

# EURL-PROFICIENCY TEST-FV-20, 2018

## Pesticide Residues in Green Bean Homogenate

### Final Report

December 2018

#### Organiser:

**Dr. Amadeo R. Fernández-Alba**

Co-Head of EURL-FV

University of Almería, Edificio Químicas CITE I

Ctra. Sacramento s/n

04120 Almería, SPAIN

Phone: +34 950015034; Fax: +34 950015008

E-mail: amadeo@ual.es

<http://www.eurl-pesticides.eu>



#### Organising team at the University of Almería:

Dr. Carmen Ferrer, Chemist.	University of Almería
Mr. Octavio Malato, Chemist.	University of Almería
Dr. Ana Lozano, Chemist.	University of Almería
Dr. M <sup>a</sup> del Mar Gómez, Agronomist.	University of Almería
Ms. Samanta Uclés, Chemist.	University of Almería
Dr. Łukasz Rajski, Chemist.	University of Almería
Dr. M <sup>a</sup> Jesús Martínez, Chemist.	University of Almería
Mr. Víctor Cutillas, Chemist.	University of Almería
Ms. Elena Hakme, Chemist.	University of Almería
Mr. Francisco José Díaz, Chemist.	University of Almería
Ms. Ana Goday, Chemist.	University of Almería
Ms. Maria Murcia, Chemist.	University of Almería

#### Scientific Committee:

Dr. Antonio Valverde, Senior Chemist (QCG).	University of Almería, Spain.
Dr. Paula Medina, Senior Chemist (QCG).	European Food Safety Authority, Italy.
Dr. Michelangelo Anastassiades, Senior Chemist (AG).	CVUA Stuttgart, Fellbach, Germany.
Dr. Miguel Gamón, Senior Chemist (AG).	Co-Head of EURL-FV. Pesticide Residue Laboratory (Agro-Food Analysis Service) of the Generalitat Valenciana, Spain.
Dr. Philippe Gros, Senior Chemist (AG).	Laboratoire du SCL, Montpellier, France.
Dr. Magnus Jezussek, Senior Chemist (AG).	Bavarian Health and Food Safety Authority, Erlangen, Germany.
Dr. André de Kok, Senior Chemist (AG).	NVWA – Netherlands Food and Consumer Product Safety Authority, Wageningen, The Netherlands.
Mr. Ralf Lippold, Senior Chemist (AG).	CVUA Freiburg, Germany.
Dr. Sonja Masselter, Senior Chemist (AG).	AGES GmbH, Institute for Food Safety Innsbruck, Austria.
Mr. Finbarr O'Regan, Senior Chemist(AG).	Pesticide Control Laboratory, Celbridge, Ireland.
Dr. Tuija Pihlström, Senior Chemist (AG).	National Food Agency, Uppsala, Sweden.
Dr. Mette Erecius Poulsen, Senior Chemist (AG).	National Food Institute, Søborg, Denmark.

QCG: Quality Control Group

AG: Advisory Group

*Authorized by: Dr. Amadeo R. Fernández-Alba  
Co-Head of EURL-FV*



## CONTENTS

1. INTRODUCTION .....	6
2. TEST ITEMS .....	7
2.1 Preparation of the treated test item .....	7
2.2 Preparation of 'blank' test item .....	7
2.3 Homogeneity test .....	8
2.4 Stability tests .....	9
2.5 Distribution of test items and protocol to participants .....	11
3. STATISTICAL METHODS .....	11
3.1 False positives and negatives .....	11
3.2 Estimation of the assigned values ( $x_{pt}$ ) .....	12
3.3 Fixed target standard deviations .....	13
3.4 z scores .....	13
3.5 Combined z scores .....	13
4. RESULTS .....	15
4.1 Summary of reported results .....	15
4.2 Assigned values and target standard deviations .....	23
4.3 Assessment of laboratory performance .....	24
5. CONCLUSIONS .....	31
6. REFERENCES .....	32
7. ACKNOWLEDGEMENTS .....	32
APPENDIX 1. Homogeneity data .....	33
APPENDIX 2. Histograms of residue data for each pesticide from EU/EFTA laboratories. ....	35
APPENDIX 3. Results (mg/Kg) and z scores for FFP RSD (25 %). ....	38
APPENDIX 4. Graphical representation of z scores for FFP RSD (25 %). ....	51
APPENDIX 5. Average of the Squared z scores (AZ <sup>2</sup> ) for laboratories in Category A. ....	70
APPENDIX 6. EUPT-FV-20- AZ <sup>2</sup> - Graphical representation for EU/EFTA laboratories in Category A ..	73
ANNEX 1. Protocols and Target list of pesticides to be sought .....	74
ANNEX 2. List of laboratories that agreed to participate in EUPT-FV-20. ....	91



**EURL-EUROPEAN UNION PROFICIENCY TEST 20**  
**FOR THE DETERMINATION OF PESTICIDES IN FRUIT AND VEGETABLES USING**  
**MULTIRESIDUE METHODS**  
**2018**

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 (EU) 2017/625<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 20 has been organised by the EURL in Fruit and Vegetables at the University of Almería, Spain<sup>4</sup>.

Participation in European Proficiency Test 20 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, Colombia, Costa Rica, Kenya, Peru, Serbia, Singapore, Thailand and Uruguay 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 OfIs within their network. This report may be presented to the European Union Standing Committee on Plants, Animals, Food and Feed (PAFF).

---

<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 (EU) 2017/625 of the European Parliament and of the Council on official controls and other official activities performed to ensure the application of food and feed law, rules on animal health and welfare, plant health and plant protection products. Published in the OJ of the EU L95 on 07.04.2017.

<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 eighty-four laboratories agreed to participate in European Union Proficiency Test 20.

The proficiency test was performed in 2018 using green beans with pods homogenate. The green beans were cultivated in a greenhouse in Almería, Spain, and were treated before harvest using commercial formulations applied by spraying with conventional diffusers and treated post-harvest with analytical standards. Nineteen mandatory pesticides and two voluntary<sup>5</sup> ones were used for the treatment. Participating laboratories were also provided with a 'blank' green bean homogenate as well as the treated green bean test item.

The test items, 200 g of green bean homogenate containing pesticide residues, together with 200 g of 'blank' green beans homogenate, were shipped to participants on 5<sup>th</sup> March 2018. The deadline for results submission to the Organiser was 26<sup>th</sup> March 2018. 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 195 target pesticides. The pesticide target list is detailed in Annex 1 together with the voluntary target list, which contained 34 pesticides. This list of target pesticides also contained the MRL for each pesticide fixed at 0.01 mg/kg, except for the following pesticides which have lower MRLs based on Regulation (EU) No. 396/2005 and EU Directive 2006/125/EC: cadusafos (0.006 mg/kg); dimethoate (0.003 mg/kg); ethoprophos (0.008 mg/kg); fipronil (0.004 mg/kg); omethoate (0.003 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

---

<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-20.

## **2. TEST ITEMS**

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

The green beans were cultivated in a greenhouse 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 formulations were buprofezin, clothianidin, etofenprox, fenpyrazamine, fenpyroximate, metaflumizone, pyridaben, spiromesifen, tau-fluvalinate, tebuconazole, tebufenpyrad and thiametoxam. The pesticides spiked as analytical standards were boscalid, carbendazim, chlorothalonil, diazinon, dimethoate, imazalil, iprodione, penthiopyrad and thiabendazole.

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 20. Approximately 440 m<sup>2</sup> of green bean plants were treated with commercial formulations, which were dissolved in water. One day after the application, a representative sample of the treated green beans was collected and analysed to check if the residue levels present were close to the target levels. As the residue levels in the green beans were close to those recommended by the QCG, the entire production (125 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 green beans were 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 green beans used for the production of the blank test item were grown in the same greenhouse as the test item. Before the treatment of the green beans, 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. (accredited under ISO/IEC 17025 by the Spanish accreditation body, ENAC) after evaluation by the organisation of the PT. 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 procedural standard calibration prepared from the 'blank' green bean 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	0.033	2.0E-6	2.0E-5	Pass
Buprofezin	0.686	2.7E-4	6.1E-3	Pass
Carbendazim	0.048	1.0E-6	2.0E-5	Pass
Chlorothalonil*	0.052	3.8E-4	1.3E-4	Fail
Chlothianidin	0.038	0	2.0E-5	Pass
Diazinon	0.027	0	1.0E-5	Pass
Dimethoate	0.048	0	3.0E-5	Pass
Ethofenprox	0.404	2.3E-4	2.0E-3	Pass
Fenpyroximate	0.382	3.0E-6	1.7E-3	Pass
Imazalil	0.034	1.0E-6	1.0E-5	Pass
Iprodione	0.051	4.0E-6	3.0E-5	Pass
Metaflumizone	0.261	8.1E-5	8.0E-4	Pass
Pyridaben	0.997	1.1E-3	1.4E-2	Pass
Spiromesifen	0.615	1.1E-4	4.1E-3	Pass
Tau-Fluvalinate	0.137	0	2.9E-4	Pass
Tebuconazole	1.220	0	2.2E-2	Pass
Tebufenpyrad	1.060	1.6E-3	1.3E-2	Pass
Thiabendazole	0.052	2.0E-6	3.0E-5	Pass
Thiamethoxam	0.373	2.6E-5	1.5E-3	Pass
<b>Voluntary Pesticides</b>				
Fenpyrazamine*	0.131	0	1.9E-4	Pass
Penthiopyrad*	0.062	1.0E-6	4.0E-5	Pass

$S_s$ : Between-Sampling Standard Deviation

\*Compound out of the accredited scope of the subcontracted laboratory  
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 green bean matrix passed the homogeneity test.

## 2.4 Stability tests

The stability tests were also subcontracted to the laboratory Eurofins-SICA AgriQ S.L. (accredited under ISO/IEC 17025 by the Spanish accreditation body, ENAC) after evaluation by the organisation of the PT. 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 separate occasions:

- Day 1: shortly before the test item shipment, this took place on March 5<sup>th</sup>, 2018.
- Day 3: shortly after the deadline for reporting results, on March 26<sup>th</sup>, 2018.

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 for 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. Tau-fluvalinate did not pass the test simulating the shipment conditions. The concentration in the second day was 0.010 mg/kg lower than in the first day, being the acceptance criteria in that case 0.009 mg/kg. Considering also that the CV of tau-fluvalinate was 26.4 % and that the uncertainty of the assigned value was acceptable, the Scientific Committee (SC) agreed to include it for 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-elapse 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 181_A	Sample 181_B	Sample 201_A	Sample 201_B	Sample 196_A	Sample 196_B	Mean 1	Sample 148_A	Sample 148_B	Sample 092_A	Sample 092_B	Sample 207_A	Sample 207_B	Mean3		
Boscalid	0.041	0.038	0.034	0.034	0.038	0.038	0.037	0.035	0.035	0.032	0.035	0.036	0.034	0.035	-0.003	Pass
Buprofezin	0.670	0.680	0.640	0.630	0.680	0.740	0.673	0.580	0.630	0.650	0.650	0.640	0.630	0.630	-0.043	Pass
Carbendazim	0.043	0.043	0.043	0.045	0.043	0.042	0.043	0.040	0.042	0.044	0.044	0.041	0.041	0.042	-0.001	Pass
Chlorothalonil*	0.043	0.038	0.042	0.035	0.050	0.043	0.042	-	-	0.230	0.220	-	-	0.225	0.183	Fail
Chlothianidin	0.039	0.039	0.037	0.037	0.038	0.039	0.038	0.038	0.038	0.037	0.038	0.037	0.037	0.038	-0.001	Pass

(mg/Kg)	Day 1							Day 3							(M3 - M1)	M3-M1 ≤ 0.3*σ
	Sample 181_A	Sample 181_B	Sample 201_A	Sample 201_B	Sample 196_A	Sample 196_B	Mean 1	Sample 148_A	Sample 148_B	Sample 092_A	Sample 092_B	Sample 207_A	Sample 207_B	Mean3		
Diazinon	0.038	0.035	0.029	0.031	0.031	0.033	0.033	0.033	0.029	0.030	0.032	0.032	0.031	0.031	-0.002	Pass
Dimethoate	0.050	0.049	0.047	0.048	0.048	0.048	0.048	0.046	0.048	0.050	0.049	0.049	0.047	0.048	0.000	Pass
Ethofenprox	0.370	0.390	0.340	0.340	0.370	0.380	0.365	0.340	0.350	0.360	0.360	0.360	0.390	0.360	-0.005	Pass
Fenpyroximate	0.370	0.370	0.360	0.360	0.350	0.360	0.362	0.360	0.370	0.360	0.380	0.360	0.360	0.365	0.003	Pass
Imazalil	0.034	0.035	0.035	0.035	0.030	0.032	0.034	0.033	0.034	0.035	0.036	0.033	0.034	0.034	0.001	Pass
Iprodione	0.060	0.053	0.047	0.049	0.052	0.057	0.053	0.054	0.051	0.048	0.053	0.054	0.050	0.052	-0.001	Pass
Metaflumizone	0.250	0.230	0.240	0.240	0.230	0.230	0.237	0.250	0.250	0.250	0.260	0.270	0.240	0.253	0.017	Pass
Pyridaben	1.000	1.100	0.890	0.900	1.100	1.000	0.998	0.920	0.930	0.970	0.930	0.960	1.000	0.952	-0.047	Pass
Spiromesifen	0.610	0.590	0.580	0.580	0.590	0.580	0.588	0.620	0.590	0.600	0.610	0.620	0.580	0.603	0.015	Pass
Tau-Fluvalinate	0.150	0.140	0.130	0.130	0.140	0.150	0.140	0.140	0.130	0.130	0.140	0.140	0.130	0.135	-0.005	Pass
Tebuconazole	1.200	1.300	1.100	1.100	1.200	1.300	1.200	1.100	1.100	1.200	1.100	1.100	1.100	1.117	-0.083	Pass
Tebufenpyrad	1.100	1.000	1.100	1.000	1.000	1.000	1.033	1.000	1.000	1.100	1.100	1.000	1.000	1.033	0.000	Pass
Thiabendazole	0.053	0.053	0.050	0.049	0.049	0.048	0.050	0.050	0.051	0.053	0.053	0.051	0.053	0.052	0.002	Pass
Thiamethoxam	0.380	0.350	0.350	0.350	0.380	0.350	0.360	0.370	0.360	0.370	0.380	0.360	0.360	0.367	0.007	Pass
<b>Voluntary Pesticides</b>																
Fenpyrazamine*	0.140	0.130	0.130	0.130	0.130	0.130	0.132	0.130	0.130	0.130	0.130	0.130	0.130	0.130	-0.002	Pass
Penthiopyrad*	0.058	0.061	0.059	0.059	0.059	0.059	0.059	0.060	0.060	0.062	0.062	0.059	0.059	0.060	0.001	Pass

The voluntary pesticides are not covered by the ISO/IEC 17043  
 \*Compound out of the accredited scope of the subcontracted laboratory

