1	0.3	0.8	0.0	0.1	-0.5	0.4	-0.2	0.0	0.9	-0.1	0.2	0.0	0.2	-0.6	0.7	0.6	-0.1	-0.1	0.0	22	0.2
61*	0.3	-0.3	0.2	0.8	0.1	1.1	0.3	0.0	-0.1	0.3	0.3	0.0	-1.4	-2.0	0.1	0.0	0.3	0.3	-0.3	0.8	-0.5	0.3	22	0.4
62	-0.5	-0.4	-1.1	-1.3	-1.1	-0.3	-1.0	-0.3	-1.0	-2.6	-1.1	-0.5	-0.7	-0.5	-0.6	-1.0	-0.7	-1.0	-0.3	-1.4	-0.3	-0.2	22	0.9
63	0.3	0.3	0.2	-1.1	0.0	0.2	0.3	-0.2	0.1	0.1	0.1	0.1	0.0	0.8	0.2	-0.2	-0.1	-0.1	1.3	0.5	0.0	0.2	22	0.2
65*	0.1	-3.8	-0.1	0.1	-0.4	-0.8	-0.2	-0.3	-0.3	0.8	0.5	0.1	0.4	2.0	-0.3	1.7	-0.5	-0.2	-0.9	-1.1	3.4	0.2	22	1.7
66	0.0	-1.2	-0.9	-1.8	-1.2	-1.4	-0.8	-0.7	-0.9	3.2	-1.1	-0.4	-0.4	-0.7	-0.9	-1.2	-0.9	-0.6	-0.2	-0.8	-0.2	-0.9	22	1.3
67	0.1	-0.2	4.1		-0.4	0.2	1.4	0.9	1.5	0.8	0.3	0.7	1.6	1.2	0.2	-1.8	0.6	0.5	0.1	0.1	0.5	0.8	21	1.5
68	0.1	-0.1	0.2	0.9	0.3	-0.3	0.2	0.1	0.2	0.2	0.4	0.4	0.2	1.3	0.4	0.1	-0.3	0.3	0.4	0.2	0.7	0.0	22	0.2
70	0.8	0.5	0.8	0.7	0.6	0.4	0.4	0.5	0.5	0.9	0.4	0.7	0.9	0.1	1.0	0.8	-0.1	0.6	0.4	0.7	0.1	-0.2	22	0.4
72	0.0	0.1	-0.2	-1.1	-0.4	0.1	0.2	-0.2	-0.4	-0.1	-0.4	-0.6	0.7	-0.5	-0.3	0.0	-0.1	-0.2	0.0	-0.1	-0.6	-0.2	22	0.2
75*	0.3	-0.1	1.3	-0.2	-0.6	0.5	1.2	0.2	0.1	0.2	0.1	0.6	0.4	0.5	0.3	0.2	0.1	0.3	0.6	0.3	0.1	0.7	22	0.3
76	0.1	-0.8	-0.4	0.1	-0.5	-0.3	-0.2	-0.5	-0.8	-1.0	-0.9	0.0	-0.3	0.0	-0.6	-0.2	-1.5	-0.3	-0.1	-0.8	-0.8	-0.6	22	0.4
77	0.1	0.0	-0.2	-0.2	-0.6	-0.2	-0.4	-0.7	-0.7	-0.2	-0.1	0.1	-0.5	-0.2	-1.3	-0.8	-0.2	-0.7	0.2	-1.4	-1.3	-0.7	22	0.4
78	-0.2	-0.8	-0.6	-1.4	-1.1	-0.4	-1.0	-0.9	-1.2	-0.6	-0.9	-0.8	-0.1	-0.8	-0.8	-0.7	-1.1	-0.9	-0.6	-1.0	-1.4	-0.4	22	0.8
79	0.1	-0.4	0.3	0.6	0.5	0.2	0.2	0.2	-0.2	0.1	-0.1	0.3	-0.1	-0.3	0.4	0.5	-0.4	0.4	0.1	0.1	0.6	0.5	22	0.1
81*	0.0	1.5	-0.2	-0.3	-1.3	-1.6	-0.4	-1.3	-1.3	-1.3	-1.2	-1.9	0.0	2.6	-0.6	-0.8	-0.8	-1.0	0.1	-1.5	0.0	-0.2	22	1.3
82	-0.1	-0.6	0.1	-0.5	-0.5	-0.7	-0.9	0.5	-0.1	-0.7	-0.6	-0.8	0.0	-0.5	0.6	-0.2	-0.1	-0.1	0.1	0.5	0.1	0.7	22	0.2
84	-0.3	-0.3	-0.2	0.8	0.0	0.8	-0.5	-0.7	0.1	-0.3	-0.2	-0.4	-0.4	-0.3	1.0	-0.2	0.6	0.8	-0.8	0.2	0.3	-0.4	22	0.3
85	1.4	0.3	0.2	0.0	0.1	0.1	0.1	0.3	0.2	0.1	0.1	0.0	-0.4	-1.3	0.3	0.0	0.1	0.9	-1.2	-0.5		0.4	21	0.3
89*	2.0	0.4	0.2	-0.2	0.6	0.3	0.4	-0.1	0.4	0.4	0.4	-0.2	-1.0	0.1	0.4	0.4	1.0	-1.3	0.2	0.1	1.8	0.0	22	0.6
90	1.0	-2.8	-0.2	-0.3	0.1	-1.2	-0.7	-0.5	1.6	-1.0	-0.6	2.2	0.9	1.2	0.1	0.4	5.0	0.3	0.8	0.0	0.3	0.2	22	2.2
91*	1.3	3.7	0.2	0.5	0.3	-0.6	0.1	0.8	0.1	0.3	0.0	0.1	0.1	0.0	-0.8	-0.4	0.3	0.6	1.5	-0.4		1.0	21	1.0
93	-1.0	-0.8	-0.7		-0.1	-1.3	0.3	0.7	0.1	0.1	0.3	0.3	0.0	0.6	-1.4	0.3	-1.1	-1.1	-0.7	-0.8	1.1	0.2	21	0.6
95	-1.5	1.4	0.2	-4.0	1.1	0.2	0.2	1.4	0.0	0.3	0.7	-0.7	-3.6	-1.4	0.7	-0.5	0.6	-0.3	0.5	-0.1	-1.4	0.3	22	1.9
96	0.6	1.3	1.3	0.1	1.1	0.7	0.9	-0.4	0.7	0.7	0.7	1.1	-0.7	0.5	1.0	1.4	0.2	1.1	0.5	-0.1	1.4	0.4	22	0.7
97*	0.0	-0.1	0.0	-0.8	-0.7	0.0	-0.8	-1.0	-0.5	-0.1	-0.1	-0.3	-2.8	0.6	0.9	-0.6	0.4	-0.8	-0.8	0.0	2.6	-0.3	22	0.9
99	1.1	0.0	0.1	-0.1	0.2	0.2	-0.3	-0.1	0.1	0.5	0.1	0.2			-0.5	0.6	-0.6	0.6	-0.5	0.8	-0.4	-0.9	20	0.3
100	1.1	0.5	0.8	-1.7	0.5	0.3	0.6	0.6	0.8	0.9	1.1	1.1	0.0	0.9	0.5	0.8	0.1	-0.9	0.2	0.6	-0.7	3.5	22	1.2
101	-0.3	-0.3	0.1	-0.4	-0.7	0.4	-0.4	0.0	-0.6	-0.2	0.3	-0.6	-0.4	-0.3	-0.3	-0.2	-0.3	-0.1	-0.6	-0.8	0.3	-0.2	22	0.2
103*	-0.3	0.1	0.5	0.8	0.5	-0.1	-0.1	0.0	0.2	0.5	0.5	0.5	-0.6	-0.4	0.5	0.6	-3.5	1.3	-0.3	0.5	0.0	0.2	22	0.8
104*	-0.3	0.2	-0.6	0.2	-0.4	-0.2	-0.3	-0.5	-0.5	-0.2	-0.2	-0.5	-0.5	-0.2	-0.4	0.0	-0.2	-0.9	0.0	0.3	-0.7	-0.5	22	0.2

