

# EURL-PROFICIENCY TEST-FV-19, 2017

## Pesticide Residues in Lemon Homogenate

### Final Report

December 2017

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**EURL-EUROPEAN UNION PROFICIENCY TEST 19**  
**FOR THE DETERMINATION OF PESTICIDES IN FRUIT AND VEGETABLES USING**  
**MULTIRESIDUE METHODS**  
**2017**

According to Article 28 of Regulation 396/2005/EC (23<sup>rd</sup> February 2005) of the European Parliament and of the Council, concerning maximum residue levels for pesticides in or on food and feed of plant and animal origin<sup>1</sup>, 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 882/2004<sup>2</sup> lays down the general tasks, duties and requirements for European Union Reference Laboratories (EURLs)<sup>3</sup> for Food, Feed and Animal Health. Among these tasks is the provision for independently-organised comparative tests. European Proficiency Test 19 has been organised by the EURL in Fruit and Vegetables at the University of Almería, Spain<sup>4</sup>.

Participation in European Proficiency Test 19 was mandatory for all National Reference Laboratories (NRLs), as well as all other EU official laboratories, involved in the determination of pesticide residues in fruit and vegetables for the EU multi-annual coordinated control programme or for their own national monitoring programmes. Additionally, laboratories from China, Costa Rica, Indonesia, Kenya, Panama, Peru, Saudi Arabia, Serbia, Singapore, Turkey, Uruguay and Zambia participated in this test.

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

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<sup>1</sup> 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.

<sup>2</sup> 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.

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

<sup>4</sup> Commission Regulation (EC) No 776/2006 of 23<sup>rd</sup> 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 seventy-four laboratories agreed to participate in European Union Proficiency Test 19.

The proficiency test was performed in 2017 using a lemon homogenate. The lemon trees were organically cultivated in a farm in Almería, Spain, and were treated before harvest using commercial formulations applied by spraying with conventional diffusers and treated post-harvest with analytical standards. Seventeen mandatory pesticides and two voluntary<sup>5</sup> ones were used for the treatment. Participating laboratories were also provided with a 'blank' lemon homogenate as well as the treated lemon test item.

The test items, 200 g of lemon homogenate containing pesticide residues, together with 200 g of 'blank' lemon homogenate, were shipped to participants on 13<sup>th</sup> February 2017. The deadline for results submission to the Organiser was 6<sup>th</sup> March 2017. The participants were asked to determine the residue levels of all the pesticides that they detected and to report the concentrations in mg/kg. The participants were provided with two target pesticide lists, one with pesticides that had to be analysed on a compulsory basis, and a second one with pesticides to be analysed voluntarily<sup>5</sup>. The compulsory list contained 192 target pesticides. The pesticide target list is detailed in Annex 1 together with the voluntary target list, which contained 35 pesticides. This list of target pesticides also contained the MRRL for each pesticide fixed at 0.01 mg/kg, except for the following pesticides which have lower MRRLs based on Regulation (EU) No. 396/2005 and EU Directive 2006/125/EC: cadusafos (0.006 mg/kg); dimethoate and omethoate (0.003 mg/kg); ethoprophos (0.008 mg/kg); fipronil (0.004 mg/kg) along with oxydemeton-methyl and demeton-S-methylsulfone (0.006 mg/kg).

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

The robust mean values of the analytical data submitted were used to obtain the assigned (true) values for each of the pesticide residues present. A fit-for-purpose relative target standard deviation (FFP RSD) of 25 % was chosen to calculate the target standard deviations ( $\sigma$ ) as well as the z scores for the individual pesticides.

For the assessment of overall laboratory performance, the Average of the squared z scores ( $AZ^2$ ) has been used. Laboratories that had 'sufficient scope' and were able to analyse at least 90 % of the compulsory pesticides in the target pesticides list, had correctly detected and quantified a sufficiently high percentage of the pesticides present in the Test Item (at least 90 %) and reported no false positives were classified into Category A. Within this category, the laboratories were also

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<sup>5</sup> The voluntary pesticides are not covered by the ISO/IEC 17043.

subclassified as 'good', 'satisfactory' or 'unsatisfactory', in relation to the overall accuracy of the results that they reported.

All the other laboratories were classified into Category B, because they demonstrated 'insufficient scope'. For laboratories in Category B, individual z scores were calculated but the overall accuracy of their results was not assessed. They have been listed in order of the number of pesticides detected (of those used for the statistical evaluation) and the number of acceptable z scores achieved.

Laboratories that did not report results have not been classified into any category and are listed in Annex 2 with the remainder of laboratories that participated in EUPT-FV-19.

## **2. TEST ITEMS**

### **2.1 Preparation of the treated test item**

The lemon trees were organically cultivated in a farm in Almería, Spain, and were treated before harvest using commercial formulations applied by spraying with conventional diffusers. Additionally, they were post-harvest treated using analytical standards. The pesticides used as commercial formulation were carbendazim, chlorantraniliprole, chlorpyrifos, imidacloprid and iprodione. The pesticides spiked as analytical standards were boscalid, chlorfenapyr, diazinon, ethoprophos, famoxadone, fipronil, flubendiamide, fluopyram, lufenuron, omethoate, propamocarb, pyraclostrobin, penflufen and sulfoxaflor.

Before preparation of the test item, the pesticides and target residue levels were selected, following recommendations made by the QCG, which had been appointed specifically for Proficiency Test 19. Eight lemon trees were treated with commercial formulations, which were dissolved in water. Seven days after the application, a representative sample of the treated lemon was collected and analysed to check if the residue levels present were close to the target levels. As the residue levels in the lemon were close to those recommended by the QCG, the entire production (120 kg) was harvested and treated post-harvest with analytical standards dissolved in ethyl acetate. Afterwards, the material was frozen and processed using liquid nitrogen and a mincer. The frozen minced lemon was mixed in a constantly-spinning container until a homogeneous material was obtained. 200 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 lemon trees used for the production of the blank test item were organically grown in the same farm as the test item. Before the treatment of the lemon trees, 100 kg were harvested in order to be used 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 Eurofins-SICA AgriQ S.L., which is accredited under ISO/IEC 17025 by the Spanish accreditation body (ENAC). Ten bottles of the treated test item were randomly chosen from those stored in the freezer and analyses were performed on duplicate portions taken from each bottle. The sequence of analyses was determined using a table of randomly-generated numbers. The injection sequence of the 20 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' lemon 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
Boscalid	0.331	0.0001	0.002	Pass
Carbendazim and benomyl (sum of benomyl and carbendazim expressed as carbendazim)	0.131	0.00001	0.0002	Pass
Chlorantraniliprole	0.188	0.00004	0.0004	Pass
Chlorfenapyr	0.047	0.00001	0.00005	Pass
Chlorpyrifos	0.150	0	0.0004	Pass
Diazinon	0.188	0.00001	0.001	Pass
Ethoprophos	0.043	0.000002	0.00002	Pass
Famoxadone	0.054	0.000002	0.00003	Pass
Fipronil (only parent compound)	0.024	0.000002	0.000011	Pass
Flubendiamide	0.059	0.000006	0.00004	Pass
Fluopyram	0.108	0.00003	0.0002	Pass
Imidacloprid	0.183	0.000023	0.0004	Pass
Iprodione	0.043	0.000003	0.00004	Pass
Lufenuron	0.677	0	0.007	Pass
Omethoate	0.025	0.0000001	0.00001	Pass
Propamocarb	0.133	0.00005	0.0002	Pass
Pyraclostrobin	0.206	0.000001	0.0005	Pass
<b>Voluntary Pesticides</b>				
Penfuflen	0.537	0.001	0.004	Pass
Sulfoxaflor	0.033	0.0000002	0.00001	Pass

$S_s$ : Between-Sampling Standard Deviation  
The voluntary pesticides are not covered by the ISO/IEC 17043

As can be seen from Table 2.1, all the pesticides evaluated in the lemon matrix passed the homogeneity test.



## 2.4 Stability tests

The stability tests were also subcontracted to the laboratory Eurofins-SICA AgriQ S.L., which is accredited under ISO/IEC 17025 by the Spanish accreditation body (ENAC). The tests were performed according to ISO 13528:2015, Annex B [2]. Three bottles that were stored in the freezer at -20°C were chosen randomly and duplicate analyses were performed for each one of them. This procedure was repeated on two different occasions:

- Day 1: shortly before the test item shipment, this took place on February 13<sup>th</sup>, 2017.
- Day 3: shortly after the deadline for reporting results, on May 6<sup>th</sup>, 2017.

A pesticide was 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  $\sigma$  the standard deviation used for proficiency assessment (typically 25 % of the assigned value).

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

Moreover, regarding the stability of the sample arriving not completely frozen, a duplicate analysis of a bottle reproducing the delivery conditions that the samples experienced during 48 hours was performed (Day 2). Laboratories could therefore be sufficiently confident in accepting the treated test item even if it was not completely frozen. Chlorfenapyr and diazinon did not pass the test simulating the shipment conditions. In the case of chlorfenapyr, the concentration in the second day was higher than in the first day. As regards diazinon, it has been used in almost all the EUPs-FV test items, passing in all cases the stability tests. Considering also that the CVs of chlorfenapyr and diazinon were 15.6 and 18.2 %, respectively, the Advisory Group agreed to include them in the evaluation of the laboratories. Results for this 48-hour stability test are indicated in Table 2.3.

Table 2.2 Statistical test for analytical precision and to demonstrate results stability after the interval of time-elapsed between the shipment of the test item and the deadline for reporting of results.

(mg/Kg)	Day 1						Day 3						(M3 - M1)	M3-M1 ≤ 0.3*σ		
	Sample 046_A	Sample 046_B	Sample 186_A	Sample 186_B	Sample 212_A	Sample 212_B	Mean 1	Sample 127_A	Sample 127_B	Sample 193_A	Sample 193_B	Sample 250_A			Sample 250_B	Mean3
Boscalid	0.340	0.340	0.380	0.380	0.320	0.350	0.352	0.360	0.340	0.350	0.350	0.350	0.370	0.353	0.002	Pass
Carbendazim and benomyl (sum of benomyl and carbendazim expressed as carbendazim)	0.140	0.140	0.150	0.150	0.140	0.140	0.143	0.150	0.140	0.140	0.140	0.140	0.140	0.142	-0.002	Pass
Chlorantraniliprole	0.180	0.190	0.190	0.190	0.180	0.180	0.185	0.190	0.180	0.190	0.190	0.180	0.190	0.187	0.002	Pass
Chlorfenapyr	0.050	0.028	0.038	0.040	0.047	0.047	0.042	0.044	0.039	0.042	0.044	0.041	0.042	0.042	0.000	Pass
Chlorpyrifos	0.160	0.110	0.140	0.160	0.160	0.120	0.142	0.140	0.140	0.150	0.150	0.150	0.150	0.147	0.005	Pass
Diazinon	0.170	0.180	0.170	0.200	0.170	0.180	0.178	0.180	0.160	0.180	0.150	0.150	0.180	0.167	-0.012	Pass

(mg/Kg)	Day 1							Day 3							(M3 - M1)	M3-M1 ≤ 0.3*σ
	Sample 046_A	Sample 046_B	Sample 186_A	Sample 186_B	Sample 212_A	Sample 212_B	Mean 1	Sample 127_A	Sample 127_B	Sample 193_A	Sample 193_B	Sample 250_A	Sample 250_B	Mean3		
Ethoprophos	0.042	0.044	0.045	0.046	0.043	0.042	0.044	0.043	0.041	0.042	0.041	0.041	0.042	0.042	-0.002	Pass
Famoxadone	0.056	0.056	0.058	0.056	0.052	0.047	0.054	0.051	0.051	0.050	0.051	0.052	0.052	0.051	-0.003	Pass
Fipronil (only parent compound)	0.019	0.013	0.017	0.019	0.019	0.013	0.017	0.018	0.017	0.018	0.018	0.017	0.018	0.018	0.001	Pass
Flubendiamide	0.060	0.064	0.064	0.064	0.059	0.056	0.061	0.064	0.058	0.057	0.059	0.059	0.058	0.059	-0.002	Pass
Fluopyram	0.120	0.087	0.110	0.130	0.120	0.093	0.110	0.110	0.100	0.110	0.110	0.110	0.100	0.107	-0.003	Pass
Imidacloprid	0.160	0.160	0.170	0.180	0.170	0.160	0.167	0.170	0.170	0.170	0.170	0.160	0.170	0.168	0.002	Pass
Iprodione	0.047	0.033	0.043	0.050	0.050	0.046	0.045	0.043	0.041	0.043	0.047	0.043	0.044	0.044	-0.001	Pass
Lufenuron	0.660	0.760	0.650	0.750	0.630	0.610	0.677	0.760	0.740	0.670	0.670	0.690	0.700	0.705	0.028	Pass
Omethoate	0.024	0.025	0.024	0.025	0.024	0.024	0.024	0.026	0.026	0.024	0.025	0.025	0.025	0.025	0.001	Pass
Propamocarb	0.140	0.140	0.140	0.140	0.130	0.130	0.137	0.140	0.130	0.140	0.140	0.140	0.130	0.137	0.000	Pass
Pyraclostrobin	0.190	0.200	0.200	0.210	0.190	0.180	0.195	0.200	0.200	0.200	0.200	0.200	0.200	0.200	0.005	Pass
<b>Voluntary Pesticides</b>																
Penfufen	0.525	0.538	0.517	0.502	0.518	0.527	0.521	0.546	0.497	0.514	0.556	0.502	0.540	0.526	0.005	Pass
Sulfoxaflor	0.029	0.029	0.030	0.031	0.030	0.029	0.030	0.033	0.031	0.030	0.032	0.031	0.031	0.031	0.001	Pass

The voluntary pesticides are not covered by the ISO/IEC 17043

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

(mg/Kg)	Day 1							Day 2							(M2 - M1)	M2-M1 ≤ 0.3*σ
	Sample 046_A	Sample 046_B	Sample 186_A	Sample 186_B	Sample 212_A	Sample 212_B	Mean 1	Sample 082_A	Sample 082_B	Sample 184_A	Sample 184_B	Sample 258_A	Sample 258_B	Mean2		
Boscalid	0.340	0.340	0.380	0.380	0.320	0.350	0.352	0.380	0.300	0.320	0.360	0.380	0.300	0.340	-0.012	Pass
Carbendazim and benomyl (sum of benomyl and carbendazim expressed as carbendazim)	0.140	0.140	0.150	0.150	0.140	0.140	0.143	0.140	0.140	0.150	0.150	0.140	0.140	0.143	0.000	Pass
Chlorantraniliprole	0.180	0.190	0.190	0.190	0.180	0.180	0.185	0.180	0.180	0.190	0.190	0.180	0.180	0.183	-0.002	Pass
Chlorfenapyr	0.050	0.028	0.038	0.040	0.047	0.047	0.042	0.041	0.049	0.046	0.054	0.059	0.042	0.049	0.007	Not Pass
Chlorpyrifos	0.160	0.110	0.140	0.160	0.160	0.120	0.142	0.140	0.140	0.120	0.160	0.170	0.130	0.143	0.002	Pass
Diazinon	0.170	0.180	0.170	0.200	0.170	0.180	0.178	0.170	0.140	0.150	0.170	0.190	0.140	0.160	-0.018	Not Pass
Ethoprophos	0.042	0.044	0.045	0.046	0.043	0.042	0.044	0.042	0.041	0.042	0.045	0.044	0.043	0.043	-0.001	Pass
Famoxadone	0.056	0.056	0.058	0.056	0.052	0.047	0.054	0.053	0.049	0.051	0.054	0.051	0.053	0.052	-0.002	Pass
Fipronil (only parent compound)	0.019	0.013	0.017	0.019	0.019	0.013	0.017	0.017	0.018	0.019	0.017	0.019	0.014	0.017	0.001	Pass
Flubendiamide	0.060	0.064	0.064	0.064	0.059	0.056	0.061	0.056	0.054	0.058	0.059	0.059	0.059	0.058	-0.004	Pass
Fluopyram	0.120	0.087	0.110	0.130	0.120	0.093	0.110	0.110	0.110	0.095	0.110	0.110	0.087	0.104	-0.006	Pass
Imidacloprid	0.160	0.160	0.170	0.180	0.170	0.160	0.167	0.160	0.170	0.170	0.180	0.170	0.170	0.170	0.003	Pass
Iprodione	0.047	0.033	0.043	0.050	0.050	0.046	0.045	0.047	0.045	0.039	0.048	0.076	0.036	0.049	0.004	Pass
Lufenuron	0.660	0.760	0.650	0.750	0.630	0.610	0.667	0.610	0.720	0.660	0.630	0.680	0.640	0.657	-0.020	Pass
Omethoate	0.024	0.025	0.024	0.025	0.024	0.024	0.024	0.023	0.022	0.024	0.025	0.024	0.025	0.024	-0.001	Pass
Propamocarb	0.140	0.140	0.140	0.140	0.130	0.130	0.137	0.140	0.140	0.130	0.140	0.140	0.140	0.138	0.002	Pass
Pyraclostrobin	0.190	0.200	0.200	0.210	0.190	0.180	0.195	0.200	0.210	0.200	0.210	0.200	0.200	0.203	0.008	Pass

(mg/Kg)	Day 1							Day 2							(M2 – M1)	M2-M1 ≤ 0.3*σ
	Sample 046_A	Sample 046_B	Sample 186_A	Sample 186_B	Sample 212_A	Sample 212_B	Mean 1	Sample 082_A	Sample 082_B	Sample 184_A	Sample 184_B	Sample 258_A	Sample 258_B	Mean2		
<b>Voluntary Pesticides</b>																
Penflufen	0.525	0.538	0.517	0.502	0.518	0.527	0.521	0.551	0.572	0.580	0.531	0.566	0.546	0.558	0.036	Pass
Sulfoxaflor	0.029	0.029	0.030	0.031	0.030	0.029	0.030	0.029	0.028	0.031	0.032	0.031	0.029	0.030	0.000	Pass

The voluntary pesticides are not covered by the ISO/IEC 17043

## 2.5 Distribution of test items and protocol to participants

One bottle of frozen treated test item and one bottle of frozen 'blank' material were shipped to each participant in boxes containing dry ice. The test items were sent out on 13<sup>th</sup> February 2017. Ninety-nine percent of the shipments to EU/EFTA countries arrived within the first 48 hours.

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 19. The Target Pesticide List and the Minimum Required Reporting Levels (MRRLs), as established by the Organiser, were uploaded onto the EURL-FV open website at least three months before the shipment of the test item to allow laboratories sufficient time to purchase standards and to validate their methods.

## 3. STATISTICAL METHODS

### 3.1 False positives and negatives

#### 3.1.1 False positives

These are results of pesticides from the Target Pesticides List, that are reported at, or above, their respective MRRLs although they were: (i) not detected by the Organiser, even after repeated analyses, and/or (ii) not detected by the overwhelming majority (e.g. > 95 %) of the participating laboratories that had targeted the specific pesticides. In certain instances, case-by-case decisions by the EUPT-Panel may be necessary.

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

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

### 3.1.2 False negatives

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

In cases of the assigned value being less than a factor of 3 times the MRRL, false negatives will typically not be assigned. The EUPT-Panel may decide to take case-by-case decisions in this respect after considering all relevant factors such as the result distribution and the reporting limits of the affected labs. z scores have been calculated for all evaluated pesticides that were detected and reported at levels at, or above, the MRRL. They have also been calculated for false negatives. However, these z scores were not taken into account in assessing the 90 %, or more, of pesticides present in the sample needed to be classified into Category A.

### **3.2 Estimation of the assigned values ( $x_{pt}$ )**

In order to minimise the influence of out-lying results on the statistical evaluation, the assigned value (= consensus concentration) was estimated using robust statistics as described in ISO 13528:2015, taking into account the results reported by EU and EFTA countries laboratories only. Individual results without any numerical values reported, such as detected (D), were not considered. The spread of results for each pesticide was tested for multimodality. In special justifiable cases, the EUPT-Panel may decide to eliminate certain results traceably associated with gross errors or to use only the results of a subgroup consisting of laboratories that have repeatedly demonstrated good performance for the specific compound in the past.

Taking into account the normative for robust analysis in ISO 13528:2015, the uncertainty was accompanying the assigned value for each pesticide was calculated according to the following equation:

$$u(x_{pt}) = 1.25 \frac{s^*}{\sqrt{p}}$$

Where:

- $u(x_{pt})$  is the uncertainty in mg/Kg.
- $s^*$  is the robust standard deviation of the results.
- $p$  is the total number of results.

### 3.3 Fixed target standard deviations

Based on the experience gained from previous EU proficiency tests and recommendations from the EURL Advisory Group, a fixed relative standard deviation (FFP-RSD) of 25 % was chosen [3]. This is in line with the internationally-accepted target Measurement Uncertainty of 50 % for multiresidue analysis of pesticides [4], which is derived from, and linked to, the EUPTs. The same target RSD has been applied to all the pesticides, independent of concentration. For informative purposes the robust relative standard deviation (CVs\*) is calculated according to ISO 13528:2015; Chapter 7.7 (Consensus value from participant results) following Algorithm A in Annex C, and it can be compared to the FFP-RSD in Table 4.4.

### 3.4 z scores

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

$$z_i = \frac{(x_i - x_{pt})}{\sigma_{pt}}$$

Where:

- $x_i$  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.
- $x_{pt}$  is the assigned value.
- $\sigma_{pt}$  is the target standard deviation (the FFP-RSD of 25 % multiplied by the assigned value).

z score classification is as follows:

$ z  \leq 2.0$	Acceptable
$2.0 <  z  < 3.0$	Questionable
$ z  \geq 3.0$	Unacceptable

- Any z score values of  $|z| > 5$  have been reported as '>5' and a value of '5' has been used to calculate combined z scores.
- No z score calculations have been performed for false positive results.
- For false negative results, the MRRL (or RL) has been used to calculate the z score. These z scores have also been included in the graphical representation, and are marked with an asterisk.

### 3.5 Combined z scores

In order to evaluate each laboratory's overall performance according to the quality of its results and its scope, two classifications - Category A and B - were used. To be classified into Category A, laboratories had to be able to analyse at least 90 % of the compulsory pesticides in the target pesticides list, 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 (for the 90 % criterion the number of pesticides needed to be correctly analysed to have sufficient scope will be calculated by multiplying the number of compulsory pesticides from the Target Pesticides List by 0.9 and rounded to the nearest full number with 0.5 decimals being rounded downwards). If these three requirements were met, then the combined z scores were calculated as the 'Average of the Squared z scores' ( $AZ^2$ ) [5].

### 3.5.1 The Average of the Squared z scores ( $AZ^2$ )

The 'Average of the Squared z scores' was introduced for the first time in EUPT-FV-12. The  $AZ^2$  is calculated as follows:

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

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

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

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

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

Laboratories that did not detect and quantify sufficient pesticides, that were not able to analyse at least 90 % of the compulsory 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 seventy-four laboratories agreed to participate in this proficiency test. Four did not submit results. The total number of laboratories submitting results was 170. 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 China, Costa Rica, Indonesia, Kenya,

Panama, Peru, Saudi Arabia, Serbia, Singapore, Turkey and Uruguay have not been included. This last group totals 17 laboratories that reported results.

Seventeen pesticides from the compulsory pesticide target list and two voluntary pesticides<sup>6</sup> were used to treat the sample. The assigned values of all the pesticides present in the test item were higher than a factor of four times the MRRL, and therefore, the Scientific Committee considered that they should be evaluated. A summary of the reported results for the pesticides evaluated can be seen below in Table 4.1.

Table 4.1 Summary of Reported Results

Pesticides	No. of Reported Results	No. of False Negative Results	No. of Not Analysed Results	Percentage of Reported Results* (out of 153)
Boscalid	149	0	4	97
Carbendazim and benomyl (sum of benomyl and carbendazim expressed as carbendazim)	134	1	18	88
Chlorantraniliprole	126	1	26	82
Chlorfenapyr	127	2	24	83
Chlorpyrifos	151	1	1	99
Diazinon	153	0	0	100
Ethoprophos	141	1	11	92
Famoxadone	117	5	31	76
Fipronil (only parent compound)	129	3	21	84
Flubendiamide	101	1	51	66
Fluopyram	117	1	35	76
Imidacloprid	137	0	16	90
Iprodione	140	2	11	92
Lufenuron	129	0	24	84
Omethoate	133	6	14	87
Propamocarb	128	5	20	84
Pyraclostrobin	141	0	12	92
<b>Voluntary Pesticides</b>				
Penflufen	43	2	108	28
Sulfoxaflor	43	3	107	28

\* The percentage of Reported Results comes from 153 laboratories. It does not take into account the seventeen laboratories from China, Costa Rica, Indonesia, Kenya, Panama, Peru, Saudi Arabia, Serbia, Singapore, Turkey and Uruguay.

The voluntary pesticides are not covered by the ISO/IEC 17043

<sup>6</sup> The voluntary pesticides are not covered by the ISO/IEC 17043

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

Seven laboratories (including non-EU countries) reported results for additional pesticides that were not present in the test item. These pesticides and the residue levels reported are presented in Table 4.2 together with the MRRLs and reporting levels (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. If the concentrations reported were below the MRRLs, or if the pesticides did not appear in the pesticide list included in Annex 1, then they were not considered to be false positives.

One out of those seven laboratories reporting a false positive result was not classified into Category A despite achieving sufficient scope. Fenpropathrin was reported by two EU/EFTA laboratories at the same concentration level than chlorpyrifos by LC-MS/MS. Both pesticides have common transitions and very similar retention times.

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)
Lab080	Thiamethoxam	0.019	GC-MS/MS (IT)	0.01	0.01
Lab107*	Diphenylamine	0.021	GC-MS/MS (QQQ)	0.01	0.01
Lab113*	Fenpropathrin	0.031	LC-MS/MS (QQQ)	0.01	0.01
Lab135	Diflubenzuron	0.241	GC-MS/MS (QQQ)	0.01	0.01
Lab139	Fenpropathrin	0.16	Both GC and LC-MS/MS (QQQ)	0.01	0.01
Lab148	Fenpropimorph	0.0476	GC-MS/MS (QQQ)	0.02	0.01
Lab159	Fenpropathrin	0.117	LC-MS/MS (QQQ)	0.01	0.01

\*Non-EU/EFTA laboratories

#### 4.1.2 False negatives

Table 4.3 summarises the results from laboratories (including non-EU 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	Boscollid	Carbendazim	Chlorantraniliprole	Chlofenapyr	Chlorpyrifos	Diazinon	Ethoprophos	Famoxadone	Fipronil (only parent compound)	Flubendiamide	Fluopyram	Imidacloprid	Iprodione	Lufenuron	Omethoate	Propamocarb	Pyraclostrobin
010													ND				
018																ND	
024		ND	ND	ND				ND								ND	
034															ND		
041				ND													
052					ND					ND							ND
069											ND						
070								ND									
084									ND								
085								ND									
103*																	ND
104*								ND									
109*			ND										ND				
111*					ND				ND								
115*				ND						ND							
117*									ND								
121															ND		
122								ND									
126															ND	ND	
128															ND		
136															ND		
144									ND								
156															ND		
159																ND	
160													ND				
165							ND										
174								ND	ND								
	<b>Voluntary Pesticides</b>																
	<b>Penflufen</b>								<b>Sulfoxaflor</b>								
058	ND								ND								
069	ND								ND								
097									ND								

\*Non-EU/EFTA laboratories

The voluntary pesticides are not covered by the ISO/IEC 17043

#### 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  $0.75 \cdot \sigma$  ( $\sigma$  is the target standard deviation (the FFP-RSD of 25 % multiplied by the assigned value) after removing outliers, if necessary. The histograms of both the compulsory and voluntary pesticides present in the test item are presented in Appendix 2.

## 4.2 Assigned values and target standard deviations

The assigned values are based on the robust mean values calculated using all the results reported by laboratories from EU and EFTA countries. The assigned values for the seventeen compulsory and the two voluntary pesticides and their uncertainties are presented in Table 4.4.

The target standard deviation was calculated using a fixed FFP-RSD value of 25 %. For comparison, a robust standard deviation (CV\*) was also calculated for informative purposes, employing also this value for the calculation of the uncertainty. These RSDs can be seen in Table 4.4.

Table 4.4 Robust mean values, uncertainty and % RSDs for all pesticides evaluated.

Pesticides	MRRL (mg/kg)	Robust mean (mg/kg)	Uncertainty (mg/kg)	Number of results (n)	FFP-RSD (%)	CV* (%)
Boscalid	0.01	0.378	0.0068	149	25.0	17.5
Carbendazim and benomyl (sum of benomyl and carbendazim expressed as carbendazim)	0.01	0.134	0.0015	134	25.0	27.0
Chlorantraniliprole	0.01	0.178	0.0037	126	25.0	18.6
Chlorfenapyr	0.01	0.047	0.0008	127	25.0	15.6
Chlorpyrifos	0.01	0.131	0.0024	151	25.0	17.8
Diazinon	0.01	0.170	0.0031	153	25.0	18.2
Ethoprophos	0.008	0.038	0.0007	141	25.0	16.4
Famoxadone	0.01	0.044	0.0009	117	25.0	17.0
Fipronil (only parent compound)	0.004	0.019	0.0004	129	25.0	20.8
Flubendiamide	0.01	0.058	0.0011	101	25.0	15.4
Fluopyram	0.01	0.128	0.0023	117	25.0	15.8
Imidacloprid	0.01	0.158	0.0029	137	25.0	17.0
Iprodione	0.01	0.051	0.0012	140	25.0	22.2
Lufenuron	0.01	0.644	0.0145	129	25.0	20.5
Omethoate	0.003	0.021	0.0006	133	25.0	28.1
Propamocarb	0.01	0.123	0.0028	128	25.0	20.9
Pyraclostrobin	0.01	0.185	0.0030	141	25.0	15.5
<b>Voluntary Pesticides</b>						
Penflufen	0.01	0.512	0.0143	43	25.0	14.6
Sulfoxaflor	0.01	0.032	0.0010	43	25.0	16.4

The voluntary pesticides are not covered by the ISO/IEC 17043

## 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 evaluated.

In Appendix 3, the individual z scores are presented for each laboratory, together with the assigned values for each pesticide. The z scores of laboratories from non-EU countries 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 (only EU/EFTA participants)

Pesticides	Acceptable (%)	Questionable (%)	Unacceptable (%)
Boscalid	97.3	1.3	1.3
Carbendazim and benomyl (sum of benomyl and carbendazim expressed as carbendazim)	86.7	5.9	7.4
Chlorantraniliprole	92.1	3.9	3.9
Chlorfenapyr	97.7	0.8	1.6
Chlorpyrifos	98.7	0.7	0.7
Diazinon	94.1	5.2	0.7
Ethoprophos	97.9	0.0	2.1
Famoxadone	94.3	0.8	4.9
Fipronil (only parent compound)	92.4	3.0	4.5
Flubendiamide	96.1	2.9	1.0
Fluopyram	95.8	2.5	1.7
Imidacloprid	95.6	1.5	2.9
Iprodione	91.5	5.6	2.8
Lufenuron	96.9	2.3	0.8
Omethoate	85.6	4.3	10.1
Propamocarb	90.2	3.8	6.0
Pyraclostrobin	100.0	0.0	0.0
<b>Voluntary Pesticides</b>			
Penflufen	93.3	0.0	6.7
Sulfoxaflor	89.1	10.9	0.0

The voluntary pesticides are not covered by the ISO/IEC 17043

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 of EU/EFTA laboratories are presented. No z scores have been calculated for false positive results. z scores for false negative results have been included on the chart and are indicated by an asterisk. The charts have been constructed using different colour bars according to the determination technique used for each particular pesticide.

#### 4.3.2 Combined z scores

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

The table in Appendix 5 shows the values of individual z scores for each compulsory pesticide and the combined 'Average of the Squared z scores' ( $AZ^2$ ) for all laboratories in Category A (including non-EU countries), which were those laboratories that were able to analyse at least 90 % of the compulsory pesticides in the target pesticides list (11), to detect and quantify at least 90 % of the pesticides present in the Test Item (171), and that do not report any false positive result. A graphical representation of those results for the EU/EFTA laboratories can be found in Appendix 6.

One hundred and one of the 153 EU and EFTA laboratories that submitted results were classified into Category A (66 %).

From the  $AZ^2$ , 92 % were classed as 'good', 5 % as 'satisfactory' and 3 % as 'unsatisfactory'.

Of the 52 EU and EFTA laboratories in Category B, one would have been in Category A if they had not reported a false positive result.

Table 4.6 shows all the laboratories in Category A, the number of pesticides reported, the percentage of pesticides analysed from the compulsory target list, 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 all the laboratories in Category B, the number and percentage of results reported, the percentage of pesticides analysed from the compulsory target list 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 EU/EFTA 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-19, out of 153 laboratories (EU and EFTA), 101 were in Category A with the following classes: 3 'unsatisfactory', 5 'satisfactory' and 93 'good'.
- For EUPT-FV-18, out of 171 laboratories (EU and EFTA), 98 were in Category A with the following classes: 3 'unsatisfactory', 9 'satisfactory' and 86 'good'.
- For EUPT-FV-17, out of 165 laboratories (EU and EFTA), 113 were in Category A with the following classes: 15 'unsatisfactory', 8 'satisfactory' and 90 'good'.
- For EUPT-FV-16, out of 169 laboratories (EU and EFTA), 100 were in Category A with the following classes: 2 'unsatisfactory', 5 'satisfactory' and 93 'good'.