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 181_A	Sample 181_B	Sample 201_A	Sample 201_B	Sample 196_A	Sample 196_B	Mean 1	Sample 176_A	Sample 176_B	Sample 209_A	Sample 209_B	Sample 175_A	Sample 175_B	Mean2		
Boscalid	0.041	0.038	0.034	0.034	0.038	0.038	0.037	0.033	0.033	0.036	0.035	0.041	0.035	0.036	-0.002	Pass
Buprofezin	0.670	0.680	0.640	0.630	0.680	0.740	0.673	0.640	0.680	0.680	0.640	0.690	0.740	0.678	0.005	Pass
Carbendazim	0.043	0.043	0.043	0.045	0.043	0.042	0.043	0.042	0.043	0.042	0.042	0.042	0.047	0.043	0.000	Pass
Chlorothalonil*	0.043	0.038	0.042	0.035	0.050	0.043	0.042	0.055	0.052	0.069	0.070	0.058	0.058	0.060	0.019	Pass
Chlothianidin	0.039	0.039	0.037	0.037	0.038	0.039	0.038	0.040	0.040	0.037	0.036	0.036	0.037	0.037	-0.001	Pass
Diazinon	0.038	0.035	0.029	0.031	0.031	0.033	0.033	0.028	0.028	0.032	0.033	0.037	0.032	0.032	-0.001	Pass
Dimethoate	0.050	0.049	0.047	0.048	0.048	0.048	0.048	0.050	0.051	0.051	0.050	0.049	0.052	0.050	0.002	Pass
Ethofenprox	0.370	0.390	0.340	0.340	0.370	0.380	0.365	0.300	0.340	0.350	0.350	0.370	0.400	0.352	-0.013	Pass
Fenpyroximate	0.370	0.370	0.360	0.360	0.350	0.360	0.362	0.360	0.360	0.350	0.350	0.340	0.370	0.355	-0.007	Pass
Imazalil	0.034	0.035	0.035	0.035	0.030	0.032	0.034	0.031	0.033	0.037	0.037	0.037	0.036	0.035	0.002	Pass
Iprodione	0.060	0.053	0.047	0.049	0.052	0.057	0.053	0.047	0.048	0.057	0.058	0.058	0.057	0.054	0.001	Pass
Metaflumizone	0.250	0.230	0.240	0.240	0.230	0.230	0.237	0.230	0.240	0.240	0.230	0.240	0.250	0.238	0.002	Pass
Pyridaben	1.000	1.100	0.890	0.900	1.100	1.000	0.998	0.800	0.940	0.940	0.940	1.000	1.100	0.953	-0.045	Pass
Spiromesifen	0.610	0.590	0.580	0.580	0.590	0.580	0.588	0.600	0.590	0.590	0.590	0.570	0.640	0.597	0.008	Pass
Tau-Fluvalinate	0.150	0.140	0.130	0.130	0.140	0.150	0.140	0.120	0.120	0.140	0.140	0.140	0.120	0.130	-0.010	Fail
Tebuconazole	1.200	1.300	1.100	1.100	1.200	1.300	1.200	0.980	1.100	1.100	1.100	1.200	1.300	1.130	-0.070	Pass
Tebufenpyrad	1.100	1.000	1.100	1.000	1.000	1.000	1.033	1.000	0.980	1.000	1.000	1.000	1.100	1.013	-0.020	Pass

(mg/Kg)	Day 1							Day 2							(M2 - M1)	M2-M1 ≤ 0.3*σ
	Sample 181_A	Sample 181_B	Sample 201_A	Sample 201_B	Sample 196_A	Sample 196_B	Mean 1	Sample 176_A	Sample 176_B	Sample 209_A	Sample 209_B	Sample 175_A	Sample 175_B	Mean2		
Thiabendazole	0.053	0.053	0.050	0.049	0.049	0.048	0.050	0.052	0.052	0.054	0.055	0.053	0.055	0.054	0.003	Pass
Thiamethoxam	0.380	0.350	0.350	0.350	0.380	0.350	0.360	0.380	0.340	0.340	0.360	0.360	0.380	0.360	0.000	Pass
<b>Voluntary Pesticides</b>																
Fenpyrazamine*	0.140	0.130	0.130	0.130	0.130	0.130	0.132	0.130	0.130	0.130	0.130	0.130	0.130	0.130	-0.002	Pass
Penthiopyrad*	0.058	0.061	0.059	0.059	0.059	0.059	0.059	0.059	0.058	0.061	0.064	0.061	0.064	0.061	0.002	Pass

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

\*Compound out of the accredited scope of the subcontracted laboratory

## 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 5<sup>th</sup> March 2018. Ninety-seven 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 20. 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 enough 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.

Considering 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-FV12. 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'.

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

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

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).

One hundred and eighty-four laboratories agreed to participate in this proficiency test. Two did not submit results. The total number of laboratories submitting results was 182. 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, Colombia, Costa Rica, Kenya, Peru, Serbia, Singapore, Thailand and Uruguay have not been included. This last group totals 15 laboratories that reported results.

Nineteen 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 three times the MRRL. However, due to the high dispersion of results (see below), the SC agreed that chlorothalonil and metaflumizone should not be considered for the evaluation of the participants.

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 167)
Boscalid	159	1	7	95
Buprofezin	158	2	7	95
Carbendazim and benomyl (sum of benomyl and carbendazim expressed as carbendazim)	140	2	25	84
Chlorothalonil**	93	46	28	56
Clothianidin	141	1	25	84
Diazinon	162	1	4	97
Dimethoate	159	2	6	95
Etofenprox	150	0	17	90
Fenpyroximate	140	3	24	84
Imazalil	154	5	8	92
Iprodione	147	6	14	88

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

Pesticides	No. of Reported Results	No. of False Negative Results	No. of Not Analysed Results	Percentage of Reported Results* (out of 167)
Metaflumizone** (sum of E- and Z- isomers)	116	3	48	69
Pyridaben	157	1	9	94
Spiromesifen	140	1	26	84
Tau-Fluvalinate	149	3	15	89
Tebuconazole	161	0	6	96
Tebufenpyrad	156	0	11	95
Thiabendazole	154	1	12	92
Thiamethoxam	147	0	20	88
<b>Voluntary Pesticides</b>				
Fenpyrazamine	74	1	92	44
Penthiopyrad	70	2	95	42

\* The percentage of Reported Results comes from 167 laboratories. It does not take into account the fifteen laboratories from China, Colombia, Costa Rica, Kenya, Peru, Serbia, Singapore, Thailand and Uruguay.

\*\* Only for informative purposes.

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

The coefficients of variation of chlorothalonil and metaflumizone were 76.4 and 64.3 %, respectively. The causes of such a high dispersion were carefully studied:

#### Chlorothalonil:

The results reported by the participants for chlorothalonil are shown in table 4.2.

Table 4.2 Summary of reported results for chlorothalonil.

Lab Code	Result reported (mg/kg)	Lab Code	Result reported (mg/kg)	Lab Code	Result reported (mg/kg)	Lab Code	Result reported (mg/kg)
Lab001	NR	Lab047	0.233	Lab095	NA	Lab142	NR
Lab002	0.309	Lab048	0.214	Lab096	NA	Lab143	0.757
Lab003	NR	Lab049	NA	Lab097	NA	Lab144	0.682
Lab004	0.364	Lab050	0.219	Lab098	NR	Lab145	NR
Lab005	0.217	Lab051	0.314	Lab099	0.714	Lab146	0.018
Lab006	0.825	Lab052	NR	Lab100	0.198	Lab147	0.656
Lab007	0.485	Lab053	0.184	Lab101	0.391	Lab148	0.350
Lab008	0.202	Lab054	0.366	Lab102	NR	Lab150	0.600
Lab009	NR	Lab055	NR	Lab103	NR	Lab151	0.480
Lab010	NR	Lab057	NR	Lab104	0.012	Lab152	NR
Lab011	0.330	Lab058	0.406	Lab105	NR	Lab153	NR
Lab012	NR	Lab059	0.179	Lab106	NA	Lab154	NA
Lab013	NA	Lab060	NA	Lab107	0.432	Lab155	1.160
Lab014	0.317	Lab061	0.160	Lab108	0.030	Lab156	0.791
Lab015	NR	Lab062	0.811	Lab109	NA	Lab157	0.460
Lab016	0.017	Lab063	1.980	Lab110	0.010	Lab158	0.576
Lab017	NR	Lab064	NR	Lab111	NR	Lab159	0.096



Lab Code	Result reported (mg/kg)	Lab Code	Result reported (mg/kg)	Lab Code	Result reported (mg/kg)	Lab Code	Result reported (mg/kg)
Lab018	0.674	Lab065	0.113	Lab112	NA	Lab160	0.046
Lab019	NA	Lab066	0.950	Lab113	NR	Lab161	0.520
Lab020	NR	Lab068	0.077	Lab114	NA	Lab162	0.146
Lab021	NR	Lab069	0.089	Lab115	0.412	Lab163	NA
Lab022	0.402	Lab070	NR	Lab116	NR	Lab164	NR
Lab023	0.705	Lab071	0.443	Lab117	NR	Lab165	NA
Lab024	0.335	Lab072	NR	Lab118	NA	Lab166	0.230
Lab025	0.319	Lab073	0.569	Lab119	0.781	Lab167	NR
Lab026	0.679	Lab074	NA	Lab120	NA	Lab168	NR
Lab027	0.089	Lab075	NR	Lab121	NR	Lab169	0.164
Lab028	0.540	Lab076	NR	Lab122	NR	Lab170	NA
Lab029	0.200	Lab077	0.322	Lab123	NA	Lab171	0.243
Lab030	NA	Lab078	NR	Lab125	NR	Lab172	NA
Lab031	0.037	Lab079	0.395	Lab126	1.270	Lab173	0.202
Lab032	0.329	Lab080	NR	Lab127	0.041	Lab174	NR
Lab033	NA	Lab081	0.310	Lab128	NR	Lab175	0.067
Lab034	NR	Lab082	NR	Lab129	NR	Lab176	0.176
Lab035	0.250	Lab083	NA	Lab130	0.217	Lab177	NA
Lab036	NA	Lab084	NR	Lab131	NA	Lab178	NA
Lab037	0.200	Lab085	0.564	Lab132	NR	Lab179	0.130
Lab038	0.870	Lab086	0.727	Lab133	NA	Lab180	NA
Lab039	NR	Lab087	0.950	Lab134	NA	Lab181	NR
Lab040	NR	Lab088	NA	Lab135	0.430	Lab182	NR
Lab041	0.189	Lab089	0.810	Lab136	0.239	Lab183	0.015
Lab042	0.197	Lab090	0.223	Lab137	NR	Lab184	NR
Lab043	0.251	Lab091	0.536	Lab138	0.020	Lab185	0.304
Lab044	NR	Lab092	0.075	Lab139	0.063	Lab186	1.007
Lab045	NA	Lab093	0.099	Lab140	0.544		
Lab046	0.593	Lab094	0.364	Lab141	0.940		

NA: Not analysed  
NR: Not reported

The number of false negative results was 46. Figure 4.1 represents the z scores of the results reported by the laboratories. The different colors represent the extraction solvent used for the extraction of chlorothalonil in green beans.

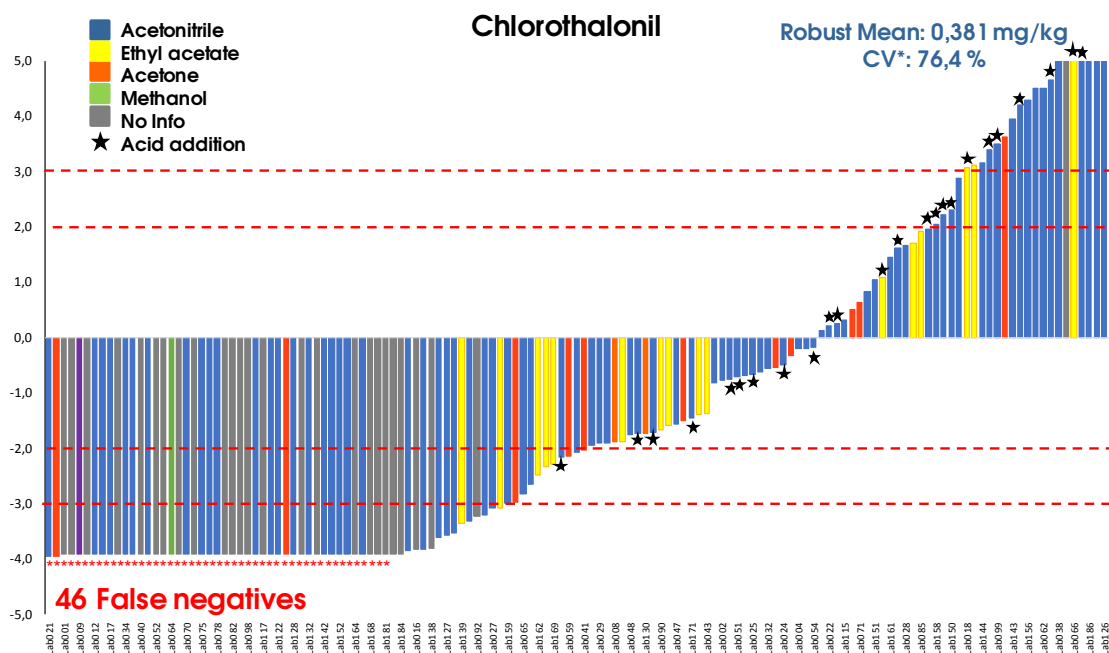


Figure 4.1. z score representation of the results reported.

In previous EUPTs in which chlorothalonil was present in the test item, it was observed that some extraction methods were not suitable for chlorothalonil in specific matrices, as was the case of chlorothalonil in leek using acetonitrile-based methods (EUPT-FV12). In EUPT-FV20, the ethyl acetate based-method seemed to be the most effective one, as there were no false negative results when chlorothalonil was extracted with that solvent.

Furthermore, it was detected that the concentration level of chlorothalonil decreased over time. So, an additional test was performed to the EUPT-FV20 test item over a nine-day period. The same sample was extracted every day and kept in the freezer in the meantime, but the concentration of chlorothalonil still decreased with time, showing stability problems.

In the case of EUPT-FV12, the problem with the extraction of chlorothalonil in leek was related to the sulphur compounds naturally present in leek<sup>7</sup>. In green beans there is no reference to its content in sulphur, but the ones used for the test item were treated with sulphur (as fungicide) in the greenhouse. In order to investigate if sulphur was the cause of the problems found, several trials were performed. Parallel analyses were made, spiking chlorothalonil to an organic sample of green beans and sulphur was added at increasing concentrations to some of them. Afterwards, these samples were extracted with the ethyl acetate method. The recoveries when no sulphur was added were around 80%, and the more sulphur was added, the lower the recoveries were. The spiked material was extracted in two consecutive days, and on the second day, in the sample containing 1000 ppm of sulphur, chlorothalonil could not be recovered at all. Additionally, in order to study if sulphur affected only the extraction or also the analysis, sulphur was spiked at increasing concentrations to analytical standards of chlorothalonil in solvent, and

<sup>7</sup> Determination of chlorothalonil in difficult-to-analyse vegetable matrices using various multiresidue methods. Belmonte Valles, N., Retamal, M., Martínez-Uroz, M.A., Mezcua M., Fernández-Alba, A.R., De Kok, A. Analyst Vol 137, Issue 10, 2012, Pp. 2513-2520

they were analysed in the same sequence. The relative responses decreased when the concentration of sulphur increased. It was also observed that sulphur was retained in the liner, and it was making chlorothalonil to be retained as well.

Chlorothalonil's metabolite, 4-hydroxychlorothalonil, was also monitored: the same analytical standard of chlorothalonil spiked with 0.050 mg/kg of sulphur was analysed six consecutive times, and in all of them the concentration of 4-hydroxychlorothalonil was stable, whereas chlorothalonil area decreased with the injections. This showed that chlorothalonil was not being degraded in the vial, but instead it was being retained in the liner during the injection. Based on the results, the SC decided to present the results of chlorothalonil only for informative purposes.

Metaflumizone:

Table 4.3 shows the results reported by the participant laboratories for metaflumizone.

Table 4.3 Summary of reported results for metaflumizone (sum of E- and Z- isomers).