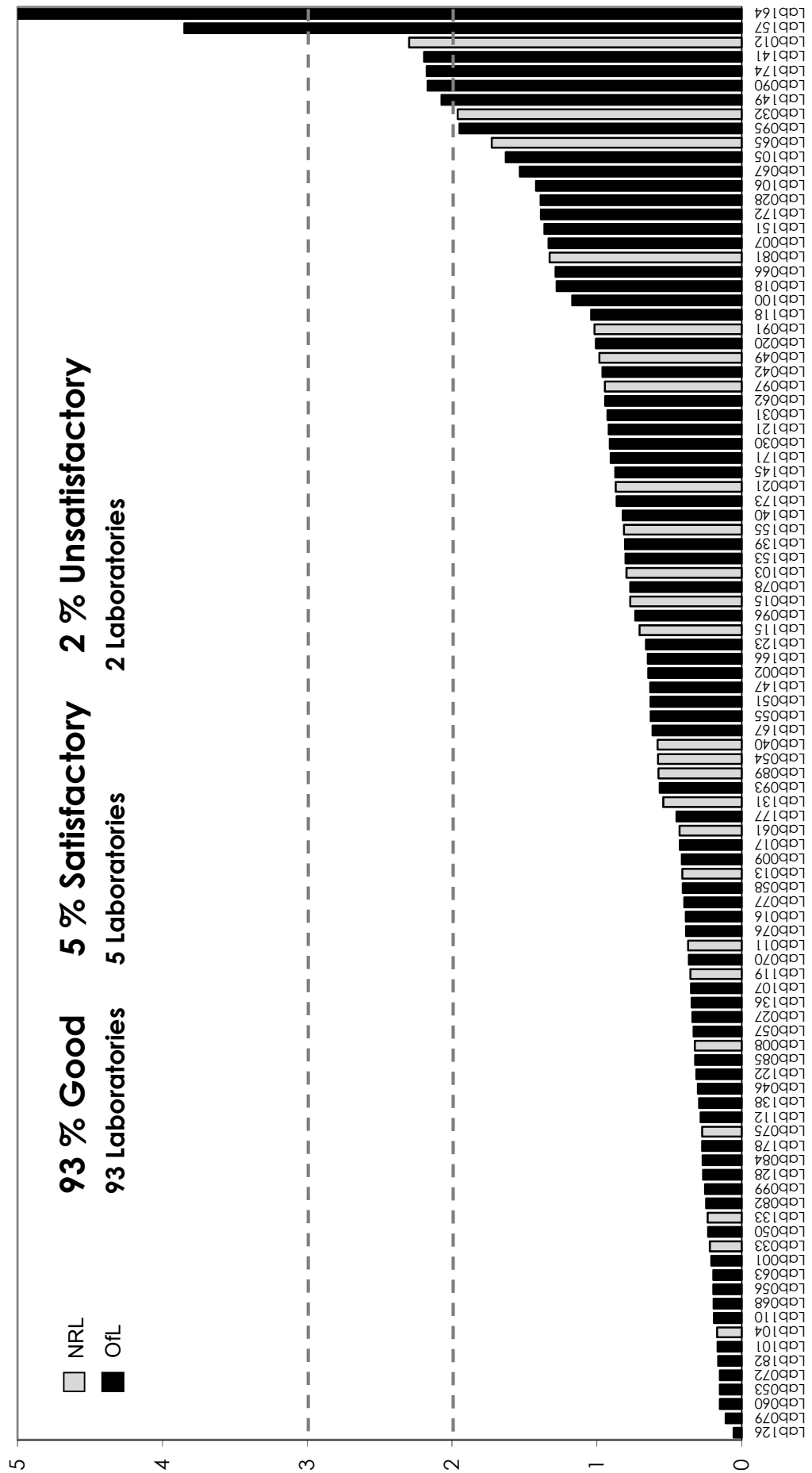
APPENDIX 5. Average of the Squared z-Score (AZ²) for laboratories in Category A.