Table 4.6 Performance and Classification of laboratories in Category A using the AZ<sup>2</sup> formula

Lab Code	No. of pesticides detected (max.17)	% of pesticides analysed from target list	AZ <sup>2</sup>	Classification
Lab002	17	100	1.0	Good
Lab004	17	99	0.4	Good
Lab005	17	99	1.4	Good
Lab006	17	100	0.3	Good
Lab007	17	98	0.2	Good
Lab009	17	100	0.2	Good
Lab010*	17	96	2.4	Satisfactory
Lab011	17	100	0.2	Good
Lab015	17	100	0.1	Good
Lab016	17	99	0.1	Good
Lab017	17	100	0.8	Good
Lab018*	17	97	1.8	Good
Lab019	17	99	0.9	Good
Lab020	17	99	0.3	Good
Lab021	17	100	0.3	Good
Lab022	17	100	1.7	Good
Lab023	17	99	0.6	Good
Lab025	17	98	1.1	Good
Lab026	17	98	0.3	Good
Lab027	17	100	0.3	Good
Lab029	17	100	0.5	Good
Lab030	17	99	0.5	Good
Lab033	17	99	0.2	Good
Lab035	17	98	1.8	Good
Lab036	16	99	0.3	Good
Lab037	16	99	1.2	Good
Lab038	16	93	0.2	Good
Lab039	17	95	0.9	Good
Lab040	15	96	3.4	Unsatisfactory
Lab041*	17	100	1.2	Good
Lab042	17	100	0.8	Good
Lab043	17	98	1.1	Good
Lab046	17	100	0.2	Good
Lab049	17	98	1.3	Good
Lab050	17	90	0.7	Good
Lab051	17	100	0.6	Good
Lab053	17	100	0.8	Good
Lab054	17	99	0.6	Good
Lab055	15	95	0.7	Good
Lab056	17	96	0.4	Good
Lab057	17	98	1.3	Good
Lab058	17	100	2.6	Satisfactory
Lab059	17	100	0.2	Good

Lab Code	No. of pesticides detected (max.17)	% of pesticides analysed from target list	AZ <sup>2</sup>	Classification
Lab061	17	96	0.7	Good
Lab062	17	100	2.1	Satisfactory
Lab063	17	97	0.2	Good
Lab065	17	100	0.4	Good
Lab066	17	100	0.2	Good
Lab067	17	100	0.4	Good
Lab068	17	100	1.7	Good
Lab069*	17	100	1.3	Good
Lab070*	17	100	1.5	Good
Lab071	16	96	0.5	Good
Lab072	17	99	0.4	Good
Lab073	17	100	0.2	Good
Lab075	17	100	0.3	Good
Lab076	17	96	0.1	Good
Lab078	17	96	0.5	Good
Lab081	16	99	4.4	Unsatisfactory
Lab082	17	96	0.2	Good
Lab083	17	98	0.3	Good
Lab084*	17	100	1.7	Good
Lab086	16	95	1.6	Good
Lab087	17	100	0.2	Good
Lab089	16	98	2.0	Good
Lab090	17	99	0.3	Good
Lab091	17	90	0.6	Good
Lab094	17	100	0.5	Good
Lab095	16	96	4.5	Unsatisfactory
Lab096	16	97	0.4	Good
Lab097	17	98	0.6	Good
Lab098	17	100	0.6	Good
Lab099	17	99	1.1	Good
Lab120	17	100	0.1	Good
Lab122*	17	95	1.1	Good
Lab123	16	91	0.7	Good
Lab124	17	100	0.3	Good
Lab129	17	100	0.4	Good
Lab130	17	100	0.9	Good
Lab132	17	100	0.3	Good
Lab133	17	100	0.1	Good
Lab136*	16	98	1.4	Good
Lab138	17	100	0.5	Good
Lab140	17	100	0.2	Good
Lab142	17	100	0.5	Good
Lab145	17	100	0.2	Good
Lab146	17	93	2.4	Satisfactory

Lab Code	No. of pesticides detected (max.17)	% of pesticides analysed from target list	AZ <sup>2</sup>	Classification
Lab147	17	92	1.4	Good
Lab149	17	100	0.1	Good
Lab150	17	99	0.4	Good
Lab151	17	96	0.6	Good
Lab154	17	99	0.6	Good
Lab160*	16	93	2.3	Satisfactory
Lab161	17	99	0.5	Good
Lab165*	17	98	1.1	Good
Lab167	16	98	0.2	Good
Lab169	17	98	0.2	Good
Lab171	17	100	1.0	Good
Lab172	16	97	1.0	Good
Lab173	17	100	0.2	Good
Lab179	17	97	0.3	Good

\* 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 (17)	No. of pesticides detected	% of pesticides analysed from target list	No. of total z scores	No. of acceptable z scores (z score ≤ 2)
Lab001	53	9	44	9	6
Lab003	65	11	84	11	10
Lab008	88	15	78	15	12
Lab012	76	13	66	13	13
Lab013	41	7	57	7	2
Lab014	82	14	71	14	12
Lab024*	65	6	71	11	6
Lab028	35	6	38	6	6
Lab031	65	11	65	11	11
Lab032	76	13	88	13	13
Lab034*	65	10	85	11	10
Lab044	82	14	91	14	13
Lab045	59	10	72	10	10
Lab047	82	14	89	14	14
Lab048	65	11	72	11	11
Lab052*	76	10	72	13	7
Lab060	76	13	62	13	11
Lab064	65	11	66	11	11
Lab077	94	16	89	16	16
Lab079	76	13	91	13	12
Lab080+	88	15	78	15	15
Lab085*	100	16	89	17	9
Lab088	76	13	84	13	13

Lab Code	% No. of pesticides detected / No. of pesticides evaluated (17)	No. of pesticides detected	% of pesticides analysed from target list	No. of total z scores	No. of acceptable z scores (z score ≤ 2)
Lab092	47	8	43	8	8
Lab093	82	14	68	14	14
Lab119	47	8	57	8	8
Lab121*	88	14	67	15	14
Lab126*	71	10	93	12	10
Lab127	82	14	64	14	13
Lab128*	53	8	67	9	8
Lab131	53	9	47	9	8
Lab135+	88	15	85	15	14
Lab139+	88	15	99	15	11
Lab141	82	14	80	14	13
Lab143	76	13	71	13	13
Lab144*	65	10	75	11	10
Lab148+	82	14	85	14	10
Lab152	71	12	66	12	12
Lab153	76	13	68	13	13
Lab155	71	12	67	12	12
Lab156*	29	4	39	5	4
Lab158	47	8	60	8	8
Lab159*+	76	12	77	13	10
Lab162	29	5	31	5	3
Lab163	35	6	31	6	6
Lab164	29	5	55	5	5
Lab166	53	9	65	9	9
Lab170	29	5	44	5	4
Lab174*	71	10	67	12	7
Lab175	82	14	95	14	14
Lab176	47	8	35	8	8
Lab177	24	4	34	4	4

\* Laboratories reporting a false negative result.

+ Laboratories reporting a false positive result.

The voluntary pesticides are not covered by the ISO/IEC 17043



## 5. CONCLUSIONS

One hundred and seventy-four laboratories agreed to participate in EUPT-FV-19. Four did not submit results for the analysis of the lemon homogenate test item. From the remaining 170 laboratories that submitted results, 17 did not belong to EU nor EFTA countries, so their results were not considered for the estimation of the assigned value.

Seventeen mandatory and two voluntary pesticides were evaluated in EUPT-FV-19, based on the analysis of lemon homogenate.

Of a total number of 2601 possible determinations from EU/EFTA laboratories (153 laboratories by 17 evaluated pesticides), 86.6 % results were reported, 12.3 % were not analysed and 1.1 % were not detected (false negative results). The false positive rate was 0.2 %.

The total number of z scores of laboratories from EU/EFTA countries was 2373, with 94.2 % of them acceptable, 2.7 % questionable and 3.1 % unacceptable.

65 % of the EU and EFTA laboratories that submitted results were classified into Category A. Of them, 92 % were classed as 'good', 5 % as 'satisfactory' and 3 % as 'unsatisfactory'.

The robust standard deviation (CV\*) was in all cases below 28.1 %, with an average value of 18.7 % for the 17 pesticides evaluated.

Participation in this year's European Proficiency Test 19 involved at least one laboratory from each Member State. Additionally, laboratories from Iceland, Norway and Switzerland participated as EFTA 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. Non-European laboratories from China, Costa Rica, Indonesia, Kenya, Panama, Peru, Saudi Arabia, Serbia, Singapore, Turkey, Uruguay and Zambia participated in EUPT-FV-19. These Non-EU laboratories, however, are official laboratories in their own countries.

## 6. REFERENCES

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## 7. ACKNOWLEDGEMENTS

The Organiser is most grateful to the European Commission for funding this European Proficiency Test FV-19.

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

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

**APPENDIX 1. Homogeneity data.**

Boscalid (mg/kg)		Carbendazim (mg/kg)		Chlorantraniliprole (mg/kg)		Chlorfenapyr (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.340	0.340	0.130	0.130	0.180	0.180	0.056	0.051
0.310	0.310	0.130	0.130	0.190	0.190	0.036	0.045
0.350	0.310	0.130	0.130	0.190	0.190	0.048	0.046
0.350	0.340	0.130	0.120	0.180	0.170	0.046	0.051
0.330	0.320	0.130	0.130	0.190	0.190	0.047	0.056
0.350	0.330	0.140	0.140	0.190	0.200	0.041	0.038
0.310	0.310	0.130	0.130	0.190	0.190	0.044	0.054
0.310	0.300	0.130	0.130	0.190	0.180	0.047	0.035
0.350	0.340	0.130	0.130	0.180	0.190	0.048	0.043
0.320	0.390	0.140	0.130	0.200	0.200	0.046	0.054

Chlorpyrifos (mg/kg)		Diazinon (mg/kg)		Ethoprophos (mg/kg)		Famoxadone (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.170	0.150	0.200	0.190	0.042	0.042	0.053	0.051
0.130	0.150	0.170	0.180	0.042	0.041	0.053	0.054
0.150	0.140	0.190	0.180	0.043	0.042	0.050	0.055
0.150	0.160	0.190	0.190	0.042	0.042	0.052	0.049
0.160	0.160	0.180	0.170	0.045	0.046	0.056	0.056
0.140	0.130	0.200	0.180	0.044	0.045	0.052	0.057
0.150	0.150	0.180	0.180	0.045	0.045	0.055	0.054
0.160	0.120	0.200	0.180	0.043	0.042	0.052	0.052
0.170	0.150	0.210	0.200	0.043	0.043	0.054	0.055
0.140	0.170	0.170	0.210	0.044	0.045	0.057	0.055

Fipronil (mg/kg)		Flubendiamide (mg/kg)		Fluopyram (mg/kg)		Imidacloprid (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.025	0.025	0.058	0.057	0.120	0.110	0.180	0.180
0.020	0.022	0.058	0.057	0.088	0.100	0.180	0.180
0.022	0.025	0.060	0.059	0.110	0.100	0.180	0.180
0.023	0.028	0.055	0.055	0.100	0.120	0.180	0.180
0.025	0.026	0.063	0.061	0.120	0.110	0.180	0.180
0.024	0.021	0.063	0.062	0.099	0.094	0.190	0.190
0.022	0.024	0.059	0.061	0.110	0.110	0.190	0.190
0.024	0.019	0.056	0.058	0.110	0.079	0.180	0.180
0.024	0.020	0.058	0.059	0.120	0.110	0.180	0.180
0.028	0.026	0.063	0.062	0.110	0.130	0.190	0.190

The sample numbers used for this test were: 1, 23, 44, 90, 116, 154, 169, 185, 202 and 229.

**APPENDIX 1. Homogeneity data.**

Iprodione (mg/kg)		Lufenuron (mg/kg)		Omethoate (mg/kg)		Propamocarb (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.049	0.043	0.740	0.660	0.025	0.025	0.120	0.120
0.038	0.041	0.610	0.680	0.025	0.024	0.130	0.130
0.049	0.044	0.670	0.710	0.024	0.024	0.140	0.150
0.040	0.043	0.720	0.710	0.024	0.025	0.130	0.120
0.044	0.048	0.680	0.620	0.025	0.025	0.130	0.130
0.040	0.041	0.710	0.650	0.026	0.025	0.140	0.140
0.040	0.041	0.610	0.680	0.025	0.025	0.130	0.130
0.043	0.033	0.650	0.730	0.025	0.025	0.140	0.130
0.046	0.039	0.670	0.660	0.024	0.025	0.130	0.130
0.043	0.055	0.710	0.660	0.025	0.025	0.140	0.140

Pyraclostrobin (mg/kg)	
Replicate 1	Replicate 2
0.200	0.200
0.200	0.210
0.210	0.200
0.200	0.200
0.210	0.200
0.210	0.210
0.210	0.210
0.210	0.200
0.210	0.200
0.210	0.210

**Voluntary Pesticides**

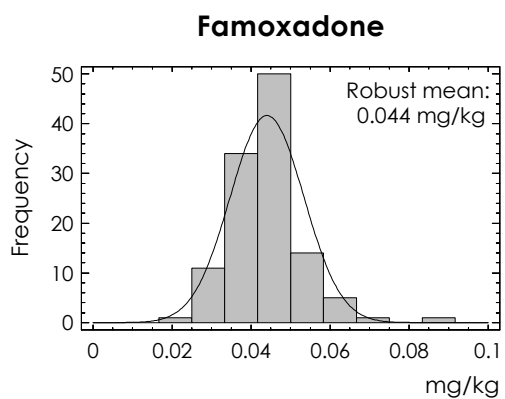
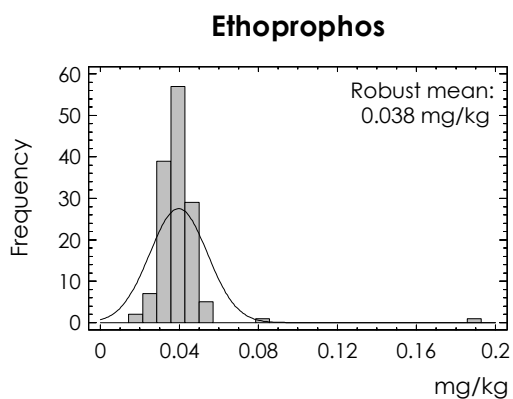
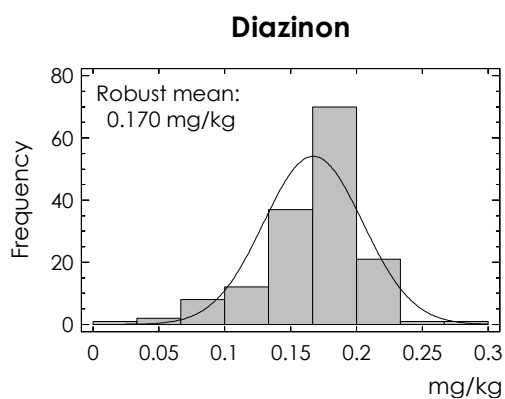
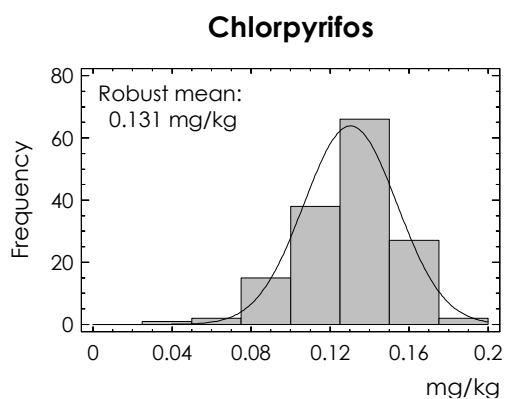
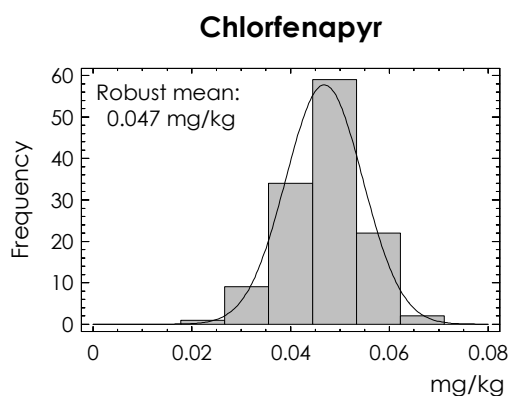
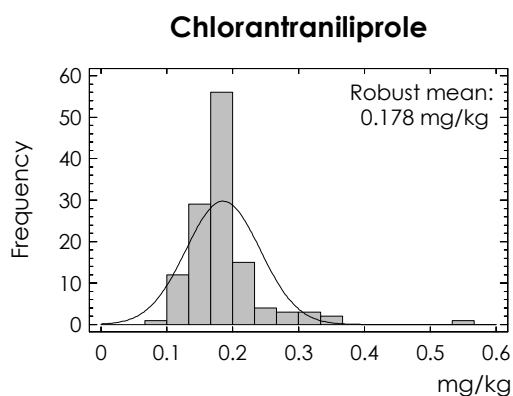
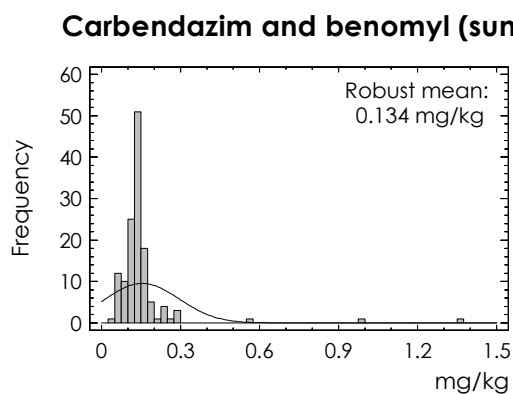
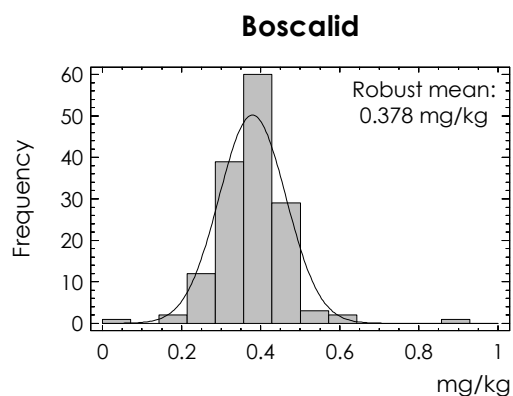
Penflufen (mg/kg)		Sulfoxaflor (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.530	0.510	0.033	0.032
0.500	0.500	0.033	0.032
0.580	0.500	0.033	0.033
0.540	0.490	0.031	0.032
0.550	0.500	0.033	0.035
0.550	0.560	0.033	0.034
0.630	0.550	0.033	0.034
0.460	0.500	0.033	0.032
0.600	0.520	0.033	0.033
0.590	0.570	0.033	0.033

The sample numbers used for this test were: 1, 23, 44, 90, 116, 154, 169, 185, 202 and 229.

The voluntary pesticides are not covered by the ISO/IEC 17043

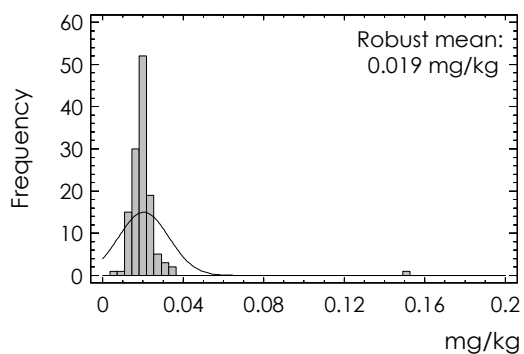
**APPENDIX 2. Histograms of residue data for each pesticide from EU/EFTA laboratories.**

Results presented as histograms.

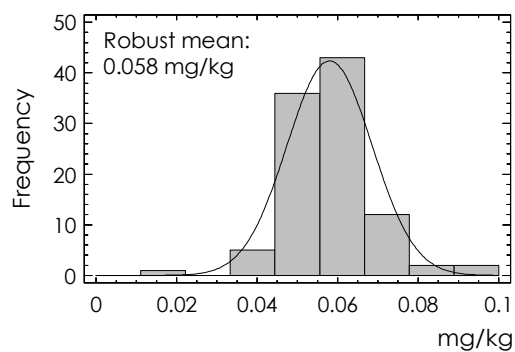


APPENDIX 2. Histograms of residue data for each pesticide from EU/EFTA laboratories.

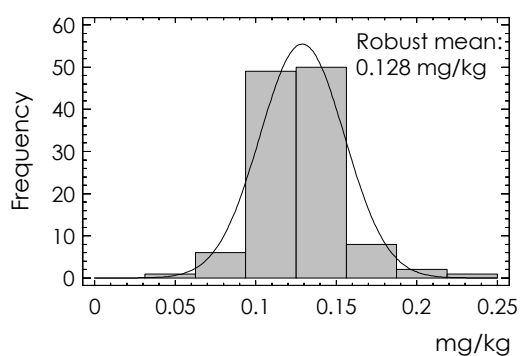
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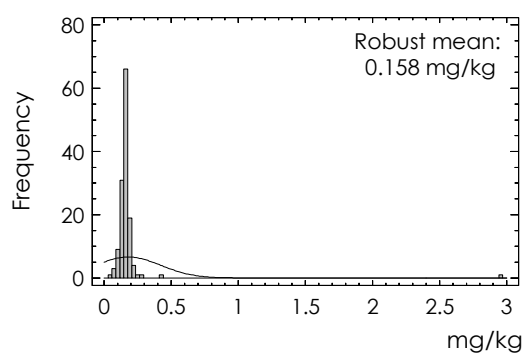
**Flubendiamide**



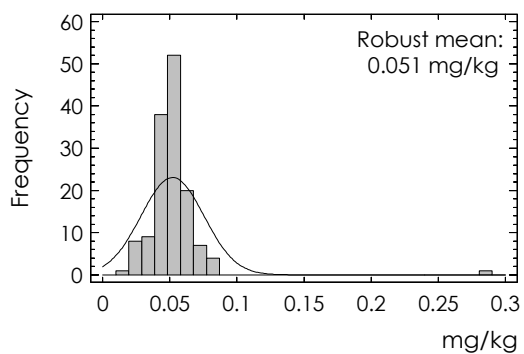
**Fluopyram**



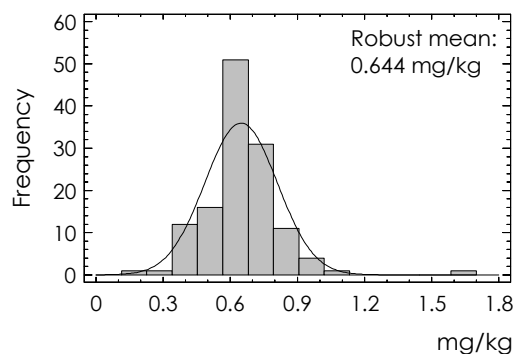
**Imidacloprid**



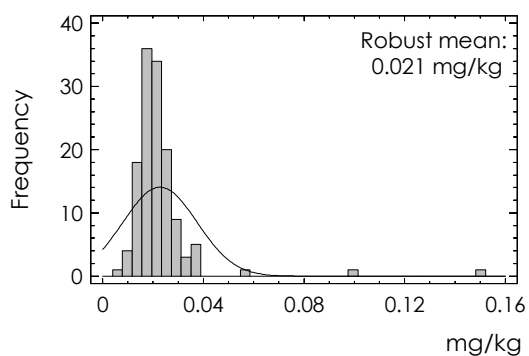
**Iprodione**



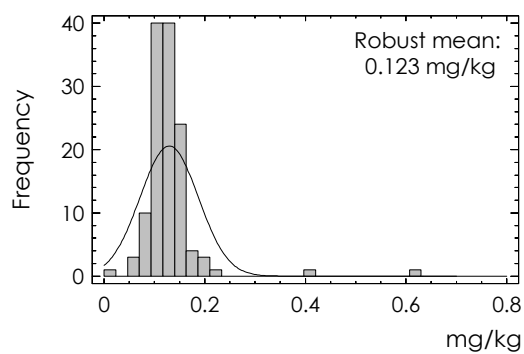
**Lufenuron**



**Omethoate**

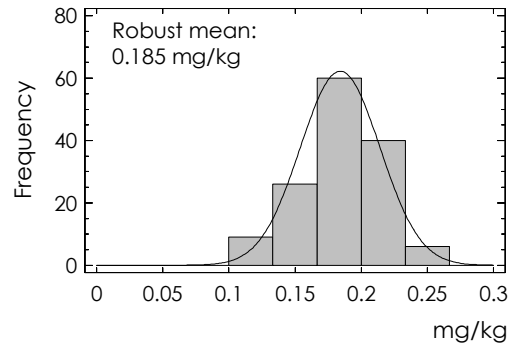


**Propamocarb**



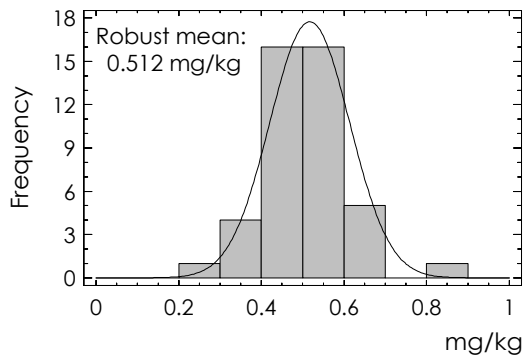
**APPENDIX 2. Histograms of residue data for each pesticide from EU/EFTA laboratories.**

**Pyraclostrobin**

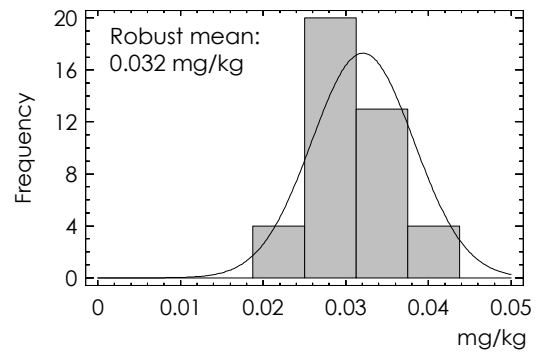


**Voluntary Pesticides**

**Penflufen**



**Sulfoxaflor**



The voluntary pesticides are not covered by the ISO/IEC 17043

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

Results reported by the laboratories for the mandatory pesticides boscalid, carbendazim and benomyl (sum of benomyl and carbendazim expressed as carbendazim), chlorantraniliprole, chlorfenapyr, chlorpyrifos, diazinon, ethoprophos, famoxadone, fipronil (only parent compound), flubendiamide, fluopyram, imidacloprid, iprodione, lufenuron, omethoate, propamocarb, and pyraclostrobin (mg/kg) and their calculated z score value using FFP RSD 25 %

Lab Code	Boscalid	z score (FFP RSD 25 %)		z score (FFP RSD 25 %)	Chlorantraniliprole		z score (FFP RSD 25 %)	Chlorfenapyr		z score (FFP RSD 25 %)	Chlorpyrifos		z score (FFP RSD 25 %)	Diazinon		z score (FFP RSD 25 %)	Ethoprophos		z score (FFP RSD 25 %)	Famoxadone		z score (FFP RSD 25 %)	Fipronil (only parent compound)		z score (FFP RSD 25 %)
		0.01	0.01		0.01	0.003		0.01	0.01		0.008	0.01		0.004											
Robust mean (mg/kg)	0.378	0.134		0.178	0.047		0.131	0.170		0.038	0.044		0.019												
Lab001	0.875	5.0	NA		0.565	5.0	NA		0.127	-0.1	0.145	-0.6	NA		NA		NA		NA		NA				
Lab002	0.432	0.6	0.14	0.1	0.23	1.2	0.0484	0.1	0.159	0.9	0.197	0.6	0.046	0.8	0.041	-0.2	0.0224	0.7							
Lab003	0.45	0.8	NA		NA		0.048	0.1	0.144	0.4	0.188	0.4	0.054	1.6	NA		0.025	1.2							
Lab004	0.379	0.0	0.13	-0.2	0.17	-0.2	0.048	0.1	0.153	0.7	0.212	1.0	0.051	1.3	0.043	-0.1	0.019	0.0							
Lab005	0.284	-1.0	0.15	0.4	0.159	-0.4	0.0391	-0.7	0.101	-0.9	0.0653	-2.5	0.0206	-1.9	0.0387	-0.4	0.0189	-0.1							
Lab006	0.41	0.3	0.13	-0.1	0.19	0.3	0.052	0.4	0.15	0.6	0.19	0.5	0.04	0.2	0.047	0.3	0.021	0.4							
Lab007	0.381	0.0	0.15	0.4	0.199	0.5	0.0563	0.8	0.15	0.6	0.17	0.0	0.0371	-0.1	0.0454	0.2	0.021	0.4							
Lab008	0.458	0.8	1.35	5.0	0.074	-2.3	0.036	-0.9	0.123	-0.2	0.069	-2.4	0.027	-1.2	0.043	-0.1	0.013	-1.3							
Lab009	0.41	0.3	0.16	0.8	0.17	-0.2	0.046	-0.1	0.14	0.3	0.18	0.2	0.042	0.4	0.041	-0.2	0.02	0.2							
Lab010	0.341	-0.4	0.24	3.2	0.164	-0.3	0.031	-1.4	0.127	-0.1	0.062	-2.5	0.034	-0.5	0.036	-0.7	0.015	-0.9							
Lab011	0.391	0.1	0.12	-0.4	0.188	0.2	0.041	-0.5	0.12	-0.3	0.174	0.1	0.042	0.4	0.048	0.4	0.014	-1.1							
Lab012	0.43	0.6	0.07	-1.8	0.203	0.6	NA		0.107	-0.7	0.147	-0.5	0.033	-0.6	0.0593	1.4	NA								
Lab013	0.022	-3.8	NA		NA		0.019	-2.4	0.05	-2.5	0.033	-3.2	NA		NA		0.022	0.6							
Lab014	NA		0.12	-0.3	NA		0.029	-1.5	0.084	-1.4	0.097	-1.7	0.033	-0.6	NA		0.01	-1.9							
Lab015	0.379	0.0	0.15	0.5	0.181	0.1	0.0395	-0.6	0.133	0.1	0.172	0.0	0.0355	-0.3	0.043	-0.1	0.0192	0.0							
Lab016	0.348	-0.3	0.13	0.0	0.176	0.0	0.044	-0.2	0.137	0.2	0.177	0.2	0.034	-0.5	0.0425	-0.1	0.0185	-0.1							
Lab017	0.501	1.3	0.15	0.4	0.214	0.8	0.0493	0.2	0.0938	-1.1	0.124	-1.1	0.0237	-1.5	0.0519	0.8	0.0204	0.2							
Lab018	0.459	0.9	0.06	-2.4	0.196	0.4	0.046	-0.1	0.163	1.0	0.186	0.4	0.04	0.2	0.053	0.9	0.021	0.4							
Lab019	0.36	-0.2	0.11	-0.7	0.254	1.7	0.044	-0.2	0.124	-0.2	0.173	0.1	0.0405	0.2	0.035	-0.8	0.03	2.2							
Lab020	0.385	0.1	0.14	0.2	0.162	-0.4	0.047	0.0	0.128	-0.1	0.16	-0.2	0.036	-0.3	0.032	-1.1	0.014	-1.1							
Lab021	0.336	-0.4	0.13	0.0	0.154	-0.5	0.0438	-0.3	0.111	-0.6	0.151	-0.5	0.0309	-0.8	0.0405	-0.3	0.0164	-0.6							
Lab022	0.311	-0.7	0.13	0.0	0.152	-0.6	0.0358	-0.9	0.133	0.1	0.168	-0.1	0.036	-0.3	0.041	-0.2	0.152	5.0							
Lab023	0.329	-0.5	0.12	-0.4	0.14	-0.9	0.0619	1.3	0.126	-0.2	0.172	0.0	0.0302	-0.9	0.0454	0.2	0.0191	0.0							
Lab024	0.241	-1.4	ND	-3.7	ND	-3.8	ND	-3.1	0.151	0.6	0.211	1.0	0.05	1.2	ND	-3.1	NA								
Lab025	0.43	0.6	0.07	-2.1	0.165	-0.3	0.0387	-0.7	0.0976	-1.0	0.145	-0.6	0.0322	-0.7	0.0429	-0.1	0.0202	0.2							
Lab026	0.43	0.6	0.14	0.1	0.19	0.3	0.055	0.7	0.151	0.6	0.213	1.0	0.043	0.5	0.05	0.6	0.022	0.6							
Lab027	0.37	-0.1	0.16	0.8	0.199	0.5	0.0444	-0.2	0.126	-0.2	0.17	0.0	0.0389	0.0	0.0376	-0.6	0.0173	-0.4							
Lab028	NA		0.07	-1.9	NA		0.04	-0.6	0.13	0.0	0.17	0.0	NA		NA		NA								
Lab029	0.333	-0.5	0.1	-1.0	0.176	0.0	0.045	-0.2	0.119	-0.4	0.167	-0.1	0.037	-0.2	0.037	-0.6	0.019	0.0							
Lab030	0.42	0.4	0.15	0.5	0.172	-0.1	0.047	0.0	0.11	-0.6	0.215	1.1	0.045	0.7	0.041	-0.2	0.025	1.2							
Lab031	0.342	-0.4	0.18	1.2	NA		NA		0.111	-0.6	0.124	-1.1	NA		0.04	-0.3	0.0258	1.4							
Lab032	0.435	0.6	0.11	-0.8	0.146	-0.7	NA		0.155	0.7	0.18	0.2	0.048	1.0	NA		0.019	0.0							
Lab033	0.411	0.3	0.12	-0.5	0.2	0.5	0.047	0.0	0.108	-0.7	0.182	0.3	0.04	0.2	0.044	0.0	0.017	-0.5							
Lab034	0.391	0.1	NA		0.134	-1.0	NA		0.15	0.6	0.151	-0.5	NA		0.03	-1.2	NA								
Lab035	0.364	-0.1	0.99	5.0	0.173	-0.1	0.055	0.7	0.155	0.7	0.196	0.6	0.042	0.4	0.051	0.7	0.022	0.6							
Lab036	0.39	0.1	0.09	-1.3	0.155	-0.5	0.044	-0.2	0.158	0.8	0.205	0.8	0.038	0.0	0.044	0.0	0.021	0.4							
Lab037	0.377	0.0	0.23	2.9	0.144	-0.8	0.057	0.9	0.13	0.0	0.17	0.0	0.0347	-0.4	0.0469	0.3	0.0297	2.2							
Lab038	0.431	0.6	0.100	-1.0	0.175	-0.1	0.05	0.3	0.142	0.3	0.166	-0.1	0.045	0.7	0.043	-0.1	0.019	0.0							
Lab039	0.49	1.2	0.1	-1.0	0.17	-0.2	0.066	1.6	0.144	0.4	0.182	0.3	0.029	-1.0	0.051	0.7	0.017	-0.5							