Lab Code	Result reported (mg/kg)	Lab Code	Result reported (mg/kg)	Lab Code	Result reported (mg/kg)	Lab Code	Result reported (mg/kg)
Lab001	0.310	Lab047	0.679	Lab095	0.276	Lab142	NA
Lab002	0.900	Lab048	0.721	Lab096	NA	Lab143	0.336
Lab003	0.200	Lab049	0.186	Lab097	NA	Lab144	0.275
Lab004	0.452	Lab050	0.672	Lab098	0.250	Lab145	0.320
Lab005	1.040	Lab051	0.215	Lab099	0.197	Lab146	0.791
Lab006	0.531	Lab052	0.290	Lab100	0.232	Lab147	NA
Lab007	0.700	Lab053	0.450	Lab101	NA	Lab148	0.290
Lab008	0.810	Lab054	NA	Lab102	NA	Lab150	NA
Lab009	1.252	Lab055	0.233	Lab103	NA	Lab151	NA
Lab010	0.670	Lab057	0.239	Lab104	0.918	Lab152	0.287
Lab011	NA	Lab058	0.266	Lab105	NA	Lab153	0.479
Lab012	0.250	Lab059	0.377	Lab106	0.692	Lab154	0.178
Lab013	NA	Lab060	0.226	Lab107	NA	Lab155	0.406
Lab014	1.095	Lab061	0.730	Lab108	NA	Lab156	NA
Lab015	ND	Lab062	0.257	Lab109	NA	Lab157	NA
Lab016	0.511	Lab063	NA	Lab110	NA	Lab158	0.845
Lab017	0.627	Lab064	0.233	Lab111	ND	Lab159	NA
Lab018	0.230	Lab065	0.246	Lab112	NA	Lab160	NA
Lab019	0.244	Lab066	1.388	Lab113	NA	Lab161	NA
Lab020	NA	Lab068	0.270	Lab114	NA	Lab162	0.293
Lab021	NA	Lab069	1.000	Lab115	0.722	Lab163	NA
Lab022	0.800	Lab070	1.050	Lab116	0.320	Lab164	0.370
Lab023	0.265	Lab071	0.450	Lab117	0.560	Lab165	0.090
Lab024	0.969	Lab072	0.283	Lab118	0.136	Lab166	0.200
Lab025	1.120	Lab073	0.793	Lab119	0.291	Lab167	0.144
Lab026	0.830	Lab074	NA	Lab120	NA	Lab168	ND
Lab027	0.285	Lab075	NA	Lab121	0.766	Lab169	NA
Lab028	0.250	Lab076	0.421	Lab122	0.280	Lab170	NA

Lab029	1.505	Lab077	0.306	Lab123	NA	Lab171	0.266
Lab030	0.317	Lab078	NA	Lab125	0.163	Lab172	0.220
Lab031	0.259	Lab079	0.461	Lab126	0.276	Lab173	NA
Lab032	0.514	Lab080	0.263	Lab127	0.176	Lab174	0.443
Lab033	0.249	Lab081	0.266	Lab128	0.326	Lab175	NA
Lab034	0.325	Lab082	NA	Lab129	0.232	Lab176	0.348
Lab035	NA	Lab083	0.670	Lab130	0.701	Lab177	0.202
Lab036	NA	Lab084	NA	Lab131	NA	Lab178	0.252
Lab037	0.920	Lab085	0.150	Lab132	0.240	Lab179	0.700
Lab038	NA	Lab086	NA	Lab133	NA	Lab180	NA
Lab039	NA	Lab087	1.650	Lab134	NA	Lab181	NA
Lab040	0.260	Lab088	NA	Lab135	NA	Lab182	NA
Lab041	NA	Lab089	0.265	Lab136	0.180	Lab183	NA
Lab042	0.717	Lab090	NA	Lab137	0.231	Lab184	0.660
Lab043	0.195	Lab091	1.240	Lab138	0.190	Lab185	NA
Lab044	0.320	Lab092	0.260	Lab139	NA	Lab186	0.808
Lab045	ND	Lab093	NA	Lab140	0.270		
Lab046	1.620	Lab094	0.680	Lab141	0.260		

NA: Not analysed  
ND: Not detected

The representation of the z scores (Figure 4.2) shows an overestimation of the concentration for a high number of results.

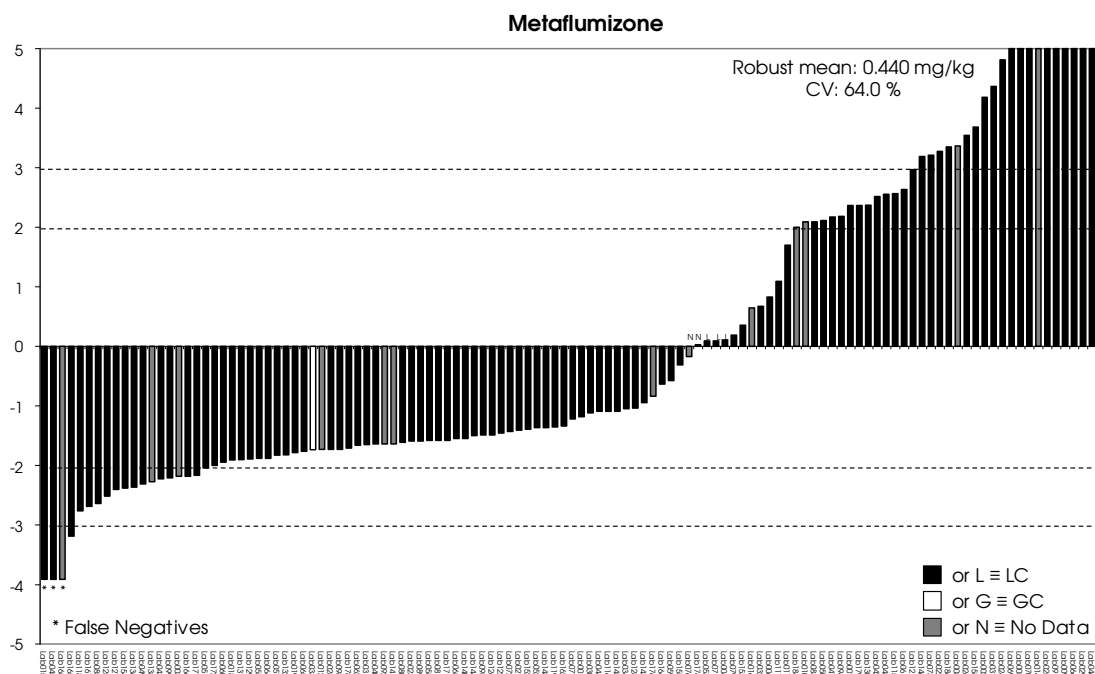


Figure 4.2. z score representation of the results reported.

When the histogram was represented and studied (see Appendix 2. Histograms of residue data for each pesticide from EU/EFTA laboratories.), the modality test showed suspicion of bimodality.

Metaflumizone is composed of two isomers: E and Z, and the residue definition is the sum of both isomers. In the PT sample, both isomers were present because it was applied as a commercial formulation. When purchasing metaflumizone, some analytical standard suppliers are not clear whether it contains both isomers or only one. If the isomer Z is purchased individually and analysed, two peaks appear in the same chromatogram (isomer E and Z, see figure 4.3) with a ratio between areas of 1.7 (isomer E/isomer Z). In the EUPT sample, the ratio between the isomers was 2.9. On the other hand, the individual analytical standard of metaflumizone E presents only one peak. That could be one of the causes of bimodality: some laboratories only had the analytical standard of isomer E, and they were quantifying both isomers E and Z in the sample.

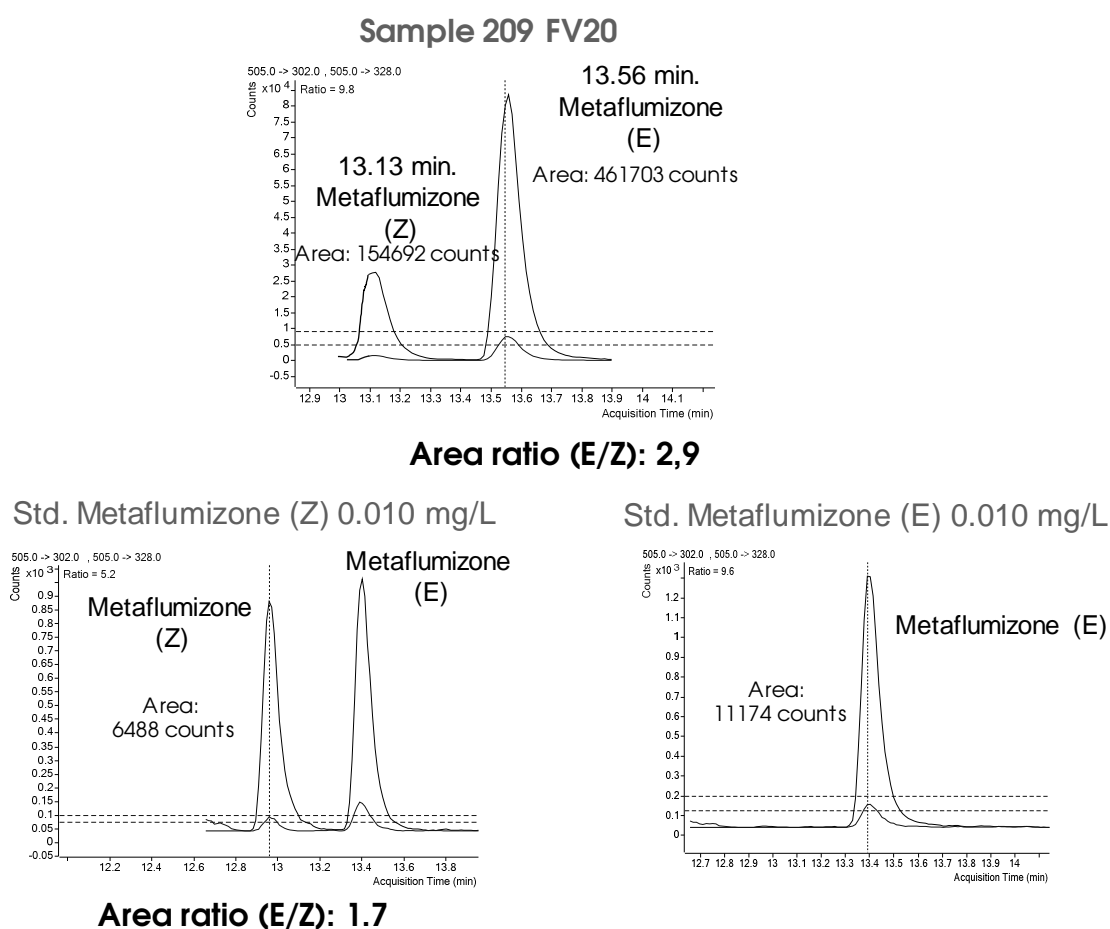


Figure 4.3. LC chromatograms of metaflumizone (E and Z) in the sample (top), of a standard of metaflumizone Z (down, left) and of a standard of metaflumizone E (down, right).

Other laboratories were not aware of the presence of the two isomers, and they had the retention time window acquisition set only at the retention time of isomer E, which is the most abundant one, so they were not able to see both isomers in the test item, and thus, they were only reporting the results for isomer E. The aforementioned situations lead to a big dispersion of the results and to bimodality, and thus, the SC decided to show the results only for informative purposes.

#### 4.1.1 False positives

Five 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 4.4 together with the MRRs and reporting levels (RLs). Where the reported concentrations of the erroneously-detected pesticide were higher than the assigned MRR value in the Target Pesticide List (Annex 1), the result has been considered as a false positive. If the concentrations reported were below the MRRs, or if the pesticides did not appear in the pesticide list included in Annex 1, then they were not considered to be false positives.

Table 4.4 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)	MRR (mg/kg)
Lab013	Cypermethrin	0.05		0.01	0.01
Lab182	Cypermethrin	0.411	GC-Ion Trap	0.01	0.01
Lab039	Epoxiconazole	0.034	GC-MS/MS (QQQ)	0.01	0.01
Lab108*	Cypermethrin	0.06	GC-MS/MS (QQQ)	0.01	0.01
Lab154	Spirodiclofen	0.0821	LC-MS/MS QQQ	0.01	0.01

\*Non-EU/EFTA laboratories

#### 4.1.2 False negatives

Table 4.5 summarises the results from laboratories (including non-EU laboratories) that reported false negatives presented as 'Not Detected' (ND).

Table 4.5 Laboratories that failed to report pesticides that were present in the treated test item.

Laboratory Code	Boscalid	Buprofezin	Carbendazim	Clothianidin	Diazinon	Dimethoate	Fenprothymate	Imazalil	Iprodione	Pyridaben	Spyromesifen	Tau-Fluvalinate	Thiabendazole
Lab015													
Lab017												ND	
Lab019									ND				
Lab020			ND			ND		ND	ND				ND
Lab021							ND	ND			ND		
Lab031									ND				
Lab044									ND				
Lab045				ND									
Lab078			ND										
Lab082							ND		ND				
Lab097												ND	
Lab102*												ND	
Lab103*												ND	
Lab104*									ND				

Laboratory Code	Boscalid	Buprofezin	Carbendazim	Clothianidin	Diazinon	Dimeethoate	Fenpyroximate	Imazalil	Iprodione	Pyridaben	Spyromesifen	Tau-Fluvalinate	Thiabendazole
Lab111*							ND						
Lab112*									ND				
Lab168							ND						
Lab173	ND	ND						ND		ND		ND	
Lab181								ND					
Lab182		ND			ND	ND		ND	ND				
Voluntary Pesticides													
Fenpyrazamine							Penthiopyrad						
Lab064							ND						
Lab176							ND						
Lab179	ND												

\*Non-EU/EFTA laboratories  
ND: Not detected

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 4.6.

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 4.6 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	0.010	0.039	0.0007	159	25	16.9
Buprofezin	0.010	0.738	0.0129	158	25	17.6
Carbendazim and benomyl (sum of benomyl and carbendazim expressed as	0.010	0.039	0.0008	140	25	20.8

Pesticides	MRRL (mg/kg)	Robust mean (mg/kg)	Uncertainty (mg/kg)	Number of results (n)	FFP-RSD (%)	CV* (%)
carbendazim)						
Clothianidin	0.010	0.036	0.0006	141	25	15.4
Diazinon	0.010	0.044	0.0008	162	25	17.7
Dimethoate	0.003	0.049	0.0008	159	25	16.2
Etofenprox	0.010	0.372	0.0069	150	25	18.1
Fenpyroximate	0.010	0.377	0.0087	140	25	21.7
Imazalil	0.010	0.038	0.0010	154	25	27.1
Iprodione	0.010	0.056	0.0014	147	25	24.5
Pyridaben	0.010	0.935	0.0164	157	25	17.6
Spiromesifen	0.010	0.603	0.0132	140	25	20.7
Tau-Fluvalinate	0.010	0.122	0.0032	149	25	26.0
Tebuconazole	0.010	1.220	0.0201	161	25	16.7
Tebufenpyrad	0.010	1.029	0.0179	156	25	17.4
Thiabendazole	0.010	0.048	0.0009	154	25	18.6
Thiamethoxam	0.010	0.367	0.0057	147	25	15.1
<b>Voluntary Pesticides</b>						
Fenpyrazamine	0.010	0.131	0.003	74	25	15.6
Penthiopyrad	0.010	0.058	0.001	70	25	17.1

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 4.7 Classification of z scores for the pesticides reported (only EU/EFTA participants)

Pesticides	Acceptable (%)	Questionable (%)	Unacceptable (%)
Boscalid	97.4	1.3	1.3
Buprofezin	98.1	0.6	1.3
Carbendazim and benomyl (sum of benomyl and carbendazim expressed as carbendazim)	95.8	1.4	2.8
Clothianidin	97.9	2.1	0.0
Diazinon	93.9	4.9	1.2
Dimethoate	95.6	2.5	1.9
Etofenprox	94.0	2.7	3.3
Fenpyroximate	92.3	2.8	4.9
Imazalil	91.2	6.3	2.5



Pesticides	Acceptable (%)	Questionable (%)	Unacceptable (%)
Iprodione	88.2	4.6	7.2
Pyridaben	96.2	1.9	1.9
Spiromesifen	90.8	7.1	2.1
Tau-Fluvalinate	88.2	5.9	5.9
Tebuconazole	98.1	1.3	0.6
Tebufenpyrad	97.4	1.3	1.3
Thiabendazole	92.3	4.5	3.2
Thiamethoxam	98.6	1.4	0.0
<b>Voluntary Pesticides</b>			
Fenpyrazamine	94.7	4.0	1.3
Penthiopyrad	94.4	1.4	4.2

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 (16), to detect and quantify at least 90 % of the pesticides present in the Test Item (195), and that did 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 eleven of the 167 EU and EFTA laboratories that submitted results were classified into Category A (66 %).

From the  $AZ^2$ , 94 % were classed as 'good', 5 % as 'satisfactory' and 1 % as 'unsatisfactory' (Only considering EU and EFTA laboratories).