Lab Code	Acetaminiprid	Acrinathrin	Buprofezin	Chlorothaloniil	Chlorpyrifos	Cypermethrin	Cyprodinil	Diazinon	Difencanazole	Endosulfan alpha	Endosulfan beta	Fenamiphos	Fenamiphos sulfone	Fenamiphos sulfoxide	Fenhexamid	Fludoxonil	Lambda-Cyhalothrin	Methoxyfenozide	Plitmicarb	Pyridaben	Sphinosad	Tetraconazole	No. of z-scores	AZ ²
	z-scores																							
105	0.3	-2.7	-0.1	3.1	0.4	-0.1	-0.1	-0.7	1.1	0.7	-0.3	0.1	0.8	1.1	-0.6	-0.3	0.4	0.8	0.7	0.1	3.6	-0.2	22	1.6
106	0.0	1.5	0.9	-2.3	-0.2	0.8	-0.1	-0.1	1.5	0.5	0.5	0.1	0.0	-0.7	0.8	4.0	0.4	-0.6	0.7	-0.6	0.2		21	1.4
107	1.3	0.3	0.2	0.0	0.3	0.4	0.4	0.5	0.1	0.4	0.2	0.0	-1.1	-1.1	0.7	0.4	0.1	1.0	-0.5	0.3	-0.1	0.8	22	0.3
110	0.1	0.3	0.4	-0.1	0.7	1.0	0.3	0.3	0.3	0.1	0.8	0.2	0.2	0.0	0.5	0.4	-0.1	0.4	0.2	0.5	-0.2	0.6	22	0.2
112	0.1	-0.2	0.6	0.1	0.8	0.3	-0.1	1.2	-0.1	0.1	0.1	0.4	1.1	0.8	0.4	0.0	-0.5	0.6	0.0	0.2	-0.6	0.6	22	0.3
115*	0.7	0.1	0.8	0.7	0.7	0.3	0.7	0.8	0.6	1.0	1.9	0.5	0.0	0.5	1.1	0.5	0.6	1.3	1.4	0.8	0.7	0.2	22	0.7
118	-0.3	-0.6	-0.2	3.0	0.4	-1.2	-0.1	0.6	-0.3	1.6	0.7	-0.9	0.5	1.6	0.1	-1.7	-0.3	0.1	-0.4	1.3	-0.1	-0.2	22	1.0
119*	-0.9	0.7	0.1	0.2	0.5	0.5	0.0	0.5	0.1	0.1	0.2	0.1	-0.2	-1.4	0.4	1.2	0.2	0.7	-0.8	0.4	-0.7	0.3	22	0.4
121	0.0	-0.5	1.2		3.0	-1.6	-0.1	-1.0	0.9	1.0	-0.7	0.5	-0.8	-0.9	0.0	0.0	-0.5	0.4	-0.4	-0.3	0.6	0.5	21	0.9
122	0.4	-0.1	-0.2	2.1	-0.1	-0.1	0.0	0.0	0.2	0.1	-0.4	-0.3	-0.3	-0.2	-0.2	-0.2	0.9	-0.1	-0.4	0.3	-0.9	-0.1	22	0.3
123	-0.8	-0.5	-0.5	1.5	-0.9	-0.9	-1.3	-0.7	-1.0	-0.4	-0.8	-0.5	-0.6	-1.2	-1.0	-0.3	-0.4	-0.6	-0.8	-1.1	-0.3	-0.5	22	0.7
126	0.1	0.2	0.2	-0.7	0.1	0.3	-0.1	0.2	0.1	0.1	0.1	-0.1	0.1	0.3	-0.3	0.1	0.3	0.2	0.0	0.0	0.0	0.3	22	0.1
128	-0.2	-0.9	-0.4	1.0	0.0	-0.4	0.1	-0.2	-0.3	-0.4	-0.1	0.3	-0.5	-1.1	0.5	0.2	-0.7	-0.1	-0.4	-0.8	0.4	0.0	22	0.3
131*	1.5	-0.7	-0.9	-0.4	-0.5	-0.1	-0.4	-0.7	-0.4	-0.6	-0.5	-0.3	-1.0	-1.5	-0.2	-0.4	-0.4	-0.7	-0.6	-0.3	-1.1	-0.8	22	0.5
133*	-0.3	-1.0	0.1	0.8	0.6	-0.2	-0.4	-1.0	-0.5	-0.7	-0.3	0.5	-0.2	-0.3	-0.4	0.5	-0.4	-0.3	0.0	0.0	-0.1	-0.3	22	0.2
136	0.0	0.0	0.3	-1.4	-0.8	0.6	-0.6	0.5	-0.7	-0.5	-0.4	0.9	0.1	-0.1	-0.2	0.0	-0.6	-0.2	-0.5	-0.9	0.9	-0.3	22	0.3
138	-0.1	0.6	-0.1	-2.0	0.0	-0.5	0.3	-0.1	-0.3	0.0	-0.2	-0.1	0.0	0.0	-0.1	-0.3	-0.9	-0.5	0.1	-0.5	-0.5	0.0	22	0.3
139	0.0	-0.3	-0.7	-0.1	-1.0	-0.4	0.1	-1.6	-0.3	-0.1	-0.8	-0.3	0.0	1.3	1.1	0.2	-1.9	-0.9	0.8	-1.4	-0.5	-1.5	22	0.8
140	0.2	-0.5	-0.8	-0.1	-0.8	-0.7	-0.5	-1.1	-0.7	-0.4	-0.3	-0.5	0.0	2.6	-0.2	-0.7	-0.9	0.1	-0.2	-1.4	-0.2	-1.7	22	0.8
141	0.4	0.1	-0.3		0.2	0.8	-0.5	0.5	0.4	0.6	0.1	0.6	3.3	0.3	-1.9	-0.3	0.0	2.8	-0.1	0.2	3.2	3.3	21	2.2
145	-0.3	-0.4	0.4	0.1	-0.4	-0.4	0.4	0.1	0.4	-2.1	-2.2	-0.5	-0.5	-0.3	0.5	-0.2	-0.9	-0.3	0.4	-0.4	2.6	0.2	22	0.9
147	-0.8	1.8	0.3	0.6	0.3	1.4	0.9	0.4	1.2	0.3	0.5	-0.1	1.1	0.8	0.3	0.1	0.2	-0.1	1.0	-0.3		1.0	21	0.6
149	-2.6	2.4	0.1	0.1	-1.8	-0.1	-0.4	0.0	-1.4	-0.6	-0.4	-2.9			-1.6	-1.2	-0.2	1.8	-2.4	-1.2	-0.9	-0.3	20	2.1
151	0.0	-0.7	-0.2	-1.9	-0.7	-0.5	-0.3	-0.4	-1.2	-0.5	-0.7	-2.3	0.1	-3.1	2.1	-0.1	-1.1	-0.6	0.9	0.1	1.1	-0.4	22	1.4
153	0.1	1.7	0.5	-1.0	0.2	-0.1	0.0	0.4	0.0	0.3	0.6	-1.0	0.4	-1.9	-0.5	-0.7	1.2	1.7	-0.1	0.2		1.3	21	0.8
155*	-0.1		-0.4	-0.4	0.7	0.1	-0.6	0.8	-0.7	-0.2	-1.8	-0.7	-0.9	0.0	-0.7	1.0	1.7	1.6	-0.1	0.8	-1.3	1.0	21	0.8
157	-0.6	-0.5	-1.6	-3.3	-1.6	-1.3	-2.4	0.3	-1.5	-2.6	-2.7	-1.8	-0.7	1.2	-2.1	-0.3	-0.9	-2.3	-0.1	1.3	-1.0	5.0	22	3.8
164	-0.5	-2.7	-0.8	-1.8	4.0	-0.6	0.1	-0.3	-0.8	-0.1	0.0	-0.2	5.0	5.0	-0.9	-3.8	0.2	-3.8	-0.4	-0.2	5.0	-0.2	22	5.0
166	0.4	0.2	-0.2	-2.0	-0.5	-0.4	0.0	0.1	1.0	-0.5	-0.2	-1.0	-2.5	0.3	0.0	0.3	-0.1	-0.1	-0.1	0.5	-0.5	0.0	22	0.6
167	-0.6	0.8	-0.7	1.5	-1.6	-1.6	-1.0	-0.2	-0.5	0.2	1.0	-0.6	0.6	-0.1	-0.9	-0.2	0.4	-0.4	-0.4	-0.4	-0.2	-0.4	22	0.6
171	-0.2	0.3	-0.1	0.7	0.3	0.4	0.7	1.7	0.6	0.1	0.1	0.3	0.0	-0.8	-0.5	1.6	-0.6	1.1	-0.4	1.0	1.5	2.5	22	0.9
172	1.7	0.3	-0.2	1.0	0.2	0.1	-0.1	0.3	-0.3	1.9	-1.0	3.3	0.0	1.5	0.6	1.1	-0.2	-0.8	2.0	-1.4	0.7	0.2	22	1.4

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

Lab Code	Acetamiprid	Acrinathrin	Buprofezin	Chlorothaloniil	Chlorpyrifos	Cypermethrin	Cyprodinil	Diazinon	Difenoconazole	Endosulfan alpha	Endosulfan beta	Fenamiphos	Fenamiphos sulfone	Fenamiphos sulfoxide	Fenhexamid	Fludoxonil	Lambda-Cyhalothrin	Methoxyfenozide	Plimicarb	Pyridaben	Spinosad	Tetraconazole	No. of z-scores	AZ ²
	z-scores																							
173	0.2	0.6	0.2	1.6	0.8	0.5	0.1	0.3	0.5	0.4	0.3	-1.0	-0.4	0.4	0.5	0.3	0.2	1.1	0.7	0.5	3.2	0.6	22	0.9
174	-1.2	3.1	-1.0	-2.4	-1.5	0.2	-0.8	-1.1	-2.2	-1.1	-1.2	-0.6	1.7	-2.1	-0.9	-1.3	-1.0	-0.7	-1.1	-1.7	-1.0	-1.3	22	2.2
177	0.8	0.2	0.2	0.5	-0.2	-1.2	0.2	0.3	0.4	-0.1	-0.2	-0.3			1.2	0.5	-0.2	1.0	0.2	0.7	0.3	-1.7	20	0.5
178	0.4	-0.6	-0.2	0.2	0.4	-0.6	-0.1	-1.1	0.5	-0.1	0.2	0.1	0.2	0.6	0.0	-0.5	-1.2	-0.7	0.3	-0.7	-0.1	-0.3	22	0.3
182	-0.2	-0.2	-0.4	0.2	-0.1	0.3	0.4	0.4	-0.1	0.0	0.3	-0.7	0.5	0.4	-0.8	0.4	0.6	0.2	0.3	-0.2	0.0	0.8	22	0.2

EUPT-FV-16 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-SANCO⁵ 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. Official labs from EFTA countries and EU-Candidate countries are also welcome to participate in the EUPTs. Laboratories from Third countries may be permitted to participate on a case-by-case basis after consultation with DG-SANCO.

The following four EURLs for pesticide residues were appointed by DG-SANCO based on regulation 882/2004/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)

NRLs are appointed at Member State level, based on the provisions of Regulation 882/2004/EC, whereas OfLs are laboratories that are actively involved in official control of pesticide residues in food or feed in the sense of Regulation 882/2004/EC or Regulation 396/2005/EC. This includes labs involved in control within the framework of national and/or EU-controlled programmes as well as labs involved in import controls according to Regulation 669/2009/EC.

According to Article 28 (3) of Regulation 396/2005/EC⁸, all laboratories analysing samples for the official control of pesticide residues are obliged to participate in Proficiency Test(s) organised by the European Union.

⁵ DG-SANCO = European Commission, Health and Consumer Protection Directorate-General

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

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

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 co-ordinated multiannual community 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.

EUPT-Organizers and External Consultants

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

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

To complement the internal expertise of the EURLs a group of external consultants named **EUPT-Scientific Committee** (EUPT-SC)¹⁰ has been established and approved by DG SANCO. The EUPTSC consists of expert scientists with long-term experience in PTs and/or pesticide residue analysis. The actual composition of the EUPT-SC, the affiliation of each member and the topics each member is consulted are listed in the EURL-Website. They 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 in the Test Item.