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

Lab Code	Boscalid	z score (FFP RSD 25 %)		Carbendazim and benomyl (sum of benomyl and carbendazim expressed as carbendazim)	z score (FFP RSD 25 %)		Chlorantranilprole	z score (FFP RSD 25 %)		Chlorfenapyr	z score (FFP RSD 25 %)		Chlorpyrifos	z score (FFP RSD 25 %)		Diazinon	z score (FFP RSD 25 %)		Ethoprophos	z score (FFP RSD 25 %)		Famoxadone	z score (FFP RSD 25 %)		Fipronil (only parent compound)	z score (FFP RSD 25 %)		
		MRRL	0.01		0.01	0.01		0.003	0.01		0.01	0.008		0.01	0.004													
		Robust mean (mg/kg)	0.378		0.134	0.178		0.047	0.131		0.170	0.038		0.044	0.019													
Lab040	0.581	2.1	0.17	1.1	0.244	1.5	0.06	1.1	0.193	1.9	0.198	0.7	0.049	1.1	0.057	1.2	0.036	3.5										
Lab041	0.387	0.1	0.19	1.8	0.208	0.7	ND	-3.1	0.133	0.1	0.172	0.0	0.041	0.3	0.04	-0.3	0.018	-0.3										
Lab042	0.421	0.5	0.12	-0.4	0.148	-0.7	0.0475	0.1	0.164	1.0	0.231	1.4	0.0456	0.7	0.0633	1.8	0.0259	1.4										
Lab043	0.191	-2.0	0.08	-1.6	0.126	-1.2	0.053	0.5	0.126	-0.2	0.169	0.0	0.034	-0.5	0.0376	-0.6	0.0143	-1.0										
Lab044	0.447	0.7	0.09	-1.2	0.162	-0.4	0.0504	0.3	0.115	-0.5	0.187	0.4	NA		0.0368	-0.6	NA											
Lab045	0.312	-0.7	0.15	0.5	NA		0.054	0.6	0.1	-0.9	0.18	0.2	0.05	1.2	NA		0.019	0.0										
Lab046	0.362	-0.2	0.13	-0.2	0.148	-0.7	0.048	0.1	0.119	-0.4	0.181	0.3	0.034	-0.5	0.033	-1.0	0.019	0.0										
Lab047	0.402	0.3	0.16	0.6	0.179	0.0	0.0535	0.6	0.142	0.3	0.228	1.4	0.051	1.3	NA		0.02	0.2										
Lab048	0.276	-1.1	NA		0.121	-1.3	NA		0.109	-0.7	0.168	-0.1	0.035	-0.4	0.035	-0.8	0.022	0.6										
Lab049	0.35	-0.3	0.1	-1.1	0.13	-1.1	0.035	-1.0	0.088	-1.3	0.16	-0.2	0.035	-0.4	0.027	-1.5	0.012	-1.5										
Lab050	0.381	0.0	0.05	-2.5	0.141	-0.8	0.0476	0.1	0.163	1.0	0.165	-0.1	0.0385	0.0	0.0448	0.1	0.0184	-0.2										
Lab051	0.315	-0.7	0.13	-0.2	0.13	-1.1	0.0439	-0.3	0.127	-0.1	0.161	-0.2	0.038	0.0	0.0318	-1.1	0.0177	-0.3										
Lab052	0.277	-1.1	0.09	-1.4	0.125	-1.2	NA		ND	-3.7	0.1003	-1.6	NA		NA		0.0069	-2.6										
Lab053	0.43	0.6	0.2	1.9	0.169	-0.2	0.068	1.8	0.162	0.9	0.206	0.8	0.034	-0.5	0.041	-0.2	0.02	0.2										
Lab054	0.386	0.1	0.12	-0.5	0.211	0.7	0.0329	-1.2	0.094	-1.1	0.185	0.3	0.035	-0.4	0.0306	-1.2	0.0195	0.1										
Lab055	0.399	0.2	0.11	-0.8	0.169	-0.2	0.0572	0.9	0.129	-0.1	0.169	0.0	0.0398	0.1	0.0469	0.3	0.0212	0.4										
Lab056	0.43	0.6	0.12	-0.4	0.206	0.6	0.0589	1.0	0.168	1.1	0.211	1.0	0.0436	0.5	0.0457	0.2	0.024	1.0										
Lab057	0.261	-1.2	0.07	-1.9	0.125	-1.2	0.035	-1.0	0.102	-0.9	0.13	-0.9	0.027	-1.2	0.028	-1.4	0.013	-1.3										
Lab058	0.323	-0.6	0.17	0.9	0.359	4.1	0.048	0.1	0.146	0.5	0.067	-2.4	0.033	-0.6	0.041	-0.2	0.023	0.8										
Lab059	0.38	0.0	0.18	1.3	0.197	0.4	0.045	-0.2	0.118	-0.4	0.176	0.1	0.036	-0.3	0.049	0.5	0.018	-0.3										
Lab060	0.323	-0.6	0.12	-0.4	0.196	0.4	NA		0.157	0.8	0.268	2.3	0.042	0.4	NA		0.0299	2.2										
Lab061	0.403	0.3	0.14	0.1	0.192	0.3	0.052	0.4	0.151	0.6	0.201	0.7	0.041	0.3	0.039	-0.4	0.023	0.8										
Lab062	0.31	-0.7	0.17	1.1	0.332	3.5	0.05	0.3	0.135	0.1	0.13	-0.9	0.027	-1.2	0.029	-1.3	0.02	0.2										
Lab063	0.443	0.7	0.14	0.1	0.189	0.2	0.053	0.5	0.155	0.7	0.203	0.8	0.04	0.2	0.046	0.2	0.021	0.4										
Lab064	0.407	0.3	0.14	0.2	0.189	0.2	0.0447	-0.2	0.143	0.4	0.176	0.1	0.035	-0.4	0.0404	-0.3	NA											
Lab065	0.334	-0.5	0.15	0.4	0.164	-0.3	0.042	-0.4	0.094	-1.1	0.163	-0.2	0.033	-0.6	0.036	-0.7	0.023	0.8										
Lab066	0.314	-0.7	0.16	0.8	0.17	-0.2	0.04	-0.6	0.106	-0.8	0.168	-0.1	0.037	-0.2	0.039	-0.4	0.02	0.2										
Lab067	0.37	-0.1	0.13	-0.1	0.17	-0.2	0.041	-0.5	0.13	0.0	0.17	0.0	0.041	0.3	0.046	0.2	0.02	0.2										
Lab068	0.466	0.9	0.17	0.9	0.188	0.2	0.057	0.9	0.156	0.8	0.211	1.0	0.044	0.6	0.048	0.4	0.021	0.4										
Lab069	0.332	-0.5	0.13	-0.2	0.149	-0.7	0.037	-0.8	0.108	-0.7	0.195	0.6	0.037	-0.2	0.044	0.0	0.014	-1.1										
Lab070	0.267	-1.2	0.07	-1.8	0.115	-1.4	0.047	0.0	0.125	-0.2	0.148	-0.5	0.03	-0.9	ND	-3.1	0.018	-0.3										
Lab071	0.322	-0.6	0.13	-0.3	0.195	0.4	0.052	0.4	0.13	0.0	0.128	-1.0	0.04	0.2	0.058	1.3	0.02	0.2										
Lab072	0.32	-0.6	0.17	1.1	0.16	-0.4	0.044	-0.2	0.13	0.0	0.18	0.2	0.042	0.4	0.044	0.0	0.015	-0.9										
Lab073	0.349	-0.3	0.13	-0.1	0.185	0.2	0.048	0.1	0.15	0.6	0.186	0.4	0.043	0.5	0.05	0.6	0.022	0.6										
Lab074	No Results Reported																											
Lab075	0.404	0.3	0.14	0.2	0.19	0.3	0.036	-0.9	0.142	0.3	0.227	1.3	0.039	0.1	0.052	0.8	0.02	0.2										
Lab076	0.338	-0.4	0.13	-0.2	0.189	0.2	0.045	-0.2	0.13	0.0	0.165	-0.1	0.042	0.4	0.042	-0.1	0.018	-0.3										
Lab077	0.406	0.3	0.12	-0.4	0.179	0.0	0.048	0.1	0.142	0.3	0.196	0.6	0.04	0.2	0.044	0.0	0.0239	1.0										
Lab078	0.415	0.4	0.11	-0.7	0.15	-0.6	0.043	-0.3	0.105	-0.8	0.151	-0.5	0.034	-0.5	0.043	-0.1	0.018	-0.3										
Lab079	0.461	0.9	0.24	3.1	0.177	0.0	0.0399	-0.6	0.117	-0.4	0.205	0.8	0.0366	-0.2	0.0515	0.7	0.0122	-1.5										
Lab080	0.42	0.4	0.08	-1.7	0.214	0.8	0.047	0.0	0.138	0.2	0.197	0.6	0.048	1.0	NA		0.02	0.2										
Lab081	0.185	-2.0	0.06	-2.2	0.11	-1.5	0.032	-1.3	0.091	-1.2	0.0978	-1.7	0.01896	5.0	0.026	-1.6	0.011	-1.7										
Lab082	0.379	0.0	0.17	1.0	0.205	0.6	0.046	-0.1	0.128	-0.1	0.173	0.1	0.0431	0.5	0.0401	-0.3	0.0201	0.2										
Lab083	0.393	0.2	0.1	-1.0	0.213	0.8	0.0456	-0.1	0.118	-0.4	0.16	-0.2	0.0358	-0.3	0.0504	0.6	0.0165	-0.6										
Lab084	0.341	-0.4	0.09	-1.3	0.25	1.6	0.061	1.2	0.089	-1.3	0.138	-0.8	0.033	-0.6	0.036	-0.7	ND	-3.2										
Lab085	0.224	-1.6	0.13	-0.1	0.268	2.0	0.03	-1.4	0.08	-1.6	0.14	-0.7	0.08	4.3	ND	-3.1	0.013	-1.3										

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

Lab Code	Boscalid	z score (FFP RSD 25 %)		z score (FFP RSD 25 %)	Chlorantranilprole	z score (FFP RSD 25 %)		z score (FFP RSD 25 %)	Chlorfenapyr	z score (FFP RSD 25 %)		z score (FFP RSD 25 %)	Diazinon	z score (FFP RSD 25 %)		z score (FFP RSD 25 %)	Ethoprophos	z score (FFP RSD 25 %)		z score (FFP RSD 25 %)	Famoxadone	z score (FFP RSD 25 %)		z score (FFP RSD 25 %)	Fipronil (only parent compound)	z score (FFP RSD 25 %)	
		MRRL	0.01			0.01	0.01			0.003	0.01			0.01	0.008			0.01	0.004								
		Robust mean (mg/kg)	0.378			0.134	0.178			0.047	0.131			0.170	0.038			0.044	0.019								
Lab086	0.364	-0.1	0.16	0.7	0.171	-0.2	0.0542	0.6	0.172	1.3	0.192	0.5	NA		0.0884	4.1	0.016	-0.7									
Lab087	0.41	0.3	0.13	-0.2	0.164	-0.3	0.049	0.2	0.159	0.9	0.183	0.3	0.0439	0.6	0.049	0.5	0.0183	-0.2									
Lab088	0.299	-0.8	0.15	0.4	0.146	-0.7	0.042	-0.4	0.142	0.3	0.15	-0.5	0.032	-0.7	NA		0.016	-0.7									
Lab089	0.387	0.1	0.15	0.4	0.188	0.2	0.046	-0.1	0.133	0.1	0.19	0.5	0.0472	0.9	0.059	1.4	0.0218	0.5									
Lab090	0.49	1.2	0.12	-0.4	0.15	-0.6	0.05	0.3	0.15	0.6	0.19	0.5	0.03	-0.9	0.04	-0.3	0.02	0.2									
Lab091	0.278	-1.1	0.11	-0.9	0.108	-1.6	0.051	0.3	0.118	-0.4	0.142	-0.7	0.043	0.5	0.035	-0.8	0.016	-0.7									
Lab092	0.45	0.8	NA		NA		NA		0.1	-0.9	0.14	-0.7	0.04	0.2	NA		NA										
Lab093	0.452	0.8	0.15	0.4	NA		0.0441	-0.2	0.126	-0.2	0.178	0.2	0.0393	0.1	NA		0.0191	0.0									
Lab094	0.399	0.2	0.14	0.2	0.204	0.6	0.062	1.3	0.139	0.2	0.181	0.3	0.03	-0.9	0.055	1.0	0.0224	0.7									
Lab095	0.574	2.1	0.29	4.7	0.307	2.9	0.0474	0.0	0.162	0.9	0.174	0.1	0.0405	0.2	NA		0.0215	0.5									
Lab096	0.364	-0.1	0.15	0.5	0.188	0.2	0.059	1.0	0.144	0.4	0.186	0.4	0.037	-0.2	0.042	-0.1	0.021	0.4									
Lab097	0.48	1.1	0.14	0.2	0.21	0.7	0.06	1.1	0.12	-0.3	0.16	-0.2	0.039	0.1	0.06	1.5	0.015	-0.9									
Lab098	0.39	0.1	0.23	2.7	0.17	-0.2	0.0448	-0.2	0.14	0.3	0.164	-0.1	0.0343	-0.4	0.0461	0.2	0.019	0.0									
Lab099	0.42	0.4	0.16	0.9	0.18	0.0	0.055	0.7	0.154	0.7	0.223	1.2	0.051	1.3	0.022	-2.0	0.019	0.0									
Lab100	0.4685	1.0	0.11	-0.8	0.195	0.4	0.0546	0.7	0.1277	-0.1	0.144	-0.6	0.0395	0.1	NA		0.0252	1.2									
Lab101	0.398	0.2	0.16	0.7	0.174	-0.1	0.0521	0.4	0.148	0.5	0.191	0.5	0.0398	0.1	0.0389	-0.4	0.0176	-0.3									
Lab102	0.4575	0.8	0.11	-0.7	0.1935	0.3	0.0493	0.2	0.1291	-0.1	0.1185	-1.2	0.0392	0.1	NA		0.0195	0.1									
Lab103	0.381	0.0	NA		NA		0.12	5.0	0.134	0.1	0.162	-0.2	0.039	0.1	0.112	5.0	0.047	5.0									
Lab104	0.42	0.4	0.18	1.4	NA		0.043	-0.3	0.12	-0.3	0.14	-0.7	0.033	-0.6	ND	-3.1	0.02	0.2									
Lab105	0.394	0.2	0.09	-1.4	NA		NA		0.191	1.8	0.063	-2.5	NA		NA		0.016	-0.7									
Lab106	0.488	1.2	0.13	-0.2	0.276	2.2	0.0548	0.7	0.127	-0.1	0.158	-0.3	0.0486	1.1	NA		0.0263	1.5									
Lab107	0.358	-0.2	0.2	2.0	0.177	0.0	0.06	1.1	0.15	0.6	0.186	0.4	0.041	0.3	0.046	0.2	0.023	0.8									
Lab108	0.45	0.8	0.51	5.0	0.045	-3.0	0.049	0.2	0.155	0.7	0.21	0.9	0.042	0.4	NA		0.022	0.6									
Lab109	0.3	-0.8	0.17	1.2	ND	-3.8	0.06	1.1	0.014	-3.6	0.056	-2.7	0.037	-0.2	0.043	-0.1	0.022	0.6									
Lab110	No Results Reported																										
Lab111	0.417	0.4	0.06	-2.2	NA		0.043	-0.3	ND	-3.7	0.148	-0.5	0.032	-0.7	NA		ND	-3.2									
Lab113	0.331	-0.5	0.13	0.0	0.167	-0.3	0.0441	-0.2	0.155	0.7	0.142	-0.7	0.0346	-0.4	NA		0.0177	-0.3									
Lab114	0.390	0.1	0.122	-0.4	NA		0.052	0.4	0.166	1.1	0.169	0.0	0.032	-0.7	NA		0.018	-0.3									
Lab115	0.354	-0.3	0.15	0.5	NA		ND	-3.1	0.116	-0.5	0.07	-2.4	0.034	-0.5	NA		0.019	0.0									
Lab116	0.36	-0.2	0.17	1.1	0.18	0.0	0.045	-0.2	0.12	-0.3	0.14	-0.7	0.03	-0.9	0.046	0.2	0.02	0.2									
Lab117	NA		0.12	-0.4	NA		NA		0.08	-1.6	0.082	-2.1	0.03	-0.9	NA		ND	-3.2									
Lab118	0.406	0.3	NA		NA		NA		0.125	-0.2	0.0719	-2.3	0.0357	-0.3	NA		0.0183	-0.2									
Lab119	0.282	-1.0	NA		NA		0.0352	-1.0	0.107	-0.7	0.128	-1.0	0.0267	-1.2	NA		NA										
Lab120	0.333	-0.5	0.13	-0.1	0.162	-0.4	0.0442	-0.2	0.132	0.0	0.172	0.0	0.0381	0.0	0.0432	0.0	0.0209	0.4									
Lab121	0.380	0.0	####	-1.9	0.180	0.0	0.0442	-0.2	0.126	-0.2	0.187	0.4	0.0303	-0.8	0.0579	1.3	0.0152	-0.8									
Lab122	0.4	0.2	0.11	-0.9	0.154	-0.5	0.052	0.4	0.148	0.5	0.18	0.2	0.048	1.0	ND	-3.1	0.015	-0.9									
Lab123	0.295	-0.9	0.12	-0.6	0.125	-1.2	NA		0.121	-0.3	0.156	-0.3	0.038	0.0	0.0351	-0.8	0.0152	-0.8									
Lab124	0.401	0.2	0.14	0.2	0.181	0.1	0.058	0.9	0.152	0.6	0.185	0.3	0.036	-0.3	0.046	0.2	0.012	-1.5									
Lab126	0.34	-0.4	0.07	-2.0	NA		0.043	-0.3	0.115	-0.5	0.15	-0.5	0.04	0.2	NA		NA										
Lab127	0.324	-0.6	0.57	5.0	0.131	-1.1	NA		0.123	-0.2	0.187	0.4	0.035	-0.4	0.037	-0.6	0.015	-0.9									
Lab128	0.291	-0.9	0.07	-1.8	NA		0.0367	-0.9	0.0998	-1.0	0.139	-0.7	0.0451	0.7	NA		NA										
Lab129	0.4	0.2	0.13	-0.1	0.2	0.5	0.051	0.3	0.13	0.0	0.2	0.7	0.038	0.0	0.047	0.3	0.025	1.2									
Lab130	0.385	0.1	0.12	-0.6	0.306	2.9	0.051	0.3	0.147	0.5	0.199	0.7	0.045	0.7	0.047	0.3	0.026	1.4									
Lab131	0.402	0.3	0.14	0.2	NA		NA		0.127	-0.1	0.113	-1.3	0.04	0.2	NA		NA										
Lab132	0.401	0.2	0.17	1.0	0.18	0.0	0.052	0.4	0.148	0.5	0.185	0.3	0.04	0.2	0.048	0.4	0.027	1.6									
Lab133	0.357	-0.2	0.13	-0.2	0.159	-0.4	0.0464	0.0	0.133	0.1	0.174	0.1	0.0371	-0.1	0.0416	-0.2	0.0193	0.0									

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

Lab Code	Boscalid	z score (FFP RSD 25 %)			Chlorantraniliprole	z score (FFP RSD 25 %)			Chlorfenapyr	z score (FFP RSD 25 %)			Diazinon	z score (FFP RSD 25 %)			Ethoprophos	z score (FFP RSD 25 %)			Famoxadone	z score (FFP RSD 25 %)			Fipronil (only parent compound)	z score (FFP RSD 25 %)		
		MRL	0.01	0.01		0.01	0.003	0.01		0.01	0.01	0.008		0.01	0.01	0.004		0.01	0.01	0.01		0.004						
Robust mean (mg/kg)	0.378	0.134			0.178	0.047			0.131	0.170			0.038	0.044			0.019											
Lab134	No Results Reported																											
Lab135	0.431	0.6	0.17	1.0	0.285	2.4	0.047	0.0	0.121	-0.3	0.12	-1.2	0.037	-0.2	0.038	-0.5	0.023	0.8										
Lab136	0.489	1.2	0.14	0.2	0.114	-1.4	0.044	-0.2	0.148	0.5	0.168	-0.1	0.043	0.5	NA		0.02	0.2										
Lab138	0.299	-0.8	0.14	0.1	0.155	-0.5	0.0392	-0.7	0.114	-0.5	0.155	-0.4	0.0364	-0.2	0.0375	-0.6	0.0155	-0.8										
Lab139	0.407	0.3	0.28	4.4	0.271	2.1	0.0517	0.4	0.163	1.0	0.209	0.9	0.0433	0.5	0.0489	0.5	NA											
Lab140	0.388	0.1	0.14	0.2	0.164	-0.3	0.051	0.3	0.16	0.9	0.169	0.0	0.035	-0.4	0.052	0.8	0.02	0.2										
Lab141	0.286	-1.0	0.07	-1.8	0.137	-0.9	0.041	-0.5	0.103	-0.9	0.121	-1.2	0.031	-0.8	NA		0.017	-0.5										
Lab142	0.28	-1.0	0.15	0.4	0.199	0.5	0.0486	0.1	0.0965	-1.1	0.157	-0.3	0.0359	-0.3	0.0405	-0.3	0.0121	-1.5										
Lab143	0.427	0.5	0.14	0.0	0.199	0.5	NA		NA		0.196	0.6	0.0423	0.4	0.0484	0.4	0.0207	0.3										
Lab144	0.35	-0.3	0.12	-0.4	NA		NA		0.14	0.3	0.16	-0.2	0.04	0.2	NA		ND	-3.2										
Lab145	0.436	0.6	0.13	-0.3	0.183	0.1	0.0503	0.3	0.147	0.5	0.196	0.6	0.0432	0.5	0.0441	0.0	0.0214	0.5										
Lab146	0.414	0.4	0.29	4.7	0.171	-0.2	0.0425	-0.4	0.073	-1.8	0.131	-0.9	0.025	-1.4	0.074	2.8	0.015	-0.9										
Lab147	0.357	-0.2	0.27	4.0	0.221	1.0	0.0454	-0.1	0.1395	0.3	0.1385	-0.7	0.0446	0.6	0.0382	-0.5	0.0231	0.8										
Lab148	0.437	0.6	0.15	0.4	0.334	3.5	0.046	-0.1	0.144	0.4	0.186	0.4	0.0353	-0.3	NA		NA											
Lab149	0.305	-0.8	0.13	-0.1	0.185	0.2	0.0471	0.0	0.136	0.2	0.185	0.3	0.0376	-0.1	0.042	-0.1	0.0222	0.6										
Lab150	0.435	0.6	0.13	-0.1	0.189	0.2	0.0387	-0.7	0.163	1.0	0.192	0.5	0.0493	1.1	0.0509	0.7	0.0179	-0.3										
Lab151	0.34	-0.4	0.2	2.0	0.18	0.0	0.038	-0.8	0.12	-0.3	0.15	-0.5	0.038	0.0	0.045	0.1	0.013	-1.3										
Lab152	0.48	1.1	0.11	-0.7	NA		NA		0.14	0.3	0.18	0.2	0.039	0.1	0.043	-0.1	0.02	0.2										
Lab153	0.52	1.5	NA		0.235	1.3	0.0562	0.8	0.15	0.6	0.195	0.6	0.0405	0.2	0.05	0.6	NA											
Lab154	0.293	-0.9	0.15	0.5	0.191	0.3	0.0424	-0.4	0.125	-0.2	0.15	-0.5	0.0277	-1.1	0.0647	1.9	0.0141	-1.1										
Lab155	0.432	0.6	0.13	-0.1	0.177	0.0	0.047	0.0	0.133	0.1	0.18	0.2	0.038	0.0	0.047	0.3	0.018	-0.3										
Lab156	NA		NA		NA		NA		0.139	0.2	0.142	-0.7	0.033	-0.6	NA		NA											
Lab158	0.432	0.6	NA		NA		0.049	0.2	0.134	0.1	0.191	0.5	0.04	0.2	0.045	0.1	0.02	0.2										
Lab159	0.363	-0.2	NA		NA		NA		0.12	-0.3	0.161	-0.2	0.0494	1.1	0.0475	0.4	0.0335	3.0										
Lab160	0.408	0.3	0.14	0.2	0.149	-0.7	0.049	0.2	0.129	-0.1	0.186	0.4	0.032	-0.7	0.046	0.2	0.018	-0.3										
Lab161	0.394	0.2	0.18	1.5	0.168	-0.2	0.0539	0.6	0.145	0.4	0.206	0.8	0.0372	-0.1	0.0401	-0.3	0.0201	0.2										
Lab162	0.295	-0.9	NA		NA		NA		0.100	-0.9	0.0824	-2.1	0.0507	1.3	NA		NA											
Lab163	0.348	-0.3	NA		NA		NA		0.163	1.0	0.209	0.9	NA		NA		NA											
Lab164	0.238	-1.5	NA		NA		NA		0.096	-1.1	0.084	-2.0	0.02	-1.9	NA		NA											
Lab165	0.409	0.3	0.2	2.1	0.198	0.4	0.05	0.3	0.13	0.0	0.116	-1.3	ND	-3.2	0.047	0.3	0.017	-0.5										
Lab166	0.38	0.0	NA		NA		0.0546	0.7	0.146	0.5	0.206	0.8	0.0382	0.0	NA		0.0222	0.6										
Lab167	0.349	-0.3	0.15	0.4	0.167	-0.3	NA		0.117	-0.4	0.135	-0.8	0.0345	-0.4	0.041	-0.2	0.0163	-0.6										
Lab169	0.409	0.3	0.15	0.6	0.177	0.0	0.0463	-0.1	0.137	0.2	0.203	0.8	0.0453	0.7	0.0431	0.0	0.0214	0.5										
Lab170	NA		NA		NA		0.035	-1.0	0.104	-0.8	0.074	-2.3	0.031	-0.8	NA		NA											
Lab171	0.55	1.8	0.09	-1.3	0.21	0.7	0.054	0.6	0.16	0.9	0.26	2.1	0.045	0.7	0.043	-0.1	0.019	0.0										
Lab172	0.261	-1.2	0.13	-0.1	0.199	0.5	0.0463	-0.1	0.112	-0.6	0.158	-0.3	0.0339	-0.5	0.0257	-1.6	0.0202	0.2										
Lab173	0.393	0.2	0.14	0.2	0.182	0.1	0.049	0.2	0.141	0.3	0.19	0.5	0.0436	0.5	0.0463	0.2	0.0218	0.5										
Lab174	0.35	-0.3	0.07	-2.1	NA		NA		0.16	0.9	0.17	0.0	0.035	-0.4	ND	-3.1	ND	-3.2										
Lab175	0.373	-0.1	0.14	0.0	NA		0.045	-0.2	0.118	-0.4	0.161	-0.2	0.035	-0.4	0.03	-1.2	0.0148	-0.9										
Lab176	0.455	0.8	NA		NA		NA		0.193	1.9	0.174	0.1	NA		NA		0.0128	-1.3										
Lab177	0.42	0.4	NA		NA		NA		0.07	-1.9	0.09	-1.9	NA		NA		NA											
Lab178	No Results Reported																											
Lab179	0.383	0.1	0.12	-0.4	0.181	0.1	0.0474	0.0	0.114	-0.5	0.161	-0.2	0.0348	-0.4	0.0561	1.1	0.0266	1.5										

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

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

Lab Code	Flubendamide	z score (FFP RSD 25 %)		z score (FFP RSD 25 %)		z score (FFP RSD 25 %)		z score (FFP RSD 25 %)		z score (FFP RSD 25 %)		z score (FFP RSD 25 %)		z score (FFP RSD 25 %)		
		0.003	0.01	0.01	0.010	0.010	0.010	0.003	0.010	0.010	0.003	0.010	0.010	0.010	0.010	
																0.058
Lab001	NA		0.24	3.5	0.087	-1.8	0.048	-0.2	NA		0.027	1.2	NA		0.199	0.3
Lab002	0.074	1.1	0.161	1.0	0.182	0.6	0.055	0.4	0.879	1.5	0.0134	-1.4	0.0573	-2.1	0.246	1.3
Lab003	NA		NA		0.193	0.9	0.05	0.0	NA		0.024	0.6	0.609	5.0	0.213	0.6
Lab004	0.076	1.3	0.157	0.9	0.156	0.0	0.05	0.0	0.537	-0.7	0.027	1.2	0.13	0.2	0.182	-0.1
Lab005	0.0527	-0.3	0.113	-0.5	0.125	-0.8	0.081	2.4	0.86	1.3	0.0286	1.5	0.126	0.1	0.131	-1.2
Lab006	0.069	0.8	0.12	-0.2	0.16	0.1	0.06	0.7	0.69	0.3	0.03	1.8	0.13	0.2	0.2	0.3
Lab007	0.0605	0.2	0.133	0.2	0.155	-0.1	0.0535	0.2	0.692	0.3	0.0254	0.9	0.135	0.4	0.216	0.7
Lab008	NA		NA		0.118	-1.0	0.031	-1.5	0.601	-0.3	0.015	-1.1	0.077	-1.5	0.189	0.1
Lab009	0.058	0.0	0.13	0.1	0.15	-0.2	0.051	0.0	0.69	0.3	0.012	-1.7	0.12	-0.1	0.2	0.3
Lab010	0.051	-0.5	0.107	-0.6	0.22	1.6	ND	-3.2	0.634	-0.1	0.018	-0.5	0.2	2.5	0.15	-0.8
Lab011	0.063	0.4	0.123	-0.1	0.153	-0.1	0.049	-0.1	0.656	0.1	0.022	0.2	0.141	0.6	0.19	0.1
Lab012	NA		NA		0.0853	-1.8	0.042	-0.7	0.82	1.1	0.011	-1.9	0.0737	-1.6	0.216	0.7
Lab013	NA		NA		NA		0.023	-2.2	NA		NA		NA		0.116	-1.5
Lab014	0.042	-1.1	0.104	-0.7	0.185	0.7	0.023	-2.2	0.173	-2.9	0.024	0.6	0.135	0.4	0.14	-1.0
Lab015	0.0618	0.3	0.122	-0.2	0.158	0.0	0.0415	-0.7	0.684	0.2	0.0192	-0.3	0.118	-0.2	0.173	-0.3
Lab016	0.0541	-0.2	0.122	-0.2	0.139	-0.5	0.0511	0.0	0.641	0.0	0.0178	-0.6	0.138	0.5	0.172	-0.3
Lab017	0.0537	-0.3	0.138	0.3	0.181	0.6	0.0363	-1.1	0.812	1.0	0.0159	-0.9	0.0908	-1.1	0.216	0.7
Lab018	0.067	0.7	0.173	1.4	0.196	1.0	0.053	0.2	0.673	0.2	0.01	-2.1	ND	-3.7	0.208	0.5
Lab019	0.0755	1.2	0.098	-0.9	0.154	-0.1	0.045	-0.4	0.889	1.5	0.0267	1.1	0.112	-0.4	0.172	-0.3
Lab020	0.054	-0.2	0.138	0.3	0.183	0.6	0.067	1.3	0.685	0.3	0.019	-0.4	0.146	0.7	0.163	-0.5
Lab021	0.0499	-0.5	0.11	-0.6	0.141	-0.4	0.0456	-0.4	0.728	0.5	0.0159	-0.9	0.15	0.9	0.16	-0.5
Lab022	0.047	-0.7	0.125	-0.1	0.151	-0.2	0.0432	-0.6	0.71	0.4	0.0168	-0.8	0.102	-0.7	0.147	-0.8
Lab023	0.0609	0.2	0.104	-0.7	0.141	-0.4	0.0585	0.6	0.367	-1.7	0.0155	-1.0	0.104	-0.6	0.158	-0.6
Lab024	NA		0.116	-0.4	NA		0.062	0.9	NA		NA		ND	-3.7	NA	
Lab025	0.052	-0.4	0.112	-0.5	0.187	0.7	0.0381	-1.0	0.62	-0.1	0.006	-2.8	0.0951	-0.9	0.214	0.6
Lab026	0.057	0.0	0.131	0.1	0.159	0.0	0.059	0.7	0.737	0.6	0.024	0.6	0.146	0.7	0.211	0.6
Lab027	0.0617	0.3	0.136	0.3	0.151	-0.2	0.0499	-0.1	0.568	-0.5	0.0304	1.8	0.14	0.5	0.184	0.0
Lab028	NA		NA		0.15	-0.2	0.04	-0.8	NA		NA		NA		NA	
Lab029	0.052	-0.4	0.119	-0.3	0.126	-0.8	0.045	-0.4	0.527	-0.7	0.019	-0.4	0.188	2.1	0.179	-0.1
Lab030	0.057	0.0	0.137	0.3	0.17	0.3	0.065	1.1	0.417	-1.4	0.019	-0.4	0.146	0.7	0.21	0.5
Lab031	NA		NA		0.187	0.7	0.04	-0.8	0.925	1.7	0.021	0.0	NA		0.177	-0.2
Lab032	0.063	0.4	0.124	-0.1	0.1	-1.5	NA		NA		0.022	0.2	0.112	-0.4	0.179	-0.1
Lab033	0.058	0.0	0.147	0.6	0.113	-1.1	0.047	-0.3	0.679	0.2	0.022	0.2	0.12	-0.1	0.185	0.0
Lab034	NA		NA		0.146	-0.3	0.029	-1.7	0.597	-0.3	ND	-3.4	0.062	-2.0	0.215	0.7
Lab035	0.063	0.4	0.135	0.2	0.168	0.3	0.067	1.3	0.625	-0.1	0.018	-0.5	0.107	-0.5	0.196	0.2
Lab036	NA		0.125	-0.1	0.15	-0.2	0.055	0.4	0.65	0.0	0.021	0.0	0.12	-0.1	0.15	-0.8
Lab037	NA		0.122	-0.2	0.163	0.1	0.066	1.2	0.397	-1.5	0.0176	-0.6	0.116	-0.2	0.177	-0.2
Lab038	NA		0.116	-0.4	0.143	-0.4	0.057	0.5	0.688	0.3	0.019	-0.4	0.096	-0.9	0.176	-0.2
Lab039	0.064	0.4	0.18	1.6	0.15	-0.2	0.072	1.7	0.8	1.0	0.017	-0.7	0.15	0.9	0.23	1.0
Lab040	NA		0.2	2.3	0.277	3.0	0.081	2.4	0.774	0.8	0.022	0.2	NA		0.249	1.4
Lab041	0.059	0.1	0.12	-0.2	0.156	0.0	0.059	0.7	0.62	-0.1	0.033	2.3	0.123	0.0	0.195	0.2
Lab042	0.0488	-0.6	0.174	1.5	0.158	0.0	0.0654	1.2	0.728	0.5	0.0245	0.7	0.111	-0.4	0.177	-0.2
Lab043	0.0476	-0.7	0.0862	-1.3	0.0992	-1.5	0.0345	-1.3	0.396	-1.5	0.0224	0.3	0.112	-0.4	0.157	-0.6
Lab044	NA		0.127	0.0	0.171	0.3	0.0435	-0.6	0.641	0.0	0.0097	-2.1	0.0949	-0.9	0.181	-0.1
Lab045	NA		0.115	-0.4	NA		0.05	0.0	NA		NA		NA		0.178	-0.1
Lab046	0.051	-0.5	0.117	-0.3	0.143	-0.4	0.051	0.0	0.614	-0.2	0.017	-0.7	0.111	-0.4	0.168	-0.4
Lab047	NA		NA		0.167	0.2	0.0275	-1.8	0.637	0.0	0.02	-0.2	0.152	0.9	0.21	0.5
Lab048	0.045	-0.9	NA		NA		0.047	-0.3	0.415	-1.4	NA		NA		0.234	1.1