Of the 56 EU and EFTA laboratories in Category B, four had reported a false positive result.

Table 4.8 shows all the laboratories in Category A, the number of pesticides reported, the percentage of pesticides analysed from the compulsory target list, the AZ<sup>2</sup> values and their subclassifications. Laboratories that reported false negative results in Category A are marked with an asterisk.

Table 4.9 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<sup>2</sup> 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 distinct colour.

Laboratory performance over the last three EUPTs using the AZ<sup>2</sup> formula has been summarized as follows:

- For EUPT-FV-20, out of 167 laboratories (EU and EFTA), 111 were in Category A with the following classes: 1 'unsatisfactory', 6 'satisfactory' and 104 'good'.
- 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'.

Table 4.8 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
Lab001	17	100	0,5	Good
Lab002	17	100	0,4	Good
Lab003	17	98	0,6	Good
Lab004	17	99	0,5	Good
Lab005	17	100	0,2	Good
Lab006	16	92	0,6	Good
Lab007	17	100	1,0	Good
Lab008	17	99	0,3	Good
Lab009	17	100	0,4	Good
Lab010	17	99	3,2	Unsatisfactory
Lab012	17	100	0,2	Good
Lab014	17	100	0,8	Good
Lab015	17	99	0,2	Good
Lab016	17	99	0,7	Good
Lab017*	16	96	1,2	Good
Lab018	17	100	0,3	Good

Lab Code	No. of pesticides detected (max. 17)	% of pesticides analysed from target list	AZ <sup>2</sup>	Classification
Lab019*	16	92	0,9	Good
Lab023	17	100	2,5	Satisfactory
Lab024	17	100	0,6	Good
Lab025	17	100	0,6	Good
Lab026	17	98	0,3	Good
Lab027	17	91	0,6	Good
Lab028	17	98	0,5	Good
Lab029	17	100	0,5	Good
Lab031*	16	99	0,7	Good
Lab032	17	98	0,9	Good
Lab033	17	99	0,4	Good
Lab034	17	93	1,3	Good
Lab037	17	97	1,5	Good
Lab038	17	98	0,3	Good
Lab040	17	100	0,7	Good
Lab042	17	100	0,2	Good
Lab043	17	100	0,8	Good
Lab044*	16	100	1,8	Good
Lab046	17	99	0,2	Good
Lab047	17	100	0,2	Good
Lab048	17	100	0,1	Good
Lab049	17	96	0,8	Good
Lab050	17	100	0,1	Good
Lab051	17	98	0,3	Good
Lab052	17	100	0,4	Good
Lab053	17	100	0,1	Good
Lab055	17	100	0,6	Good
Lab057	17	100	0,2	Good
Lab058	17	99	0,3	Good
Lab059	17	99	1,4	Good
Lab061	17	100	0,2	Good
Lab062	17	99	0,3	Good
Lab064	17	98	0,7	Good
Lab065	17	98	1,7	Good
Lab066	17	100	0,3	Good
Lab068	17	96	1,0	Good
Lab069	17	100	0,2	Good
Lab070	17	100	1,4	Good
Lab071	17	91	0,7	Good
Lab072	17	97	0,3	Good
Lab073	17	100	0,5	Good
Lab076	17	96	1,9	Good
Lab077	17	100	1,4	Good
Lab078*	15	91	2,5	Satisfactory

Lab Code	No. of pesticides detected (max. 17)	% of pesticides analysed from target list	AZ <sup>2</sup>	Classification
Lab079	17	100	0,2	Good
Lab080	17	99	0,5	Good
Lab081	17	99	0,3	Good
Lab083	17	99	0,6	Good
Lab085	17	96	0,3	Good
Lab087	17	100	0,5	Good
Lab089	17	100	0,4	Good
Lab091	17	97	0,6	Good
Lab092	17	100	0,3	Good
Lab093	17	97	0,6	Good
Lab094	17	99	1,8	Good
Lab095	17	97	0,6	Good
Lab098	17	94	1,6	Good
Lab099	17	98	0,6	Good
Lab100	17	99	1,9	Good
Lab102*	16	98	2,6	Satisfactory
Lab103*	16	98	3,5	Unsatisfactory
Lab104*	16	100	1,1	Good
Lab106	17	98	2,2	Satisfactory
Lab115	17	93	0,6	Good
Lab116	17	99	0,2	Good
Lab117	17	100	0,3	Good
Lab119	17	97	0,8	Good
Lab121	17	100	0,2	Good
Lab122	17	100	0,4	Good
Lab123	17	93	0,4	Good
Lab125	17	96	0,1	Good
Lab126	17	91	1,7	Good
Lab127	17	99	0,5	Good
Lab128	17	99	2,3	Satisfactory
Lab130	17	99	0,3	Good
Lab136	17	98	1,6	Good
Lab137	17	96	0,8	Good
Lab138	17	99	0,8	Good
Lab140	17	100	0,2	Good
Lab141	17	98	0,5	Good
Lab143	17	97	0,5	Good
Lab144	17	100	0,6	Good
Lab145	17	100	0,3	Good
Lab146	17	100	1,1	Good
Lab148	17	100	1,1	Good
Lab152	17	99	2,1	Satisfactory
Lab155	17	95	1,4	Good
Lab158	17	98	0,1	Good

Lab Code	No. of pesticides detected (max. 17)	% of pesticides analysed from target list	AZ <sup>2</sup>	Classification
Lab162	17	97	1,0	Good
Lab164	17	98	2,8	Satisfactory
Lab166	17	91	0,5	Good
Lab167	17	99	2,4	Satisfactory
Lab171	17	100	0,2	Good
Lab172	17	92	0,2	Good
Lab174	17	100	1,8	Good
Lab176	17	94	0,6	Good
Lab177	16	93	0,4	Good
Lab179	17	95	0,2	Good
Lab184	17	100	0,3	Good
Lab186	17	100	0,5	Good

\* Laboratories reporting a false negative result.

Table 4.9 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)
Lab011	76	13	63	13	13
Lab013+	53	9	34	9	7
Lab020*	47	8	69	13	7
Lab021*	76	13	89	16	13
Lab022	100	17	85	17	16
Lab030	88	15	59	15	15
Lab035	94	16	65	16	15
Lab036	94	16	78	16	16
Lab039+	82	14	77	14	14
Lab041	100	17	68	17	17
Lab045*	88	15	71	16	14
Lab054	100	17	85	17	17
Lab060	94	16	76	16	15
Lab063	94	16	81	16	16
Lab074	71	12	78	12	11
Lab075	88	15	79	15	14
Lab082*	59	10	52	12	8
Lab084	76	13	59	13	13
Lab086	100	17	88	17	17
Lab088	24	4	30	4	4
Lab090	65	11	51	11	10
Lab096	24	4	16	4	3
Lab097*	71	12	63	13	12
Lab101	88	15	82	15	15
Lab105	100	17	85	17	16

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)
Lab107	100	17	84	17	17
Lab108+	82	14	64	14	11
Lab109	0	0	14	0	0
Lab110	76	13	59	13	11
Lab111*	94	16	90	17	13
Lab112*	29	5	17	6	2
Lab113	82	14	67	14	12
Lab114	24	4	19	4	4
Lab118	76	13	71	13	10
Lab120	53	9	26	9	8
Lab129	82	14	78	14	14
Lab131	82	14	65	14	14
Lab132	88	15	87	15	14
Lab133	100	17	87	17	14
Lab134	53	9	34	9	9
Lab135	94	16	79	16	16
Lab139	82	14	72	14	8
Lab142	82	14	70	14	14
Lab147	76	13	66	13	13
Lab150	82	14	67	14	14
Lab151	29	5	29	5	5
Lab153	100	17	82	17	14
Lab154+	88	15	75	15	13
Lab156	82	14	69	14	14
Lab157	65	11	56	11	11
Lab159	88	15	49	15	14
Lab160	41	7	42	7	6
Lab161	76	13	72	13	12
Lab163	94	16	87	16	15
Lab165	88	15	84	15	13
Lab168*	94	16	76	17	15
Lab169	59	10	54	10	10
Lab170	35	6	44	6	6
Lab173*	65	11	87	16	5
Lab175	18	3	19	3	0
Lab178	94	16	89	16	15
Lab180	6	1	14	1	1
Lab181*	29	5	28	6	4
Lab182*+	41	7	61	12	0
Lab183	88	15	73	15	14
Lab185	88	15	68	15	13

\* 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 eighty-four laboratories agreed to participate in EUPT-FV-20. Two did not submit results for the analysis of the green bean homogenate test item. From the remaining 182 laboratories that submitted results, 15 did not belong to EU nor EFTA countries, so their results were not considered for the estimation of the assigned value.

From the total 21 pesticides used to treat the test item, 17 mandatory and two voluntary pesticides were evaluated in EUPT-FV-20, based on the analysis of green beans with pods homogenate. Two of the mandatory pesticides (chlorothalonil and metaflumizone) were excluded of the data treatment by the SC due to the high dispersion of the results caused by difficulties in their analysis.

Of a total number of 2839 possible determinations from EU/EFTA laboratories (167 laboratories by 17 evaluated pesticides), 90.7 % results were reported, 8.3 % were not analysed and 1.0 % were not detected (false negative results). The false positive rate was 0.1 %.

The total number of z scores of laboratories from EU/EFTA countries was 2603, with 94.5 % of them acceptable, 3.1 % questionable and 2.4 % unacceptable.

66 % of the EU and EFTA laboratories that submitted results were classified into Category A. Of them, 94 % were classed as 'good', 5 % as 'satisfactory' and 1 % as 'unsatisfactory'.

The robust standard deviation (CV\*) was in all cases below 27.1 %, with an average value of 19.3 % for the 17 pesticides evaluated.

Participation in this year's European Proficiency Test 20 involved at least one laboratory from each Member State. Additionally, laboratories from Iceland, Norway and Switzerland participated as EFTA countries. As laid down in paragraph 2 (h) of Article 94 of Regulation (EU) 2017/625, 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, Colombia, Costa Rica, Kenya, Peru, Serbia, Singapore, Thailand and Uruguay participated in EUPT-FV-20.

## 6. REFERENCES

1. M. Thompson, S. L. R. Ellison, and R. Wood. The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories. *Pure Appl. Chem.*, 2006, 78 (1), 145–196.
2. ISO 13528:2015. Statistical methods for use in proficiency testing by interlaboratory comparison. International Organization for Standardization.
3. P. Medina-Pastor, C. Rodriguez-Torreblanca, A. Andersson, A. R. Fernandez-Alba. European Commission proficiency tests for pesticide residues in fruits and vegetables. *Trends in Analytical Chemistry*, 2010, 29 (1), 70-83.
4. P. Medina Pastor, A. Valverde, T. Pihlström, S. Masselter, M. Gamón, M. Mezcua, C. Rodríguez Torreblanca, A. R. Fernández-Alba. Comparative Study of the Main Top-down Approaches for the Estimation of Measurement Uncertainty in Multiresidue Analysis of Pesticides in Fruits and Vegetables. *J. Agric. Food Chem.*, 2011, 59 (14), 7609-7619.
5. P. Medina-Pastor, M. Mezcua, C. Rodríguez-Torreblanca, A. R. Fernández-Alba. Laboratory assessment by combined z-score values in proficiency tests: experience gained through the European Union proficiency tests for pesticide residues in fruits and vegetables. *Anal. Bioanal. Chem.*, 2010, 397, 3061–3070.

## 7. ACKNOWLEDGEMENTS

The Organiser is most grateful to the European Commission for funding this European Proficiency Test FV-20.

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)		Burpofezin (mg/kg)		Carbendazim (mg/kg)		Chlorothalonil (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.033	0.033	0.710	0.730	0.047	0.045	0.019	0.034
0.031	0.034	0.670	0.730	0.049	0.048	0.064	0.065
0.032	0.034	0.680	0.690	0.049	0.049	0.047	0.053
0.033	0.036	0.770	0.670	0.049	0.048	0.058	0.058
0.038	0.035	0.640	0.670	0.049	0.049	0.110	0.075
0.033	0.030	0.720	0.640	0.047	0.048	0.055	0.035
0.034	0.033	0.710	0.710	0.048	0.048	0.080	0.070
0.029	0.033	0.690	0.690	0.047	0.047	0.036	0.038
0.033	0.034	0.650	0.670	0.048	0.048	0.032	0.040
0.032	0.027	0.630	0.640	0.049	0.047	0.031	0.036

Clothianidin (mg/kg)		Diazinon (mg/kg)		Dimethoate (mg/kg)		Ethofenprox (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.038	0.038	0.027	0.028	0.047	0.048	0.400	0.420
0.039	0.038	0.026	0.027	0.050	0.048	0.410	0.420
0.039	0.038	0.027	0.029	0.050	0.050	0.400	0.410
0.038	0.037	0.027	0.029	0.048	0.047	0.450	0.420
0.038	0.038	0.030	0.027	0.049	0.049	0.390	0.400
0.037	0.037	0.026	0.026	0.047	0.048	0.430	0.380
0.038	0.038	0.027	0.026	0.048	0.048	0.420	0.400
0.038	0.038	0.024	0.028	0.048	0.048	0.410	0.420
0.037	0.038	0.028	0.029	0.048	0.048	0.370	0.410
0.039	0.038	0.026	0.024	0.049	0.048	0.360	0.360

Fenpyroximate (mg/kg)		Imazalil (mg/kg)		Fluopyram (mg/kg)		Metaflumizone (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.400	0.390	0.033	0.033	0.054	0.054	0.280	0.260
0.370	0.380	0.035	0.034	0.046	0.049	0.250	0.240
0.390	0.380	0.036	0.036	0.050	0.055	0.270	0.270
0.380	0.390	0.035	0.034	0.048	0.054	0.260	0.260
0.390	0.380	0.033	0.034	0.054	0.051	0.270	0.260
0.360	0.380	0.034	0.035	0.048	0.047	0.250	0.260
0.360	0.390	0.035	0.035	0.052	0.048	0.260	0.280
0.370	0.380	0.033	0.032	0.047	0.051	0.230	0.250
0.380	0.390	0.033	0.034	0.054	0.054	0.270	0.270
0.390	0.380	0.032	0.035	0.048	0.047	0.270	0.260

The sample numbers used for this test were: 8, 20, 52, 64, 106, 164, 180, 211, 212 and 248.