⁸ 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.

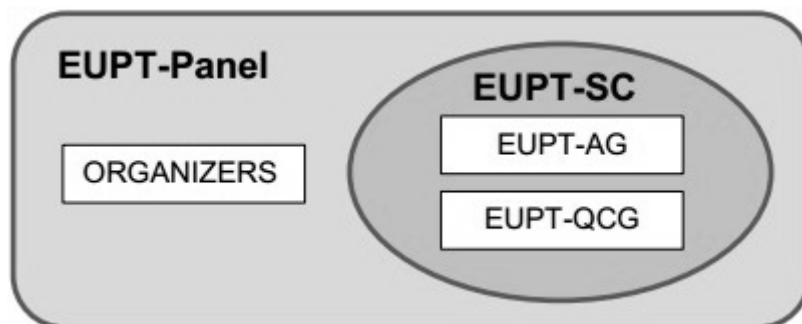
⁹ European Commission Proficiency Tests for Pesticide Residues in Fruits and Vegetables, Trends in Analytical Chemistry, 2010, 29 (1), 70-83.

¹⁰ Link to the List of current members of the EUPT Scientific Committee: <http://www.eurl-pesticides.eu/library/docs/allcrl/EUPT-SC>.

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

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 decisions. Upcoming EUPTs are also planned during these meetings.

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



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-SANCO.

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 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. This "List of Obligated Labs" is to be considered as tentative as it is based only on information submitted by OfLs concerning their commodity scope and status. The legal obligation of NRLs and OfLs to participate in EUPTs arises from:

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

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

NRLs are responsible for checking whether all relevant OfLs within their network are included in the list of obligated laboratories and whether the contact information and commodity-scope is 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 intending to participate, must provide the reasons for their non-participation without prejudice of any legal action taken

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

against them for not participating. This also applies to initially participating laboratories that fail to report results.

Confidentiality and Communication

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

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

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 EUPT-report.

For each EUPT the organizing 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 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 contains the commodity to be used as the 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).

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

In some cases, that will be clearly marked, residue definitions in the Target Pesticide List may differ from the legal ones. 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 laboratories. The Specific Protocol contains 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 other relevant information.

Homogeneity of the Test Item

In order for the Test Item to be suitable for the proficiency test the pesticides contained in it must be uniformly distributed within the bulk Test Item. The variation of the pesticide concentration between the different sub-sample test portions should not significantly impact the performance of the participating labs. To check for homogeneity, two analytical portions, taken from at least ten randomly chosen units of treated Test Item, are analysed in duplicate. 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. $\sigma_{all}^2 = 0.3 \times \text{FFP-RSD (25 \%)} \times$ 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 behavior 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

To make sure that any pesticide losses occurring during the PT at the recommended storage conditions will not significantly impact the performance of the labs a stability test according to ISO 13528, Annex B is conducted by the Organizers. 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 second one shortly after the deadline for submission of results. To better recognize trends and gain additional certainty one or more additional tests may be conducted by the Organizers. At least 6 sub-samples (analytical portions)

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

should be analyzed on each test day (e.g. 2 analytical portions withdrawn from three randomly chosen containers OR 6 portions withdrawn from one 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 (e.g. inorganic bromide) is very unlikely to be significantly affected during storage (e.g. based on past stability tests or based on knowledge of its physicochemical properties) the Organizers, after consultation with the EUPT-QCG, may decide to omit a specific stability test. The final decision on whether analytes for which the stability test was not undertaken must be included in the final evaluation after consideration of the distribution of results (Qn-RSD) and following consultation with EUPT-SC.

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 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 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

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

analyte detected in the Test Item. The concentrations of the pesticides detected should be expressed in 'mg/ kg' and in some cases in 'µg/kg fat' for products of animal origin.

The Test Item is intentionally treated with pesticides whereas the Blank Item is analysed to ensure that it does not contain any of the pesticides in the Target Pesticides List at or above the specified MRRLs. Both the Test Item and Blank Item 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 Feed¹¹, 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 with good precision). Other approaches for recovery correction explicitly allowed in the SANCO document are the use of isotope labelled analogues of the target analytes as Internal Standards (ISTDs) as well as the approach of 'standard addition' with additions of analyte(s) being made to analytical portions. Where reported residue data have been adjusted for recovery, this must be indicated on the specific field of the 'Result Submission Form'. 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 results automatically from using the 'standard addition(s)' approach, or isotopically-labelled internal standards (in both cases with spiking of the Test Item at the beginning of the extraction procedures). In these cases, the laboratories should report the recovery adjustment 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. Qn-RSD>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

¹¹ Document N° SANCO/12495/2011

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

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.

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 (μ)

The assigned value (= consensus concentration or 'true' concentration) will be typically estimated using the median of all the results after excluding outliers. 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, outliers¹² and other results associated with obvious errors have to be removed from the population.

Where the Organizers (e.g. after the publication of the preliminary report) receive information that indicates gross errors, which have a strong impact on the generated result, such as:

- a) incorrect recording (e.g. due to transcription errors by the participant, decimal point faults or transposed digits),
- b) calculation errors (e.g. missing factors),
- c) analysis of a wrong sample,
- d) use of wrong concentrations of standard solutions,
- e) incorrect data processing (e.g. integration of wrong peak),
- f) major deviation from the analytical procedure

¹² Reported results that are numerically so distant from the rest of the results within a dataset, that they are considered irreconcilable with them.

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

- g) inappropriate storage or transport conditions (in case of susceptible compounds),
- h) use of inappropriate procedures that demonstrably lead to significantly biased results (e.g. due to degradation or incomplete extraction).

Particular results will be examined on a case-by-case basis to decide whether or not they should be excluded from the population used to determine the assigned value. 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. An omission of a result from the calculation of the assigned value does, however, not necessarily mean that z-scores will not be calculated.

Where results are to be omitted/excluded based on the use of a biased methodology, the number of results associated with this type of methodology as well as the overall distribution of results (kernel density histogram) will be taken into account. The Organizers may furthermore conduct experiments demonstrating that a certain methodology leads to significantly biased results.

After eliminating obvious errors, all results with z-scores > 5 will be regarded as outliers and excluded from the assigned value calculation. After exclusion of these results, the assigned value of the remaining results and the corresponding z-scores will be recalculated.

Following the omission/exclusion of all values with z-scores > 5 an appropriate outlier test (e.g. the Grubbs' test) may be applied, if this is deemed necessary, to identify any statistical outliers within the remaining data-set. The Grubbs' test should be applied following the instructions in ISO 5725-2 and only where the distribution of the results appears to be Gaussian (no clear indications for bimodality). The significance level (α) for identifying statistical outliers using the Grubbs' test will be set at 0.01 (=99% confidence level). Omitted results might be interesting as they might give indications about possible source(s) of errors. The Organizers will thus ask the relevant lab(s) to provide feedback on possible sources of errors (see also "follow-up activities").

Uncertainty of the assigned value

The uncertainty¹³ of the assigned values μ_i is calculated as:

$$\mu_i = 1.25 * \frac{Qn \text{ SD}}{\sqrt{n}}$$

Where $Qn \text{ SD}$ 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 by the participants), the EUPT-Panel may consider the assigned value of a specific analyte to be too uncertain and decide that

¹³ ISO 13528:2005, Statistical methods for use in proficiency testing by interlaboratory comparisons, International Organization for Standardization.

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

the results should not be evaluated, or only evaluated for informative purposes. The provisions of ISO 13528:2005 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 (δ) of the assigned value will be calculated using a Fit-For-Purpose Relative Standard Deviation (FFP-RSD) approach, as follows:

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

The percentage FFP-RSD is set at 25% based on experience from previous EUPTs¹⁴. The EUPTPanel reserves the right to also employ other approaches on a case-by-case basis considering analytical difficulties and experience gained from previous proficiency tests.