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

Lab Code	Flubendiamide	z score (FFP RSD 25 %)		Fluopyram		Imidacloprid		Iprodione		Lufenuron		Omethoate		Pyraclostrobin		z score (FFP RSD 25 %)		
		MRRL	0.003	0.01	0.01	0.01	0.010	0.010	0.010	0.003	0.010	0.003	0.010	0.010	0.010	0.010	0.010	z score (FFP RSD 25 %)
			Robust mean (mg/kg)															
Lab049	0.056	-0.1	0.092	-1.1	0.12	-1.0	0.037	-1.1	0.43	-1.3	0.012	-1.7	0.18	1.8	0.14	-1.0		
Lab050	0.0464	-0.8	0.108	-0.6	0.108	-1.3	0.0489	-0.1	0.658	0.1	0.0224	0.3	0.111	-0.4	0.156	-0.6		
Lab051	0.0461	-0.8	0.0725	-1.7	0.134	-0.6	0.0558	0.4	0.439	-1.3	0.0269	1.2	0.114	-0.3	0.165	-0.4		
Lab052	ND	-3.3	NA		0.133	-0.6	0.0125	-3.0	0.415	-1.4	0.15	5.0	ND	-3.7	0.183	0.0		
Lab053	0.053	-0.3	0.137	0.3	0.166	0.2	0.037	-1.1	0.841	1.2	0.015	-1.1	0.103	-0.7	0.185	0.0		
Lab054	0.079	1.5	0.123	-0.1	0.172	0.4	0.039	-0.9	0.649	0.0	0.029	1.6	0.126	0.1	0.191	0.1		
Lab055	NA		NA		0.148	-0.2	0.0519	0.1	0.678	0.2	0.0209	0.0	0.211	2.8	0.202	0.4		
Lab056	0.068	0.7	0.147	0.6	0.174	0.4	0.0518	0.1	0.722	0.5	0.02	-0.2	0.115	-0.3	0.216	0.7		
Lab057	0.042	-1.1	0.08	-1.5	0.132	-0.6	0.033	-1.4	0.608	-0.2	0.02	-0.2	0.123	0.0	0.116	-1.5		
Lab058	0.06	0.2	0.111	-0.5	0.18	0.6	0.07	1.5	0.918	1.7	0.039	3.5	0.162	1.3	0.179	-0.1		
Lab059	0.055	-0.2	0.124	-0.1	0.167	0.2	0.054	0.3	0.633	-0.1	0.025	0.8	0.132	0.3	0.203	0.4		
Lab060	NA		0.15	0.7	0.161	0.1	0.0687	1.4	0.721	0.5	0.0194	-0.3	NA		0.225	0.9		
Lab061	0.078	1.4	0.156	0.9	0.167	0.2	0.047	-0.3	0.643	0.0	0.034	2.5	0.129	0.2	0.212	0.6		
Lab062	0.054	-0.2	0.141	0.4	0.112	-1.2	0.075	1.9	0.851	1.3	0.037	3.1	0.135	0.4	0.152	-0.7		
Lab063	0.061	0.2	0.138	0.3	0.175	0.4	0.055	0.4	0.721	0.5	0.018	-0.5	0.113	-0.3	0.202	0.4		
Lab064	NA		0.112	-0.5	NA		NA		NA		0.0169	-0.8	0.09	-1.1	NA			
Lab065	0.067	0.7	0.105	-0.7	0.143	-0.4	0.053	0.2	0.536	-0.7	0.022	0.2	0.092	-1.0	0.15	-0.8		
Lab066	0.061	0.2	0.13	0.1	0.17	0.3	0.052	0.1	0.72	0.5	0.017	-0.7	0.15	0.9	0.19	0.1		
Lab067	0.063	0.4	0.12	-0.2	0.16	0.1	0.056	0.4	0.61	-0.2	0.032	2.1	0.14	0.5	0.16	-0.5		
Lab068	0.096	2.7	0.147	0.6	0.186	0.7	0.056	0.4	0.649	0.0	0.039	3.5	0.163	1.3	0.109	-1.6		
Lab069	0.053	-0.3	ND	-3.7	0.125	-0.8	0.047	-0.3	0.69	0.3	0.011	-1.9	0.094	-0.9	0.177	-0.2		
Lab070	0.063	0.4	0.127	0.0	0.0968	-1.5	0.04	-0.8	0.512	-0.8	0.0145	-1.2	0.135	0.4	0.122	-1.4		
Lab071	NA		0.156	0.9	0.157	0.0	0.053	0.2	0.523	-0.8	0.027	1.2	0.125	0.1	0.255	1.5		
Lab072	0.06	0.2	0.13	0.1	0.16	0.1	0.049	-0.1	0.47	-1.1	0.028	1.4	0.11	-0.4	0.16	-0.5		
Lab073	0.06	0.2	0.112	-0.5	0.175	0.4	0.049	-0.1	0.67	0.2	0.022	0.2	0.14	0.5	0.205	0.4		
Lab074		No results reported																
Lab075	0.053	-0.3	0.146	0.6	0.147	-0.3	0.056	0.4	0.65	0.0	0.017	-0.7	0.149	0.8	0.194	0.2		
Lab076	0.062	0.3	0.1	-0.9	0.158	0.0	0.052	0.1	0.65	0.0	0.018	-0.5	0.114	-0.3	0.199	0.3		
Lab077	0.063	0.4	0.134	0.2	0.161	0.1	NA		0.695	0.3	0.019	-0.4	0.099	-0.8	0.205	0.4		
Lab078	0.054	-0.2	0.174	1.5	0.161	0.1	0.071	1.6	0.7	0.3	0.017	-0.7	0.126	0.1	0.193	0.2		
Lab079	NA		NA		0.193	0.9	NA		0.806	1.0	NA		0.185	2.0	0.253	1.5		
Lab080	NA		0.139	0.4	0.176	0.5	0.047	-0.3	0.758	0.7	0.019	-0.4	0.123	0.0	0.166	-0.4		
Lab081	NA		0.066	-1.9	0.115	-1.1	0.021	-2.3	0.273	-2.3	0.014	-1.3	0.077	-1.5	0.115	-1.5		
Lab082	0.0574	0.0	0.149	0.7	0.174	0.4	0.0523	0.1	0.648	0.0	0.025	0.8	0.15	0.9	0.198	0.3		
Lab083	0.06	0.2	0.127	0.0	0.136	-0.5	0.044	-0.5	0.789	0.9	0.0153	-1.1	0.135	0.4	0.208	0.5		
Lab084	0.053	-0.3	0.083	-1.4	0.205	1.2	0.067	1.3	0.4	-1.5	0.028	1.4	0.109	-0.5	0.192	0.2		
Lab085	0.022	-2.5	0.06	-2.1	2.97	5.0	0.034	-1.3	1.6	5.0	0.1	5.0	0.02	-3.4	0.175	-0.2		
Lab086	0.053	-0.3	0.112	-0.5	0.196	1.0	0.0693	1.5	0.651	0.0	0.028	1.4	0.132	0.3	0.171	-0.3		
Lab087	0.0542	-0.2	0.136	0.3	0.154	-0.1	0.0396	-0.9	0.666	0.1	0.0216	0.1	0.0884	-1.1	0.209	0.5		
Lab088	NA		0.094	-1.1	NA		0.044	-0.5	0.445	-1.2	NA		0.092	-1.0	0.178	-0.1		
Lab089	NA		0.168	1.3	0.16	0.1	0.286	5.0	0.665	0.1	0.0156	-1.0	0.104	-0.6	0.196	0.2		
Lab090	0.07	0.9	0.13	0.1	0.14	-0.4	0.05	0.0	0.64	0.0	0.02	-0.2	0.11	-0.4	0.16	-0.5		
Lab091	0.054	-0.2	0.095	-1.0	0.115	-1.1	0.051	0.0	0.472	-1.1	0.017	-0.7	0.103	-0.7	0.173	-0.3		
Lab092	NA		NA		NA		0.06	0.7	0.57	-0.5	0.02	-0.2	NA		0.2	0.3		
Lab093	0.0581	0.0	NA		0.181	0.6	0.0462	-0.3	0.595	-0.3	0.0172	-0.7	0.105	-0.6	0.226	0.9		
Lab094	0.0655	0.5	0.118	-0.3	0.156	0.0	0.0656	1.2	0.475	-1.0	0.0225	0.3	0.114	-0.3	0.204	0.4		
Lab095	0.0531	-0.3	0.177	1.5	0.429	5.0	0.0702	1.6	0.724	0.5	0.029	1.6	0.182	1.9	0.213	0.6		
Lab096	NA		0.13	0.1	0.139	-0.5	0.051	0.0	0.615	-0.2	0.012	-1.7	0.103	-0.7	0.212	0.6		

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

Lab Code	Flubendiamide	z score (FFP RSD 25 %)	Fluopyram	z score (FFP RSD 25 %)	Imidacloprid	z score (FFP RSD 25 %)	Iprodione	z score (FFP RSD 25 %)	Lufenuron	z score (FFP RSD 25 %)	Omethoate	z score (FFP RSD 25 %)	Propamocarb	z score (FFP RSD 25 %)	Pyraclostrobin	z score (FFP RSD 25 %)
Robust mean (mg/kg)	0.058		0.128		0.158		0.051		0.644		0.021		0.123		0.185	
Lab097	0.062	0.3	0.15	0.7	0.17	0.3	0.065	1.1	0.47	-1.1	0.022	0.2	0.13	0.2	0.23	1.0
Lab098	0.0733	1.1	0.131	0.1	0.169	0.3	0.0543	0.3	0.63	-0.1	0.0176	-0.6	0.115	-0.3	0.17	-0.3
Lab099	0.057	0.0	0.147	0.6	0.215	1.5	0.052	0.1	0.928	1.8	0.026	1.0	0.179	1.8	0.217	0.7
Lab100	0.0698	0.8	0.1532	0.8	0.151	-0.2	NA		NA		0.0333	2.4	0.1191	-0.1	0.1928	0.2
Lab101	0.058	0.0	0.127	0.0	0.151	-0.2	0.0524	0.1	0.493	-0.9	0.0262	1.0	0.143	0.6	0.179	-0.1
Lab102	0.0685	0.8	0.1426	0.5	0.1753	0.5	NA		NA		0.0306	1.9	0.1216	-0.1	0.2037	0.4
Lab103	NA		NA		0.143	-0.4	0.208	5.0	NA		NA		ND	-3.7	NA	
Lab104	NA		NA		0.22	1.6	0.037	-1.1	NA		0.034	2.5	0.22	3.1	0.2	0.3
Lab105	NA		NA		0.252	2.4	0.057	0.5	0.619	-0.2	0.019	-0.4	NA		0.125	-1.3
Lab106	0.0893	2.2	0.161	1.0	0.174	0.4	0.0792	2.3	0.475	-1.0	0.0339	2.5	0.147	0.8	0.262	1.7
Lab107	0.063	0.4	0.126	-0.1	0.164	0.2	0.052	0.1	0.653	0.1	0.022	0.2	0.146	0.7	0.189	0.1
Lab108	NA		0.064	-2.0	0.217	1.5	0.055	0.4	NA		0.027	1.2	0.133	0.3	0.037	-3.2
Lab109	0.066	0.6	NA		0.095	-1.6	ND	-3.2	0.5	-0.9	0.023	0.4	0.267	4.7	0.126	-1.3
Lab110	No results reported															
Lab111	NA		NA		NA		0.042	-0.7	0.53	-0.7	0.018	-0.5	NA		0.177	-0.2
Lab113	NA		NA		0.149	-0.2	0.0312	-1.5	0.646	0.0	0.0111	-1.9	0.0792	-1.4	0.17	-0.3
Lab114	NA		NA		0.146	-0.3	0.055	0.4	0.658	0.1	0.016	-0.9	0.121	-0.1	0.179	-0.1
Lab115	ND	-3.3	0.123	-0.1	0.158	0.0	0.046	-0.4	0.649	0.0	0.029	1.6	NA		0.156	-0.6
Lab116	0.057	0.0	0.1	-0.9	0.166	0.2	0.045	-0.4	0.69	0.3	0.021	0.0	0.13	0.2	0.19	0.1
Lab117	NA		NA		0.12	-1.0	NA		NA		NA		NA		NA	
Lab118	NA		NA		NA		0.0467	-0.3	NA		NA		NA		NA	
Lab119	NA		NA		NA		0.0403	-0.8	NA		0.0143	-1.3	NA		0.158	-0.6
Lab120	0.0525	-0.4	0.125	-0.1	0.146	-0.3	0.0458	-0.4	0.695	0.3	0.0204	-0.1	0.132	0.3	0.179	-0.1
Lab121	0.0563	-0.1	NA		0.133	-0.6	0.0593	0.7	0.574	-0.4	ND	-3.4	NA		0.181	-0.1
Lab122	0.035	-1.6	0.105	-0.7	0.16	0.1	0.064	1.1	0.54	-0.6	0.023	0.4	0.137	0.4	0.14	-1.0
Lab123	0.072	1.0	0.125	-0.1	0.11	-1.2	0.0453	-0.4	0.475	-1.0	0.018	-0.5	0.0691	-1.8	0.185	0.0
Lab124	0.047	-0.7	0.125	-0.1	0.167	0.2	0.05	0.0	0.51	-0.8	0.024	0.6	0.131	0.3	0.199	0.3
Lab126	NA		0.105	-0.7	0.146	-0.3	0.055	0.4	NA		ND	-3.4	ND	-3.7	0.149	-0.8
Lab127	NA		NA		0.136	-0.5	0.05	0.0	0.693	0.3	0.02	-0.2	0.079	-1.4	0.177	-0.2
Lab128	NA		NA		0.137	-0.5	NA		NA		ND	-3.4	NA		0.142	-0.9
Lab129	0.072	1.0	0.125	-0.1	0.19	0.8	0.052	0.1	0.45	-1.2	0.015	-1.1	0.12	-0.1	0.19	0.1
Lab130	0.06	0.2	0.137	0.3	0.155	-0.1	0.062	0.9	0.83	1.2	0.023	0.4	0.14	0.5	0.191	0.1
Lab131	NA		NA		0.15	-0.2	NA		NA		0.038	3.3	0.118	-0.2	0.189	0.1
Lab132	0.064	0.4	0.115	-0.4	0.164	0.2	0.054	0.3	0.614	-0.2	0.023	0.4	0.143	0.6	0.188	0.1
Lab133	0.0527	-0.3	0.117	-0.3	0.154	-0.1	0.0448	-0.5	0.684	0.2	0.0199	-0.2	0.127	0.1	0.173	-0.3
Lab134	No Results Reported															
Lab135	NA		0.147	0.6	0.206	1.2	0.047	-0.3	NA		0.022	0.2	0.129	0.2	0.144	-0.9
Lab136	0.047	-0.7	0.126	-0.1	0.252	2.4	0.045	-0.4	0.673	0.2	ND	-3.4	0.111	-0.4	0.204	0.4
Lab138	0.0469	-0.7	0.107	-0.6	0.171	0.3	0.0398	-0.8	0.6	-0.3	0.027	1.2	0.156	1.1	0.151	-0.7
Lab139	NA		0.126	-0.1	0.161	0.1	0.0527	0.2	0.758	0.7	0.0373	3.2	0.417	5.0	0.2	0.3
Lab140	0.053	-0.3	0.14	0.4	0.175	0.4	0.06	0.7	0.55	-0.6	0.021	0.0	0.141	0.6	0.179	-0.1
Lab141	0.042	-1.1	NA		0.035	-3.1	0.034	-1.3	NA		0.019	-0.4	0.098	-0.8	0.128	-1.2
Lab142	0.0582	0.0	0.11	-0.6	0.146	-0.3	0.0455	-0.4	0.585	-0.4	0.0235	0.5	0.139	0.5	0.122	-1.4
Lab143	NA		0.14	0.4	0.198	1.0	NA		0.725	0.5	0.017	-0.7	0.12	-0.1	0.221	0.8
Lab144	NA		NA		0.17	0.3	0.048	-0.2	NA		0.02	-0.2	0.11	-0.4	0.17	-0.3
Lab145	0.0536	-0.3	0.13	0.1	0.149	-0.2	0.0538	0.3	0.573	-0.4	0.0217	0.2	0.0989	-0.8	0.206	0.5
Lab146	0.0615	0.3	0.144	0.5	0.148	-0.2	0.0425	-0.6	0.41	-1.5	0.018	-0.5	0.11	-0.4	0.19	0.1
Lab147	0.0501	-0.5	0.152	0.8	0.143	-0.4	0.0594	0.7	0.589	-0.3	0.0283	1.4	0.155	1.0	0.218	0.7

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

Lab Code	Flubendiamide	z score (FFP RSD 25 %)		Fluopyram	z score (FFP RSD 25 %)		Imidacloprid	z score (FFP RSD 25 %)		Iprodione	z score (FFP RSD 25 %)		Lufenuron	z score (FFP RSD 25 %)		Omethoate	z score (FFP RSD 25 %)		Propamocarb	z score (FFP RSD 25 %)		Pyraclostrobin	z score (FFP RSD 25 %)	
		MRRL	0.003		0.01	0.01		0.010	0.010		0.003	0.010		0.010	0.021		0.123	0.010		0.185				
Robust mean (mg/kg)	0.058		0.128		0.158		0.051		0.644		0.021		0.123		0.185									
Lab148	0.093	2.5	0.217	2.8	0.232	1.9	0.0556	0.4	1.024	2.4	0.0246	0.7	0.143	0.6	NA									
Lab149	0.0567	-0.1	0.134	0.2	0.169	0.3	0.046	-0.4	0.753	0.7	0.0214	0.1	0.134	0.3	0.206	0.5								
Lab150	0.0571	0.0	0.147	0.6	0.176	0.5	0.0491	-0.1	0.742	0.6	0.0197	-0.2	0.135	0.4	0.249	1.4								
Lab151	0.045	-0.9	0.13	0.1	0.14	-0.4	0.05	0.0	0.56	-0.5	0.017	-0.7	0.084	-1.3	0.17	-0.3								
Lab152	NA		NA		0.13	-0.7	NA		0.81	1.0	0.013	-1.5	0.13	0.2	0.22	0.8								
Lab153	0.065	0.5	NA		0.19	0.8	NA		0.574	-0.4	0.027	1.2	0.15	0.9	0.2	0.3								
Lab154	0.0436	-1.0	0.107	-0.6	0.149	-0.2	0.0388	-0.9	0.556	-0.5	0.0242	0.6	0.132	0.3	0.18	-0.1								
Lab155	NA		0.132	0.1	NA		0.055	0.4	NA		NA		0.098	-0.8	NA									
Lab156	NA		NA		NA		0.05	0.0	NA		ND	-3.4	NA		NA									
Lab158	NA		NA		NA		0.055	0.4	NA		NA		NA		NA									
Lab159	NA		0.131	0.1	0.165	0.2	0.0833	2.6	0.652	0.1	0.0218	0.2	ND	-3.7	0.209	0.5								
Lab160	NA		0.121	-0.2	0.15	-0.2	ND	-3.2	0.606	-0.2	0.056	5.0	0.148	0.8	0.178	-0.1								
Lab161	0.0591	0.1	0.123	-0.1	0.202	1.1	0.0397	-0.9	0.655	0.1	0.0147	-1.2	0.0952	-0.9	0.165	-0.4								
Lab162	NA		NA		NA		0.0800	2.3	NA		NA		NA		NA									
Lab163	NA		NA		0.164	0.2	0.0574	0.5	0.659	0.1	NA		NA		NA									
Lab164	NA		NA		NA		0.044	-0.5	NA		NA		NA		NA									
Lab165	0.056	-0.1	0.123	-0.1	0.13	-0.7	0.05	0.0	0.634	-0.1	0.017	-0.7	0.138	0.5	0.179	-0.1								
Lab166	NA		NA		NA		0.0414	-0.7	NA		0.0135	-1.4	NA		0.183	0.0								
Lab167	0.059	0.1	0.111	-0.5	0.163	0.1	0.0466	-0.3	0.576	-0.4	0.0225	0.3	0.096	-0.9	0.174	-0.2								
Lab169	0.061	0.2	0.142	0.4	0.159	0.0	0.047	-0.3	0.624	-0.1	0.0186	-0.4	0.145	0.7	0.182	-0.1								
Lab170	NA		NA		NA		0.028	-1.8	NA		NA		NA		NA									
Lab171	0.047	-0.7	0.14	0.4	0.176	0.5	0.053	0.2	0.93	1.8	0.026	1.0	0.12	-0.1	0.21	0.5								
Lab172	0.0657	0.6	NA		0.12	-1.0	0.0225	-2.2	0.476	-1.0	0.0152	-1.1	0.131	0.3	0.106	-1.7								
Lab173	0.0705	0.9	0.138	0.3	0.158	0.0	0.0492	-0.1	0.792	0.9	0.022	0.2	0.101	-0.7	0.198	0.3								
Lab174	NA		NA		0.06	-2.5	NA		0.79	0.9	0.013	-1.5	0.21	2.8	0.18	-0.1								
Lab175	NA		NA		0.147	-0.3	0.026	-1.9	0.678	0.2	0.0165	-0.8	0.138	0.5	0.166	-0.4								
Lab176	NA		NA		0.2	1.1	NA		NA		0.0172	-0.7	0.111	-0.4	0.135	-1.1								
Lab177	NA		NA		NA		0.06	0.7	NA		NA		NA		NA									
Lab178							No Results Reported																	
Lab179	0.0592	0.1	0.138	0.3	0.159	0.0	0.0532	0.2	0.696	0.3	0.0182	-0.5	0.105	-0.6	0.195	0.2								

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

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

**Results reported by the laboratories for the voluntary pesticides penflufen and sulfoxaflor (mg/kg) and their calculated z score value using FFP RSD 25 %. The voluntary pesticides are not covered by the ISO/IEC 17043**

Lab Code	Penflufen	z score (FFP RSD 25 %)	Sulfoxaflor	z score (FFP RSD 25 %)
MRL	0.01		0.01	
Robust mean (mg/kg)	0.512		0.032	
Lab001	NA		NA	
Lab002	NA		NA	
Lab003	NA		NA	
Lab004	0.605	0.7	NA	
Lab005	NA		NA	
Lab006	0.5	-0.1	0.039	0.9
Lab007	NA		NA	
Lab008	NA		NA	
Lab009	NA		0.031	-0.1
Lab010	NA		0.033	0.2
Lab011	0.38	-1.0	0.028	-0.5
Lab012	NA		NA	
Lab013	NA		NA	
Lab014	NA		NA	
Lab015	0.495	-0.1	0.03	-0.2
Lab016	0.509	0.0	NA	
Lab017	0.675	1.3	0.0385	0.9
Lab018	NA		NA	
Lab019	0.468	-0.3	NA	
Lab020	0.894	3.0	0.021	-1.3
Lab021	NA		0.0269	-0.6
Lab022	NA		NA	
Lab023	NA		NA	
Lab024	NA		NA	
Lab025	NA		0.0264	-0.7
Lab026	0.57	0.5	0.03	-0.2
Lab027	0.478	-0.3	0.0368	0.7
Lab028	NA		NA	
Lab029	0.4	-0.9	0.027	-0.6
Lab030	0.534	0.2	0.038	0.8
Lab031	NA		NA	
Lab032	NA		NA	
Lab033	NA		0.031	-0.1
Lab034	NA		NA	
Lab035	NA		0.041	1.2
Lab036	0.46	-0.4	NA	
Lab037	0.459	-0.4	0.025	-0.8
Lab038	NA		NA	
Lab039	NA		NA	
Lab040	NA		NA	
Lab041	0.51	0.0	0.034	0.3
Lab042	NA		0.03	-0.2
Lab043	NA		NA	
Lab044	NA		NA	
Lab045	NA		NA	
Lab046	NA		NA	



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

Lab Code	Penflufen	z score (FFP RSD 25 %)	Sulfoxaflor	z score (FFP RSD 25 %)
			0.01	
Robust mean (mg/kg)	0.512		0.032	
Lab047	NA		NA	
Lab048	NA		NA	
Lab049	NA		NA	
Lab050	NA		NA	
Lab051	0.396	-0.9	0.0252	-0.8
Lab052	NA		NA	
Lab053	0.658	1.1	0.029	-0.3
Lab054	NA		NA	
Lab055	NA		NA	
Lab056	NA		NA	
Lab057	0.283	-1.8	0.035	0.4
Lab058	ND	-3.9	ND	-2.7
Lab059	0.38	-1.0	0.031	-0.1
Lab060	NA		NA	
Lab061	NA		NA	
Lab062	NA		NA	
Lab063	0.574	0.5	0.051	2.5
Lab064	NA		NA	
Lab065	NA		NA	
Lab066	0.56	0.4	0.029	-0.3
Lab067	0.47	-0.3	0.023	-1.1
Lab068	NA		NA	
Lab069	ND	-3.9	ND	-2.7
Lab070	0.523	0.1	NA	
Lab071	NA		NA	
Lab072	0.52	0.1	0.035	0.4
Lab073	NA		0.033	0.2
Lab074	No Results Reported			
Lab075	0.569	0.4	NA	
Lab076	0.48	-0.3	0.029	-0.3
Lab077	0.642	1.0	0.033	0.2
Lab078	NA		0.034	0.3
Lab079	NA		NA	
Lab080	NA		NA	
Lab081	NA		NA	
Lab082	NA		NA	
Lab083	0.466	-0.4	NA	
Lab084	NA		NA	
Lab085	NA		NA	
Lab086	NA		0.035	0.4
Lab087	0.578	0.5	NA	
Lab088	NA		NA	
Lab089	0.508	0.0	NA	
Lab090	NA		NA	
Lab091	NA		NA	
Lab092	NA		NA	
Lab093	NA		NA	
Lab094	NA		NA	
Lab095	NA		0.036	0.6
Lab096	NA		NA	

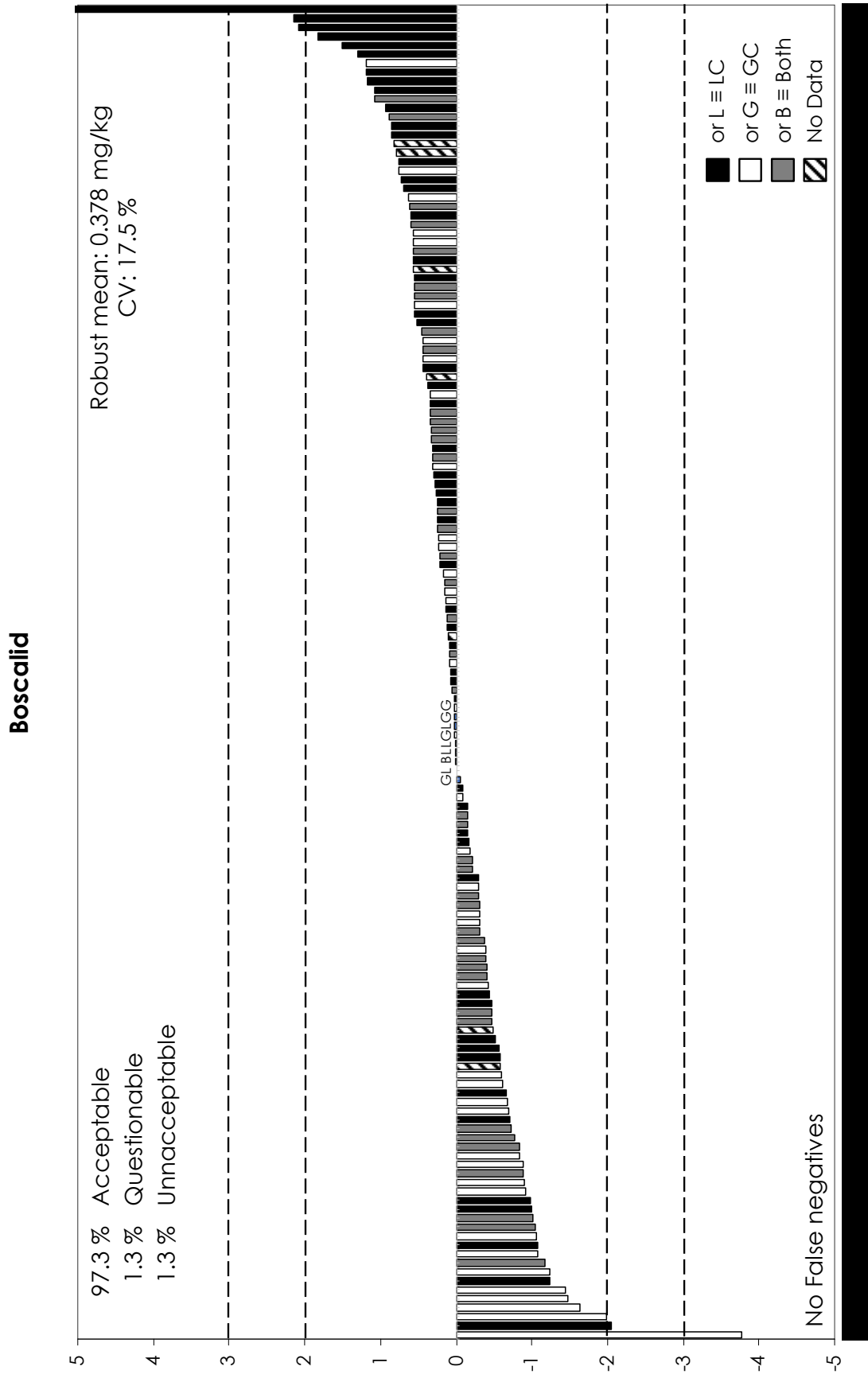
APPENDIX 3. Results (mg/Kg) and z scores for FFP RSD (25 %).

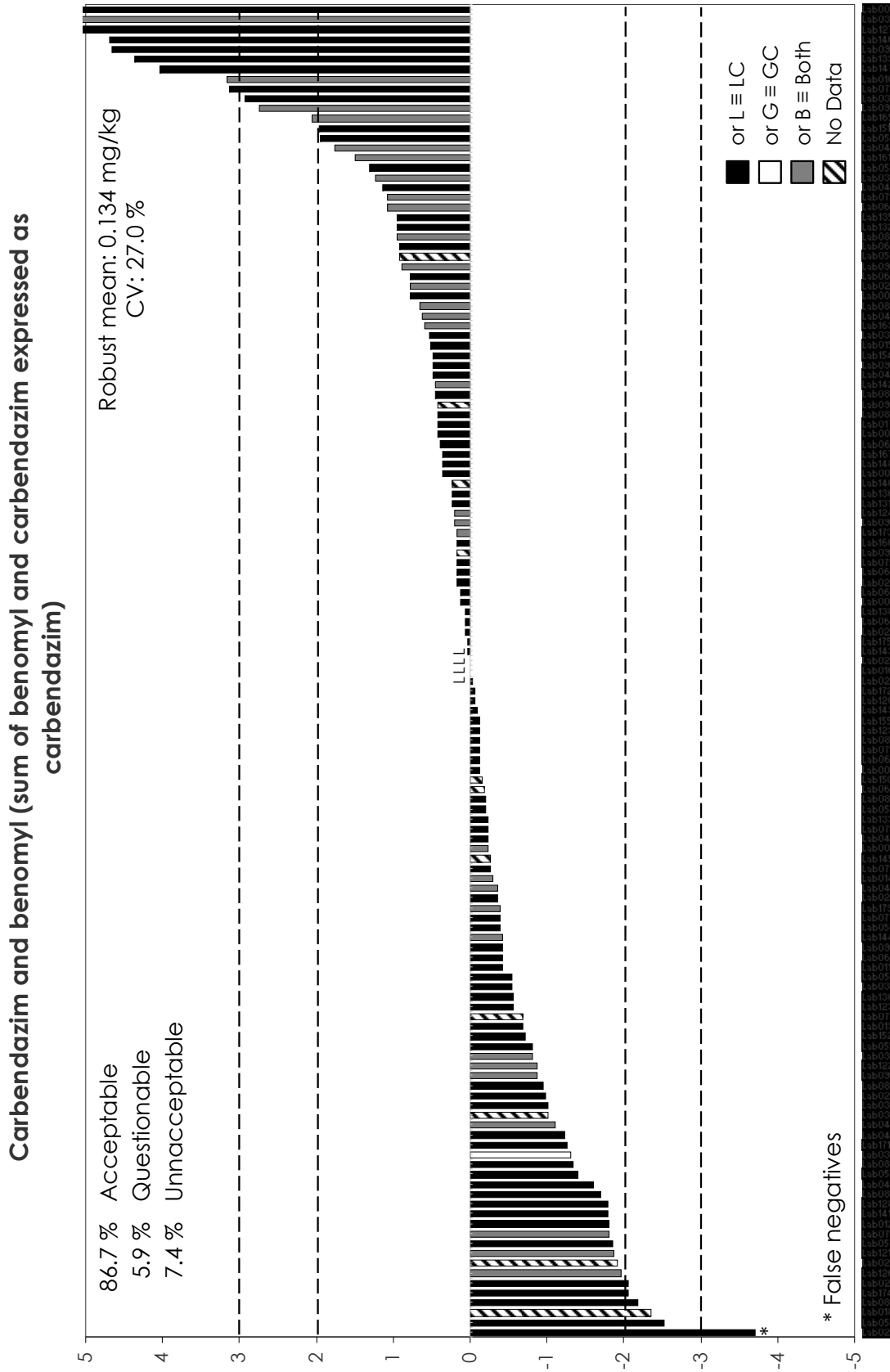
Lab Code	Penflufen	z score (FFP RSD 25 %)	Sulfoxafior	z score (FFP RSD 25 %)
MRRL	0.01		0.01	
Robust mean (mg/kg)	0.512		0.032	
Lab097	NA		ND	-2.7
Lab098	0.487	-0.2	0.0312	0.0
Lab099	NA		0.023	-1.1
Lab100	NA		NA	
Lab101	NA		0.0309	-0.1
Lab102	NA		NA	
Lab103	NA		NA	
Lab104	NA		NA	
Lab105	NA		NA	
Lab106	NA		0.0437	1.5
Lab107	NA		NA	
Lab108	NA		NA	
Lab109	NA		NA	
Lab110	No Results Reported			
Lab111	NA		NA	
Lab113	NA		NA	
Lab114	NA		NA	
Lab115	NA		0.044	1.6
Lab116	NA		NA	
Lab117	NA		NA	
Lab118	NA		NA	
Lab119	NA		NA	
Lab120	0.495	-0.1	NA	
Lab121	NA		NA	
Lab122	NA		NA	
Lab123	NA		NA	
Lab124	0.602	0.7	0.03	-0.2
Lab126	NA		NA	
Lab127	NA		NA	
Lab128	NA		NA	
Lab129	NA		NA	
Lab130	0.587	0.6	0.031	-0.1
Lab131	NA		NA	
Lab132	0.561	0.4	0.03	-0.2
Lab133	0.488	-0.2	NA	
Lab134	No Results Reported			
Lab135	NA		NA	
Lab136	0.516	0.0	0.026	-0.7
Lab138	NA		NA	
Lab139	NA		NA	
Lab140	0.451	-0.5	0.034	0.3
Lab141	NA		NA	
Lab142	NA		NA	
Lab143	NA		NA	
Lab144	NA		NA	
Lab145	0.535	0.2	0.0285	-0.4
Lab146	NA		NA	
Lab147	0.546	0.3	0.0512	2.5
Lab148	NA		NA	
Lab149	0.489	-0.2	NA	