**APPENDIX 1. Homogeneity data.**

Pyridaben (mg/kg)		Spiromesifen (mg/kg)		Tau-Fluvalinate (mg/kg)		Tebuconazole (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
1.000	1.000	0.650	0.630	0.140	0.140	1.200	1.200
1.100	1.000	0.600	0.620	0.130	0.150	1.300	1.300
0.980	1.000	0.630	0.620	0.130	0.140	1.200	1.300
1.100	1.000	0.600	0.610	0.130	0.140	1.400	1.200
1.000	0.990	0.640	0.610	0.150	0.140	1.200	1.200
1.100	0.940	0.600	0.600	0.140	0.130	1.300	1.100
1.100	0.990	0.600	0.620	0.140	0.140	1.300	1.200
1.000	1.000	0.590	0.600	0.130	0.140	1.200	1.200
0.890	0.990	0.620	0.610	0.130	0.140	1.100	1.200
0.890	0.870	0.630	0.610	0.140	0.110	1.200	1.100

Tebufenpyrad (mg/kg)		Thiabendazole (mg/kg)		Thiametoxam (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
1.100	1.100	0.050	0.051	0.380	0.380
1.000	1.000	0.052	0.051	0.360	0.360
1.000	1.100	0.056	0.055	0.380	0.370
1.100	1.100	0.051	0.051	0.370	0.380
1.100	1.100	0.052	0.052	0.360	0.380
1.000	1.000	0.051	0.050	0.370	0.380
1.000	1.100	0.051	0.052	0.360	0.370
1.100	1.100	0.050	0.052	0.370	0.380
1.000	1.000	0.050	0.050	0.370	0.370
1.100	1.100	0.052	0.051	0.380	0.390

**Voluntary Pesticides**

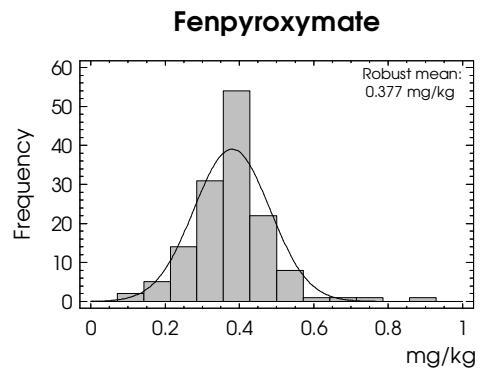
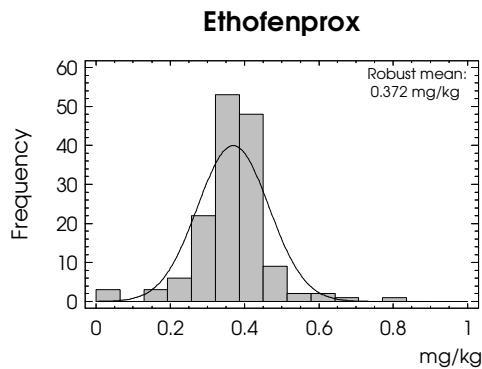
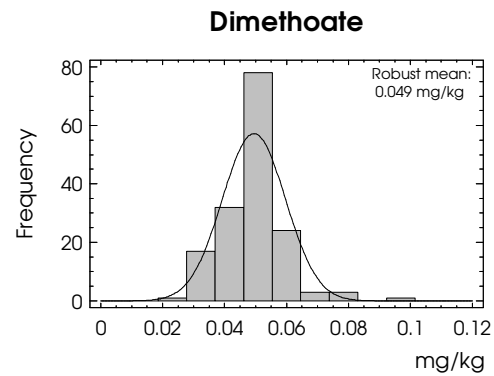
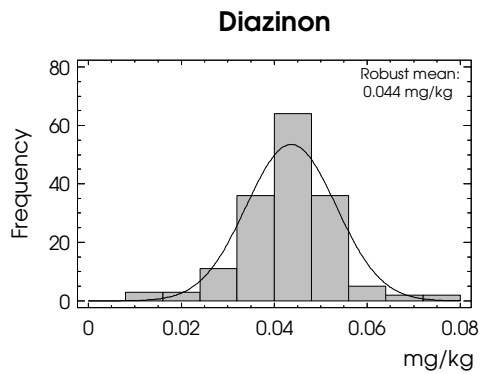
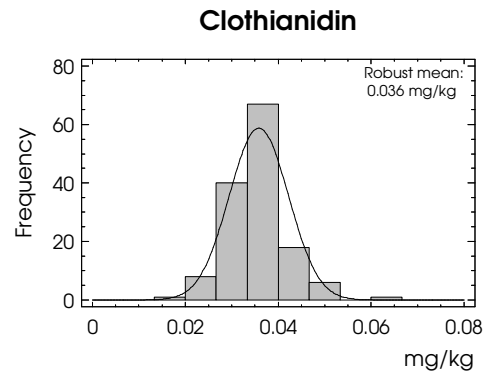
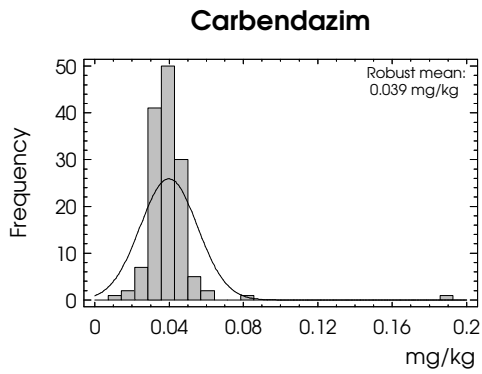
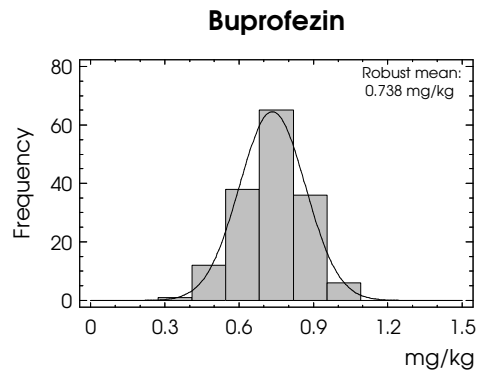
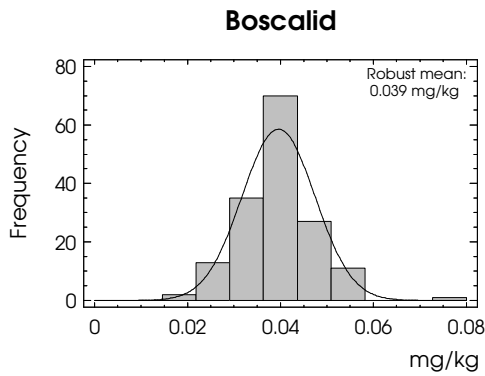
Fenpyrazamine (mg/kg)		Penthiopyrad (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.140	0.130	0.060	0.060
0.130	0.130	0.064	0.061
0.130	0.130	0.064	0.063
0.130	0.130	0.064	0.063
0.140	0.130	0.061	0.062
0.130	0.130	0.062	0.062
0.130	0.130	0.063	0.063
0.130	0.130	0.062	0.060
0.130	0.130	0.062	0.064
0.130	0.130	0.063	0.061

The sample numbers used for this test were: 8, 20, 52, 64, 106, 164, 180, 211, 212 and 248.

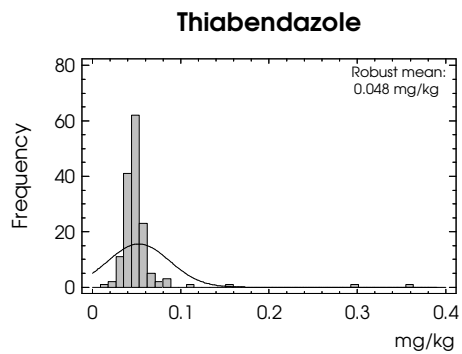
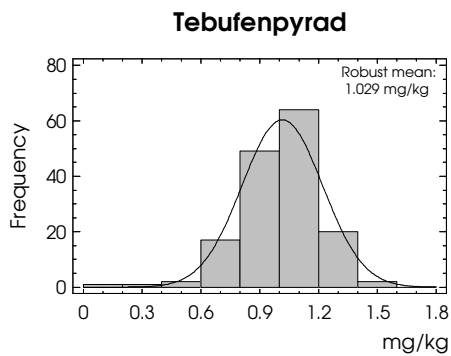
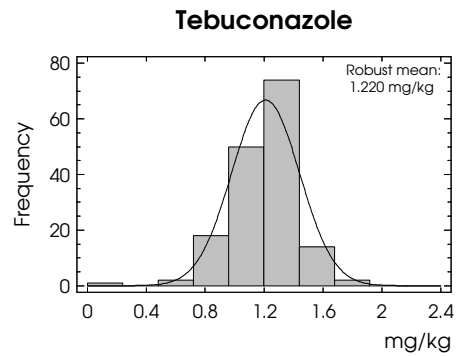
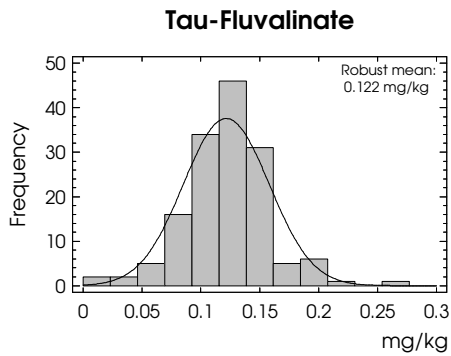
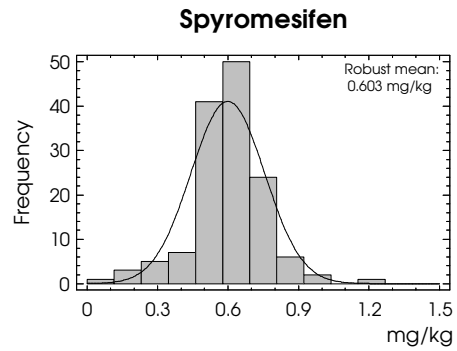
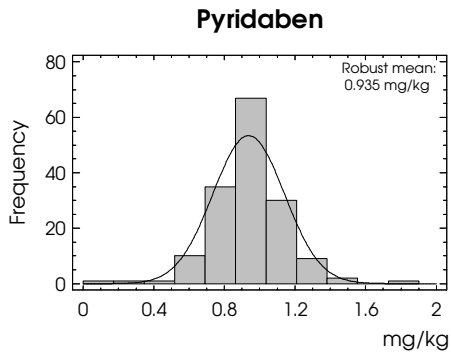
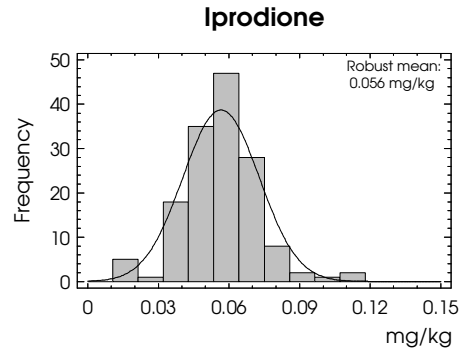
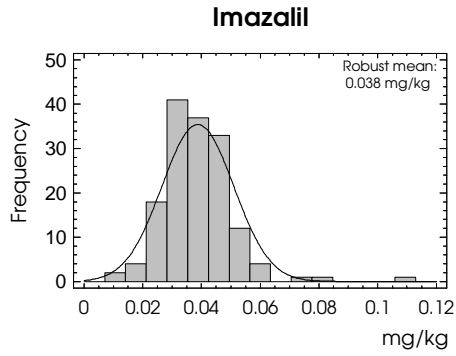
The voluntary pesticides are not covered by the ISO/IEC 17043

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

Results presented as histograms.

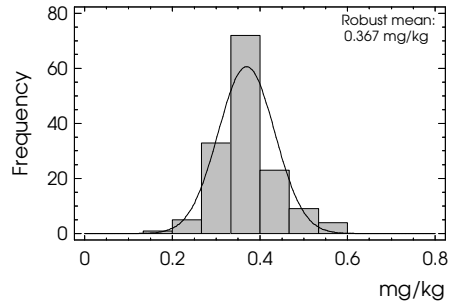


APPENDIX 2. Histograms of residue data for each pesticide from EU/EFTA laboratories.



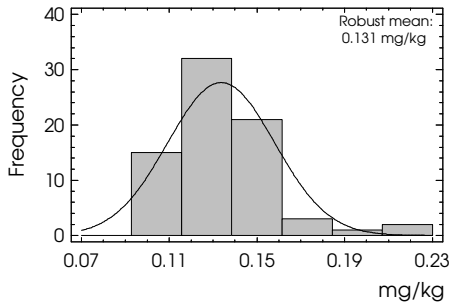
**APPENDIX 2. Histograms of residue data for each pesticide from EU/EFTA laboratories.**

**Thiamethoxam**

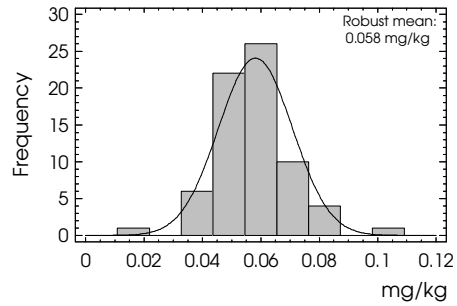


**Voluntary pesticides**

**Fenpyrazamine**



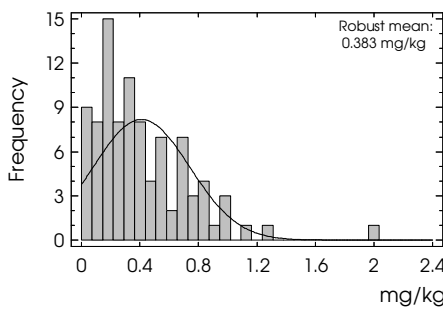
**Penthiopyrad**



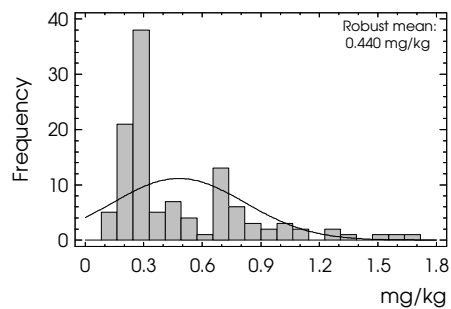
The voluntary pesticides are not covered by the ISO/IEC 17043

**Only for informative purposes**

**Chlorothalonil**



**Metaflumizone**



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

Results reported by the laboratories for the mandatory pesticides boscalid, buprofezin, carbendazim and benomyl (sum of benomyl and carbendazim expressed as carbendazim), clothianidin, diazinon, dimethoate, ethofenprox, fenpyroximate, imazalil, iprodione, pyridaben, spiromesifen, tau-fluvalinate, tebuconazole, tebufenpyrad, thiabendazole, and thiamethoxam (mg/kg) and their calculated z score value using FFP RSD 25 %