– **z-scores**

This parameter is calculated using the following formula:

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

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

Any z-scores > 5 will be 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:

$ z \leq 2$	Acceptable
$2 < z \leq 3$	Questionable
$ z > 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.

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

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

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

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

– 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 (AZ^2)^{15,16} (see below) will be used. The AZ^2 is calculated as follows:

¹⁵ Formerly named "Sum of squared z-scores (SZ2)"

¹⁶ 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.

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

Where n is the number of z-scores to be considered in the calculation. In the calculation of the AZ^2 , zscores higher than 5 will be set at 5. Based on the AZ^2 achieved, the laboratories are classified as follows:

$AZ^2 \leq 2$	Good
$2 < AZ^2 \leq 3$	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 AZ^2 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 with 5 or more z-scores available. For the calculation of the AAZ, z-scores higher than 5 will be set at 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 (zscores) are available.

Publication of results

The EURLs will publish a preliminary report, containing tentative medians and z-score values for all pesticides present in the Test Item, within 2 months from 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.

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 zscores 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,

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

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 - including all false positives and false negatives, along with results with $|z| > 2$.

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 $|z| > 3$. Concerning z-scores between 2 and 3 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.

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



EUPF-V-16 SPECIFIC PROTOCOL

European Union Proficiency Test for Pesticide Residues in Fruits and Vegetables (2014)

Introduction

This protocol is complementary to the General Protocol of EU Proficiency Tests (EUPF) for Pesticide Residues in Food and Feed (4th Edition, Approved: May 2013). This Proficiency Test is organised by the EURL for Pesticide Residues in Fruits and Vegetables covering Multiresidue Methods (MRM) of analysis.

Test Item

This proficiency test is based on the analysis of incurred pesticide residues in green pepper. The peppers 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, homogenized 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. Two bottles, again chosen randomly, will be analysed over a period of time to confirm the stability of the pesticides in the test item (firstly, when the test items are shipped, then a few days after the receipt deadline for participants' results). There will be one further analysis during this period reproducing the sample shipment to see if there is any degradation of any of the pesticides present in the test item.

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

Steps to follow

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

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

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 peppers.

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 **175 Euros** for EU and EFTA laboratories and **250 Euros** for any other participants. The payment procedure must have started before 24th February 2014. An e-mail showing the bank transfer confirmation, or similar, must have been sent beforehand; or 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 3rd – 24th February 2014, 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.

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 have accepted the test item. This Form has a deadline: 28th February 2014, 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 - 17th March 2014 - 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 24th – 28th March 2014. 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.

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

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. It is possible that the laboratory, after receipt of the test item, performs further validations for some of the pesticides and then wants to report results for these pesticides. In that case, the laboratory will have to inform the organisers so they can include the new pesticides in their scope. This year again no residue definition needs to be followed so only individual contributions will be requested.

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 pepper test item treated with pesticides.
- Approximately 300 g of 'blank' pepper 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 24th February 2014. 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 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.

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

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 28th February 2014. 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 28th February, 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.

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.

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

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 17th March 2014. 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 peppers 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 24th – 28th March 2014.

Calendar

ACTIVITY	DATE
Publishing the Target Pesticide List, Calendar and Matrix on the Web page.	15 th November 2013
Receiving Application Form from invited laboratories.	23 rd December 2013 - 31 st January 2014
Specific Protocol published on the Web site.	3 rd February 2014 at the latest
Deadline for receiving Laboratory scope: Form 0	3 rd -14 th February 2014
Sample distribution.	24 th February 2014
Deadline for receiving sample acceptance: Form 1	28 th February 2014
Deadline for receiving results: Form 2, Form 3 and Form 4	17 th March 2014
Filling in Form 5	24 th – 28 th March 2014
Preliminary Report: only results, no statistical treatment	End of April 2014
Final Report distributed to the Laboratories	November 2014

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

Cost of test item shipment.

EU and EFTA laboratories will be charged **175€** 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. 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

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
Dr. Milagros Mezcuca Peral	EURL-FV mmezcuca@ual.es +34 950014102
Ms. Carmen Ferrer Amate	EURL-FV cferrer@ual.es +34 950014102
Mr. Octavio Malato Rodríguez	EURL-FV omalato@ual.es +34 950214423
Ms. Noelia Belmonte Valles	EURL-FV nbelmonte@ual.es +34 950015645
Ms. Ana Lozano Fernández	EURL-FV analozano@ual.es +34 950015645
Ms. M ^o del Mar Gómez Ramos	EURL-FV mgr337@ual.es +34 950015645
Ms. Samanta Uclés Duque	EURL-FV samantaucles@ual.es +34 950015645
Ms. Ana Uclés Moreno	EURL-FV anauclesm@ual.es +34 950015645

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

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

Advisory Group

- Dr. Michelangelo Anastassiades, Senior Chemist, CVUA, Stuttgart, Germany.
- Mr. Richard Fussell, Senior Chemist, FERA, York, United Kingdom.
- Dr. Miguel Gamón, Senior Chemist, Laboratorio Agroalimentario, Valencia, Spain.
- 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, Institute for Food Safety, Innsbruck, Austria.
- Dr. Tuija Pihlström, Senior Chemist NFA, Uppsala, Sweden.
- Dr. Mette Erecius Poulsen, Senior Chemist, NFI, Copenhagen, Denmark.
- Dr. Darinka Štajnbaher, Senior Chemist, Maribor, Slovenia.

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

TARGET PESTICIDE LIST FOR THE EUPT-FV-16

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
Amitraz	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
Captan	0.01
Carbaryl	0.01
Carbendazim (sum of benomyl and carbendazim expressed as 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
Dichlofluaniid (only parent compound)	0.01
Dichlorvos	0.01
Dicloran	0.01
Dicofol	0.01
Difenoconazole	0.01
Diflubenzuron	0.01
Dimethoate	0.003
Dimethomorph	0.01
Dimethylaminosulfotoluidide (DMST)	0.01
Diphenylamine	0.01
DMF (2,4-Dimethylformanilide)	0.01
DMPF (N-2,4-Dimethylphenyl-N-Methyl-formamidine)	0.01
Endosulfan alpha	0.01
Endosulfan beta	0.01
Endosulfan sulfate	0.01
EPN	0.01
Epoxiconazole	0.01
Ethion	0.01
Ethoprophos	0.008

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

Pesticide	MRRL (mg/Kg)
Etofenprox	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
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
Fipronil (only parent compound)	0.004
Fludioxonil	0.01
Flufenoxuron	0.01
Fluopicolide	0.01
Fluquinconazole	0.01
Flusilazole	0.01
Flutolanil	0.01
Flutriafol	0.01
Folpet	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
Isofenphos-methyl	0.01
Kresoxim-methyl	0.01
Lambda-Cyhalothrin	0.01
Linuron	0.01
Lufenuron	0.01
Malaoxon	0.01
Malathion	0.01
Mepanipyrim (only parent compound)	0.01
Metaflumizone	0.01
Metalaxyl and metalaxyl-M	0.01
Metconazole	0.01
Methamidophos	0.01
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
Paclobutrazole	0.01
Paraoxon-methyl	0.01
Parathion-ethyl	0.01
Parathion-methyl	0.01

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

Pesticide	MRRL (mg/Kg)
Penconazole	0.01
Pencycuron	0.01
Pendimethalin	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
Tetraconazole	0.01
Tetradifon	0.01
Thiabendazole	0.01
Thiacloprid	0.01
Thiamethoxam	0.01
Thiodicarb	0.01
Thiophanate-methyl	0.01
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
Triticonazole	0.01
Vinclazolin (only parent compound)	0.01
Zoxamide	0.01

This list is based on Commission Regulation (EU) No No 788/2012
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-16.