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

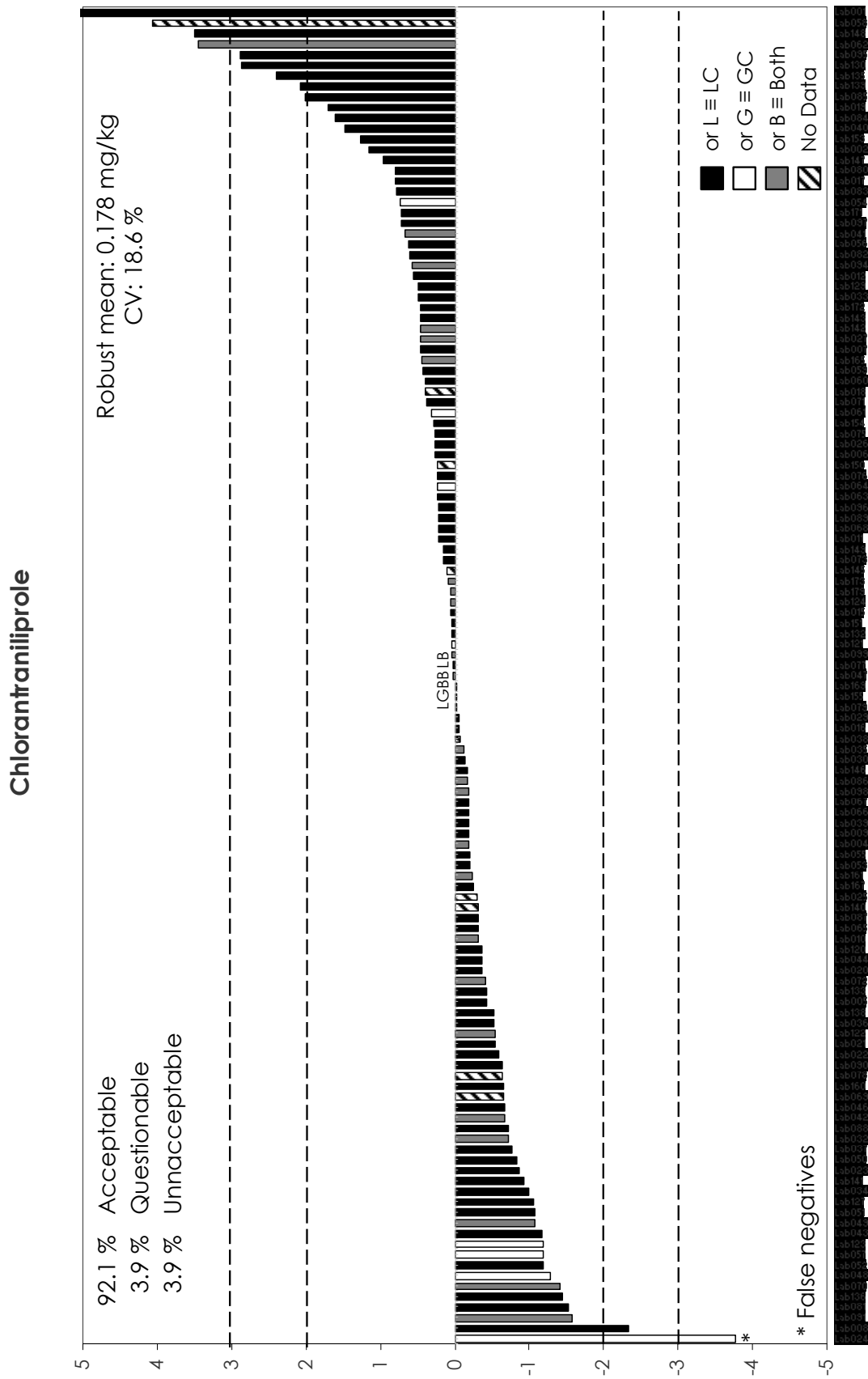
Lab Code	Penflufen	z score (FFP RSD 25 %)	Sulfoxafior	z score (FFP RSD 25 %)
MRRL	0.01		0.01	
Robust mean (mg/kg)	0.512		0.032	
Lab150	NA		NA	
Lab151	NA		0.033	0.2
Lab152	NA		NA	
Lab153	NA		NA	
Lab154	NA		NA	
Lab155	NA		NA	
Lab156	NA		NA	
Lab158	NA		NA	
Lab159	NA		NA	
Lab160	0.44	-0.6	NA	
Lab161	NA		NA	
Lab162	NA		NA	
Lab163	NA		NA	
Lab164	NA		NA	
Lab165	NA		NA	
Lab166	NA		NA	
Lab167	NA		0.0363	0.6
Lab169	NA		NA	
Lab170	NA		NA	
Lab171	NA		NA	
Lab172	NA		NA	
Lab173	NA		NA	
Lab174	NA		NA	
Lab175	NA		NA	
Lab176	NA		NA	
Lab177	NA		NA	
Lab178	No Results Reported			
Lab179	0.491	-0.2	NA	

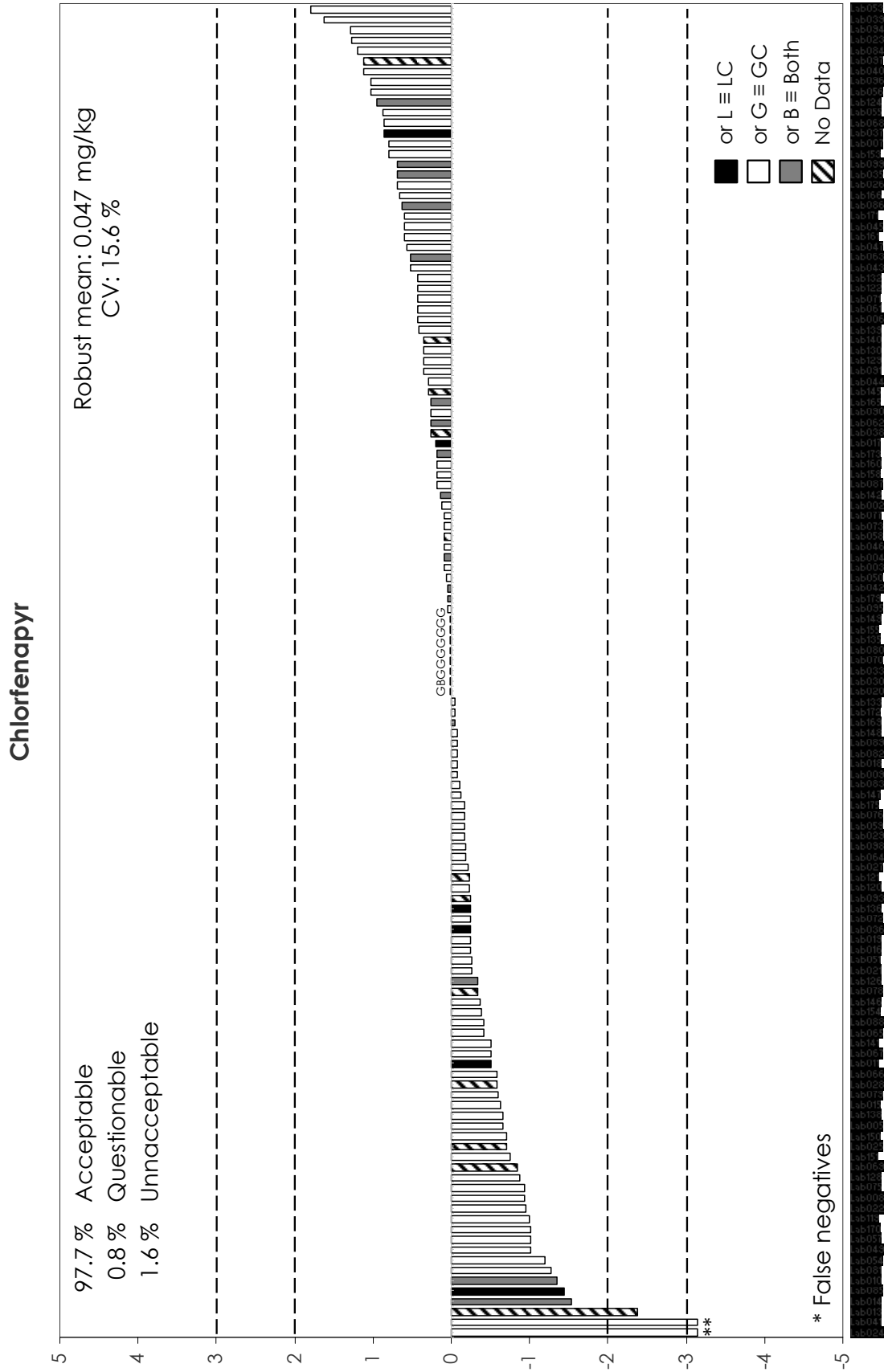
NA: Not analysed  
 ND: Not detected (False negative)



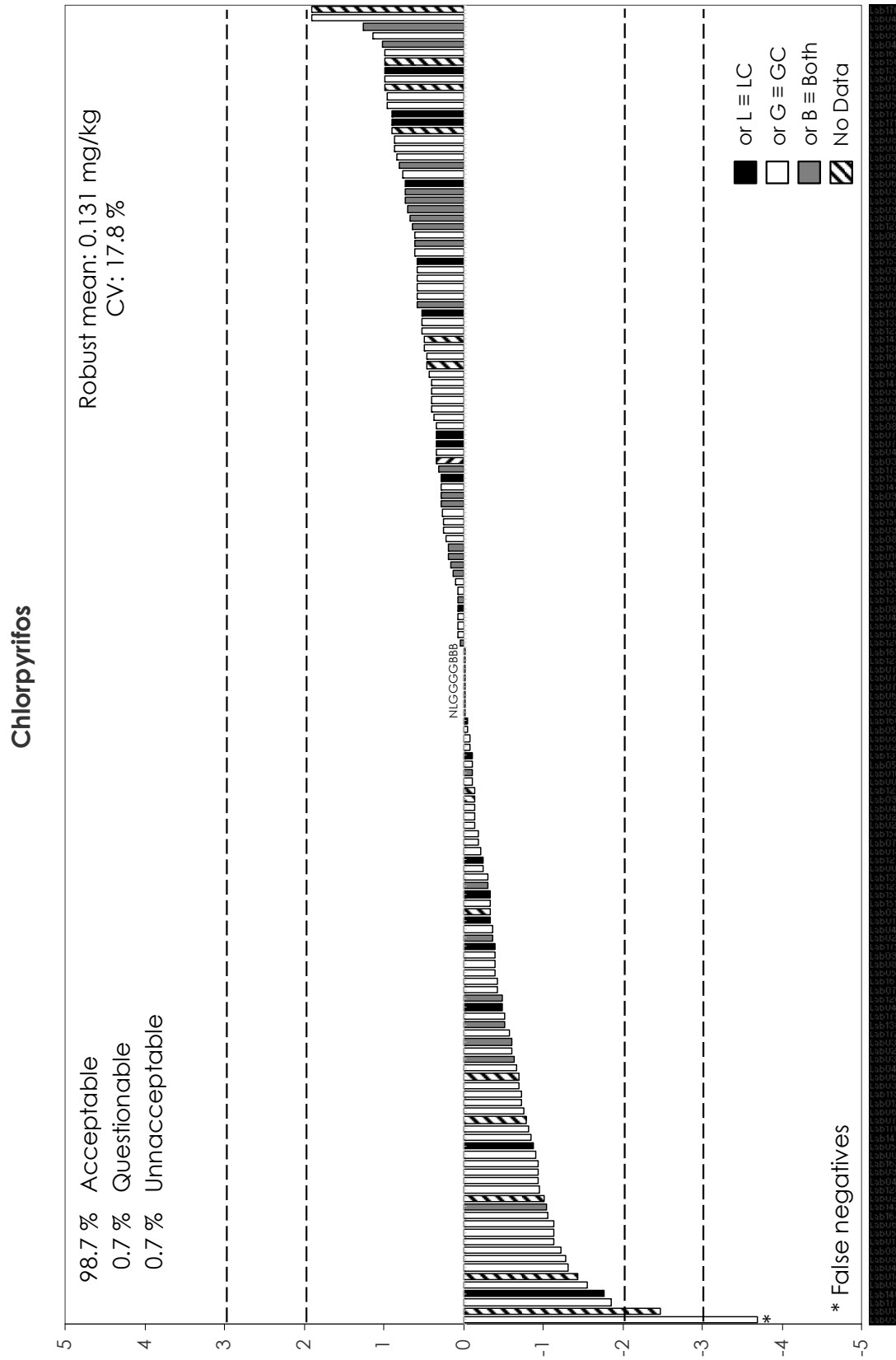


APPENDIX 4. Graphical representation of z scores for FFP RSD (25 %).

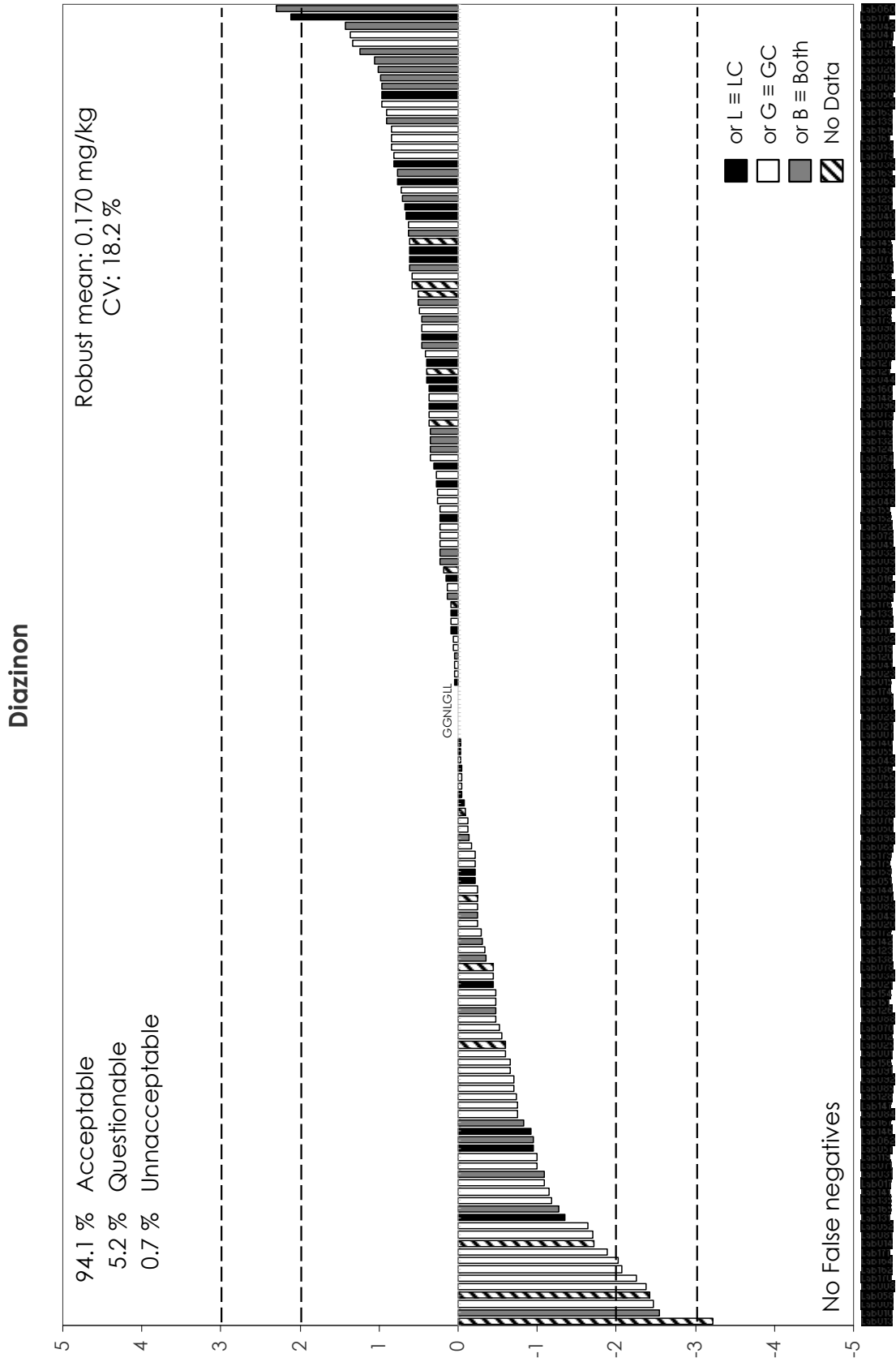




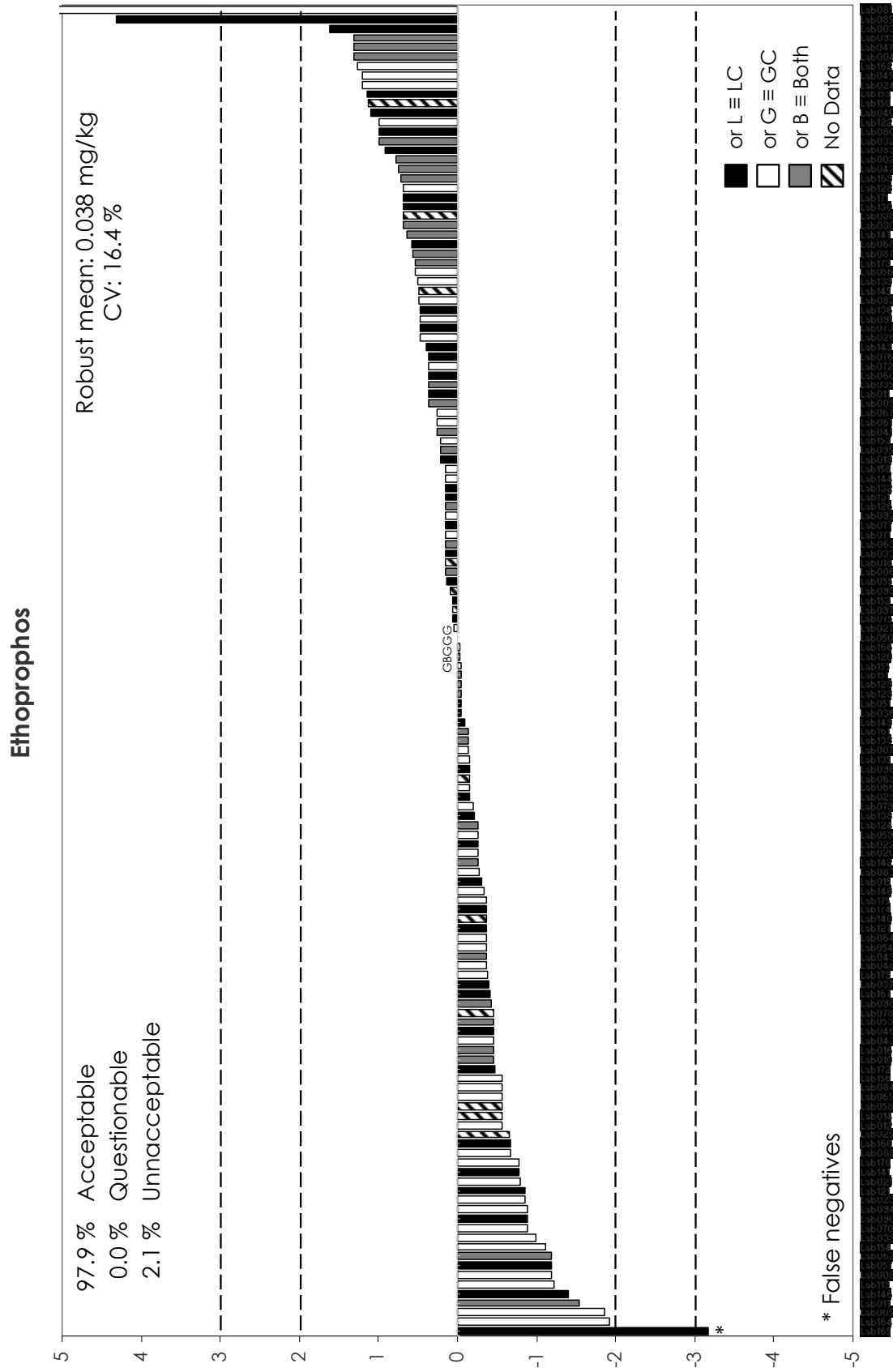
APPENDIX 4. Graphical representation of z scores for FFP RSD (25 %).

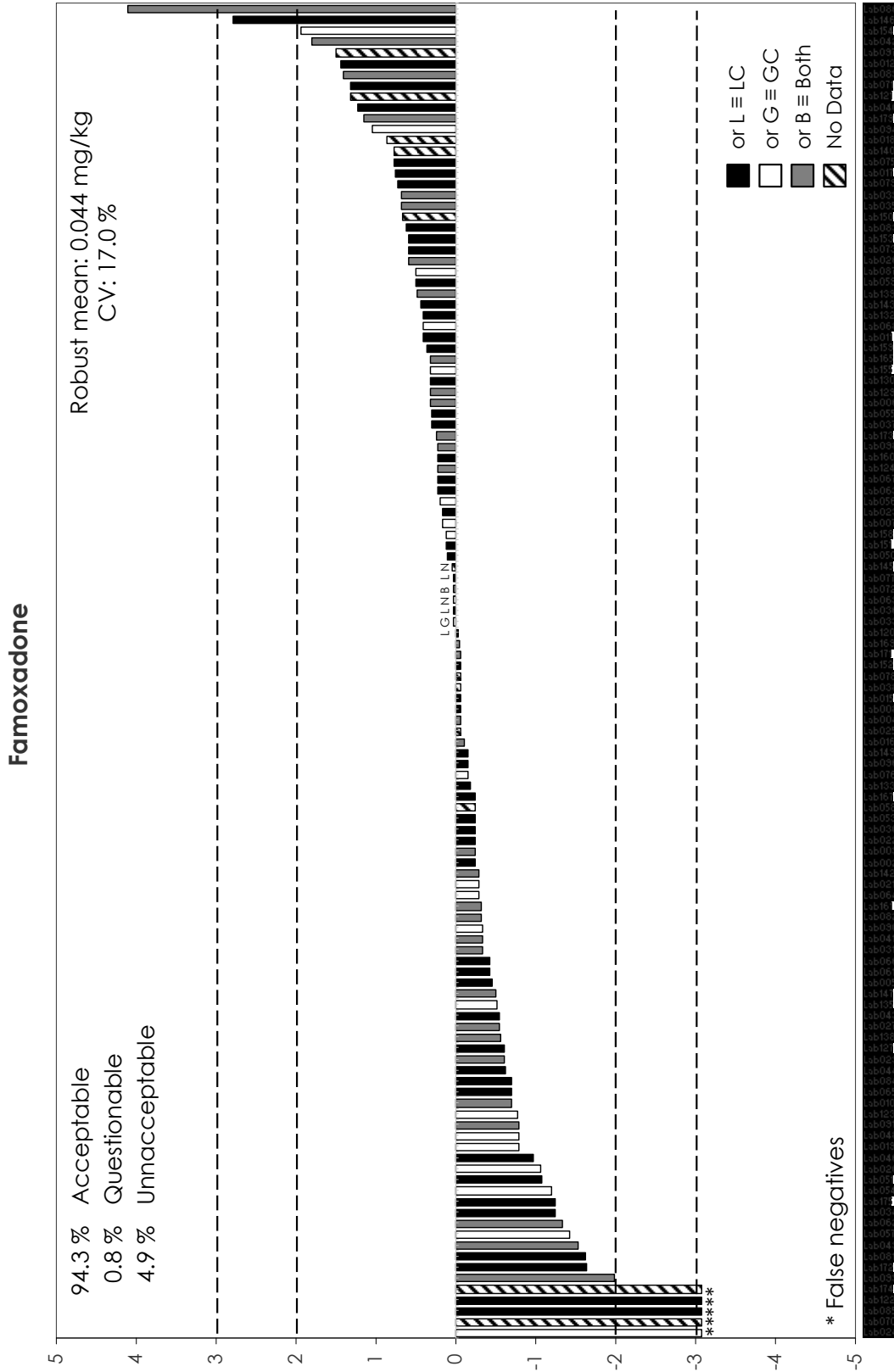




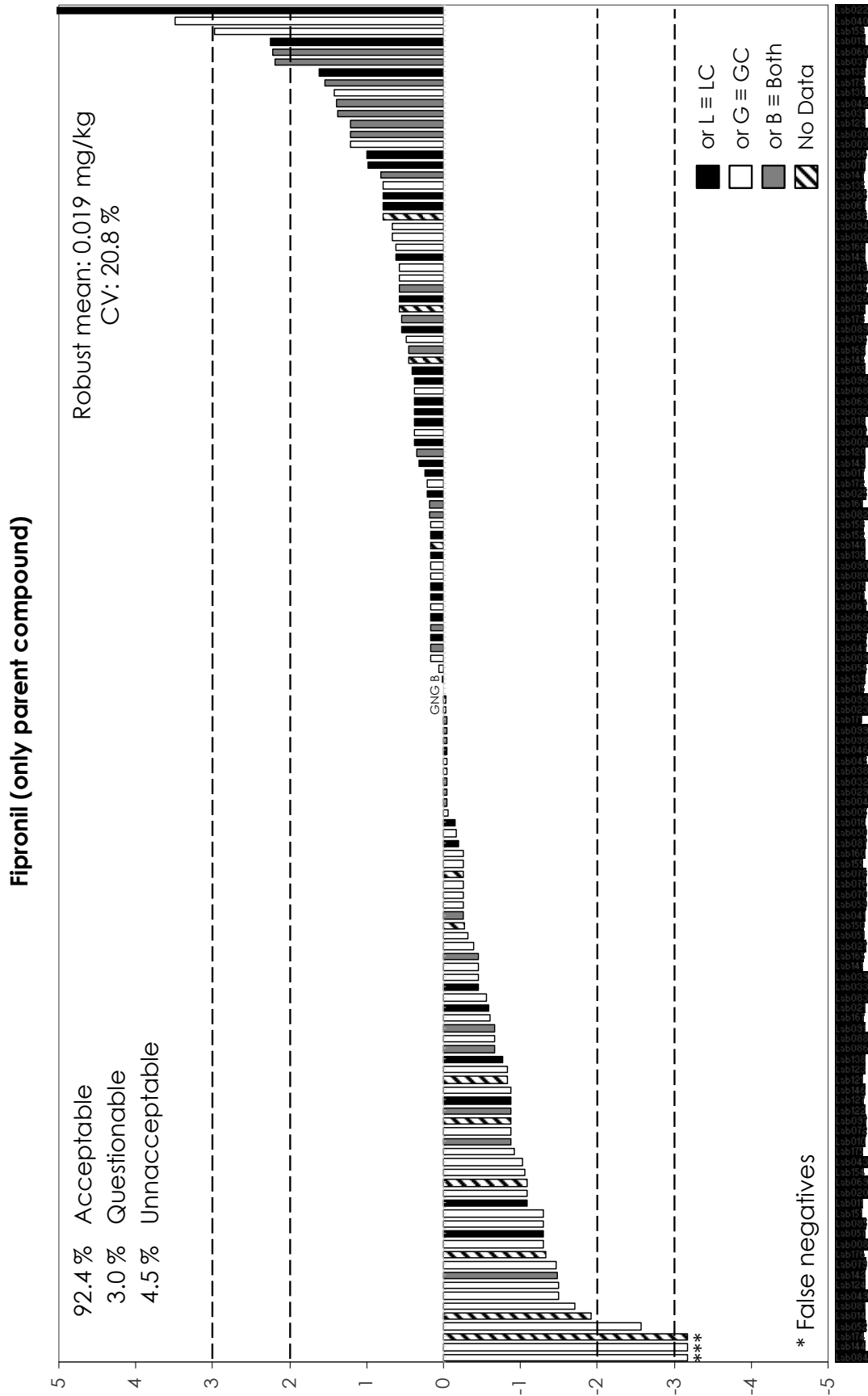


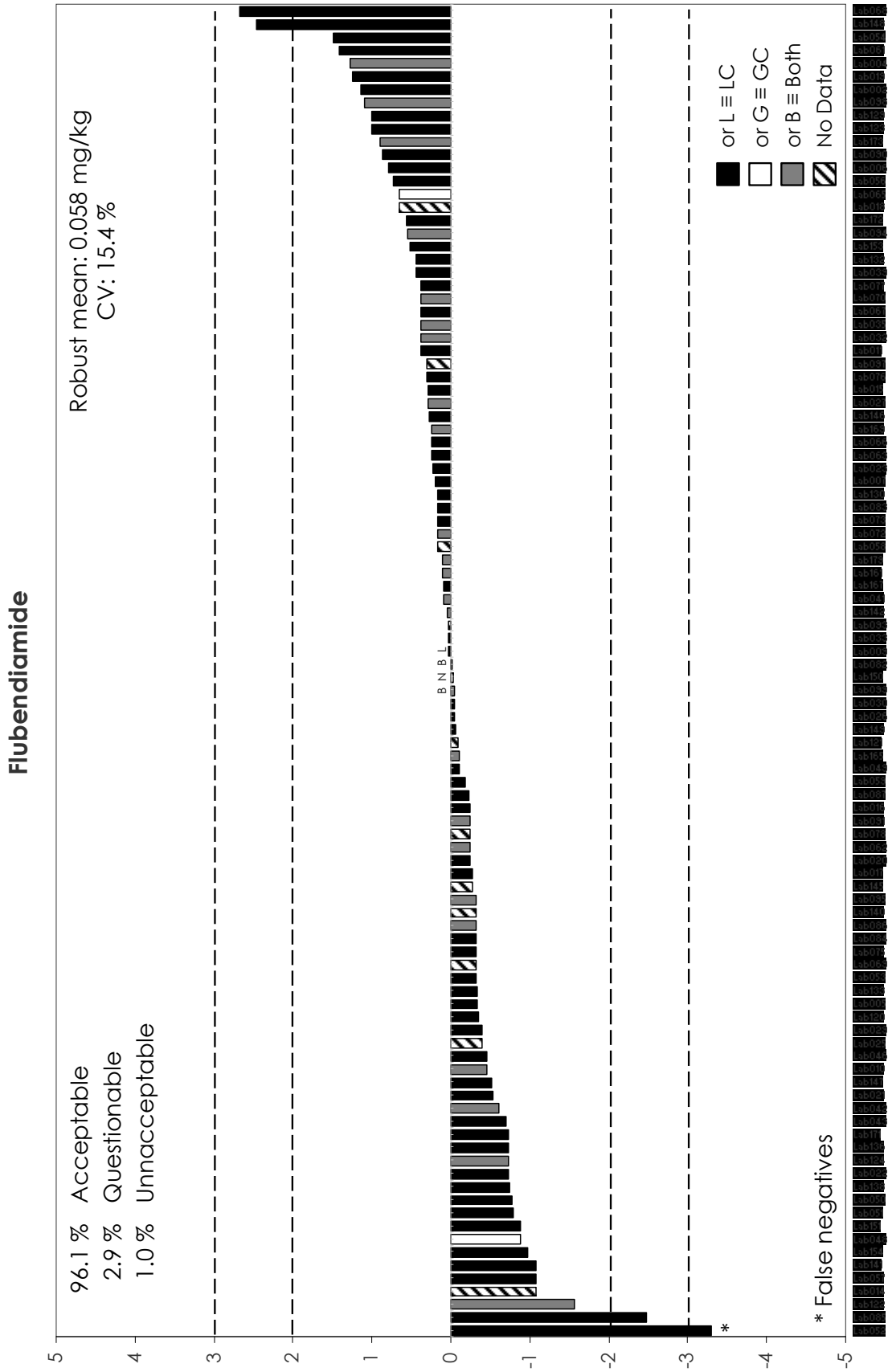
APPENDIX 4. Graphical representation of z scores for FFP RSD (25 %).



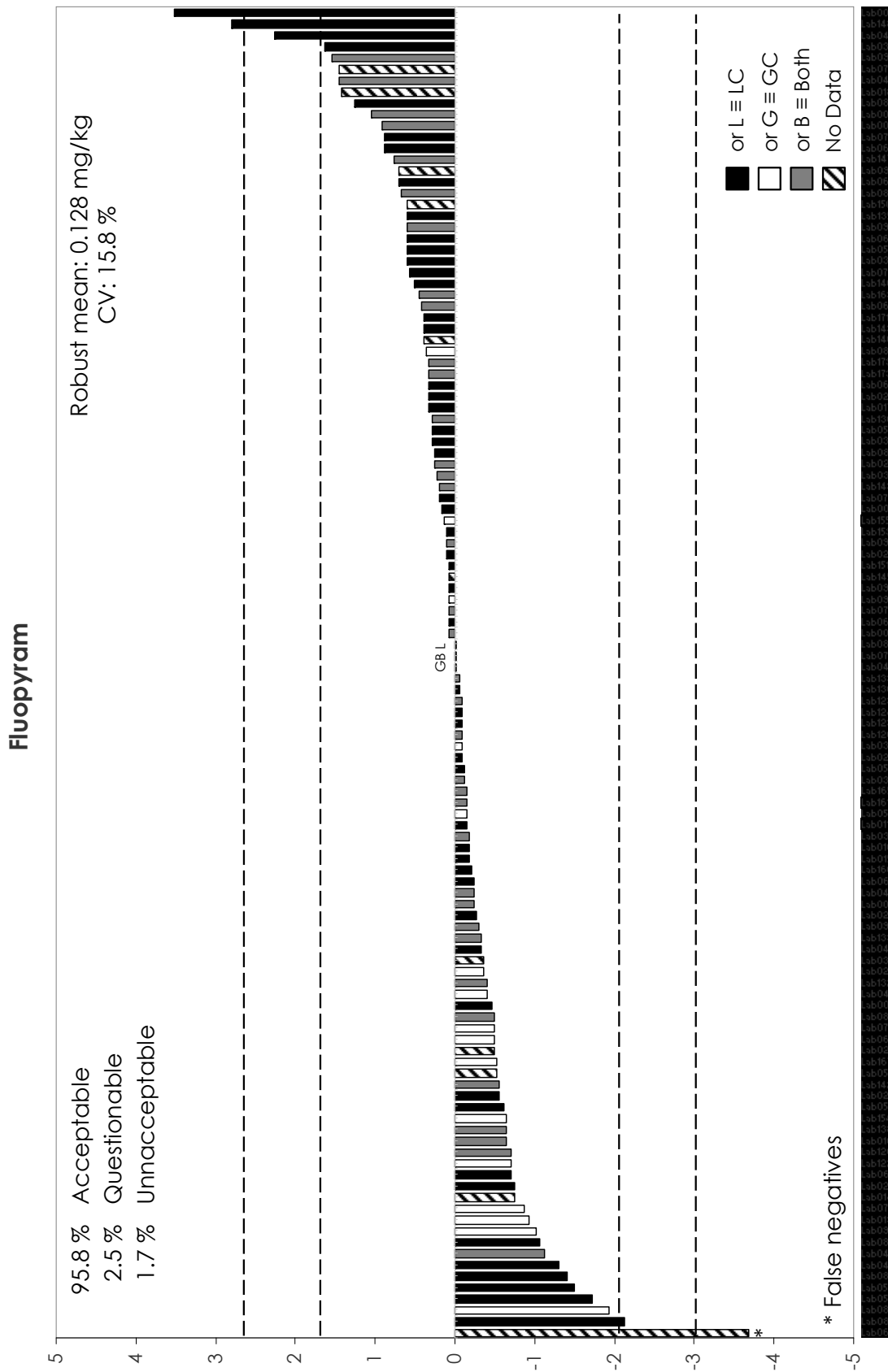


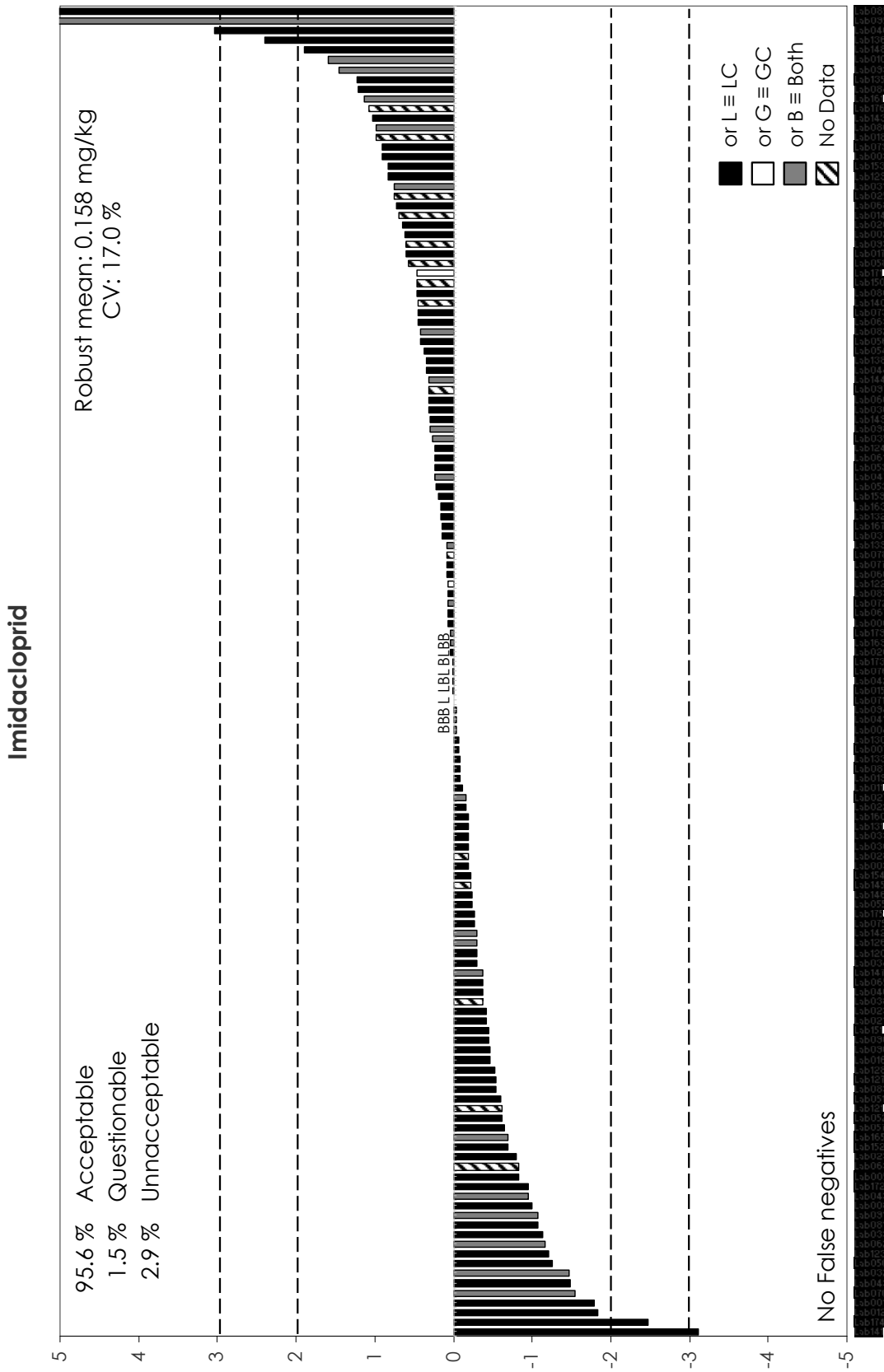
APPENDIX 4. Graphical representation of z scores for FFP RSD (25 %).