Lab Code	Boscalid		Buprofezin		Carbendazim and benomyl (sum of benomyl and carbendazim expressed as carbendazim)		Clothianidin		Diazinon		Dimethoate		Ethofenprox		Fenpyroximate		Imazalil	
	MRRL (mg/kg)	z score (FFP RSD 25 %)	z score (FFP RSD 25 %)	z score (FFP RSD 25 %)	z score (FFP RSD 25 %)	z score (FFP RSD 25 %)	z score (FFP RSD 25 %)	z score (FFP RSD 25 %)	z score (FFP RSD 25 %)	z score (FFP RSD 25 %)	z score (FFP RSD 25 %)	z score (FFP RSD 25 %)	z score (FFP RSD 25 %)	z score (FFP RSD 25 %)	z score (FFP RSD 25 %)	z score (FFP RSD 25 %)	z score (FFP RSD 25 %)	z score (FFP RSD 25 %)
Robust mean (mg/kg)	0.01	0.039	0.01	0.738	0.01	0.039	0.01	0.036	0.01	0.044	0.003	0.049	0.01	0.372	0.01	0.377	0.01	0.038
Lab001	0.042	0.3	0.940	1.1	0.038	-0.1	0.039	0.4	0.048	0.4	0.052	0.2	0.410	0.4	0.530	1.6	0.026	-1.3
Lab002	0.044	0.5	0.874	0.7	0.033	-0.6	0.037	0.1	0.056	1.1	0.047	-0.2	0.399	0.3	0.445	0.7	0.047	0.9
Lab003	0.045	0.6	0.804	0.4	0.048	1.0	0.040	0.5	0.048	0.4	0.057	0.6	0.460	0.9	0.519	1.5	0.046	0.8
Lab004	0.044	0.5	0.782	0.2	0.049	1.1	0.031	-0.5	0.045	0.1	0.051	0.1	0.412	0.4	0.398	0.2	0.051	1.4
Lab005	0.037	-0.2	0.806	0.4	0.034	-0.5	0.037	0.2	0.044	0.0	0.051	0.1	0.408	0.4	0.398	0.2	0.034	-0.4
Lab006	0.043	0.3	0.801	0.3	0.033	-0.5	0.036	0.0	0.050	0.5	0.049	0.0	0.316	-0.6	0.361	-0.2	0.047	1.0
Lab007	0.031	-0.9	0.666	-0.4	0.049	1.1	0.041	0.6	0.036	-0.7	0.054	0.3	0.407	0.4	0.439	0.7	0.048	1.0
Lab008	0.033	-0.7	0.820	0.4	0.041	0.2	0.039	0.4	0.046	0.2	0.049	0.0	0.364	-0.1	0.418	0.4	0.032	-0.7
Lab009	0.046	0.7	0.677	-0.3	0.033	-0.6	0.039	0.4	0.040	-0.4	0.052	0.2	0.302	-0.8	0.313	-0.7	0.039	0.1
Lab010	0.015	-2.5	0.430	-1.7	0.036	-0.3	0.037	0.1	0.047	0.3	0.037	-1.0	0.300	-0.8	0.240	-1.5	0.029	-0.9
Lab011	0.042	0.3	0.650	-0.5	0.049	1.1	NA	NA	0.035	-0.8	0.052	0.2	0.330	-0.5	NA	NA	0.034	-0.4
Lab012	0.040	0.1	0.770	0.2	0.042	0.4	0.037	0.1	0.045	0.1	0.050	0.1	0.410	0.4	0.390	0.1	0.047	0.9
Lab013	NA	NA	0.600	-0.7	0.050	1.2	NA	NA	0.060	1.5	0.050	0.1	NA	NA	NA	NA	0.110	5.0
Lab014	0.040	0.1	0.838	0.5	0.032	-0.7	0.025	-1.2	0.041	-0.3	0.041	-0.7	0.590	2.3	0.487	1.2	0.024	-1.5
Lab015	0.043	0.4	0.707	-0.2	0.036	-0.3	0.038	0.2	0.043	-0.1	0.059	0.8	0.349	-0.2	0.379	0.0	0.030	-0.8
Lab016	0.039	0.0	0.661	-0.4	0.038	-0.1	0.029	-0.8	0.045	0.1	0.052	0.2	0.427	0.6	0.365	-0.1	0.034	-0.4
Lab017	0.054	1.5	0.815	0.4	0.038	-0.1	0.032	-0.4	0.046	0.2	0.063	1.1	0.352	-0.2	0.342	-0.4	0.046	0.8
Lab018	0.034	-0.6	0.710	-0.2	0.033	-0.6	0.030	-0.6	0.035	-0.8	0.045	-0.3	0.378	0.1	0.385	0.1	0.042	0.4
Lab019	0.040	0.1	0.852	0.6	0.039	0.0	0.032	-0.4	0.052	0.7	0.055	0.5	0.360	-0.1	0.414	0.4	0.049	1.2
Lab020	0.024	-1.6	0.582	-0.8	ND	-3.0	NA	NA	0.033	-1.0	ND	-3.8	0.359	-0.1	NA	NA	ND	-2.9
Lab021	0.033	-0.7	0.753	0.1	NA	NA	0.045	1.0	0.049	0.5	0.046	-0.3	0.347	-0.3	ND	-3.9	ND	-3.5
Lab022	0.045	0.5	0.692	-0.2	0.046	0.8	0.038	0.2	0.050	0.6	0.054	0.4	0.471	1.1	0.528	1.6	0.063	2.6
Lab023	0.043	0.4	0.738	0.0	0.059	2.1	0.062	2.9	0.039	-0.4	0.083	2.7	0.493	1.3	0.368	-0.1	0.050	1.3
Lab024	0.041	0.2	0.826	0.5	0.042	0.4	0.034	-0.2	0.066	2.0	0.049	0.0	0.394	0.2	0.429	0.6	0.048	1.1
Lab025	0.043	0.4	0.804	0.4	0.047	0.8	0.033	-0.3	0.051	0.6	0.052	0.2	0.400	0.3	0.418	0.4	0.057	2.0
Lab026	0.045	0.6	0.708	-0.2	0.035	-0.4	0.029	-0.7	0.047	0.2	0.050	0.1	0.263	-1.2	0.297	-0.8	0.031	-0.7
Lab027	0.036	-0.4	0.707	-0.2	0.035	-0.4	0.031	-0.5	0.052	0.8	0.040	-0.7	0.403	0.3	0.497	1.3	0.026	-1.3
Lab028	0.043	0.3	0.585	-0.8	0.041	0.2	0.031	-0.5	0.037	-0.6	0.039	-0.9	0.356	-0.2	0.270	-1.1	0.041	0.3
Lab029	0.036	-0.4	0.825	0.5	0.047	0.9	0.042	0.7	0.051	0.7	0.054	0.4	0.380	0.1	0.595	2.3	0.036	-0.2
Lab030	0.028	-1.2	0.631	-0.6	0.037	-0.2	0.047	1.3	0.052	0.7	0.055	0.5	NA	NA	0.398	0.2	0.048	1.1
Lab031	0.036	-0.4	0.726	-0.1	0.032	-0.7	0.040	0.5	0.041	-0.3	0.045	-0.3	0.378	0.1	0.379	0.0	0.041	0.3
Lab032	0.034	-0.6	0.603	-0.7	0.054	1.6	0.041	0.5	0.039	-0.5	0.061	1.0	0.520	1.6	0.509	1.4	0.053	1.6
Lab033	0.043	0.4	0.698	-0.2	0.030	-0.9	0.037	0.1	0.050	0.6	0.067	1.4	0.384	0.1	0.355	-0.2	0.034	-0.4
Lab034	0.034	-0.6	0.669	-0.4	0.029	-1.0	0.033	-0.3	0.036	-0.7	0.048	-0.1	0.422	0.5	0.742	3.9	0.030	-0.8
Lab035	0.035	-0.5	0.620	-0.6	0.039	0.0	0.032	-0.4	0.033	-1.0	0.041	-0.7	0.370	0.0	0.320	-0.6	0.029	-0.9
Lab036	0.040	0.1	0.740	0.0	0.020	-1.9	0.040	0.5	0.040	-0.4	0.050	0.1	0.350	-0.2	0.350	-0.3	0.050	1.3
Lab037	0.054	1.5	0.850	0.6	0.050	1.2	0.034	-0.2	0.073	2.7	0.050	0.1	0.520	1.6	0.450	0.8	0.055	1.8
Lab038	0.043	0.4	0.790	0.3	0.038	-0.1	0.037	0.1	0.040	-0.4	0.050	0.1	0.360	-0.1	0.280	-1.0	0.048	1.1
Lab039	0.042	0.3	0.950	1.2	NA	NA	NA	NA	0.051	0.7	0.052	0.2	0.441	0.7	NA	NA	0.035	-0.3
Lab040	0.047	0.8	0.915	1.0	0.049	1.1	0.036	0.0	0.053	0.8	0.053	0.3	0.461	1.0	0.405	0.3	0.060	2.3

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

Lab Code	Boscalid	z score (FFP RSD 25 %)		Buprofezin	z score (FFP RSD 25 %)		Carbendazim and benomyl (sum of benomyl and carbendazim expressed as carbendazim)	z score (FFP RSD 25 %)		Clothianidin	z score (FFP RSD 25 %)		Diazinon	z score (FFP RSD 25 %)		Dimethoate	z score (FFP RSD 25 %)		Ethofenprox	z score (FFP RSD 25 %)		Fenpyroximate	z score (FFP RSD 25 %)		Imazalil	z score (FFP RSD 25 %)	
		MRL (mg/kg)	Robust mean (mg/kg)		0.01	0.039		0.01	0.738		0.01	0.039		0.01	0.036		0.01	0.044		0.003	0.049		0.01	0.372		0.01	0.377
Lab041	0.045	0.5	0.719	-0.1	0.033	-0.6	0.036	0.0	0.034	-0.9	0.059	0.8	0.256	-1.2	0.278	-1.1	0.045	0.7									
Lab042	0.039	0.0	0.741	0.0	0.038	0.0	0.034	-0.2	0.044	0.0	0.050	0.1	0.275	-1.0	0.329	-0.5	0.046	0.8									
Lab043	0.026	-1.4	0.568	-0.9	0.035	-0.4	0.031	-0.5	0.030	-1.2	0.041	-0.7	0.300	-0.8	0.291	-0.9	0.038	0.0									
Lab044	0.035	-0.5	0.890	0.8	0.034	-0.5	0.034	-0.2	0.046	0.2	0.043	-0.5	0.370	0.0	0.320	-0.6	0.032	-0.6									
Lab045	0.031	-0.9	0.568	-0.9	0.029	-1.0	ND	-2.9	0.036	-0.7	0.053	0.3	NA	NA	0.171	-2.2	0.033	-0.5									
Lab046	0.038	-0.1	0.695	-0.2	0.037	-0.1	0.036	0.1	0.042	-0.1	0.048	-0.1	0.374	0.0	0.374	0.0	0.032	-0.6									
Lab047	0.037	-0.3	0.699	-0.2	0.036	-0.3	0.027	-1.0	0.045	0.1	0.040	-0.7	0.396	0.3	0.377	0.0	0.033	-0.5									
Lab048	0.039	0.0	0.712	-0.1	0.039	0.0	0.033	-0.3	0.046	0.2	0.059	0.8	0.302	-0.8	0.362	-0.2	0.037	-0.1									
Lab049	0.036	-0.4	0.603	-0.7	0.027	-1.2	0.024	-1.3	0.039	-0.4	0.032	-1.4	0.352	-0.2	0.329	-0.5	0.045	0.7									
Lab050	0.043	0.3	0.711	-0.1	0.034	-0.4	0.034	-0.2	0.046	0.2	0.056	0.6	0.321	-0.5	0.343	-0.4	0.039	0.1									
Lab051	0.033	-0.7	0.699	-0.2	0.030	-0.9	0.029	-0.8	0.046	0.1	0.044	-0.4	0.312	-0.6	0.338	-0.4	0.029	-1.0									
Lab052	0.037	-0.3	0.710	-0.2	0.030	-0.9	0.037	0.1	0.044	0.0	0.046	-0.3	0.205	-1.8	0.340	-0.4	0.041	0.3									
Lab053	0.041	0.1	0.626	-0.6	0.038	-0.1	0.037	0.1	0.043	-0.1	0.052	0.2	0.318	-0.6	0.348	-0.3	0.038	0.0									
Lab054	0.041	0.1	0.870	0.7	0.056	1.8	0.042	0.7	0.059	1.4	0.052	0.2	0.404	0.3	0.488	1.2	0.046	0.9									
Lab055	0.044	0.4	0.787	0.3	0.039	0.0	0.037	0.1	0.051	0.7	0.052	0.2	0.385	0.1	0.382	0.1	0.047	0.9									
Lab056	No results reported																										
Lab057	0.037	-0.3	0.804	0.4	0.041	0.2	0.035	0.0	0.040	-0.4	0.049	-0.1	0.434	0.7	0.411	0.4	0.041	0.3									
Lab058	0.057	1.7	0.755	0.1	0.040	0.1	0.040	0.4	0.049	0.4	0.052	0.2	0.414	0.5	0.401	0.3	0.037	-0.1									
Lab059	0.042	0.2	0.842	0.6	0.041	0.2	0.035	-0.1	0.048	0.4	0.050	0.0	0.448	0.8	0.430	0.6	0.036	-0.2									
Lab060	0.035	-0.4	0.638	-0.5	0.034	-0.4	NA	NA	0.036	-0.7	0.043	-0.5	0.333	-0.4	0.268	-1.2	0.028	-1.0									
Lab061	0.045	0.6	0.770	0.2	0.040	0.1	0.033	-0.3	0.050	0.6	0.045	-0.3	0.330	-0.5	0.310	-0.7	0.037	-0.1									
Lab062	0.040	0.1	0.807	0.4	0.043	0.5	0.033	-0.3	0.060	1.5	0.056	0.6	0.404	0.3	0.438	0.6	0.042	0.4									
Lab063	0.048	0.9	0.840	0.6	0.048	1.0	0.034	-0.2	0.044	0.0	0.047	-0.2	0.396	0.3	0.390	0.1	0.044	0.6									
Lab064	0.049	1.0	0.699	-0.2	0.029	-1.0	0.027	-0.9	0.043	-0.1	0.041	-0.7	0.401	0.3	0.379	0.0	0.039	0.1									
Lab065	0.053	1.4	0.735	0.0	0.037	-0.2	0.033	-0.3	0.046	0.2	0.063	1.1	0.393	0.2	0.390	0.1	0.032	-0.6									
Lab066	0.038	-0.2	0.776	0.2	0.044	0.6	0.039	0.4	0.044	0.0	0.049	0.0	0.435	0.7	0.425	0.5	0.047	0.9									
Lab067	No results reported																										
Lab068	0.055	1.5	0.900	0.9	0.031	-0.8	0.042	0.6	0.049	0.5	0.050	0.0	0.422	0.5	0.395	0.2	0.041	0.3									
Lab069	0.041	0.1	0.699	-0.2	0.038	-0.1	0.039	0.3	0.044	0.0	0.050	0.1	0.396	0.3	0.473	1.0	0.037	-0.1									
Lab070	0.038	-0.2	0.760	0.1	0.057	1.9	0.040	0.5	0.046	0.2	0.057	0.6	0.481	1.2	0.501	1.3	0.073	3.7									
Lab071	0.042	0.3	0.651	-0.5	0.046	0.8	0.037	0.1	0.032	-1.1	0.059	0.8	0.265	-1.2	0.370	-0.1	0.040	0.2									
Lab072	0.035	-0.4	0.831	0.5	0.037	-0.2	0.033	-0.3	0.039	-0.4	0.060	0.9	0.381	0.1	0.426	0.5	0.030	-0.9									
Lab073	0.044	0.4	0.908	0.9	0.043	0.4	0.040	0.5	0.052	0.7	0.055	0.5	0.418	0.5	0.472	1.0	0.043	0.5									
Lab074	0.037	-0.3	0.895	0.9	NA	NA	NA	NA	0.051	0.6	0.053	0.3	0.358	-0.2	0.858	5.0	NA	NA									
Lab075	0.040	0.0	0.846	0.6	NA	NA	0.038	0.3	0.047	0.3	0.051	0.1	0.396	0.3	NA	NA	0.038	0.0									
Lab076	0.028	-1.2	0.544	-1.1	0.023	-1.7	0.029	-0.8	0.030	-1.3	0.044	-0.4	0.302	-0.8	0.331	-0.5	0.019	-2.0									
Lab077	0.042	0.3	0.990	1.4	0.048	0.9	0.048	1.3	0.049	0.5	0.065	1.3	0.492	1.3	0.476	1.1	0.024	-1.5									
Lab078	0.024	-1.6	0.473	-1.4	ND	-3.0	0.026	-1.1	0.027	-1.5	0.030	-1.6	0.220	-1.6	0.201	-1.9	0.025	-1.4									
Lab079	0.036	-0.4	0.705	-0.2	0.045	0.7	0.038	0.2	0.042	-0.2	0.062	1.0	0.371	0.0	0.437	0.6	0.045	0.7									
Lab080	0.040	0.1	0.807	0.4	0.031	-0.7	0.032	-0.5	0.045	0.1	0.049	0.0	0.315	-0.6	0.446	0.7	0.034	-0.4									
Lab081	0.045	0.6	0.768	0.2	0.040	0.1	0.038	0.3	0.042	-0.2	0.051	0.1	0.387	0.2	0.433	0.6	0.031	-0.8									
Lab082	0.036	-0.4	0.560	-1.0	NA	NA	NA	NA	0.075	2.8	0.028	-1.7	NA	NA	ND	-3.9	0.024	-1.5									
Lab083	0.046	0.7	0.740	0.0	0.051	1.3	0.039	0.4	0.032	-1.1	0.047	-0.2	0.360	-0.1	0.370	-0.1	0.047	0.9									
Lab084	0.038	-0.2	0.830	0.5	NA	NA	NA	NA	0.043	-0.1	0.041	-0.7	0.420	0.5	NA	NA	0.031	-0.7									
Lab085	0.041	0.2	0.807	0.4	0.032	-0.7	0.038	0.2	0.048	0.4	0.049	0.0	0.419	0.5	0.424	0.5	0.033	-0.5									
Lab086	0.055	1.6	0.680	-0.3	0.058	2.0	0.044	0.9	0.045	0.1	0.057	0.6	0.364	-0.1	0.192	-2.0	0.032	-0.6									