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
AUSTRIA	AUSTRIAN AGENCY FOR HEALTH AND FOOD SAFETY (AGES), INSTITUTE FOR FOOD SAFETY INNSBRUCK, DEPARTMENT FOR PESTICIDE AND FOOD ANALYTICS (PLMA)	Innsbruck	YES
AUSTRIA	MA 38 - LEBENSMITTELUNTERSUCHUNGSANSTALT DER STADT WIEN	Wien	YES
BELGIUM	SCIENTIFC INSTITUTE OF PUBLIC HEALTH	Bruxelles	YES
BELGIUM	LOVAP	Geel	YES
BELGIUM	FYTOLAB CVBA	Zwijnaarde	YES
BRASIL	PESTICIDE RESIDUE ANALYSIS LABORATORY - ITEP/LABTOX	Recife	YES
BRASIL	CENTER OF RESEARCH AND ANALYSIS OF RESIDUES AND CONTAMINANTS (CEPARC)	Santa Maria	YES
BULGARIA	CENTRAL LABORATORY FOR CHEMICAL TESTING AND CONTROL	Sofia	YES
BULGARIA	EURO LAB	Svilengrad	YES
CHINA	KEY LABORATORY OF FOOD SAFETY RISK ASSESSMENT	Beijing	YES
CHINA	SHANGHAI MUNICIPAL CENTER FOR DISEASE CONTROL AND PREVENTION	Shanghai	YES
CROATIA	TEACHING INSTITUTE OF PUBLIC HEALTH OF PRIMORSKO-GORANSKA COUNTY, DEPARTMENT OF HEALTH ECOLOGY	Rijeka	YES
CROATIA	INSTITUTE OF PUBLIC HEALTH SPLIT	Split	YES
CROATIA	CROATIAN NATIONAL INSTITUTE OF PUBLIC HEALTH, ECOLOGY SERVICE, PESTICIDE UNIT	Zagreb	YES
CROATIA	E.C. INSPEKT D.O.O.	Zagreb	YES
CROATIA	EUROINSPEKT CROATIAKONTROLA D.O.O. - LABORATORY	Zagreb	YES
CROATIA	FOOD CONTROL CENTER	Zagreb	YES
CROATIA	PUBLIC HEALTH INSTITUTE "DR ANDRIJA ŠTAMPAR"	Zagreb	YES
CYPRUS	PESTICIDE RESIDUES LABORATORY OF THE STATE GENERAL LABORATORY	Nicosia	YES
CZECH REPUBLIC	CENTRAL INSTITUTE FOR SUPERVISING AND TESTING IN AGRICULTURE (UNITED KINGDOMZUZ)	Brno	YES
CZECH REPUBLIC	INSTITUTE OF CHEMICAL TECHNOLOGY, DEPT. OF FOOD ANALYSIS AND NUTRITION	Prague	YES
CZECH REPUBLIC	CZECH AGRICULTURE AND FOOD INSPECTION AUTHORITY	Praha	YES
DENMARK	DANISH VETERINARY AND FOOD ADMINISTRATION	Ringsted	YES
DENMARK	NATIONAL FOOD INSTITUTE, TECHNICAL UNIVERSITY OF DENMARK	Soeborg	YES
EGYPT	CENTRAL LAB OF RESIDUE ANALYSIS OF PESTICIDES AND HEAVY METALS IN FOODS	Dokki, Giza	YES
ESTONIA	LABORATORY FOR RESIDUES AND CONTAMINANTS, AGRICULTURAL RESEARCH CENTRE	Saku	YES
ESTONIA	TARTU LABORATORY OF HEALTH BOARD	Tartu	YES

ANNEX 2. List of laboratories that agreed to participate in EUPT-FV-16.

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
FINLAND	FINNISH CUSTOMS LABORATORY	Espoo	YES
FINLAND	METROPOLILAB OY	Helsinki	YES
FRANCE	GIRPA	Beaucouze	YES
FRANCE	CERECO SUD	Garons	YES
FRANCE	CAPINOV - TRISKALIA	Landerneau	YES
FRANCE	LABORATOIRE DEPARTEMENTAL DE LA SARTHE	Le Mans	YES
FRANCE	LABORATOIRE DU SCL DE MASSY	Massy Cedex	YES
FRANCE	LABORATOIRE DU SCL DE MONTPELLIER	Montpellier	YES
FRANCE	IDAC	Nantes	YES
FRANCE	CENTRE ANALYSE MÉDITERRANÉE PYRÉNÉES	Perpignan	YES
GERMANY	THÜRINGER LANDESAMT FÜR VERBRAUCHERSCHUTZ	Bad Langensalza	YES
GERMANY	FEDERAL OFFICE OF CONSUMER PROTECTION AND FOOD SAFETY (BVL)	Berlin	YES
GERMANY	CVUA WESTFALEN	Bochum	YES
GERMANY	CHEMISCHES UND VETERINÄRUNTERSUCHUNGSAMT RHEINLAND	Bonn	YES
GERMANY	LANDESUNTERSUCHUNGSAMT FÜR CHEMIE, HYGIENE UND VETERINÄRMEDIZIN BREMEN	Bremen	YES
GERMANY	CHEMISCHES UND VETERINÄRUNTERSUCHUNGSAMT OSTWESTFALEN-LIPPE (CVUA-OWL)	Detmold	YES
GERMANY	LUA SACHSEN, DEUTSCHLAND	Dresden	YES
GERMANY	AMT FÜR VERBRAUCHERSCHUTZ DUESSELDORF - CHEMISCHE UND LEBENSMITTELUNTERSUCHUNG	Duesseldorf	YES
GERMANY	BAYERISCHES LANDESAMT FUER GESUNDHEIT UND LEBENSMITTELSICHERHEIT	Erlangen	YES
GERMANY	CHEMISCHES UND VETERINÄRUNTERSUCHUNGSAMT STUTTART	Fellbach	YES
GERMANY	CHEMISCHES LABOR DR. MANG	Frankfurt	YES
GERMANY	LANDESLABOR BERLIN-BRANDENBURG	Frankfurt, Oder	YES
GERMANY	LANDESAMT FÜR VERBRAUCHERSCHUTZ SACHSEN-ANHALT (LAV)	Halle/Saale	YES
GERMANY	EUROFINS DR. SPECHT LABORATORIEN GmbH	Hamburg	YES
GERMANY	GALAB LABORATORIES GmbH	Hamburg	YES
GERMANY	INSTITUT FUER HYGIENE UND UMWELT	Hamburg	YES
GERMANY	LITZ AUGUSTENBERG	Karlsruhe	YES
GERMANY	LANDESBETRIEB HESSISCHES LANDESLABOR	Kassel	YES
GERMANY	LUFA-ITL GmbH	Kiel	YES
GERMANY	CHEMISCHES UND VETERINÄRUNTERSUCHUNGSAMT RHEIN-RUHR-WUPPER (CVUA-RRW)	Krefeld	YES