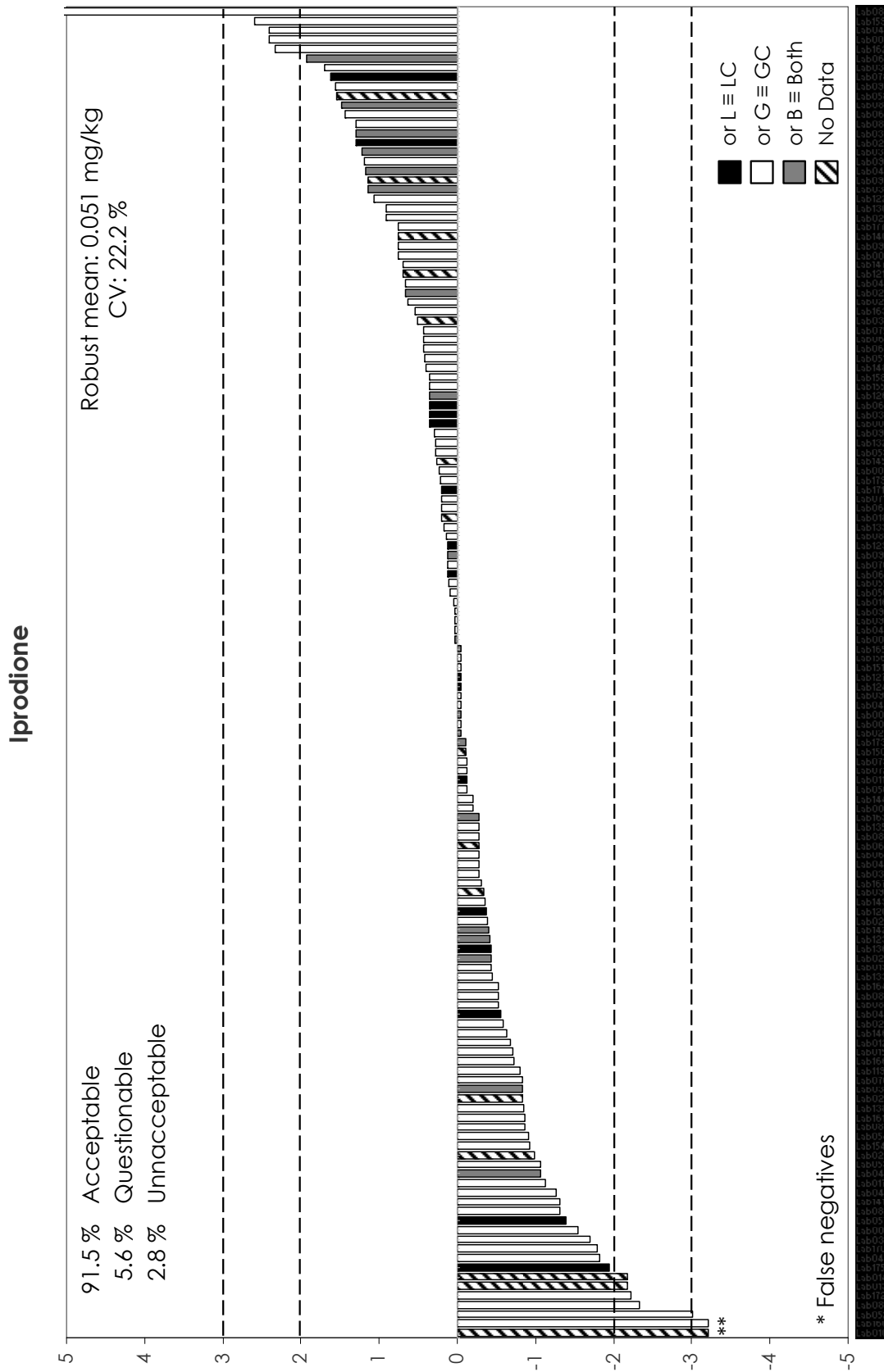


APPENDIX 4. Graphical representation of z scores for FFP RSD (25 %).



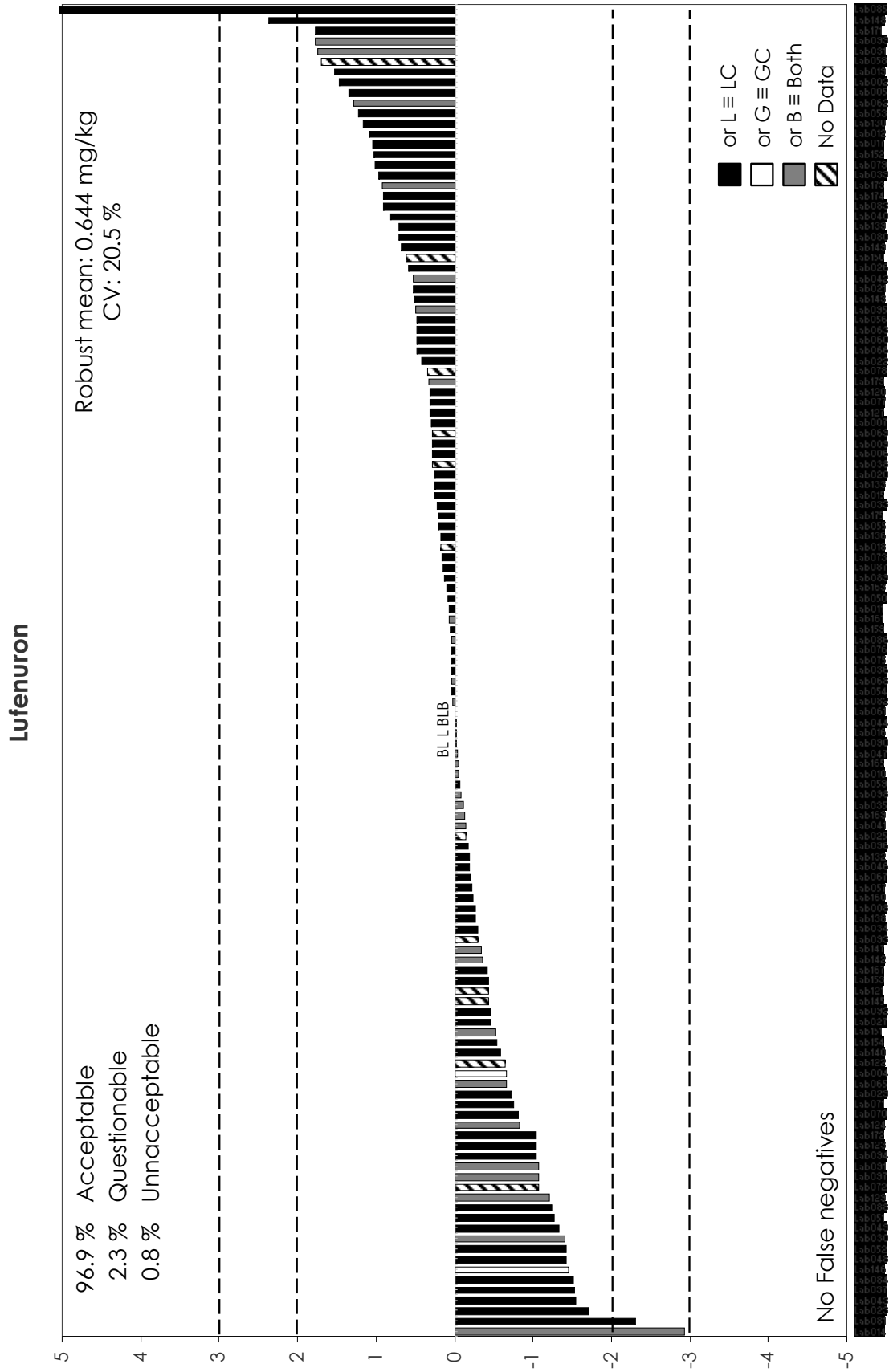


APPENDIX 4. Graphical representation of z scores for FFP RSD (25 %).

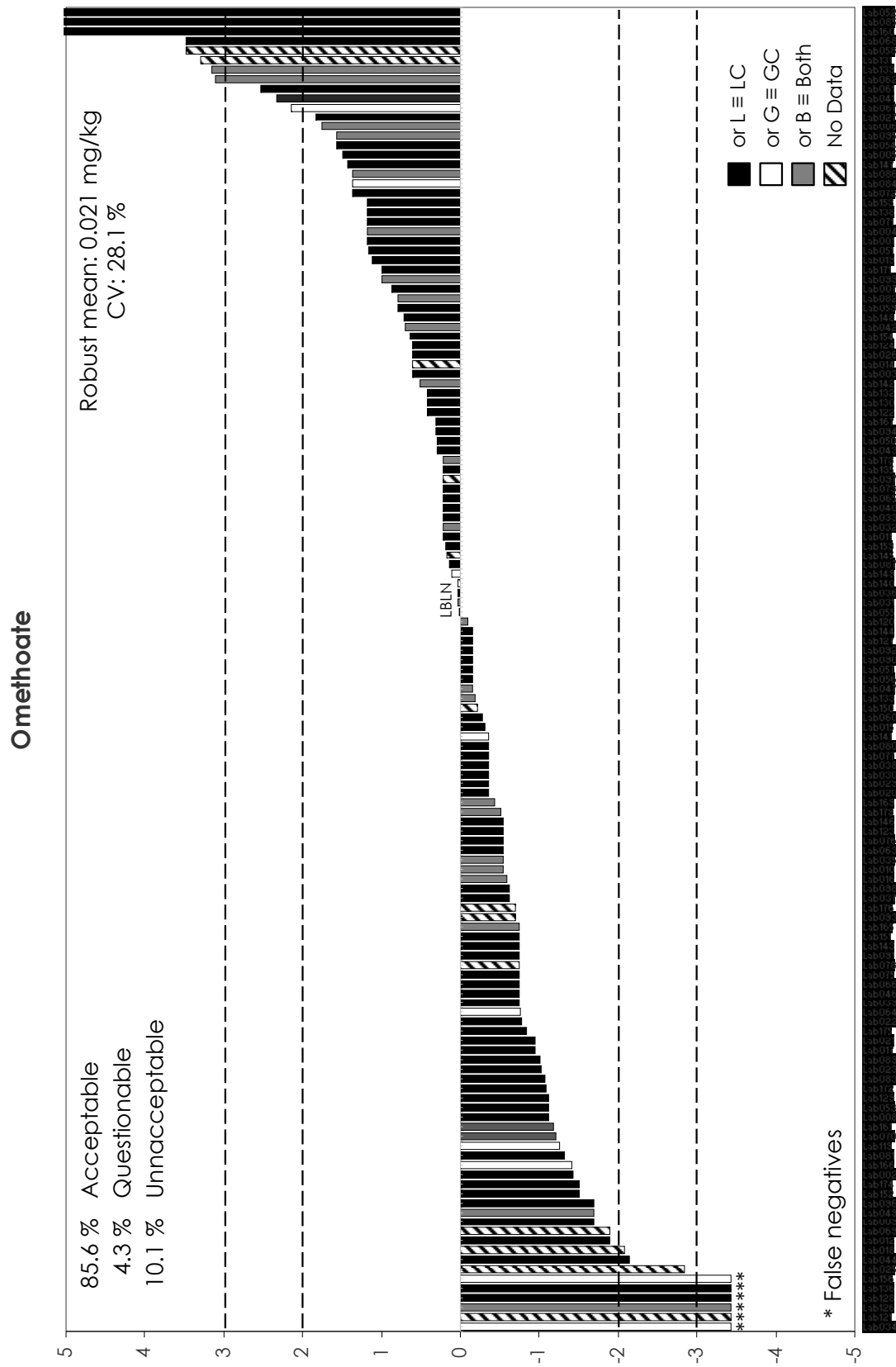




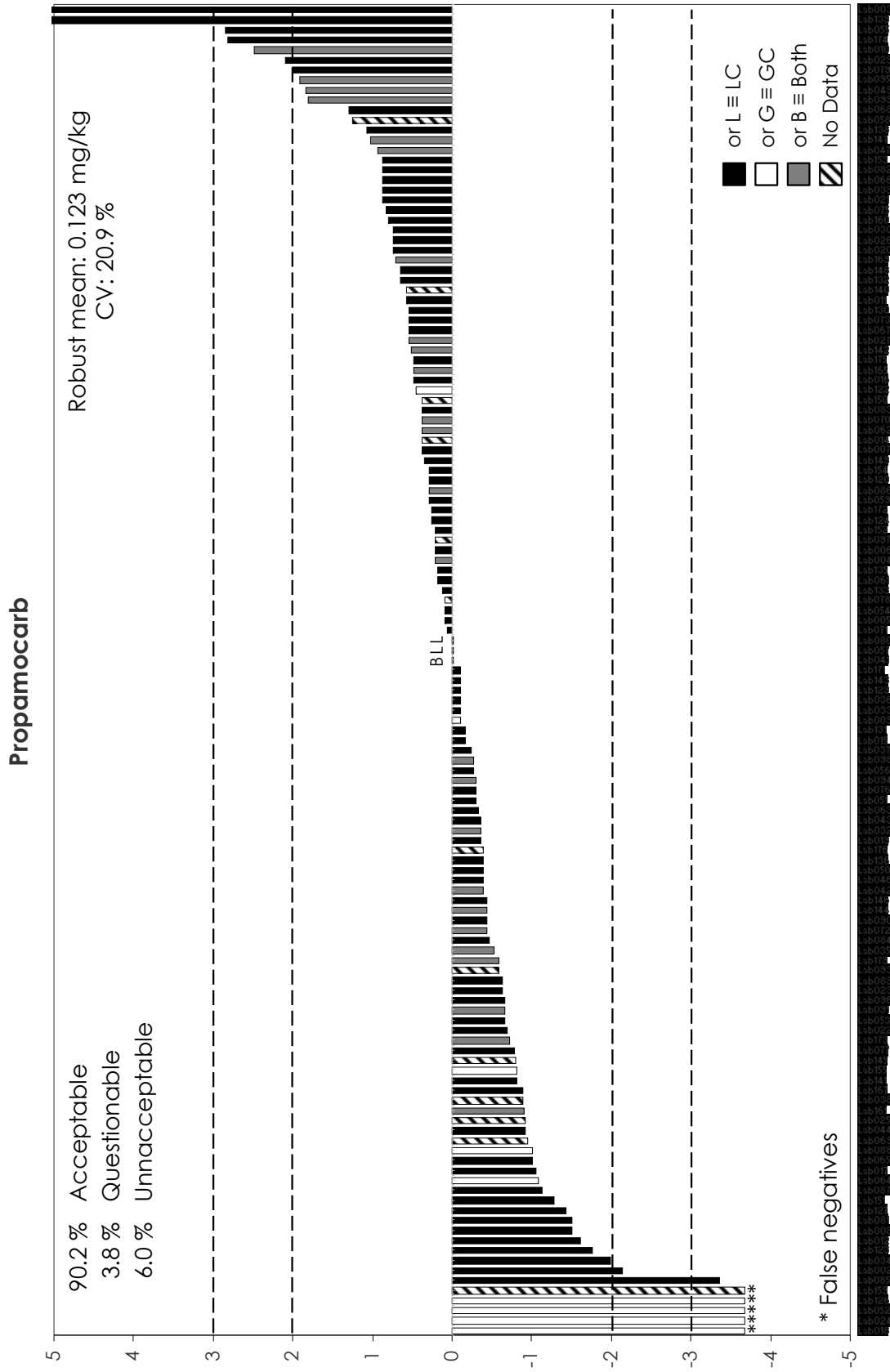
APPENDIX 4. Graphical representation of z scores for FFP RSD (25 %).



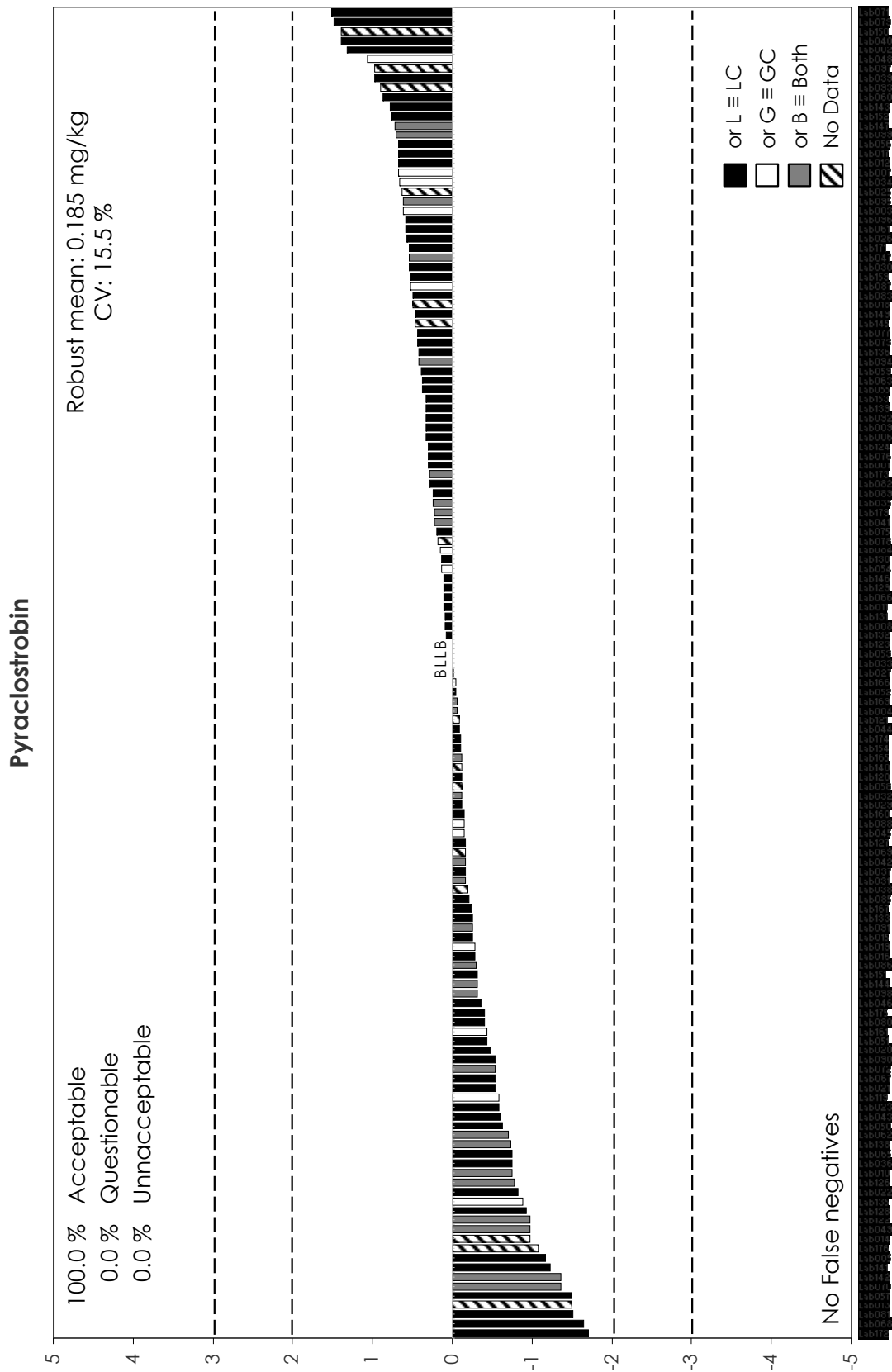
APPENDIX 4. Graphical representation of z scores for FFP RSD (25 %).



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

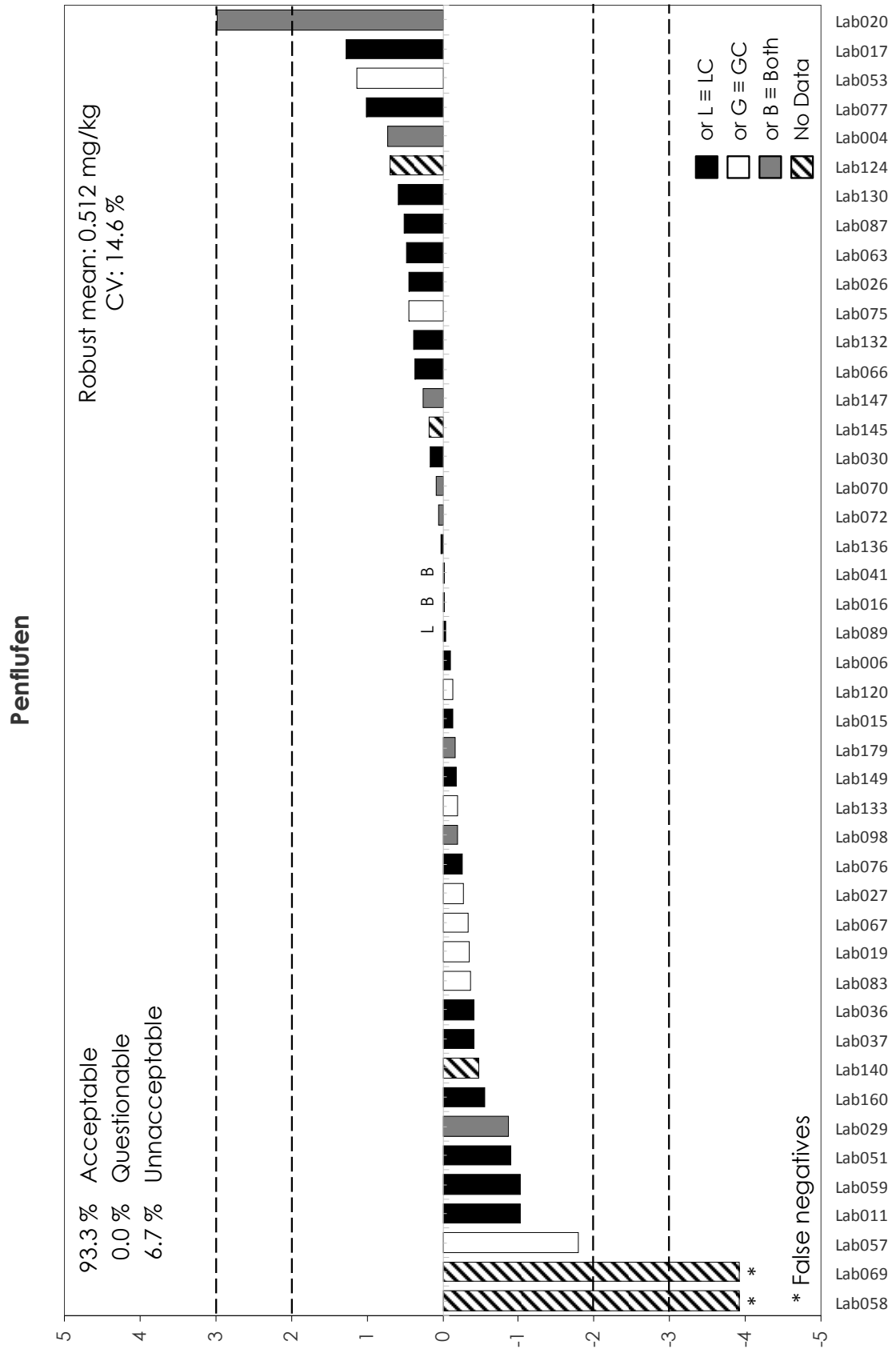


APPENDIX 4. Graphical representation of z scores for FFP RSD (25 %).

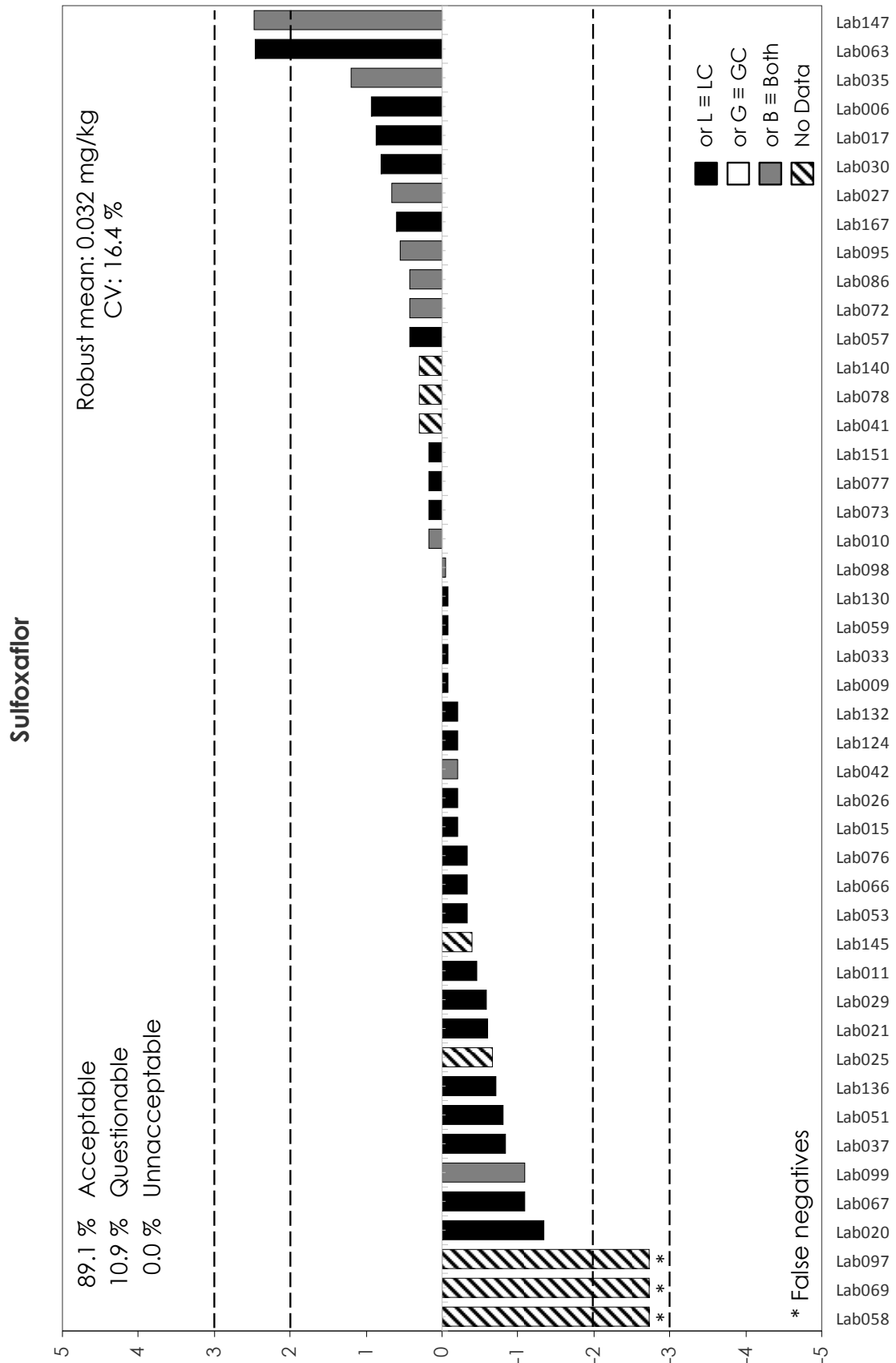


APPENDIX 4. Graphical representation of z scores for FFP RSD (25 %).