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

Lab Code	Bossalid	z score (FFP RSD 25 %)		Buprofezin	z score (FFP RSD 25 %)		Carbendazim and benomyl (sum of benomyl and carbendazim expressed as carbendazim)	z score (FFP RSD 25 %)		Clothianidin	z score (FFP RSD 25 %)		Diazinon	z score (FFP RSD 25 %)		Dimethoate	z score (FFP RSD 25 %)		Eihofenprox	z score (FFP RSD 25 %)		Fenpyroximate	z score (FFP RSD 25 %)		Imazalil	z score (FFP RSD 25 %)	
		MRL (mg/kg)	Robust mean (mg/kg)		0.01	0.039		0.01	0.738		0.01	0.039		0.01	0.036		0.01	0.044		0.003	0.049		0.01	0.372		0.01	0.377
Lab087	0.040	0.1	0.825	0.5	0.038	-0.1	0.034	-0.2	0.055	1.0	0.046	-0.3	0.450	0.8	0.510	1.4	0.035	-0.3									
Lab088	0.026	-1.4	NA	NA	NA	NA	NA	NA	0.030	-1.3	NA	NA	NA	NA	NA	NA	0.020	-1.9									
Lab089	0.036	-0.4	0.852	0.6	0.043	0.5	0.041	0.5	0.060	1.5	0.054	0.4	0.399	0.3	0.412	0.4	0.048	1.1									
Lab090	0.018	-2.2	0.859	0.7	NA	NA	NA	NA	0.032	-1.1	0.060	0.9	NA	NA	NA	NA	0.025	-1.4									
Lab091	0.027	-1.3	0.733	0.0	0.031	-0.8	0.031	-0.5	0.040	-0.3	0.043	-0.5	0.347	-0.3	0.385	0.1	0.025	-1.3									
Lab092	0.035	-0.5	0.670	-0.4	0.049	1.1	0.038	0.2	0.027	-1.5	0.049	0.0	0.400	0.3	0.380	0.0	0.034	-0.4									
Lab093	0.033	-0.6	0.701	-0.2	0.038	0.0	0.023	-1.5	0.039	-0.5	0.053	0.3	0.299	-0.8	0.295	-0.9	0.039	0.1									
Lab094	0.034	-0.5	0.817	0.4	0.032	-0.7	0.036	0.0	0.049	0.5	0.049	0.0	0.438	0.7	0.426	0.5	0.044	0.6									
Lab095	0.037	-0.3	0.609	-0.7	0.042	0.4	0.048	1.4	0.042	-0.2	0.061	1.0	0.408	0.4	0.425	0.5	0.047	0.9									
Lab096	NA	NA	0.855	0.6	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA									
Lab097	0.043	0.4	0.450	-1.6	NA	NA	0.031	-0.5	0.040	-0.4	0.033	-1.3	NA	NA	0.330	-0.5	0.039	0.1									
Lab098	0.028	-1.2	0.610	-0.7	0.044	0.6	0.024	-1.3	0.030	-1.3	0.035	-1.2	0.270	-1.1	0.270	-1.1	0.016	-2.3									
Lab099	0.042	0.2	0.556	-1.0	0.045	0.6	0.035	-0.1	0.045	0.1	0.048	-0.1	0.400	0.3	0.301	-0.8	0.049	1.2									
Lab100	0.031	-0.8	0.591	-0.8	0.104	5.0	0.039	0.3	0.040	-0.4	0.047	-0.1	0.300	-0.8	0.364	-0.1	0.041	0.3									
Lab101	0.041	0.1	0.695	-0.2	0.044	5.0	NA	NA	0.045	0.1	0.056	0.5	0.363	-0.1	0.342	-0.4	0.050	1.3									
Lab102	0.047	0.8	0.892	0.8	0.044	0.5	0.052	1.8	0.058	1.2	0.060	0.9	0.315	-0.6	0.364	-0.1	0.025	-1.4									
Lab103	0.041	0.2	0.891	0.8	0.067	2.9	0.070	3.8	0.059	1.4	0.084	2.8	0.356	-0.2	0.346	-0.3	0.025	-1.3									
Lab104	0.045	0.6	0.655	-0.4	0.057	1.9	0.034	-0.2	0.045	0.1	0.046	-0.3	0.334	-0.4	0.435	0.6	0.034	-0.4									
Lab105	0.033	-0.7	0.956	1.2	0.089	5.0	0.035	-0.1	0.048	0.4	0.047	-0.2	0.423	0.5	0.308	-0.7	0.027	-1.1									
Lab106	0.030	-1.0	0.552	-1.0	0.082	4.5	0.030	-0.7	0.054	0.9	0.045	-0.4	0.305	-0.7	0.266	-1.2	0.024	-1.5									
Lab107	0.041	0.2	0.711	-0.1	0.048	1.0	0.034	-0.2	0.047	0.3	0.056	0.5	0.360	-0.1	0.467	1.0	0.054	1.7									
Lab108	NA	NA	0.660	-0.4	0.020	-1.9	NA	NA	0.030	-1.3	0.210	5.0	0.270	-1.1	0.310	-0.7	NA	NA									
Lab109	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA									
Lab110	0.024	-1.6	0.670	-0.4	0.020	-1.9	0.330	5.0	0.016	-2.5	0.034	-1.2	0.215	-1.7	NA	NA	0.023	-1.6									
Lab111	0.044	0.5	1.100	2.0	0.062	2.4	0.040	0.5	0.053	0.8	0.061	1.0	0.383	0.1	ND	-3.9	0.063	2.6									
Lab112	NA	NA	NA	NA	0.100	5.0	NA	NA	0.068	2.2	0.057	0.6	NA	NA	NA	NA	NA	NA									
Lab113	0.041	0.2	0.670	-0.4	0.077	4.0	0.034	-0.2	0.039	-0.4	0.046	-0.3	NA	NA	0.338	-0.4	0.014	-2.5									
Lab114	NA	NA	0.876	0.7	NA	NA	NA	NA	0.053	0.9	0.058	0.7	NA	NA	NA	NA	NA	NA									
Lab115	0.035	-0.5	0.708	-0.2	0.033	-0.6	0.041	0.6	0.038	-0.5	0.050	0.1	0.372	0.0	0.456	0.8	0.030	-0.8									
Lab116	0.040	0.0	0.732	0.0	0.026	-1.4	0.037	0.1	0.045	0.1	0.044	-0.4	0.332	-0.4	0.390	0.1	0.040	0.2									
Lab117	0.038	-0.2	0.804	0.4	0.037	-0.2	0.039	0.3	0.043	-0.1	0.051	0.1	0.372	0.0	0.413	0.4	0.022	-1.7									
Lab118	0.032	-0.8	0.469	-1.5	NA	NA	0.024	-1.4	NA	NA	0.034	-1.3	0.136	-2.5	0.275	-1.1	0.026	-1.2									
Lab119	0.040	0.1	0.604	-0.7	0.030	-0.9	0.044	0.9	0.045	0.1	0.051	0.2	0.443	0.8	0.401	0.3	0.035	-0.3									
Lab120	0.054	1.5	NA	NA	NA	NA	NA	NA	0.047	0.3	0.035	-1.2	0.461	1.0	NA	NA	0.083	4.7									
Lab121	0.047	0.8	0.769	0.2	0.042	0.4	0.039	0.4	0.051	0.7	0.050	0.1	0.392	0.2	0.407	0.3	0.027	-1.2									
Lab122	0.052	1.3	0.876	0.7	0.044	0.6	0.038	0.2	0.051	0.7	0.056	0.6	0.364	-0.1	0.431	0.6	0.048	1.1									
Lab123	0.036	-0.3	0.577	-0.9	0.047	0.9	0.034	-0.2	0.040	-0.3	0.049	0.0	0.342	-0.3	0.376	0.0	0.027	-1.2									
Lab124	Participation cancelled																										
Lab125	0.042	0.2	0.788	0.3	0.037	-0.2	0.035	-0.1	0.047	0.3	0.054	0.4	0.377	0.1	0.344	-0.4	0.034	-0.4									
Lab126	0.048	0.9	0.779	0.2	0.038	-0.1	0.040	0.5	0.051	0.7	0.054	0.4	0.450	0.8	0.397	0.2	0.054	1.7									
Lab127	0.056	1.6	0.925	1.0	0.042	0.3	0.046	1.1	0.050	0.5	0.048	-0.1	0.440	0.7	0.439	0.7	0.031	-0.7									
Lab128	0.040	0.1	0.877	0.8	0.035	-0.4	0.033	-0.3	0.047	0.3	0.049	0.0	0.828	4.9	0.662	3.0	0.043	0.5									
Lab129	0.028	-1.2	0.881	0.8	0.039	0.0	0.037	0.1	0.036	-0.7	0.064	1.2	0.369	0.0	0.387	0.1	NA	NA									
Lab130	0.040	0.1	0.764	0.1	0.040	0.1	0.032	-0.4	0.040	-0.4	0.042	-0.6	0.366	-0.1	0.460	0.9	0.035	-0.3									
Lab131	0.044	0.5	0.651	-0.5	0.029	-1.0	0.030	-0.6	0.046	0.2	0.034	-1.2	0.421	0.5	0.381	0.0	0.033	-0.5									
Lab132	0.038	-0.1	0.725	-0.1	0.187	5.0	0.030	-0.7	0.050	0.6	0.041	-0.7	0.232	-1.5	0.441	0.7	0.029	-1.0									



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

Lab Code	Boscalid	z score (FFP RSD 25 %)		Buprofezin	z score (FFP RSD 25 %)		Carbendazim and benomyl (sum of benomyl and carbendazim expressed as carbendazim)	z score (FFP RSD 25 %)		Clothianidin	z score (FFP RSD 25 %)		Diazinon	z score (FFP RSD 25 %)		Dimethoate	z score (FFP RSD 25 %)		Ethofenprox	z score (FFP RSD 25 %)		Fenpyroximate	z score (FFP RSD 25 %)		Imazalil	z score (FFP RSD 25 %)	
		MRRL (mg/kg)	Robust mean (mg/kg)		0.01	0.039		0.01	0.738		0.01	0.039		0.01	0.036		0.01	0.044		0.003	0.049		0.01	0.372		0.01	0.377
Lab133	0.025	-1.5	0.680	-0.3	0.040	0.1	0.027	-1.0	0.020	-2.2	0.036	-1.1	0.190	-2.0	0.400	0.2	0.013	-2.6									
Lab134	0.030	-0.9	0.493	-1.3	NA	NA	0.048	1.4	NA	NA	0.055	0.5	NA	NA	0.301	-0.8	0.041	0.4									
Lab135	0.040	0.1	0.810	0.4	0.040	0.1	0.030	-0.6	0.040	-0.4	0.050	0.1	0.370	0.0	0.340	-0.4	0.040	0.2									
Lab136	0.042	0.3	0.816	0.4	0.085	4.8	0.039	0.4	0.048	0.4	0.052	0.2	0.349	-0.2	0.354	-0.2	0.043	0.5									
Lab137	0.042	0.3	0.637	-0.5	0.025	-1.4	0.039	0.4	0.048	0.4	0.036	-1.1	0.378	0.1	0.365	-0.1	0.054	1.7									
Lab138	0.040	0.1	0.780	0.2	0.030	-0.9	0.030	-0.6	0.040	-0.4	0.040	-0.7	0.320	-0.6	0.290	-0.9	0.010	-2.9									
Lab139	NA	NA	0.298	-2.4	0.031	-0.8	0.043	0.8	0.020	-2.2	0.035	-1.2	NA	NA	NA	NA	0.015	-2.4									
Lab140	0.035	-0.4	0.728	-0.1	0.040	0.2	0.031	-0.5	0.044	0.0	0.049	-0.1	0.375	0.0	0.387	0.1	0.049	1.1									
Lab141	0.043	0.4	0.950	1.2	0.041	0.2	0.030	-0.6	0.043	-0.1	0.045	-0.3	0.420	0.5	0.450	0.8	0.039	0.1									
Lab142	0.035	-0.5	0.594	-0.8	NA	NA	NA	NA	0.047	0.3	0.035	-1.1	0.332	-0.4	0.269	-1.1	0.051	1.4									
Lab143	0.031	-0.9	0.862	0.7	0.050	1.2	0.032	-0.4	0.042	-0.2	0.057	0.6	0.275	-1.0	0.379	0.0	0.031	-0.7									
Lab144	0.046	0.7	0.961	1.2	0.040	0.2	0.042	0.7	0.052	0.7	0.050	0.1	0.450	0.8	0.454	0.8	0.046	0.9									
Lab145	0.036	-0.4	0.770	0.2	0.041	0.2	0.036	0.0	0.050	0.6	0.051	0.1	0.400	0.3	0.280	-1.0	0.041	0.3									
Lab146	0.038	-0.2	1.001	1.4	0.044	0.6	0.043	0.8	0.055	1.0	0.054	0.4	0.470	1.1	0.540	1.7	0.047	0.9									
Lab147	0.041	0.2	0.677	-0.3	0.029	-1.0	NA	NA	0.044	0.0	0.034	-1.2	0.414	0.5	NA	NA	0.042	0.4									
Lab148	0.049	1.0	0.900	0.9	0.040	0.1	0.050	1.6	0.064	1.8	0.071	1.8	0.450	0.8	0.420	0.5	0.047	0.9									
Lab149	Participation cancelled																										
Lab150	0.034	-0.6	0.683	-0.3	0.031	-0.8	NA	NA	0.037	-0.7	0.042	-0.6	0.370	0.0	NA	NA	0.036	-0.3									
Lab151	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.324	-0.5	NA	NA	NA	NA									
Lab152	0.047	0.8	0.899	0.9	0.057	1.9	0.037	0.1	0.053	0.8	0.056	0.6	0.352	-0.2	0.152	-2.4	0.040	0.2									
Lab153	0.022	-1.8	0.498	-1.3	0.033	-0.6	0.036	0.0	0.011	-3.0	0.052	0.3	0.219	-1.6	0.255	-1.3	0.030	-0.9									
Lab154	0.040	0.0	0.774	0.2	0.033	-0.6	0.044	0.9	0.016	-2.5	0.051	0.2	NA	NA	0.427	0.5	0.024	-1.5									
Lab155	0.052	1.2	0.661	-0.4	0.029	-1.0	0.032	-0.5	0.047	0.3	0.047	-0.2	0.425	0.6	0.417	0.4	0.053	1.6									
Lab156	0.049	1.0	0.851	0.6	0.043	0.5	0.021	-1.7	0.055	1.0	0.046	-0.3	NA	NA	0.310	-0.7	0.053	1.6									
Lab157	0.041	0.2	0.620	-0.6	NA	NA	NA	NA	0.045	0.1	NA	NA	0.392	0.2	NA	NA	0.051	1.4									
Lab158	0.039	-0.1	0.797	0.3	0.042	0.4	0.034	-0.2	0.046	0.2	0.050	0.0	0.372	0.0	0.365	-0.1	0.040	0.2									
Lab159	0.030	-1.0	0.751	0.1	NA	NA	0.030	-0.7	0.042	-0.1	0.032	-1.4	0.338	-0.4	0.264	-1.2	0.022	-1.7									
Lab160	NA	NA	NA	NA	NA	NA	NA	NA	0.043	-0.1	0.049	0.0	0.348	-0.3	NA	NA	NA	NA									
Lab161	0.028	-1.2	0.492	-1.3	0.042	0.4	NA	NA	0.041	-0.3	0.046	-0.3	0.173	-2.1	NA	NA	0.036	-0.2									
Lab162	0.045	0.6	0.465	-1.5	0.023	-1.6	0.039	0.4	0.036	-0.7	0.059	0.8	0.216	-1.7	0.403	0.3	0.037	-0.1									
Lab163	0.039	0.0	0.854	0.6	0.049	1.1	0.045	1.0	0.050	0.6	0.032	-1.4	0.595	2.4	NA	NA	0.034	-0.4									
Lab164	0.029	-1.1	0.460	-1.5	0.028	-1.1	0.032	-0.4	0.033	-1.0	0.100	4.1	0.290	-0.9	0.300	-0.8	0.031	-0.7									
Lab165	0.039	-0.1	0.925	1.0	0.031	-0.8	0.043	0.8	0.043	-0.1	0.054	0.4	0.061	-3.3	0.100	-2.9	0.042	0.4									
Lab166	0.030	-1.0	0.620	-0.6	0.040	0.1	0.030	-0.6	0.040	-0.4	0.050	0.1	0.360	-0.1	0.250	-1.3	0.030	-0.8									
Lab167	0.050	1.1	1.080	1.9	0.014	-2.5	0.014	-2.5	0.053	0.8	0.032	-1.4	0.504	1.4	0.428	0.5	0.034	-0.5									
Lab168	0.039	0.0	0.858	0.7	0.042	0.4	0.039	0.4	0.053	0.8	0.057	0.6	0.651	3.0	ND	-3.9	0.049	1.2									
Lab169	0.043	0.4	0.580	-0.9	0.041	0.2	NA	NA	0.040	-0.4	0.048	-0.1	NA	NA	NA	NA	0.036	-0.2									
Lab170	0.044	0.4	NA	NA	0.026	-1.4	NA	NA	0.045	0.1	0.056	0.5	NA	NA	NA	NA	NA	NA									
Lab171	0.038	-0.2	0.722	-0.1	0.037	-0.2	0.040	0.5	0.043	-0.1	0.049	0.0	0.396	0.3	0.386	0.1	0.026	-1.3									
Lab172	0.038	-0.2	0.743	0.0	0.033	-0.6	0.035	-0.1	0.047	0.3	0.046	-0.3	0.350	-0.2	0.330	-0.5	0.043	0.5									
Lab173	ND	-3.0	ND	-3.9	NA	NA	0.032	-0.4	0.015	-2.6	0.028	-1.7	0.036	-3.6	0.096	-3.0	ND	-2.9									
Lab174	0.049	1.0	0.492	-1.3	0.020	-1.9	0.052	1.8	0.028	-1.4	0.079	2.4	0.318	-0.6	0.286	-1.0	0.030	-0.8									
Lab175	NA	NA	NA	NA	NA	NA	NA	NA	0.020	-2.2	0.021	-2.3	NA	NA	NA	NA	NA	NA									
Lab176	0.040	0.0	1.000	1.4	0.034	-0.5	0.035	-0.1	0.050	0.6	0.052	0.2	0.339	-0.4	0.266	-1.2	0.037	-0.1									
Lab177	0.037	-0.2	0.759	0.1	0.045	0.7	0.034	-0.2	0.045	0.1	0.057	0.6	0.307	-0.7	0.353	-0.3	0.051	1.4									
Lab178	0.044	0.5	0.982	1.3	0.050	1.2	0.024	-1.3	0.067	2.1	0.047	-0.2	0.385	0.1	0.322	-0.6	0.034	-0.4									