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GERMANY	ZENTRALES INSTITUT DES SANITÄTSDIENSTES DER BUNDESWEHR KIEL	Kronshagen	YES
GERMANY	CVUA-MEL CHEMISCHES UND VETERINÄRUNTERSUCHUNGSAMT MÜNSTERLAND-EMSCHER-LIPPE	Münster	YES
GERMANY	STATE LABORATORY SCHLESWIG-HOLSTEIN	Neumünster	YES
GERMANY	NIEDERSAECHSISCHES LANDESAMT FUER VERBRAUCHERSCHUTZ UND LEBENSMITTELSICHERHEIT, LEBENSMITTEL- UND VETERINAERINSTITUT OLDENBURG	Oldenburg	YES
GERMANY	LANDESAMT FÜR LANDWIRTSCHAFT, LEBENSMITTELSICHERHEIT UND FISCHEREI MECKLENBURG-VORPOMMERN	Rostock	YES
GERMANY	LANDESAMT FÜR VERBRAUCHERSCHUTZ	Saarbruecken	YES
GERMANY	LANDESUNTERSUCHUNGSAMT RHEINLAND-PFALZ INSTITUT FÜR LEBENSMITTELCHEMIE SPEYER	Speyer	YES
GERMANY	LABOR FRIEDLE GMBH	Tegernheim	YES
GREECE	PESTICIDE RESIDUES LABORATORY, D CHEMICAL DIVISION OF ATHENS, GENERAL CHEMICAL STATE LABORATORY	Athens	YES
GREECE	LABORATORY OF PESTICIDE RESIDUE ANALYSIS IN FRUITS & VEGETABLES, REGIONAL CENTRE OF CROP PROTECTION& QUALITY CONTROL OF IOANNINA	Ioannina	YES
GREECE	REGIONAL CENTER OF PLANT PROTECTION AND QUALITY CONTROL OF IRAKLION, LABORATORY OF PESTICIDE RESIDUES	Iraklion, Crete	YES
GREECE	PERIFERAL CENTER OF PLANT PROTECTION AND QYALITY CONTROL OF KAVALA-MINISTRY OF RURAL DEVELOPMENT & FOOD	Kavala	YES
GREECE	PESTICIDE RESIDUES LABORATORY, BENAKI PHYTOPATHOLOGICAL INSTITUTE	Kiphissia	YES
GREECE	PESTICIDE RESIDUE LABORATORY OF REGIONAL CENTRE OF PLANT PROTECTION AND QUALITY CONTROL OF PIRAEUS	Lykovrissi, Athens	YES
GREECE	REGIONAL CENTER OF PLANT PROTECTION AND QUALITY CONTROL OF NAFPLIO, LABORATORY OF PESTICIDE RESIDUES	Nafplio	YES
GREECE	PESTICIDE RESIDUES LAB. OF REGIONAL CENTER OF PLANT PROTECTION AND QUALITY CONTROL OF ACHAIA	Patra	YES
GREECE	REGIONAL CENTRE OF PLANT PROTECTION AND QUALITY CONTROL OF THESSALONIKI	Thessaloniki	YES
GREECE	REGIONAL CENTER OF PLANT PROTECTION & QUALITY CONTROL OF MAGNESIA, VOLOS	Volos	YES
HUNGARY	NATIONAL FOOD CHAIN SAFETY OFFICE, PESTICIDE RESIDUE ANALYTICAL LABORATORY OF HODMEZOVASARHELY	Hodmezovasarhely	YES
HUNGARY	NATIONAL FOOD CHAIN SAFETY OFFICE, DPPSCA PESTICIDE RESIDUE ANALYTICAL LABORATORY, MISKOLC	Miskolc	YES
HUNGARY	NATIONAL FOOD CHAIN OFFICE DPPSCA PESTICIDE RESIDUE ANALYTICAL LABORATORY SZOLNOK	Szolnok	YES
HUNGARY	NFCISO PESTICIDE ANALYTICAL LABORATORY, VELENCE	Velence	YES
ICELAND	MATIS OHF.	Reykjavík	YES
IRELAND	THE PESTICIDE CONTROL LABORATORY	Celbridge	YES
ISRAEL	PESTICIDE RESIDUES LABORATORY, PPIS, MINISTRY OF AGRICULTURE	Beit-Dagan	YES

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ITALIA	LABORATORIO DI SANITA' PUBBLICA ASL PROVINCIA DI BERGAMO	Bergamo	YES
ITALY	ARPA PUGLIA - POLO DI SPECIALIZZAZIONE "ALIMENTI" - BARI	Bari	YES
ITALY	LANDESAGENTUR FÜR UMWELT - LABOR FÜR CHROMATOGRAPHIE	Bozen	YES
ITALY	LABORATORIO CONTAMINANTI AMBIENTALI - ISTITUTO ZOOPROFILATTICO SPERIMENTALE LOMBARDIA EMILIA ROMAGNA - IZSLER	Brescia	YES
ITALY	SERVIZIO LABORATORIO CHIMICO ARPACAL	Cosenza	YES
ITALY	ARPA EMILIA ROMAGNA, AREA FITOFARMACI	Ferrara	YES
ITALY	LABORATORIO DI SANITA' PUBBLICA AZIENDA SANITARIA DI FIRENZE	Firenze	YES
ITALY	ARPA PIEMONTE POLO ALIMENTI	La Loggia (To)	YES
ITALY	ARPAL	La Spezia	YES
ITALY	ARPALAZIO SEZIONE DI LATINA	Latina	YES
ITALY	ARPA MARCHE - DIP. MACERATA	Macerata	YES
ITALY	A.R.P.A.B. (AGENZIA REGIONALE PER LA PROTEZIONE DELL'AMBIENTE DELLA BASILICATA) - DIPARTIMENTO PROVINCIALE DI MATERA	Matera	YES
ITALY	LABORATORIO PREVENZIONE ASL MILANO	Milano	YES
ITALY	ARPA CAMPANIA - DIPARTIMENTO DI NAPOLI - LABORATORIO REGIONALE FITOFARMACI E MICOTOSSINE	Naples	YES
ITALY	ISTITUTO ZOOPROFILATTICO SPERIMENTALE DELLA SICILIA	Palermo	YES
ITALY	ISTITUTO ZOOPROFILATTICO SPERIMENTALE UMBRIA MARCHE - LABORATORIO CONTAMINANTI AMBIENTALI	Perugia	YES
ITALY	ARPA FVG LABORATORIO UNICO MULTISITO - SEDE DI PORDENONE	Pordenone	YES
ITALY	IZSLT - CHEMISTRY LABORATORY	Rome	YES
ITALY	ARPA VALLE D'AOSTA	Saint Christophe	YES
ITALY	ISTITUTO ZOOPROFILATTICO SPERIMENTALE DELLA SARDEGNA	Sassari	YES
ITALY	ISTITUTO ZOOPROFILATTICO SPERIMENTALE DELL'ABRUZZO E DEL MOLISE "G. CAPORALE"	Teramo	YES
ITALY	APPA TRENTO	Trento	YES
ITALY	A.R.P.A. VENETO - SERVIZIO LABORATORI VERONA	Verona	YES
KENYA	KENYA PLANT HEALTH INSPECTORATE SERVICE (KEPHIS)	Nairobi	YES
LATVIA	INSTITUTE OF FOOD SAFETY, ANIMAL HEALTH AND ENVIRONMENT "BIOR"	Riga	YES
LITHUANIA	NATIONAL FOOD AND VETERINARY RISK ASSESSMENT INSTITUTE	Vilnius	YES
LUXEMBOURG	LABORATOIRE NATIONAL DE SANTÉ - ALIMENTAIRE	Dudelange	YES
NORWAY	BIOFORSK, PLANT HEALTH AND PLANT PROTECTION, PESTICIDE CHEMISTRY SECTION	Aas	YES