Voluntary Pesticides (not covered by the ISO/IEC 17043)



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



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

Lab Code	Boscalid	Carbendazim and benomyl (expressed as carbendazim)	Chlorantranilprole	Chlorfenapyr	Chlorpyrifos	Diazinon	Ethiofophos	Famoxadone	Fipronil (only parent compound)	Flubendiamide	Fluopyram	Imidacloprid	Iprodione	Lufenuron	Ometoate	Propamocarb	Pyraclostrobin	No. of z scores	AZ <sup>2</sup>
	z scores																		
Lab002	0.6	0.1	1.2	0.1	0.9	0.6	0.8	-0.2	0.7	1.1	1.0	0.6	0.4	1.5	-1.4	-2.1	1.3	17	1.0
Lab004	0.0	-0.2	-0.2	0.1	0.7	1.0	1.3	-0.1	0.0	1.3	0.9	0.0	0.0	-0.7	1.2	0.2	-0.1	17	0.4
Lab005	-1.0	0.4	-0.4	-0.7	-0.9	-2.5	-1.9	-0.4	-0.1	-0.3	-0.5	-0.8	2.4	1.3	1.5	0.1	-1.2	17	1.4
Lab006	0.3	-0.1	0.3	0.4	0.6	0.5	0.2	0.3	0.4	0.8	-0.2	0.1	0.7	0.3	1.8	0.2	0.3	17	0.3
Lab007	0.0	0.4	0.5	0.8	0.6	0.0	-0.1	0.2	0.4	0.2	0.2	-0.1	0.2	0.3	0.9	0.4	0.7	17	0.2
Lab009	0.3	0.8	-0.2	-0.1	0.3	0.2	0.4	-0.2	0.2	0.0	0.1	-0.2	0.0	0.3	-1.7	-0.1	0.3	17	0.2
Lab010	-0.4	3.2	-0.3	-1.4	-0.1	-2.5	-0.5	-0.7	-0.9	-0.5	-0.6	1.6	-3.2	-0.1	-0.5	2.5	-0.8	17	2.4
Lab011	0.1	-0.4	0.2	-0.5	-0.3	0.1	0.4	0.4	-1.1	0.4	-0.1	-0.1	-0.1	0.1	0.2	0.6	0.1	17	0.2
Lab015	0.0	0.5	0.1	-0.6	0.1	0.0	-0.3	-0.1	0.0	0.3	-0.2	0.0	-0.7	0.2	-0.3	-0.2	-0.3	17	0.1
Lab016	-0.3	0.0	0.0	-0.2	0.2	0.2	-0.5	-0.1	-0.1	-0.2	-0.2	-0.5	0.0	0.0	-0.6	0.5	-0.3	17	0.1
Lab017	1.3	0.4	0.8	0.2	-1.1	-1.1	-1.5	0.8	0.2	-0.3	0.3	0.6	-1.1	1.0	-0.9	-1.1	0.7	17	0.8
Lab018	0.9	-2.4	0.4	-0.1	1.0	0.4	0.2	0.9	0.4	0.7	1.4	1.0	0.2	0.2	-2.1	-3.7	0.5	17	1.8
Lab019	-0.2	-0.7	1.7	-0.2	-0.2	0.1	0.2	-0.8	2.2	1.2	-0.9	-0.1	-0.4	1.5	1.1	-0.4	-0.3	17	0.9
Lab020	0.1	0.2	-0.4	0.0	-0.1	-0.2	-0.3	-1.1	-1.1	-0.2	0.3	0.6	1.3	0.3	-0.4	0.7	-0.5	17	0.3
Lab021	-0.4	0.0	-0.5	-0.3	-0.6	-0.5	-0.8	-0.3	-0.6	-0.5	-0.6	-0.4	-0.4	0.5	-0.9	0.9	-0.5	17	0.3
Lab022	-0.7	0.0	-0.6	-0.9	0.1	-0.1	-0.3	-0.2	5.0	-0.7	-0.1	-0.2	-0.6	0.4	-0.8	-0.7	-0.8	17	1.7
Lab023	-0.5	-0.4	-0.9	1.3	-0.2	0.0	-0.9	0.2	0.0	0.2	-0.7	-0.4	0.6	-1.7	-1.0	-0.6	-0.6	17	0.6
Lab025	0.6	-2.1	-0.3	-0.7	-1.0	-0.6	-0.7	-0.1	0.2	-0.4	-0.5	0.7	-1.0	-0.1	-2.8	-0.9	0.6	17	1.1
Lab026	0.6	0.1	0.3	0.7	0.6	1.0	0.5	0.6	0.6	0.0	0.1	0.0	0.7	0.6	0.6	0.7	0.6	17	0.3
Lab027	-0.1	0.8	0.5	-0.2	-0.2	0.0	0.0	-0.6	-0.4	0.3	0.3	-0.2	-0.1	-0.5	1.8	0.5	0.0	17	0.3
Lab029	-0.5	-1.0	0.0	-0.2	-0.4	-0.1	-0.2	-0.6	0.0	-0.4	-0.3	-0.8	-0.4	-0.7	-0.4	2.1	-0.1	17	0.5
Lab030	0.4	0.5	-0.1	0.0	-0.6	1.1	0.7	-0.2	1.2	0.0	0.3	0.3	1.1	-1.4	-0.4	0.7	0.5	17	0.5
Lab033	0.3	-0.5	0.5	0.0	-0.7	0.3	0.2	0.0	-0.5	0.0	0.6	-1.1	-0.3	0.2	0.2	-0.1	0.0	17	0.2
Lab035	-0.1	5.0	-0.1	0.7	0.7	0.6	0.4	0.7	0.6	0.4	0.2	0.3	1.3	-0.1	-0.5	-0.5	0.2	17	1.8
Lab036	0.1	-1.3	-0.5	-0.2	0.8	0.8	0.0	0.0	0.4		-0.1	-0.2	0.4	0.0	0.0	-0.1	-0.8	16	0.3
Lab037	0.0	2.9	-0.8	0.9	0.0	0.0	-0.4	0.3	2.2		-0.2	0.1	1.2	-1.5	-0.6	-0.2	-0.2	16	1.2
Lab038	0.6	-1.0	-0.1	0.3	0.3	-0.1	0.7	-0.1	0.0		-0.4	-0.4	0.5	0.3	-0.4	-0.9	-0.2	16	0.2
Lab039	1.2	-1.0	-0.2	1.6	0.4	0.3	-1.0	0.7	-0.5	0.4	1.6	-0.2	1.7	1.0	-0.7	0.9	1.0	17	0.9
Lab040	2.1	1.1	1.5	1.1	1.9	0.7	1.1	1.2	3.5		2.3	3.0	2.4	0.8	0.2		1.4	15	3.4
Lab041	0.1	1.8	0.7	-3.1	0.1	0.0	0.3	-0.3	-0.3	0.1	-0.2	0.0	0.7	-0.1	2.3	0.0	0.2	17	1.2
Lab042	0.5	-0.4	-0.7	0.1	1.0	1.4	0.7	1.8	1.4	-0.6	1.5	0.0	1.2	0.5	0.7	-0.4	-0.2	17	0.8
Lab043	-2.0	-1.6	-1.2	0.5	-0.2	0.0	-0.5	-0.6	-1.0	-0.7	-1.3	-1.5	-1.3	-1.5	0.3	-0.4	-0.6	17	1.1
Lab046	-0.2	-0.2	-0.7	0.1	-0.4	0.3	-0.5	-1.0	0.0	-0.5	-0.3	-0.4	0.0	-0.2	-0.7	-0.4	-0.4	17	0.2
Lab049	-0.3	-1.1	-1.1	-1.0	-1.3	-0.2	-0.4	-1.5	-1.5	-0.1	-1.1	-1.0	-1.1	-1.3	-1.7	1.8	-1.0	17	1.3
Lab050	0.0	-2.5	-0.8	0.1	1.0	-0.1	0.0	0.1	-0.2	-0.8	-0.6	-1.3	-0.1	0.1	0.3	-0.4	-0.6	17	0.7
Lab051	-0.7	-0.2	-1.1	-0.3	-0.1	-0.2	0.0	-1.1	-0.3	-0.8	-1.7	-0.6	0.4	-1.3	1.2	-0.3	-0.4	17	0.6
Lab053	0.6	1.9	-0.2	1.8	0.9	0.8	-0.5	-0.2	0.2	-0.3	0.3	0.2	-1.1	1.2	-1.1	-0.7	0.0	17	0.8
Lab054	0.1	-0.5	0.7	-1.2	-1.1	0.3	-0.4	-1.2	0.1	1.5	-0.1	0.4	-0.9	0.0	1.6	0.1	0.1	17	0.6
Lab055	0.2	-0.8	-0.2	0.9	-0.1	0.0	0.1	0.3	0.4			-0.2	0.1	0.2	0.0	2.8	0.4	15	0.7
Lab056	0.6	-0.4	0.6	1.0	1.1	1.0	0.5	0.2	1.0	0.7	0.6	0.4	0.1	0.5	-0.2	-0.3	0.7	17	0.4
Lab057	-1.2	-1.9	-1.2	-1.0	-0.9	-0.9	-1.2	-1.4	-1.3	-1.1	-1.5	-0.6	-1.4	-0.2	-0.2	0.0	-1.5	17	1.3
Lab058	-0.6	0.9	4.1	0.1	0.5	-2.4	-0.6	-0.2	0.8	0.2	-0.5	0.6	1.5	1.7	3.5	1.3	-0.1	17	2.6
Lab059	0.0	1.3	0.4	-0.2	-0.4	0.1	-0.3	0.5	-0.3	-0.2	-0.1	0.2	0.3	-0.1	0.8	0.3	0.4	17	0.2
Lab061	0.3	0.1	0.3	0.4	0.6	0.7	0.3	-0.4	0.8	1.4	0.9	0.2	-0.3	0.0	2.5	0.2	0.6	17	0.7
Lab062	-0.7	1.1	3.5	0.3	0.1	-0.9	-1.2	-1.3	0.2	-0.2	0.4	-1.2	1.9	1.3	3.1	0.4	-0.7	17	2.1

**APPENDIX 5. Average of the Squared z scores (AZ<sup>2</sup>) for laboratories in Category A.**

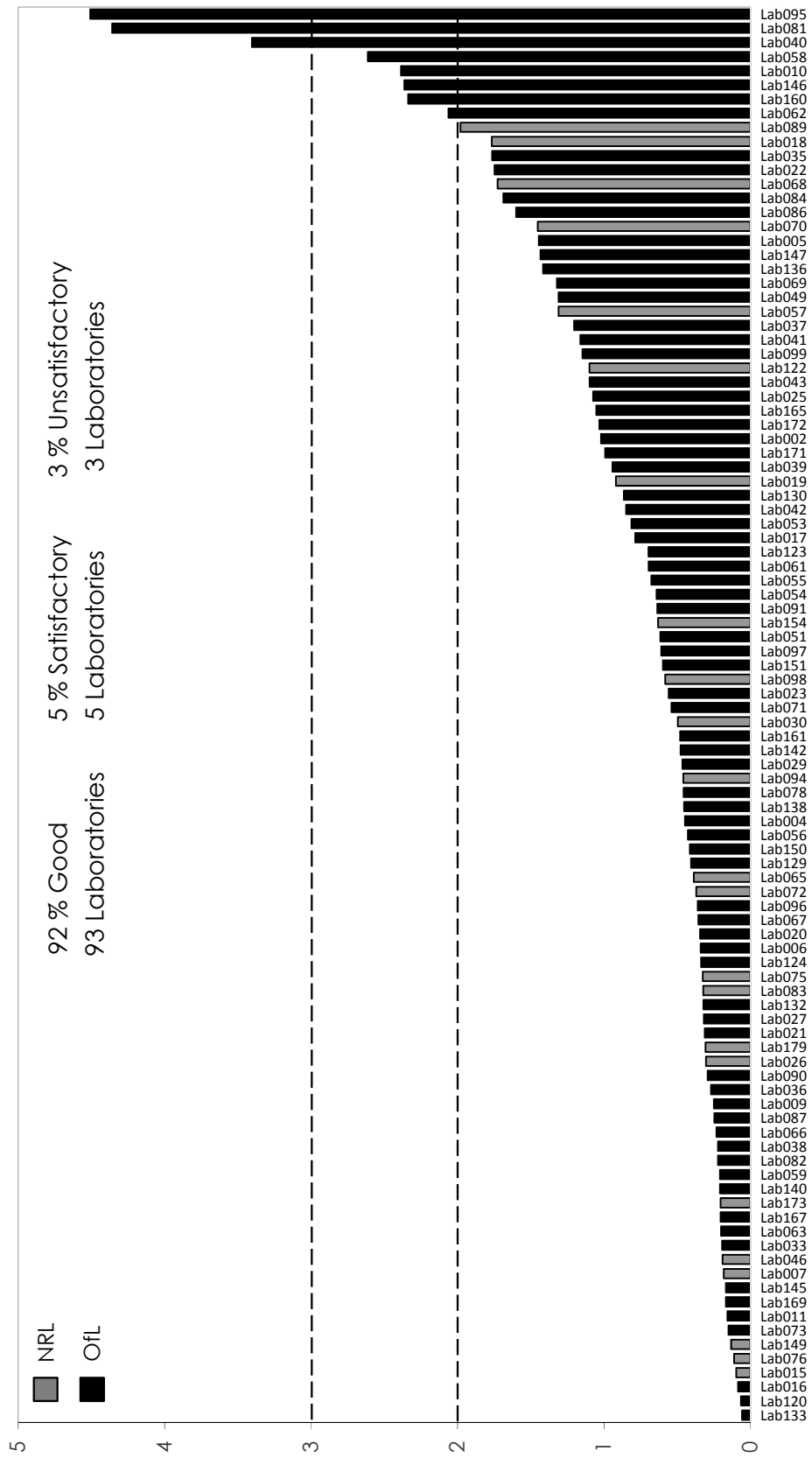
Lab Code	Boscalid	Carbendazim and benomyl (expressed as carbendazim)	Chlorantraniliprole	Chlorfenapyr	Chlorpyrifos	Diazinon	Ethiofiprophos	Famoxadone	Fipronil (only parent compound)	Flubendiamide	Fluopyram	Imidacloprid	Iprodione	Lufenuron	Omethoate	Propamocarb	Pyraclostrobin	No. of z scores	AZ <sup>2</sup>
Lab063	0.7	0.1	0.2	0.5	0.7	0.8	0.2	0.2	0.4	0.2	0.3	0.4	0.4	0.5	-0.5	-0.3	0.4	17	0.2
Lab065	-0.5	0.4	-0.3	-0.4	-1.1	-0.2	-0.6	-0.7	0.8	0.7	-0.7	-0.4	0.2	-0.7	0.2	-1.0	-0.8	17	0.4
Lab066	-0.7	0.8	-0.2	-0.6	-0.8	-0.1	-0.2	-0.4	0.2	0.2	0.1	0.3	0.1	0.5	-0.7	0.9	0.1	17	0.2
Lab067	-0.1	-0.1	-0.2	-0.5	0.0	0.0	0.3	0.2	0.2	0.4	-0.2	0.1	0.4	-0.2	2.1	0.5	-0.5	17	0.4
Lab068	0.9	0.9	0.2	0.9	0.8	1.0	0.6	0.4	0.4	2.7	0.6	0.7	0.4	0.0	3.5	1.3	-1.6	17	1.7
Lab069	-0.5	-0.2	-0.7	-0.8	-0.7	0.6	-0.2	0.0	-1.1	-0.3	-3.7	-0.8	-0.3	0.3	-1.9	-0.9	-0.2	17	1.3
Lab070	-1.2	-1.8	-1.4	0.0	-0.2	-0.5	-0.9	-3.1	-0.3	0.4	0.0	-1.5	-0.8	-0.8	-1.2	0.4	-1.4	17	1.5
Lab071	-0.6	-0.3	0.4	0.4	0.0	-1.0	0.2	1.3	0.2		0.9	0.0	0.2	-0.8	1.2	0.1	1.5	16	0.5
Lab072	-0.6	1.1	-0.4	-0.2	0.0	0.2	0.4	0.0	-0.9	0.2	0.1	0.1	-0.1	-1.1	1.4	-0.4	-0.5	17	0.4
Lab073	-0.3	-0.1	0.2	0.1	0.6	0.4	0.5	0.6	0.6	0.2	-0.5	0.4	-0.1	0.2	0.2	0.5	0.4	17	0.2
Lab075	0.3	0.2	0.3	-0.9	0.3	1.3	0.1	0.8	0.2	-0.3	0.6	-0.3	0.4	0.0	-0.7	0.8	0.2	17	0.3
Lab076	-0.4	-0.2	0.2	-0.2	0.0	-0.1	0.4	-0.1	-0.3	0.3	-0.9	0.0	0.1	0.0	-0.5	-0.3	0.3	17	0.1
Lab078	0.4	-0.7	-0.6	-0.3	-0.8	-0.5	-0.5	-0.1	-0.3	-0.2	1.5	0.1	1.6	0.3	-0.7	0.1	0.2	17	0.5
Lab081	-2.0	-2.2	-1.5	-1.3	-1.2	-1.7	5.0	-1.6	-1.7		-1.9	-1.1	-2.3	-2.3	-1.3	-1.5	-1.5	16	4.4
Lab082	0.0	1.0	0.6	-0.1	-0.1	0.1	0.5	-0.3	0.2	0.0	0.7	0.4	0.1	0.0	0.8	0.9	0.3	17	0.2
Lab083	0.2	-1.0	0.8	-0.1	-0.4	-0.2	-0.3	0.6	-0.6	0.2	0.0	-0.5	-0.5	0.9	-1.1	0.4	0.5	17	0.3
Lab084	-0.4	-1.3	1.6	1.2	-1.3	-0.8	-0.6	-0.7	-3.2	-0.3	-1.4	1.2	1.3	-1.5	1.4	-0.5	0.2	17	1.7
Lab086	-0.1	0.7	-0.2	0.6	1.3	0.5		4.1	-0.7	-0.3	-0.5	1.0	1.5	0.0	1.4	0.3	-0.3	16	1.6
Lab087	0.3	-0.2	-0.3	0.2	0.9	0.3	0.6	0.5	-0.2	-0.2	0.3	-0.1	-0.9	0.1	0.1	-1.1	0.5	17	0.2
Lab089	0.1	0.4	0.2	-0.1	0.1	0.5	0.9	1.4	0.5		1.3	0.1	5.0	0.1	-1.0	-0.6	0.2	16	2.0
Lab090	1.2	-0.4	-0.6	0.3	0.6	0.5	-0.9	-0.3	0.2	0.9	0.1	-0.4	0.0	0.0	-0.2	-0.4	-0.5	17	0.3
Lab091	-1.1	-0.9	-1.6	0.3	-0.4	-0.7	0.5	-0.8	-0.7	-0.2	-1.0	-1.1	0.0	-1.1	-0.7	-0.7	-0.3	17	0.6
Lab094	0.2	0.2	0.6	1.3	0.2	0.3	-0.9	1.0	0.7	0.5	-0.3	0.0	1.2	-1.0	0.3	-0.3	0.4	17	0.5
Lab095	2.1	4.7	2.9	0.0	0.9	0.1	0.2		0.5	-0.3	1.5	5.0	1.6	0.5	1.6	1.9	0.6	16	4.5
Lab096	-0.1	0.5	0.2	1.0	0.4	0.4	-0.2	-0.1	0.4		0.1	-0.5	0.0	-0.2	-1.7	-0.7	0.6	16	0.4
Lab097	1.1	0.2	0.7	1.1	-0.3	-0.2	0.1	1.5	-0.9	0.3	0.7	0.3	1.1	-1.1	0.2	0.2	1.0	17	0.6
Lab098	0.1	2.7	-0.2	-0.2	0.3	-0.1	-0.4	0.2	0.0	1.1	0.1	0.3	0.3	-0.1	-0.6	-0.3	-0.3	17	0.6
Lab099	0.4	0.9	0.0	0.7	0.7	1.2	1.3	-2.0	0.0	0.0	0.6	1.5	0.1	1.8	1.0	1.8	0.7	17	1.1
Lab120	-0.5	-0.1	-0.4	-0.2	0.0	0.0	0.0	0.0	0.4	-0.4	-0.1	-0.3	-0.4	0.3	-0.1	0.3	-0.1	17	0.1
Lab122	0.2	-0.9	-0.5	0.4	0.5	0.2	1.0	-3.1	-0.9	-1.6	-0.7	0.1	1.1	-0.6	0.4	0.4	-1.0	17	1.1
Lab123	-0.9	-0.6	-1.2		-0.3	-0.3	0.0	-0.8	-0.8	1.0	-0.1	-1.2	-0.4	-1.0	-0.5	-1.8	0.0	16	0.7
Lab124	0.2	0.2	0.1	0.9	0.6	0.3	-0.3	0.2	-1.5	-0.7	-0.1	0.2	0.0	-0.8	0.6	0.3	0.3	17	0.3
Lab129	0.2	-0.1	0.5	0.3	0.0	0.7	0.0	0.3	1.2	1.0	-0.1	0.8	0.1	-1.2	-1.1	-0.1	0.1	17	0.4
Lab130	0.1	-0.6	2.9	0.3	0.5	0.7	0.7	0.3	1.4	0.2	0.3	-0.1	0.9	1.2	0.4	0.5	0.1	17	0.9
Lab132	0.2	1.0	0.0	0.4	0.5	0.3	0.2	0.4	1.6	0.4	-0.4	0.2	0.3	-0.2	0.4	0.6	0.1	17	0.3
Lab133	-0.2	-0.2	-0.4	0.0	0.1	0.1	-0.1	-0.2	0.0	-0.3	-0.3	-0.1	-0.5	0.2	-0.2	0.1	-0.3	17	0.1
Lab136	1.2	0.2	-1.4	-0.2	0.5	-0.1	0.5		0.2	-0.7	-0.1	2.4	-0.4	0.2	-3.4	-0.4	0.4	16	1.4
Lab138	-0.8	0.1	-0.5	-0.7	-0.5	-0.4	-0.2	-0.6	-0.8	-0.7	-0.6	0.3	-0.8	-0.3	1.2	1.1	-0.7	17	0.5
Lab140	0.1	0.2	-0.3	0.3	0.9	0.0	-0.4	0.8	0.2	-0.3	0.4	0.4	0.7	-0.6	0.0	0.6	-0.1	17	0.2
Lab142	-1.0	0.4	0.5	0.1	-1.1	-0.3	-0.3	-0.3	-1.5	0.0	-0.6	-0.3	-0.4	-0.4	0.5	0.5	-1.4	17	0.5
Lab145	0.6	-0.3	0.1	0.3	0.5	0.6	0.5	0.0	0.5	-0.3	0.1	-0.2	0.3	-0.4	0.2	-0.8	0.5	17	0.2
Lab146	0.4	4.7	-0.2	-0.4	-1.8	-0.9	-1.4	2.8	-0.9	0.3	0.5	-0.2	-0.6	-1.5	-0.5	-0.4	0.1	17	2.4
Lab147	-0.2	4.0	1.0	-0.1	0.3	-0.7	0.6	-0.5	0.8	-0.5	0.8	-0.4	0.7	-0.3	1.4	1.0	0.7	17	1.4
Lab149	-0.8	-0.1	0.2	0.0	0.2	0.3	-0.1	-0.1	0.6	-0.1	0.2	0.3	-0.4	0.7	0.1	0.3	0.5	17	0.1
Lab150	0.6	-0.1	0.2	-0.7	1.0	0.5	1.1	0.7	-0.3	0.0	0.6	0.5	-0.1	0.6	-0.2	0.4	1.4	17	0.4
Lab151	-0.4	2.0	0.0	-0.8	-0.3	-0.5	0.0	0.1	-1.3	-0.9	0.1	-0.4	0.0	-0.5	-0.7	-1.3	-0.3	17	0.6
Lab154	-0.9	0.5	0.3	-0.4	-0.2	-0.5	-1.1	1.9	-1.1	-1.0	-0.6	-0.2	-0.9	-0.5	0.6	0.3	-0.1	17	0.6
Lab160	0.3	0.2	-0.7	0.2	-0.1	0.4	-0.7	0.2	-0.3		-0.2	-0.2	-3.2	-0.2	5.0	0.8	-0.1	16	2.3



**APPENDIX 5. Average of the Squared z scores (AZ<sup>2</sup>) for laboratories in Category A.**

Lab Code	Boscalid	Carbendazim and benomyl (expressed as carbendazim)	Chlorantraniliprole	Chlorfenapyr	Chlorpyrifos	Diazinon	Ethiofiprophos	Famoxadone	Fipronil (only parent compound)	Flubendiamide	Fluopyram	Imidacloprid	Iprodione	Lufenuron	Omethoate	Propamocarb	Pyraclostrobin	No. of z scores	AZ <sup>2</sup>
Lab161	0.2	1.5	-0.2	0.6	0.4	0.8	-0.1	-0.3	0.2	0.1	-0.1	1.1	-0.9	0.1	-1.2	-0.9	-0.4	17	0.5
Lab165	0.3	2.1	0.4	0.3	0.0	-1.3	-3.2	0.3	-0.5	-0.1	-0.1	-0.7	0.0	-0.1	-0.7	0.5	-0.1	17	1.1
Lab167	-0.3	0.4	-0.3		-0.4	-0.8	-0.4	-0.2	-0.6	0.1	-0.5	0.1	-0.3	-0.4	0.3	-0.9	-0.2	16	0.2
Lab169	0.3	0.6	0.0	-0.1	0.2	0.8	0.7	0.0	0.5	0.2	0.4	0.0	-0.3	-0.1	-0.4	0.7	-0.1	17	0.2
Lab171	1.8	-1.3	0.7	0.6	0.9	2.1	0.7	-0.1	0.0	-0.7	0.4	0.5	0.2	1.8	1.0	-0.1	0.5	17	1.0
Lab172	-1.2	-0.1	0.5	-0.1	-0.6	-0.3	-0.5	-1.6	0.2	0.6		-1.0	-2.2	-1.0	-1.1	0.3	-1.7	16	1.0
Lab173	0.2	0.2	0.1	0.2	0.3	0.5	0.5	0.2	0.5	0.9	0.3	0.0	-0.1	0.9	0.2	-0.7	0.3	17	0.2
Lab179	0.1	-0.4	0.1	0.0	-0.5	-0.2	-0.4	1.1	1.5	0.1	0.3	0.0	0.2	0.3	-0.5	-0.6	0.2	17	0.3

EUPT-FV19 AZ<sup>2</sup> - 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-SANTE<sup>7</sup> 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 Network<sup>8</sup> of National Reference Laboratories (NRLs) and Official Laboratories (OfLs) of the EU Member States. OfLs from EFTA countries and EU-Candidate countries are also welcome to participate in the EUPTs. OfLs from Third countries may be permitted to participate on a case-by-case basis.

The following four EURLs for pesticide residues were appointed by DG-SANTE based on regulation 882/2004/EC<sup>9</sup>:

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

The aim of these EUPTs is to obtain information regarding the quality, accuracy and comparability of pesticide residue data in food and feed reported to the European Union within the framework of the national control programmes and the EU multiannual co-ordinated control programme<sup>10</sup>. 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 Scientific Committee*

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

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

To complement the internal expertise of the EURLs, a group of external consultants that form the **EUPT-Scientific Committee** (EUPT-SC)<sup>11</sup> has been established and approved by DG-SANTE. The EUPT-SC consists of expert scientists with many years of experience in PTs and/or pesticide residue analysis. The actual composition of the EUPT-SC, the affiliation of each member is shown on the EURL-Website. The members of the EUPT-SC will also be listed in the Specific Protocol and the Final Report of each EUPT. The EUPT-SC is made up of the following two subgroups:

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

The EUPT-SC's role is to help the Organisers make decisions regarding the EUPT design: the selection of the commodity, the selection of pesticides to be included in the Target Pesticide List (see below), the establishment of the Minimum Required Reporting Levels (MRRLs), the statistical treatment and evaluation of participants results (in anonymous form), and the drafting and updating of documents such as the General and Specific PT Protocols and the Final EUPT-Reports.

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<sup>7</sup> DG-SANTE = European Commission, Health and Food Safety Directorate-General

<sup>8</sup> For more information about the EURL/NRL/OfL-Network please refer to the EURL-Web-portal under: <http://www.eurl-pesticides.eu>

<sup>9</sup> 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

<sup>10</sup> European Commission Proficiency Tests for Pesticide Residues in Fruits and Vegetables, Trends in Analytical Chemistry, 2010, 29 (1), 70 – 83.

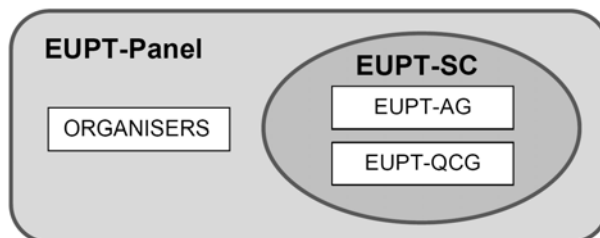
<sup>11</sup> Link to the List of current members of the EUPT Scientific Committee: <http://www.eurl-pesticides.eu/library/docs/allcrl/EUPT-SC.pdf>

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

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

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

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



The decisions of the EUPT-Panel will be documented.

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

### ***EUPT Participants***

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

- Art. 28 of Reg. 396/2005/EC<sup>12</sup> (for all OfLs analysing for pesticide residues within the framework of official controls<sup>13</sup> of food or feed)
- Art. 33 of Reg. 882/2004/EC (for all NRLs)

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

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

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

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

### ***Confidentiality and Communication***

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

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

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

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<sup>12</sup> 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.

<sup>13</sup> Official controls in the sense of Reg. 882/2004/EC, this includes labs involved in controls within the framework of national and/or EU-controlled programmes as well as labs involved in import controls according to Regulation 669/2009/EC.

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

Communication between participating laboratories during the test on matters concerning a PT exercise is not permitted from the start of the PT exercise until the distribution of the preliminary report. For each EUPT the organising EURL prepares a specific EUPT-Website where all relevant documents in their latest version are linked.

The official language used in all EUPTs is English.

### ***Announcement / Invitation Letter***

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

### ***Target Pesticide List***

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

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

### ***Specific Protocol***

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

### ***Homogeneity of the Test Item***

The Test Item will be tested for homogeneity typically before distribution to participants. The homogeneity tests involve the analysis of two replicate analytical portions, taken from at least ten randomly chosen units of treated Test Item. Both sample preparation and measurements should be conducted in random order.

The homogeneity test data are statistically evaluated according to ISO 13528, Annex B or to the International Harmonized Protocols jointly published by ISO, AOAC and IUPAC. 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 of the participants' results, the analytical difficulties faced during the test, knowledge of the analytical behaviour of the pesticide question) may decide to overrule the test. The reasons of this overruling have to be transparently explained in the Final EUPT-Report.

### ***Stability of the analytes contained in the Test Item***

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

A pesticide is considered to be adequately stable if  $|y_i - y| \leq 0.3 \times \sigma_{pt}$ , where  $y_i$  the mean value of the last period of the stability test,  $y$  is the mean value of the first period of the stability test and  $\sigma_{pt}$  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 of the participants' results, the measurement variability, analytical difficulties faced during the test and knowledge about the analytical behaviour 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 Organisers 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 Organisers will choose the shipment conditions to be such that pesticide losses are minimised (e.g. shipment of frozen samples, addition of dry ice). As shipment time can differ between labs/countries it is recommended

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that the Organisers conduct additional stability tests at conditions simulating shipment. Should critical losses be detected for certain pesticides the EUPT-SC will 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 method has not yet been established routinely this should be stated.

### *General procedures for reporting results*

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

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

### *Correction of results for recovery*

According to the Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed<sup>14</sup>, it is common practice that pesticide analysis results are not corrected for recovery if the recovery rates range between 70 and 120 %. Correction of results for recovery is recommended if the average recovery is significantly different from 100 % (typically if outside the 70 – 120 % range). Approaches for recovery correction explicitly stated in the DG-SANTE document are the use of recovery correction factors, the use of stable isotope labelled analogues of the target analytes as Internal Standards (ILISs), the 'procedural calibration' approach as well as the approach of 'standard addition' with additions of analyte(s) being made to analytical portions. Results may be corrected for recovery only in cases where this correction is applied in routine practice (including cases of MRL-violations). Laboratories are required to report whether their results were adjusted for recovery and, if a recovery factor was used, the recovery rate (in percentage) must also be reported. No recovery data are required where correction for recovery is automatic by adding amounts of analytes to the test portion for using the 'standard addition' approach, or isotopically-labelled internal standards (in both cases with spiking into the Test Item at the beginning of the extraction procedures) or procedural calibration. In these cases, the laboratories should report the actual approach that was followed.

### *Methodology information*

All laboratories are requested to provide information on the analytical method(s) they have used. A compilation of the methodology information submitted by all participants is presented in an Annex of the final report or in a separate 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. CV\* > 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 or even refuse participation in the following PT.

### *Results evaluation*

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

#### *– False Positives results*

These are results of pesticides from the Target Pesticides List, that are reported, at or above, their respective MRRL although they were: (i) not detected by the Organiser, even after repeated analyses, and/or (ii) not detected by the overwhelming majority (e.g. > 95 %) of the participating laboratories that had targeted the specific pesticides. In certain instances, case-by-case decisions by the EUPT-Panel may be necessary.

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

#### *– False Negative 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 MRRs. Results reported as '< RL' (RL= Reporting Limit of the laboratory) will be

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<sup>14</sup> Document N° SANTE/11945/2015; Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed

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

### – *Estimation of the assigned value ( $x_{pt}$ )*

In order to minimise the influence of out-lying results on the statistical evaluation, the assigned value  $x_{pt}$  (= consensus concentration) will typically be estimated using robust estimate of the participant's mean ( $x^*$ ) as described in ISO 13528:2015<sup>15</sup>, taking into account the results reported by EU and EFTA countries laboratories only. In special justifiable cases, the EUPT-Panel may decide to eliminate certain results traceably associated with gross errors (see "Omission or Exclusion of results" below) or to use only the results of a subgroup consisting of laboratories that have repeatedly demonstrated good performance for the specific compound in the past.

### – *Omission or Exclusion of results*

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

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

### – *Uncertainty of the assigned value*

The uncertainty of the assigned values  $u(x_{pt})$  is calculated according to ISO 13528:2015 as:

$$u(x_{pt}) = 1.25 \frac{s^*}{\sqrt{p}}$$

where  $s^*$  is the robust standard deviation and  $p$  is the number of results.

In certain cases and considering all relevant factors (e.g. the result distribution, multimodality), the number of submitted results, information regarding analyte homogeneity/stability, information regarding the use of methodologies that might produce a bias that were used by the participants), the EUPT-Panel may consider the assigned value of a specific analyte to be too uncertain and decide that the results should not be evaluated, or only evaluated for informative purposes. The provisions of ISO 13528:2015 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 of the assigned value ( $FFP-\sigma_{pt}$ ) will be calculated using a Fit-For-Purpose approach with a fixed Relative Standard Deviation (FFP-RSD) of 25 % as follows:

$$FFP-\sigma_{pt} = 0.25 \times x_{pt}$$

The percentage FFP-RSD is set at 25 % based on experience from results of previous EUPTs<sup>16</sup>. The EUPT-Panel reserves the right to also employ other approaches on a case-by-case basis considering analytical difficulties and experience gained from previous proficiency tests.

For informative purposes the robust relative standard deviation ( $CV^*$ ) is calculated according to ISO 13528:2015; Chapter 7.7 (Consensus value from participant results) following Algorithm A in Annex C.

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<sup>15</sup> DIN ISO 13528:2015, Statistical methods for use in proficiency testing by interlaboratory comparisons, International Organization for Standardization. Therein a specific robust method for determination of the consensus mean and standard deviation without the need for removal of deviating results is described (Algorithm A in Annex C).

<sup>16</sup> 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.

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### – z scores

This parameter is calculated using the following formula:

$$z_i = \frac{(x_i - x_{pi})}{FFP - \sigma_{pi}}$$

where  $x_i$  is the value reported by the laboratory,  $x_{pi}$  is the assigned value, and  $FFP - \sigma_{pi}$  is the standard deviation using FFP approach. Z scores will be rounded to one decimal place. For the calculation of combined z scores (see below) the original z scores will be used and rounded to one decimal place after calculation.

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

Z scores will be interpreted in the following way, as is set in the ISO 17043:2010<sup>17</sup>:

$ z  \leq 2.0$	Acceptable
$2.0 <  z  < 3.0$	Questionable
$ z  \geq 3.0$	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 if and how to classify the laboratories into two categories - A or B. Currently, laboratories that are able to analyse at least 90% of the compulsory pesticides in the target pesticides list, have correctly detected and quantified a sufficiently high percentage of the pesticides present in the Test Item (at least 90 %) and reported no false positives will have demonstrated 'sufficient scope' and can therefore be classified into Category A. For the 90% criterion the number of pesticides needed to be correctly analysed to have sufficient scope will be calculated by multiplying the number of compulsory pesticides from the Target Pesticides List by 0.9 and rounding to the nearest full number with 0.5 decimals being rounded downwards (see some examples in Table 1).

**Table 1. No. of pesticides from the target pesticides list needed to be targeted or pesticides present in the Test Item that need to be correctly detected and quantified to have sufficient scope.**

No. of compulsory pesticides present in the Test Item / Target Pesticides List (N)	90 %	No. of pesticides needed to be correctly detected and quantified / targeted to have sufficient scope (n)	n
3	2.7	3	N
4	3.6	4	
5	4.5	4	
6	5.4	5	N - 1
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	N - 2
15	13.5	13	
16	14.4	14	
17	15.3	15	
18	16.2	16	
19	17.1	17	
20	18	18	
21	18.9	19	
22	19.8	20	
23	20.7	21	

<sup>17</sup> ISO/IEC 17043:2010. Conformity assessment – General requirements for proficiency testing



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No. of compulsory pesticides present in the Test Item / Target Pesticides List (N)	90 %	No. of pesticides needed to be correctly detected and quantified / targeted to have sufficient scope (n)	n
24	21.6	22	N - 3
25	22.5	22	
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<sup>2</sup>)<sup>18,19</sup> (see below) will be used. The AZ<sup>2</sup> is calculated as follows:

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

Where *n* is the number of z scores to be considered in the calculation. In the calculation of the AZ<sup>2</sup>, z scores higher than 5 will be classified as 5. Based on the AZ<sup>2</sup> achieved, the laboratories are classified as follows:

AZ <sup>2</sup> ≤ 2.0	Good
2.0 < AZ <sup>2</sup> < 3.0	Satisfactory
AZ <sup>2</sup> ≥ 3.0	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<sup>2</sup> if it is considered as not being useful or if the number of results reported by any participant is considered to be too low.

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

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

### Publication of results

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

The Final EUPT Report will be published after the EUPT-Panel has discussed the results. Taking into account that the EUPT-Panel meets normally only once a year (typically in late summer or autumn) to discuss the results of all EUPTs organised by the EURLs earlier in the year, the final report may be published up to 10 months after the deadline for results submission. Results submitted by non-EU/EFTA laboratories might not always be used in the tables or figures in the final report.

### Certificates of participation

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

### Feedback

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

### Correction of errors

Should errors be discovered in any of the documents issued prior to the EUPT (Calendar, Target Pesticides List, Specific Protocol, General 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.

<sup>18</sup> Formerly named "Sum of squared z scores (SZ<sup>2</sup>)"

<sup>19</sup> 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.

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If substantial errors are discovered in the Preliminary EUPT-Report the Organisers will distribute a new corrected version, where it will be stated that the previous version is no longer valid.