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PERU	CENTRO DE CONTROL DE INSUMOS Y RESIDUOS TOXICOS	Lima	YES
POLAND	WOJEWÓDZKA STACJA SANITARNO_EPIDEMIOLOGICZNA W ŁODZI	Łódź	YES
POLAND	PLANT PROTECTION INSTITUTE-NATIONAL RESEARCH INSTITUTE, PESTICIDE LABORATORY IN BIALYSTOK	Białystok	YES
POLAND	WOJEWODZKA STACJA SANITARNO-EPIDEMIOLOGICZNA W OPOLU	Opole	YES
POLAND	DEPARTMENT OF PESTICIDE RESIDUE RESEARCH, INSTITUTE OF PLANT PROTECTION - NATIONAL RESEARCH INSTITUTE	Poznan	YES
POLAND	INSTITUTE OF PLANT PROTECTION – NATIONAL RESEARCH INSTITUTE, REGIONAL EXPERIMENTAL STATION IN RZESZOW	Rzeszow	YES
POLAND	INSTITUTE OF HORTICULTURE, FOOD SAFETY LABORATORY	Skierniewice	YES
POLAND	INSTITUTE OF PLANT PROTECTION-NATIONAL INSTITUTE SOSNICOWICE BRANCH, LABORATORY OF PESTICIDE RESIDUE RESEARCH	Sosnicowice	YES
POLAND	MAIN INSPECTORATE OF PLANT HEALTH AND SEED INSPECTION, CENTRAL LABORATORY	Torun	YES
POLAND	LABORATORY OF VOIVODSHIP SANITARY-EPIDEMIOLOGICAL STATION	Warsaw	YES
POLAND	WOJEWÓDZKA STACJA SANITARNO-EPIDEMIOLOGICZNA WE WROCŁAWIU - DZIAŁ LABORATORYJNY	Wrocław	YES
PORTUGAL	LABORATORIO REGIONAL DE VETERINARIA E SEGURANÇA ALIMENTAR - MADEIRA	Funchal	YES
PORTUGAL	INIAV - UEISTSA - LABORATÓRIO DE RESÍDUOS DE PESTICIDAS - OEIRAS (LRP - INIAV)	Oeiras	YES
ROMANIA	LABORATORY FOR PESTICIDES RESIDUES CONTROL IN PLANTS AND VEGETABLES	Bucharest	YES
ROMANIA	SANITARY VETERINARY AND FOOD SAFETY DIRECTORATE	Bucharest	YES
ROMANIA	SANITARY VETERINARY AND FOOD SAFETY LABORATORY IASI	Iasi	YES
ROMANIA	REGIONAL LABORATORY FOR DETERMINATION OF PESTICIDE RESIDUES IN PLANT AND PLANT PRODUCTS MURES	Targu Mures	YES
SAUDI ARABIA	NATIONAL CENTER FOR MONITORING FOOD CONTAMINANTS	Riyadh	YES
SERBIA	SP LABORATORIJA	Becej	YES
SINGAPORE	VETERINARY PUBLIC HEALTH LABORATORY	Singapore	YES
SLOVAK REPUBLIC	NATIONAL REFERENCE CENTRE FOR PESTICIDE RESIDUES, PUBLIC HEALTH AUTHORITY OF SLOVAK REPUBLIC	Bratislava	YES
SLOVAKIA	VETERINARY AND FOOD INSTITUTE IN BRATISLAVA	Bratislava	YES
SLOVENIA	KMETIJSKI INŠTITUT SLOVENIJE (AGRICULTURAL INSTITUTE OF SLOVENIA)	Ljubljana	YES
SLOVENIA	NATIONAL LABORATORY OF HEALTH, ENVIRONMENT AND FOODSTUFFS (NLZOH), DEPARTMENT OF CHEMICAL ANALYSIS, LOCATION LJUBLJANA	Ljubljana	YES
SLOVENIA	NATIONAL LABORATORY OF HEALTH, ENVIRONMENT AND FOODSTUFFS (DEPARTMENT FOR CHEMICAL ANALYSIS MARIBOR)	Maribor	YES
SPAIN	AGRICULTURAL AND PHYTOPATHOLOGICAL LABORATORY OF GALICIA	Abegondo, A Coruna	YES
SPAIN	LABORATORIO DE RESIDUOS-DEPARTAMENTO DE ANÁLISIS AMBIENTAL-INSTITUTO TECNOLÓGICO DE CANARIAS, S. A.	Agüimes	YES

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SPAIN	ANALYTICA ALIMENTARIA GMBH, SUCURSAL EN ESPAÑA	Almeria	YES
SPAIN	LABORATORIO DE SALUD PUBLICA ALMERIA	Almería	YES
SPAIN	LABORATORIO DEL SOIVRE. DIRECCIÓN PROVINCIAL DE COMERCIO DE ALMERÍA	Almería	YES
SPAIN	LABORATORIO AGROALIMENTARIO DE GRANADA	Atarfe	YES
SPAIN	LABORATORIO DE SALUD PÚBLICA DE BADAJOZ	Badajoz	YES
SPAIN	LABORATORY OF BARCELONA PUBLIC HEALTH AGENCY	Barcelona	YES
SPAIN	LABORATORIO AGRARIO REGIONAL DE LA JUNTA DE CASTILLA Y LEÓN	Burgos	YES
SPAIN	LABS & TECHNOLOGICAL SERVICES AGQ, S.L.	Burguillos, Sevilla	YES
SPAIN	LABORATORIO AGROALIMENTARIO DE VALENCIA	Burjassot	YES
SPAIN	LABORATORI AGROALIMENTARI - DAAM	Cabrils	YES
SPAIN	LABORATORIO AGROALIMENTARIO DE EXTREMADURA	Cáceres	YES
SPAIN	LABORATORIO DE SALUD PUBLICA DE CUENCA	Cuenca	YES
SPAIN	LABORATORIO AGROALIMENTARIO Y DE SANIDAD ANIMAL	El Palmar, Murcia	YES
SPAIN	LABORATORIO DE PRODUCCIÓN Y SANIDAD VEGETAL DE HUELVA	Huelva	YES
SPAIN	LABORATORIO DE PRODUCCIÓN Y SANIDAD VEGETAL DE ALMERÍA	La Mojonera, Almería	YES
SPAIN	LABORATORIO REGIONAL DE LA CCAA DE LA RIOJA	Logroño	YES
SPAIN	LABORATORIOS ECOSUR, S.A.	Lorqui, Murcia	YES
SPAIN	CNA (AESAN)	Madrid	YES
SPAIN	LABORATORIO ARBITRAL AGROALIMENTARIO	Madrid	YES
SPAIN	MADRID SALUD - LABORATORIO DE SALUD PUBLICA	Madrid	YES
SPAIN	LABORATORIO DE PRODUCCION Y SANIDAD VEGETAL JAEN. AGAPA	Mengibar, Jaen	YES
SPAIN	LABORATORIO QUÍMICO MICROBIOLÓGICO, S.A	Murcia	YES
SPAIN	LABORATORIO DE SANIDAD VEGETAL	Oviedo	YES
SPAIN	SALUD PÚBLICA DE PALMA	Palma	YES
SPAIN	AINIA	Paterna	YES
SPAIN	LABORATORIO KUDAM, S.L.	Pilar de la Horadada	YES
SPAIN	CENTRO NACIONAL DE TECNOLOGÍA Y SEGURIDAD ALIMENTARIA (CNTA)	San Adrián	YES
SPAIN	DIRECCION TERRITORIAL DE COMERCIO DE VALENCIA(SOIVRE)	Valencia	YES
SPAIN	NASERTIC	Villava	YES

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SPAIN	LABORATORIO AGROAMBIENTAL DE ZARAGOZA	Zaragoza	YES
SWEDEN	EUROFINS FOOD & AGRO TESTING SWEDEN AB	Lidköping	YES
SWEDEN	SWEDISH NATIONAL FOOD AGENCY, SCIENCE DEPARTMENT, CHEMISTRY DIVISION 1	Uppsala	YES
SWITZERLAND	AMT FÜR VERBRAUCHERSCHUTZ AARGAU (CANTONAL OFFICE OF CONSUMER PROTECTION AARGAU)	Aarau	YES
SWITZERLAND	SERVICE DE LA CONSOMMATION ET DES AFFAIRES VETERINAIRES (SCAV)	Geneve	YES
SWITZERLAND	KANTONALES LABOR ZÜRICH	Zurich	YES
THAILAND	CENTRAL LABORATORY (THAILAND) CO., LTD. BANGKOK BRANCH	Bangkok	YES
THE NETHERLANDS	GROEN AGRO CONTROL	Delfgauw	YES
THE NETHERLANDS	LABORATORIUM ZEEUWS-VLAANDEREN BV	Graauw	YES
THE NETHERLANDS	LAB DR A VERWEY AGROGROUP	Rotterdam	YES
THE NETHERLANDS	NVWA - NETHERLANDS FOOD AND CONSUMER PRODUCT SAFETY AUTHORITY	Wageningen	YES
TURKEY	OZEL MSM GIDA KONTROL LABORATUVARI VE DAN. HIZ. TIC. A.S.	Mersin	YES
UNITED KINGDOM	SASA (SCIENCE AND ADVICE FOR SCOTTISH AGRICULTURE)	Edinburgh	YES
UNITED KINGDOM	LABORATORY OF THE GOVERNMENT CHEMIST	Teddington	YES
UNITED KINGDOM	EUROFINS FOOD TESTING UNITED KINGDOM LTD	Wolverhampton	YES
UNITED KINGDOM	THE FOOD AND ENVIRONMENT RESEARCH AGENCY (FERA)	York	YES
URUGUAY	PHARMACOGNOSY& NATURAL PRODUCTS GACT	Montevideo	YES