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

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

### *Follow-up activities*

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

Upon request, the laboratory's corresponding NRL and EURL are to be informed of the outcome of any investigative activities for false positives, false negatives and for results with  $|z| \geq 3.0$ . Concerning z scores between 2.0 and 3.0 the communication of the outcome of follow-up 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-SANTE, the "Protocol for management of underperformance in comparative testing and/or lack of collaboration of National Reference Laboratories (NRLs) with EU Reference Laboratories (EURLs) activities" is to be followed.

NRLs will be considered as underperforming in relation to scope if in at least two of the last four EUPTs falling within their responsibility area if they: a) haven't participated, or b) targeted less than 90% of the compulsory pesticides in the target lists (80% for SRM-compounds), or c) detected less than 90% of the compulsory compounds present in the test items (80% for SRM-compounds). Additionally, NRLs that obtained AZ2 higher than 3 in two consecutive EUPTs of the last four EUPTs, will be considered as underperforming in accuracy. A two-step protocol established by DG-SANTE will be applied as soon as underperformance of an NRL is detected<sup>20</sup>:

#### Phase 1:

- Identifying the origin of the bad results (failure in EUPTs).
- Actions: On the spot visits and training if necessary and repetition of the comparative test if feasible and close the assessment of results by the EURL.

#### Phase 2:

- If the results still reveal underperformance the Commission shall be informed officially by the EURL including a report of the main findings and corrective actions.
- The Commission shall inform the Competent Authority and require that appropriate actions are taken.

Underperformance rules for the OfLs will be established at a later stage.

### *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.

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<sup>20</sup> Article 32 of the Regulation 882/2004



## **EUPT-FV-19 SPECIFIC PROTOCOL**

### **European Union Proficiency Test for Pesticide Residues in Fruits and Vegetables (2017)**

#### **Introduction**

This protocol is complementary to the General Protocol of EU Proficiency Tests (EUPT) for Pesticide Residues in Food and Feed (7th Edition). This Proficiency Test is organised by the EURL for Pesticide Residues in Fruits and Vegetables covering Multiresidue Methods (MRM) of analysis.

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

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

#### **Test Item**

This proficiency test is based on the analysis of incurred pesticide residues in **lemon**. The lemons were grown in a field in Almería's province. The pesticide treatments carried out were pre-harvest using commercial formulations and post-harvest using analytical standards. The test item was frozen (using liquid nitrogen), chopped, homogenised and sub-sampled into polyethylene bottles that had previously been coded. Ten of these bottles containing the test item were chosen randomly, and analysed to check for homogeneity. The test item is stored frozen (-20°C) prior to shipment to participants.

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

#### **Subcontracting**

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

#### **Target List**

In addition to the pesticide target list of mandatory compounds, a "voluntary target list" containing pesticides which might be present in the test item will be published. Those voluntary pesticides will not be used for the evaluation of the laboratories into Category A or B, and a separate statistical evaluation will be made for them.

#### **Assigned value and robust relative standard deviation**

In order to minimise the influence of out-lying results on the statistical evaluation, the assigned value will be estimated using the robust statistics as described in ANNEX C of ISO 13258:2015, where the robust mean ( $x^*$ ) according algorithm A is defined. For the calculation of the assigned value only results reported by EU and EFTA countries laboratories will be taken into account.

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

#### **Laboratory assessment**

For the assessment of the overall laboratory performance, the Average of the Squared z-Score ( $AZ^2$ ) will be used as in the last Proficiency Test, but only for those laboratories in Category A, which will be those laboratories that are able to analyse at least 90% of the pesticides in the target list, that are able to detect at least 90% of the pesticides present in the test material and that report no false positives. Within Category A, the laboratories will be sub-classified as "good", "satisfactory" or "unsatisfactory". All the other laboratories will be classified in Category B. This information will be available in the General Protocol.

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

### Steps to follow

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

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

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 be **250 euros** for EU/EFTA official laboratories and **350 euros** for official laboratories from other countries. The laboratories will receive an invoice and after that they can start the payment procedure. An e-mail showing the bank transfer confirmation, or similar, may be requested at any time by the Organiser. **Payments without a Laboratory Code 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. **Scope Form** will be placed in the restricted area and will be open to participants from the 6<sup>th</sup> - 13<sup>th</sup> February 2017, prior to test item shipment. The aim is that laboratories provide information regarding their scope of analysis before receipt of the test item and detailed information regarding which pesticide is within the accredited scope of the lab and which is not. After the deadline it will not be possible to change the scope.

6. When the participant laboratories receive the test item (and not before), they must enter the restricted area again and submit **Test Item Receipt Form** to inform the Organiser that they have accepted the test item. This Form has a deadline: 17<sup>th</sup> February 2017, 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. If form 1 is not filled in, the Organiser will consider that the participant has accepted the test item.

7. The participant laboratories must respect the deadline for submitting their results – 6<sup>th</sup> March 2017- using **Identified Pesticides Form, Results Form** and the **Methods Form** on-line.

For each pesticide included in the laboratory scope, the Reporting Limit (RL) will be requested. The MRRRL and the participant's own RL will be used to help identify false negative results. This form will also request information on which of the pesticides sought by the laboratory is within the laboratory's routine scope and whether it is accredited.

The laboratories will be also asked to report any pesticide that may have been detected in the blank test item.

All concentrations must be expressed in mg/kg together with the recovery as a percentage. The actual results/residue levels measured must be reported as numbers. Symbols (>, <, ±, ≥, ≤, ...) will not be accepted.

**IMPORTANT:** If your result is not correctly expressed it will be considered as 'ND' (Not Detected).

The number of significant figures should be based on the procedures provided in SANTE/11945/2015. 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'. If a pesticide was not sought, it will be recorded as 'NA' (Not Analysed).

The laboratory will also be asked to 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.

These forms 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 stage, always considering the deadline to submit results, which is 6<sup>th</sup> March 2017. 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 lemons are present in the test item.

8. One final form, **Additional Information Requested** will be accessible after the deadline has passed. This Form will be available only for those laboratories that reported that they analysed a pesticide present in the test item but they did not detect it. 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 from 7<sup>th</sup>-13<sup>th</sup> March 2017. 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 send all the participants the preliminary results, containing all the reported concentrations, in order to make sure that there was not any mistake from the Organisation side. After a stipulated deadline, the results will be evaluated and a preliminary report containing the preliminary assigned values and z scores will be sent to the participants. Finally, after evaluation by the Scientific

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Committee, the Final Report will be published online and a hard copy will be sent 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. Results submitted by non-EU/EFTA laboratories might not always be used in the tables or figures in the final report. Further relevant information considered to be of value may also be included.

### Amount of Test Item

Participants will receive:

- Approximately 200 g of lemon test item treated with pesticides.
- Approximately 200 g of 'blank' lemon 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 13<sup>th</sup> February 2017. 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.

### Calendar

ACTIVITY	DATE
Publishing the Calendar and Matrix on the Web page.	13th November 2016
Receiving Application Form from invited laboratories.	9th January -27th January 2017
Specific Protocol published on the Web site.	27th January 2017 at the latest
Sample distribution.	13th February 2017
Deadline for receiving sample acceptance	17th February 2017
Deadline for receiving results	6th March 2017
Filling in additional information, if necessary.	7th -13th March 2017
Preliminary Report: only results, no statistical treatment.	March 2017
Final Report distributed to the Laboratories.	December 2017

### Cost of test item shipment.

EU/EFTA laboratories will be charged **250 €** for the shipment cost, for **non-EU/EFTA** laboratories the amount will be **350 €**. Regarding payment procedures - each laboratory can specify their details and invoice requests when applying for the test.

**Please, do not pay for this EUPT until we send you the invoice.  
Remember to include your Laboratory Code in the subject of the bank transfer.**

Payment details are as follows:

**BANK NAME: CAJAMAR - Caja Rural Sociedad Corporativa de Crédito**  
**BANK ACCOUNT HOLDER: Universidad de Almería**  
**BANK ADDRESS: Office Number 990. Universidad de Almería. Spain**  
**ACCOUNT NUMBER: ES0730580130172731005000**  
**SWIFT: CCRIES2A**  
**REFERENCE GIVEN: Lab Code**

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

### Contact information

The official organising group details are as follows:

Universidad de Almería. Edificio Químicas CITE I  
Ctra. Sacramento s/n  
04120, La Cañada de San Urbano  
Almería - Spain  
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### Organising team (e-mail and phone no.):

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### Quality Control Group

Dr. Antonio Valverde, University of Almería, Spain  
Dr. Paula Medina, European Food Safety Authority, Italy.

### Advisory Group

Dr. Michelangelo Anastassiades, CVUA, Stuttgart, Germany.  
Dr. Miguel Gamón, Laboratorio Agroalimentario, Valencia, Spain.  
Dr. Philippe Gros, Laboratoire du SCL, Montpellier, France.  
Dr. Magnus Jezussek, Bavarian Health and Food Safety Authority, Erlangen, Germany.  
Dr. André de Kok, NVWA, Wageningen, The Netherlands.  
Mr. Ralf Lippold, CVUA, Freiburg, Germany.  
Dr. Sonja Masselter, AGES GmbH Institute for Food Safety, Innsbruck, Austria.  
Mr. Finbarr O'Regan, Pesticide Control Laboratory, Celbridge, Ireland.  
Dr. Tuija Pihlström, National Food Agency, Uppsala, Sweden.  
Dr. Mette Erecius Poulsen, DTU, Copenhagen, Denmark.

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

**TARGET PESTICIDE LIST FOR THE EUPT-FV-19**

Pesticide	MRRL (mg/Kg)
Acephate	0.01
Acetamiprid	0.01
Acrinathrin	0.01
Aldicarb	0.01
Aldicarb Sulfone	0.01
Aldicarb Sulfoxide	0.01
Aldrin	0.01
Azinphos-methyl	0.01
Azoxystrobin	0.01
Benfuracarb	0.01
Bifenthrin	0.01
Biphenyl	0.01
Bitertanol	0.01
Boscalid	0.01
Bromopropylate	0.01
Bromuconazole	0.01
Bupirimate	0.01
Buprofezin	0.01
Cadusafos	0.006
Carbaryl	0.01
Carbendazim and benomyl (sum of benomyl and carbendazim expressed as carbendazim)	0.01
Carbofuran	0.01
Carbofuran-3-hydroxy	0.01
Carbosulfan	0.01
Chlorantraniliprole	0.01
Chlorfenapyr	0.01
Chlorfenvinphos	0.01
Chlorobenzilate	0.01
Chlorothalonil	0.01
Chlorpropham	0.01
Chlorpyrifos	0.01
Chlorpyrifos-methyl	0.01
Clofentezine	0.01
Clothianidin	0.01
Cyfluthrin (cyfluthrin incl. other mixtures of constituent isomers (sum of isomers))	0.01
Cymoxanil	0.01
Cypermethrin (cypermethrin incl. other mixtures of constituent isomers (sum of isomers))	0.01
Cyproconazole	0.01
Cyprodinil	0.01
Deltamethrin (cis-deltamethrin)	0.01
Demeton-S-methylsulfone	0.006
Diazinon	0.01
Dichlofluanid	0.01
Dichlorvos	0.01
Dicloran	0.01
Dicofol (sum of p, p' and o,p' isomers)	0.01
Dieldrin	0.01
Diethofencarb	0.01
Difenoconazole	0.01
Diflubenzuron	0.01
Dimethoate	0.003
Dimethomorph	0.01
Dimethylaminosulfotoluidide (DMST)	0.01
Diniconazole	0.01
Diphenylamine	0.01
Endosulfan alpha	0.01
Endosulfan beta	0.01
Endosulfan sulfate	0.01
EPN	0.01
Epoxiconazole	0.01
Ethion	0.01
Ethirimol	0.01
Ethoprophos	0.008
Etofenprox	0.01

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

Pesticide	MRRL (mg/Kg)
Famoxadone	0.01
Fenamidone	0.01
Fenamiphos	0.01
Fenamiphos sulfone	0.01
Fenamiphos sulfoxide	0.01
Fenarimol	0.01
Fenazaquin	0.01
Fenbuconazole	0.01
Fenhexamid	0.01
Fenitrothion	0.01
Fenoxycarb	0.01
Fenpropathrin	0.01
Fenpropidin	0.01
Fenpropimorph	0.01
Fenpyroximate	0.01
Fenthion	0.01
Fenthion oxon	0.01
Fenthion oxon sulfone	0.01
Fenthion oxon sulfoxide	0.01
Fenthion sulfone	0.01
Fenthion sulfoxide	0.01
Fenvalerate (any ratio of constituent isomers (RR, SS, RS & SR) including esfenvalerate)	0.01
Fipronil (only parent compound)	0.004
<b>Flubendiamide</b>	0.01
Fludioxonil	0.01
Flufenoxuron	0.01
Fluopicolide	0.01
Fluopyram	0.01
Fluquinconazole	0.01
Flusilazole	0.01
Flutolanil	0.01
Flutriafol	0.01
Fosthiazate	0.01
Hexaconazole	0.01
Hexythiazox	0.01
Imazalil	0.01
Imidacloprid	0.01
Indoxacarb (sum of indoxacarb and its R enantiomer)	0.01
Iprodione	0.01
Iprovalicarb	0.01
Isocarbophos	0.01
Isofenphos-methyl	0.01
Isoprothiolane	0.01
Kresoxim-methyl	0.01
Lambda-Cyhalothrin	0.01
Linuron	0.01
Lufenuron	0.01
Malaoxon	0.01
Malathion	0.01
Mandipropamid	0.01
Mepanipyrim	0.01
Metaflumizone (sum of E- and Z- isomers)	0.01
Metalaxyl and metalaxyl-M	0.01
Metconazole (sum of isomers)	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
Myclobutanyl	0.01
Omethoate	0.003
Orthophenylphenol	0.01



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

Pesticide	MRRL (mg/Kg)
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
Penconazole	0.01
Pencycuron	0.01
Pendimethalin	0.01
Permethrin (sum of isomers)	0.01
Phenthoate	0.01
Phosalone	0.01
Phosmet	0.01
Phosmet oxon	0.01
Phoxim	0.01
Pirimicarb	0.01
Pirimicarb-desmethyl	0.01
Pirimiphos-methyl	0.01
Prochloraz (only parent compound)	0.01
Procymidone	0.01
Profenofos	0.01
Propamocarb	0.01
Propargite	0.01
Propiconazole	0.01
Propyzamide	0.01
Prothioconazole (Prothioconazole-desthio)	0.01
Prothiofos	0.01
Pyraclastrobin	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
Spiromesifen	0.01
Spiroxamine	0.01
Tau-Fluvalinate	0.01
Tebuconazole	0.01
Tebufenozide	0.01
Tebufenpyrad	0.01
Teflubenzuron	0.01
Tefluthrin	0.01
Terbutylazine	0.01
Tetraconazole	0.01
Tetradifon	0.01
Thiabendazole	0.01
Thiacloprid	0.01
Thiamethoxam	0.01
Thiodicarb	0.01
Thiophanate-methyl	0.01
Tolclofos-methyl	0.01
Tolyfluanid	0.01
Triadimefon	0.01
Triadimenol	0.01
Triazophos	0.01
Trichlorfon	0.01
Trifloxystrobin	0.01
Triflumuron	0.01
Trifluralin	0.01
Triticonazole	0.01
Vinclozolin (only parent compound)	0.01
Zoxamide	0.01

**In bold: new pesticides this year**

This list is based on Commission implementing Regulation (EU) 2015/595 of 15 April 2015  
The MRRLs are based on Regulation (EC) No. 396/2005 and Commission Directive 2006/125/EC.

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

**VOLUNTARY PESTICIDE LIST FOR THE EUPT-FV-19**

Pesticide	MRRL (mg/Kg)
Ametoctradin	0.01
Benalaxyl including other mixtures of constituent isomers including benalaxyl-M (sum of isomers)	0.01
Benzovindiflupyr	0.01
<b>Chlorfluazurone</b>	<b>0.01</b>
Clomazone	0.01
Cyazofamid	0.01
<b>Cyflufenamid</b>	<b>0.01</b>
Emamectin benzoate B1a, expressed as emamectin	0.01
Etoxazole	0.01
Fenpyrazamine	0.01
Fluxapyroxad	0.01
Heptachlor	0.01
Heptachlor epoxide	0.01
Isopyrazam	0.01
<b>Metrafenone</b>	<b>0.01</b>
<b>Novaluron</b>	<b>0.01</b>
Penflufen	0.01
Penthiopyrad	0.01
Prosulfocarb	0.01
Pyrethrins	0.01
Quintozene	0.01
Pentachloro-aniline	0.01
<b>Proquinazid</b>	<b>0.01</b>
<b>Pyridalil</b>	<b>0.01</b>
<b>Pyriofenone</b>	<b>0.01</b>
Rotenone	0.01
<b>Spinetoram</b>	<b>0.01</b>
Spirotetramat	0.01
Spirotetramat metabolite BY108330-enol	0.01
Spirotetramat metabolite BY108330-ketohydroxy	0.01
Spirotetramat metabolite BY108330-mono-hydroxy	0.01
Spirotetramat metabolite BY108330 enol-glucoside	0.01
<b>Sulfoxaflor</b>	<b>0.01</b>
Tetramethrin	0.01
<b>Tricyclazole</b>	<b>0.01</b>

In bold: new pesticides this year

This list is based on the working document SANTE/12745/2013

**ANNEX 2. List of laboratories that agreed to participate in EUPT-FV-19.**

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
Austria	Austrian Agency For Health And Food Safety, Institute for Food Safety, Department for Pesticide and Food Analysis (PLMA)	Innsbruck	Yes
Austria	MA 38 - Lebensmitteluntersuchung Wien	Wien	Yes
Belgium	Primoris Belgium - Belgium, Gent (Zwijnaarde)	Gent - Zwijnaarde	Yes
Belgium	LOVAP (Laboratorium voor Onderzoek Van levensmiddelen en Aanverwante Produkten) NV	Geel	Yes
Belgium	Scientific Institute of Public Health	Brussels	Yes
Bulgaria	Central Laboratory for Chemical Testing and Control	Sofia	Yes
Bulgaria	Primoris - Bulgaria, Plovdiv	Plovdiv	Yes
Bulgaria	Euro Lab.	Svilengrad	Yes
China	Agro-product Safety Research Center, Chinese Academy of Inspection and Quarantine	Beijing	Yes
China	Beijing Uni-Star Inspection Technology Co., Ltd.	Beijing	Yes
China	Inspection and Quarantine Technique Center of Qinhuangdao Entry-Exit inspection and Quarantine Bureau of P.R. China	Qinhuangdao	Yes
China	Shanghai Municipal Center For Disease Control and Prevention	Shanghai	Yes
Costa Rica	Laboratorio de Residuos	San José	Yes
Croatia	Euroinspekt - Croatiakontrola d.o.o.	Zagreb	Yes
Croatia	Institute of Public Health, Dr. Andrija štampar	Zagreb	Yes
Croatia	Croatian National Institute of Public Health	Zagreb	Yes
Croatia	Faculty of Food Technology and Biotechnology, Food Control Center - Croatia, Zagreb	Zagreb	Yes
Croatia	Inspecto d.o.o. Laboratorij	Osijek	Yes
Cyprus	Laboratory of Pesticide Residues Analysis, State General Laboratory	Nicosia	Yes
Czech Republic	University of Chemical Technology, Dept. of Food Chemistry and Analysis - Prague	Praha	Yes
Czech Republic	Czech Agriculture and Food Inspection Authority	Praha	Yes
Denmark	Danish Veterinary and Food Administration, Department of Residues, Ringsted	Ringsted	Yes
Denmark	National Food Institute, Technical University of Denmark	Søborg	Yes
Estonia	Agricultural Research Centre, Saku, Lab for Residues and Contaminants	Saku	Yes
Estonia	Health Board - Tartu Laboratory	Tartu	Yes
Finland	Finnish Customs Laboratory	Espoo	Yes

**ANNEX 2. List of laboratories that agreed to participate in EUPT-FV19**

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
Finland	Metropolilab Oy	Helsinki	Yes
France	GIRPA - FREDON Pays de la Loire	Beaucouzé	Yes
France	Service Commun des Laboratoires / Laboratoire de Montpellier	Montpellier	Yes
France	Service Commun des Laboratoires / Laboratoire Ile de France - Massy	Massy Cedex	Yes
France	INOVALYS Le Mans	Le Mans	Yes
France	CERECO SUD	Garons	Yes
France	Analysis Center Mediterranean Pyrenees	Perpignan	Yes
France	Capinov	Landerneau	Yes
France	Phytocontrol - France, Nimes	Nimes	Yes
Germany	State Laboratory for Chemistry, Hygiene and Veterinary Medicine of the Free Hanseatic Town Bremen	Bremen	Yes
Germany	Berlin-Brandenburg State Laboratory, Frankfurt (Oder)	Frankfurt (Oder)	Yes
Germany	Labor Friedle GmbH	Tegernheim	Yes
Germany	Eurofins - Dr. Specht Laboratorien GmbH	Hamburg	Yes
Germany	LUFA-ITL GmbH	Kiel	Yes
Germany	Central Institute of the Bundeswehr Medical Service MUNICH	Garching	Yes
Germany	Chemical and Veterinary Analytical Institute Rhine-Ruhr-Wupper	Krefeld	Yes
Germany	ILAU GmbH	Anzing	Yes
Germany	Chemical and Veterinary Analytical Institute Muensterland-Emscher Lippe	Münster	Yes
Germany	Chemisches Labor Dr. Mang	Frankfurt Am Main	Yes
Germany	Landesuntersuchungsamt Institut für Lebensmittelchemie Speyer	Speyer	Yes
Germany	Thüringer Landesamt für Lebensmittelsicherheit und Verbraucherschutz	Bad Langensalza	Yes
Germany	Landesamt für Verbraucherschutz - Sachsen-Anhalt	Halle/Saale	Yes
Germany	Landesamt für Landwirtschaft, Lebensmittelsicherheit und Fischerei Mecklenburg-Vorpommern	Rostock	Yes
Germany	Federal Office of Consumer Protection and Food Safety, NRL for Pesticide Residues	Berlin	Yes
Germany	Landesamt für Umwelt- und Arbeitsschutz, Saarland	Saarbrücken	Yes
Germany	State Investigation Institute of Health and Veterinary Saxony	Dresden	Yes
Germany	Food and Veterinary Institute Oldenburg	Oldenburg	Yes
Germany	Bavarian Health and Food Safety Authority Office Erlangen	Erlangen	Yes

**ANNEX 2. List of laboratories that agreed to participate in EUPT-FV-19.**

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
Germany	Landwirtschaftliches Technologiezentrum Augustenberg, Karlsruhe	Karlsruhe	Yes
Germany	GALAB Laboratory GmbH	Hamburg	Yes
Germany	State Laboratory Schleswig-Holstein	Neumünster	Yes
Germany	Chemical and Veterinary Investigations Office, Stuttgart (CVUAS ), (Residues)	Fellbach	Yes
Germany	Zentrales Institut des Sanitätsdienstes der Bundeswehr KIEL	Kronshagen	Yes
Germany	Institut für Hygiene und Umwelt Hamburg	Hamburg	Yes
Germany	Landesbetrieb Hessisches Landeslabor, Kassel	Kassel	Yes
Germany	Amt für Verbraucherschutz Düsseldorf - 39/2 Chemische und Lebensmitteluntersuchung	Düsseldorf	Yes
Greece	General Chemical State Laboratory, Pesticide Residues Laboratory	Athens	Yes
Greece	Benaki Phytopathological Institute, Pesticide Residues Laboratory	Kifissia	Yes
Greece	Regional Center of Plant Protection and Quality Control of Thessaloniki, Pesticide Residues Laboratory	Thessaloniki	Yes
Hungary	National Food Chain Safety Office, Food Chain Safety Centre Non-profit Ltd., Pesticide Residue Analytical Laboratory, Szolnok	Hódmezovásárhely	Yes
Hungary	National Food Chain Safety Office, Directorate of Plant Protection, Soil Conservation and Agri-environment - Pesticide Analytical Laboratory, Velence	Velence	Yes
Hungary	National Food Chain Safety Office, Food Chain Safety Centre Non-profit Ltd., Pesticide Residue Analytical Laboratory, Szolnok	Szolnok	Yes
Hungary	National Food Chain Safety Office, Food Chain Safety Centre Non-profit Ltd., Pesticide Residue Analytical Laboratory, Miskolc	Miskolc	Yes
Iceland	Mafís - Icelandic Food Research	Reykjavík	Yes
Indonesia	Angler Biochemlab, PT.	Surabaya	Yes
Ireland	Pesticide Control Laboratory, Department of Agriculture, Fisheries and Food	Co. Kildare	Yes
Italy	Istituto Zooprofilattico Sperimentale Lazio e Toscana - Roma	Roma	Yes
Italy	Laboratorio analisi acque e cromatografia	Bolzano	Yes
Italy	Environement protection regional agency Aosta Valley	Saint Christophe	Yes
Italy	ARPA Puglia - Dipartimento di Bari	Bari	Yes
Italy	Laboratorio di Sanità Pubblica ASL Bergamo	Bergamo	Yes
Italy	ARPAL Sez. di La Spezia	La Spezia	Yes
Italy	Istituto Zooprofilattico Sperimentale Umbria e Marche, Perugia	Perugia	Yes
Italy	Istituto Zooprofilattico Sperimentale Abruzzo e Molise	Teramo	Yes

**ANNEX 2. List of laboratories that agreed to participate in EUPT-FV19**

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
Italy	ARPAE Emilia Romagna, Sezione Provinciale di Ferrara, Laboratorio Tematico Fitofarmaci	Ferrara	Yes
Italy	Arpalazio Sezione P.Le Di Latina - Servizio Laboratorio Ambiente E Salute, Unità di Chimica Inorganica	Latina	Yes
Italy	Istituto Zooprofilattico Sperimentale Sicilia	Palermo	Yes
Italy	ARPA FVG Settore Laboratorio Unico - Laboratorio di Pordenone	Pordenone	Yes
Italy	Istituto Superiore di Sanità, Pesticide Section	Roma	Yes
Italy	ATS Città Metropolitana di Milano - Laboratorio di Prevenzione	Milano	Yes
Italy	Istituto Zooprofilattico Sperimentale Sardegna	Sassari	Yes
Italy	ARPAM Dipartimento di Macerata	Macerata	No
Italy	Istituto Zooprofilattico Sperimentale Lombardia ed Emilia Romagna	Brescia	Yes
Italy	ARPA Veneto Dip.Reg.Lab. S.L. Verona	Verona	Yes
Italy	ARPAC-Dipartimento Provinciale di Napoli-L.S. Fitofarmaci	Napoli	Yes
Italy	APPA Trento Settore Laboratorio	Trento	Yes
Italy	Public Health Laboratory - Florence	Firenze	Yes
Kenya	SGS Kenya Laboratory	Mombasa	Yes
Kenya	KEPHIS Analytical Chemistry Laboratory	Nairobi	Yes
Latvia	Institute of Food Safety, Animal Health and Environment (BIOR) - Riga	Riga	Yes
Lithuania	National Food and Veterinary Risk Assessment Institute (Lithuania, Vilnius)	Vilnius	Yes
Luxembourg	National Health Laboratory Luxembourg (Food Laboratory)	Dudelange	Yes
Netherlands	Eurofins Lab Zeeuws-Vlaanderen (LZV) B.V.	Graauw	Yes
Netherlands	Handelslaboratorium Dr. Verwey	Rotterdam	Yes
Netherlands	Groen Agro Control	Delfgauw	Yes
Netherlands	NVWA - Netherlands Food and Consumer Product Safety Authority (Wageningen, The Netherlands)	Wageningen	Yes
Norway	Norwegian Institute for Agricultural and Environmental Research, Plant Health and Plant Protection Division, Pesticide Chemistry Section	Aas	Yes
Panama	Laboratorio de Análisis de Residuos de Plaguicidas en Plantas y Productos Vegetales	Panama	Yes
Peru	Servicio Nacional De Sanidad Agraria - Unidad Del Centro De Control De Insumos Y Residuos Toxicos	Lima	Yes
Poland	Institute of Plant Protection - National Research Institute	Rzeszow	Yes

**ANNEX 2. List of laboratories that agreed to participate in EUPT-FV-19.**

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
Poland	Voievodship Sanitary - Epidemiological Station in Wroclaw	Wroclaw	Yes
Poland	Voievodship Sanitary - Epidemiological Station in Warszaw	Warszaw	Yes
Poland	Institute of Plant Protection - National Research Institute, Branch Sosnicowice	Sosnicowice	Yes
Poland	Institute of Plant Protection, Department of Pesticide Residue Research - Poznan	Poznan	Yes
Poland	Voievodship Sanitary - Epidemiological Station in Lodz	Lodz	Yes
Poland	Institute of Plant Protection Pesticide Residue Laboratory, Bialystok	Bialystok	Yes
Poland	Main Inspectorate of Plant Health And Seed Inspection, Central Laboratory	Torun	Yes
Poland	Institute of Horticulture, Food Safety Laboratory	Skierniewice	Yes
Poland	UO-Technologia Laboratorium Grójec	Grójec	Yes
Poland	Voievodship Sanitary - Epidemiological Station in Opole	Opole	Yes
Portugal	Regional Laboratory of Veterinary and Food Safety - Madeira Island	Funchal - Madeira Island	Yes
Portugal	INIAV- Pesticide Residues Laboratory	Oeiras	Yes
Portugal	Vairão - Pesticide Lab (Plant Origin Products)	Vairão - Vila Do Conde	No
Romania	Sanitary Veterinary and Food Safety Directorate, Bucharest	Bucharest	Yes
Romania	Central Laboratory for Pesticides Residues Control in Plants and Vegetable Products - Bucharest	Bucharest	Yes
România	Regional Laboratory for Pesticide Residues Control in Plant and Plant Products Mures	Targu Mures	Yes
Saudi Arabia	Laboratory of Monitoring Food Contaminants	Riyadh	Yes
Serbia	Center for Food Analysis	Belgrade	Yes
Serbia	SP Laboratory	Becej	Yes
Singapore	Veterinary Public Health Centre	Singapore	Yes
Slovakia	State Veterinary and Food Institute - Veterinary and Food Institute in Bratislava	Bratislava	Yes
Slovenia	National Laboratory of Health, Environment and Foodstuffs - Maribor	Maribor	Yes
Slovenia	National Laboratory for Health, Environment and Food - Maribor (Location Ljubljana)	Ljubljana	Yes
Spain	Laboratori Agroalimentari de la Generalitat de Catalunya	Cabrils	Yes
Spain	Laboratorio de Producción y Sanidad Vegetal de Huelva	Huelva	Yes
Spain	Labs & Technological Services AGQ, S.L. - Spain, Burguillos	Burguillos	Yes
Spain	Laboratorio Agroalimentario de Zaragoza	Zaragoza	Yes
Spain	Soivre Valencia	Valencia	Yes

**ANNEX 2. List of laboratories that agreed to participate in EUPT-FV19**

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
Spain	Laboratorio de Salud Pública de Almería, Ministry of Health	Almería	Yes
Spain	Laboratorio Agroalimentario de Granada	Granada	Yes
Spain	Navarra de Servicios y Tecnologías, S.A.	Villava	Yes
Spain	Laboratorio KUDAM S.L.	Pilar De La Horadada (Alicante)	Yes
Spain	National Centre for Technology and Food Safety (CNTA) - Spain, San Adrián	San Adrián (Navarra)	Yes
Spain	Analytica Alimentaria GmbH Sucursal España	Almería	Yes
Spain	Laboratorio Químico Microbiológico, S.A.	San Gines-Murcia	Yes
Spain	Laboratorio Arbitral Agroalimentario, Madrid	Madrid	Yes
Spain	EUROFINS SiCA AgriQ S.L.- Spain, Almería	Almería	Yes
Spain	Laboratorio de Salud Pública de Badajoz	Badajoz	No
Spain	Agrofood Laboratory of the Comunidad Valenciana	Valencia	Yes
Spain	Laboratorio Regional CC.AA. La Rioja	Logroño	Yes
Spain	Laboratorio de Producción y Sanidad Vegetal de Jaén	Mengibar (Jaén)	Yes
Spain	AINIA Centro tecnológico	Valencia	Yes
Spain	Laboratorio Agroalimentario y de Sanidad Animal de Murcia	Murcia	Yes
Spain	Instituto Tecnológico de Canarias, División de Investigación y Desarrollo Tecnológico - Laboratorio de Residuos	Agüimes, Gran Canaria	Yes
Spain	Laboratorio Agroalimentario de Extremadura (Cáceres)	Cáceres	Yes
Spain	Laboratory of Barcelona Public Health Agency	Barcelona	Yes
Spain	Laboratorio de Producción y Sanidad Vegetal de Almería, Ministry of Agriculture	La Mojonera (Almería)	Yes
Spain	National Centre for Food - Spain, Majadahonda	Majadahonda	Yes
Spain	Agricultural and Phytopathological Laboratory of Galicia	Abegondo. A Coruña	Yes
Spain	Laboratorio Agrario Regional - Junta de Castilla y Leon	Burgos	Yes
Spain	Laboratorio Regional Agroalimentario y Ambiental de Castilla la Mancha (LARAGA), Toledo	Toledo	Yes
Spain	Laboratorios Ecosur, S.A.L.	Lorquí (Murcia)	Yes
Spain	Laboratorio de Salud Pública Madrid Salud Ayuntamiento de Madrid	Madrid	Yes
Spain	Soivre Almería. Dirección Provincial de Comercio	Almería	Yes
Spain	Laboratorio de Sanidad Vegetal de Asturias	Oviedo (Asturias)	Yes
Sweden	National Food Agency, Chemistry Department	Uppsala	Yes



**ANNEX 2. List of laboratories that agreed to participate in EUPT-FV-19.**

<b>COUNTRY</b>	<b>LABORATORY NAME</b>	<b>CITY</b>	<b>REPORTED RESULTS</b>
Sweden	Eurofins - Food&Agro Sweden, Lidköping	Lidköping	Yes
Switzerland	Kantonales Laboratorium Zürich	Zürich	Yes
Switzerland	Amt für Verbraucherschutz Aargau (Cantonal Office of Consumer Protection Aargau)	Aargau	Yes
Switzerland	Kantonales Laboratorium Bern	Bern	Yes
Thailand	Central Laboratory (Thailand) Co.,Ltd Bangkok branch	Bangkok	Yes
Türkiye	ÖZEL MSM Gıda Kontrol Lab. ve dan. Hiz. Tic. As. (Private Msm Food Control Laboratory)	Mersin	Yes
United Kingdom	Science and Advice for Scottish Agriculture	Edinburgh	Yes
United Kingdom	Scientific Analysis Laboratories - United Kingdom, Cambridge	Cambridge	Yes
United Kingdom	Eurofins Food Testing - UK, Wolverhampton	Wolverhampton	Yes
United Kingdom	The Food and Environment Research Agency - York	York	Yes
Uruguay	Grupo de Analisis de Compuestos Trazas/ Pharmacognosy& Natural Products	Montevideo	Yes
Zambia	Zambia Bureau of Standards	Lusaka	No