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

Lab Code	Boscalid	z score (FFP RSD 25 %)		Buprofezin	z score (FFP RSD 25 %)		Carbendazim and benomyl (sum of benomyl and carbendazim expressed as carbendazim)	z score (FFP RSD 25 %)		Clothianidin	z score (FFP RSD 25 %)		Diazinon	z score (FFP RSD 25 %)		Dimethoate	z score (FFP RSD 25 %)		Ethofenprox	z score (FFP RSD 25 %)		Fenpyroximate	z score (FFP RSD 25 %)		Imazalil	z score (FFP RSD 25 %)	
		MRRL (mg/kg)	0.01		0.01	0.01		0.01	0.01		0.01	0.01		0.01	0.01		0.01	0.01		0.01	0.01		0.01	0.01		0.01	0.01
Robust mean (mg/kg)	0.039	0.738		0.039		0.036		0.044		0.049		0.372		0.377		0.038											
Lab179	0.035	-0.5	0.750	0.1	0.048	1.0	0.038	0.2	0.038	-0.5	0.045	-0.3	0.370	0.0	0.330	-0.5	0.040	0.2									
Lab180	NA	NA	NA	NA	NA	NA	NA	NA	0.035	-0.8	NA	NA	NA	NA	NA	NA	NA	NA									
Lab181	0.055	1.6	0.493	-1.3	NA	NA	NA	NA	0.035	-0.8	NA	NA	NA	NA	NA	NA	ND	-2.9									
Lab182	0.075	3.6	ND	-3.9	NA	NA	NA	NA	ND	-3.1	ND	-3.8	0.019	-3.8	NA	NA	ND	-2.9									
Lab183	0.032	-0.8	0.588	-0.8	NA	NA	0.032	-0.5	0.036	-0.7	0.040	-0.8	0.294	-0.8	0.278	-1.1	0.028	-1.1									
Lab184	0.042	0.3	0.630	-0.6	0.035	-0.4	0.041	0.6	0.050	0.6	0.047	-0.2	0.270	-1.1	0.370	-0.1	0.031	-0.7									
Lab185	0.038	-0.2	0.638	-0.5	NA	NA	0.038	0.2	0.026	-1.6	0.083	2.7	0.339	-0.4	0.200	-1.9	0.057	2.0									
Lab186	0.039	0.0	0.667	-0.4	0.035	-0.4	0.034	-0.2	0.041	-0.2	0.041	-0.6	0.328	-0.5	0.531	1.6	0.025	-1.3									

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

Lab Code	Iprodione	z score (FFP RSD 25 %)		Pyridaben	z score (FFP RSD 25 %)		Spiromesifen	z score (FFP RSD 25 %)		Tau-fluvalinate	z score (FFP RSD 25 %)		Tebuconazole	z score (FFP RSD 25 %)		Tebufenpyrad	z score (FFP RSD 25 %)		Thiabendazole	z score (FFP RSD 25 %)		Thiamethoxam	z score (FFP RSD 25 %)	
		MRRL (mg/kg)	0.01		0.01	0.01		0.01	0.01		0.01	0.01		0.01	0.01		0.01	0.01		0.01	0.01		0.01	0.01
Robust mean (mg/kg)	0.056	0.935		0.603		0.122		1.220		1.029		0.048		0.367										
Lab001	0.064	0.6	1.060	0.5	0.640	0.2	0.150	0.9	1.360	0.5	1.050	0.1	0.050	0.2	0.400	0.4								
Lab002	0.063	0.5	0.886	-0.2	0.723	0.8	0.137	0.5	1.430	0.7	1.150	0.5	0.051	0.3	0.413	0.5								
Lab003	0.037	-1.4	0.985	0.2	0.517	-0.6	0.098	-0.8	1.221	0.0	0.998	-0.1	0.050	0.2	0.500	1.4								
Lab004	0.071	1.1	1.090	0.7	0.671	0.5	0.150	0.9	1.280	0.2	1.290	1.0	0.053	0.4	0.376	0.1								
Lab005	0.066	0.7	0.979	0.2	0.660	0.4	0.154	1.0	1.420	0.7	1.110	0.3	0.045	-0.3	0.369	0.0								
Lab006	NA	NA	0.840	-0.4	0.688	0.6	0.053	-2.3	1.520	1.0	1.130	0.4	0.045	-0.3	0.340	-0.3								
Lab007	0.040	-1.1	0.919	-0.1	0.472	-0.9	0.187	2.1	0.939	-0.9	0.906	-0.5	0.059	0.9	0.552	2.0								
Lab008	0.056	0.0	0.877	-0.2	0.501	-0.7	0.132	0.3	1.560	1.1	1.300	1.1	0.054	0.5	0.361	-0.1								
Lab009	0.064	0.6	0.677	-1.1	0.526	-0.5	0.098	-0.8	0.859	-1.2	0.870	-0.6	0.051	0.3	0.442	0.8								
Lab010	0.011	-3.2	0.310	-2.7	0.180	-2.8	0.014	-3.5	0.770	-1.5	0.810	-0.9	0.048	0.0	0.300	-0.7								
Lab011	NA	NA	0.740	-0.8	NA	NA	0.103	-0.6	1.350	0.4	0.880	-0.6	0.051	0.3	0.307	-0.7								
Lab012	0.047	-0.6	0.920	-0.1	0.590	-0.1	0.080	-1.4	1.100	-0.4	1.000	-0.1	0.053	0.4	0.390	0.3								
Lab013	0.060	0.3	0.770	-0.7	NA	NA	NA	NA	NA	NA	NA	NA	0.080	2.7	0.330	-0.4								
Lab014	0.063	0.5	1.126	0.8	0.725	0.8	0.146	0.8	1.248	0.1	1.183	0.6	0.044	-0.3	0.356	-0.1								
Lab015	0.040	-1.1	1.020	0.4	0.555	-0.3	0.109	-0.4	1.300	0.3	0.975	-0.2	0.043	-0.4	0.382	0.2								
Lab016	0.057	0.1	0.912	-0.1	0.675	0.5	0.025	-3.2	1.130	-0.3	0.963	-0.3	0.042	-0.5	0.388	0.2								
Lab017	0.073	1.2	0.914	-0.1	0.614	0.1	ND	-3.7	1.397	0.6	1.169	0.5	0.049	0.1	0.409	0.5								
Lab018	0.043	-1.0	0.938	0.0	0.633	0.2	0.140	0.6	1.160	-0.2	0.940	-0.3	0.038	-0.9	0.313	-0.6								
Lab019	ND	-3.3	1.100	0.7	0.700	0.6	0.109	-0.4	1.280	0.2	1.260	0.9	0.048	0.0	0.382	0.2								
Lab020	ND	-3.3	0.753	-0.8	NA	NA	0.085	-1.2	0.502	-2.4	0.932	-0.4	ND	-3.2	NA	NA								
Lab021	0.049	-0.5	0.898	-0.2	ND	-3.9	0.074	-1.6	1.200	-0.1	1.150	0.5	0.053	0.4	0.447	0.9								

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

Lab Code	Iprodione		Pyridaben		Spiromesifen		Tau-fluvalinate		Tebuconazole		Tebufenpyrad		Thiabendazole		Thiamethoxam	
	MRRL (mg/kg)	z score (FFP RSD 25 %)	MRRL (mg/kg)	z score (FFP RSD 25 %)	MRRL (mg/kg)	z score (FFP RSD 25 %)	MRRL (mg/kg)	z score (FFP RSD 25 %)	MRRL (mg/kg)	z score (FFP RSD 25 %)	MRRL (mg/kg)	z score (FFP RSD 25 %)	MRRL (mg/kg)	z score (FFP RSD 25 %)	MRRL (mg/kg)	z score (FFP RSD 25 %)
	0.01		0.01		0.01		0.01		0.01		0.01		0.01		0.01	
Robust mean (mg/kg)	0.056		0.935		0.603		0.122		1.220		1.029		0.048		0.367	
Lab022	0.065	0.7	1.330	1.7	0.741	0.9	0.120	-0.1	1.100	-0.4	1.230	0.8	0.048	0.0	0.371	0.0
Lab023	0.056	0.0	1.010	0.3	0.908	2.0	0.159	1.2	1.190	-0.1	1.020	0.0	0.083	2.9	0.570	2.2
Lab024	0.059	0.2	0.861	-0.3	0.729	0.8	0.119	-0.1	1.440	0.7	1.190	0.6	0.057	0.8	0.466	1.1
Lab025	0.056	0.0	0.664	-1.2	0.732	0.9	0.088	-1.1	1.540	1.0	0.865	-0.6	0.050	0.2	0.404	0.4
Lab026	0.054	-0.2	0.890	-0.2	0.493	-0.7	0.119	-0.1	1.176	-0.1	0.956	-0.3	0.049	0.1	0.299	-0.7
Lab027	0.065	0.6	0.817	-0.5	0.411	-1.3	0.153	1.0	1.398	0.6	1.178	0.6	0.043	-0.4	0.273	-1.0
Lab028	0.069	0.9	0.725	-0.9	0.435	-1.1	0.118	-0.1	1.365	0.5	0.901	-0.5	0.041	-0.6	0.271	-1.0
Lab029	0.054	-0.2	1.013	0.3	0.618	0.1	0.129	0.2	1.247	0.1	1.082	0.2	0.050	0.2	0.432	0.7
Lab030	0.066	0.7	1.060	0.5	0.650	0.3	NA	NA	1.380	0.5	1.180	0.6	0.043	-0.4	0.339	-0.3
Lab031	ND	-3.3	0.950	0.1	0.597	0.0	0.123	0.0	1.186	-0.1	0.974	-0.2	0.041	-0.6	0.335	-0.3
Lab032	0.054	-0.2	1.060	0.5	0.479	-0.8	0.095	-0.9	1.340	0.4	1.360	1.3	0.055	0.6	0.398	0.3
Lab033	0.071	1.1	0.998	0.3	0.666	0.4	0.100	-0.7	1.096	-0.4	0.875	-0.6	0.057	0.8	0.352	-0.2
Lab034	0.059	0.2	0.906	-0.1	0.558	-0.3	0.159	1.2	1.076	-0.5	0.919	-0.4	0.067	1.6	0.407	0.4
Lab035	0.100	3.1	0.930	0.0	NA	NA	0.150	0.9	1.100	-0.4	1.200	0.7	0.045	-0.3	0.350	-0.2
Lab036	NA	NA	0.930	0.0	0.590	-0.1	0.140	0.6	1.010	-0.7	0.970	-0.2	0.040	-0.7	0.390	0.3
Lab037	0.082	1.9	1.300	1.6	0.600	0.0	0.150	0.9	1.300	0.3	1.000	-0.1	0.052	0.3	0.360	-0.1
Lab038	0.049	-0.5	0.940	0.0	0.750	1.0	0.130	0.3	1.280	0.2	0.940	-0.3	0.054	0.5	0.340	-0.3
Lab039	0.055	-0.1	0.861	-0.3	0.648	0.3	0.142	0.7	1.046	-0.6	1.049	0.1	0.048	0.0	0.468	1.1
Lab040	0.066	0.7	1.094	0.7	0.706	0.7	0.131	0.3	1.379	0.5	1.216	0.7	0.053	0.4	0.387	0.2
Lab041	0.051	-0.4	0.887	-0.2	0.805	1.3	0.104	-0.6	0.830	-1.3	0.703	-1.3	0.041	-0.6	0.296	-0.8
Lab042	0.051	-0.4	0.840	-0.4	0.623	0.1	0.126	0.1	1.250	0.1	1.180	0.6	0.041	-0.6	0.411	0.5
Lab043	0.043	-1.0	0.738	-0.8	0.482	-0.8	0.107	-0.5	0.809	-1.3	0.757	-1.1	0.036	-1.0	0.324	-0.5
Lab044	ND	-3.3	1.170	1.0	1.160	3.7	0.130	0.3	1.240	0.1	1.190	0.6	0.034	-1.2	0.300	-0.7
Lab045	0.066	0.7	0.674	-1.1	0.480	-0.8	0.079	-1.4	1.055	-0.5	0.733	-1.2	0.032	-1.3	0.336	-0.3
Lab046	0.051	-0.4	0.828	-0.5	0.465	-0.9	0.107	-0.5	0.970	-0.8	0.915	-0.4	0.042	-0.5	0.323	-0.5
Lab047	0.050	-0.4	0.915	-0.1	0.604	0.0	0.154	1.0	1.209	0.0	1.010	-0.1	0.050	0.2	0.304	-0.7
Lab048	0.055	-0.1	0.864	-0.3	0.639	0.2	0.118	-0.1	1.320	0.3	1.170	0.5	0.053	0.4	0.380	0.1
Lab049	0.043	-0.9	0.776	-0.7	0.534	-0.5	0.076	-1.5	1.031	-0.6	0.864	-0.6	0.030	-1.5	0.341	-0.3
Lab050	0.057	0.0	0.876	-0.3	0.616	0.1	0.118	-0.1	1.250	0.1	1.160	0.5	0.052	0.3	0.378	0.1
Lab051	0.062	0.4	1.039	0.4	0.502	-0.7	0.129	0.2	1.060	-0.5	0.902	-0.5	0.043	-0.4	0.345	-0.2
Lab052	0.070	1.0	0.850	-0.4	0.525	-0.5	0.086	-1.2	1.225	0.0	0.950	-0.3	0.046	-0.2	0.360	-0.1
Lab053	0.058	0.2	0.871	-0.3	0.643	0.3	0.113	-0.3	1.230	0.0	1.290	1.0	0.053	0.4	0.376	0.1
Lab054	0.066	0.7	1.030	0.4	0.624	0.1	0.115	-0.2	1.530	1.0	1.150	0.5	0.054	0.5	0.490	1.3
Lab055	0.095	2.8	0.994	0.3	0.638	0.2	0.135	0.4	1.160	-0.2	0.984	-0.2	0.056	0.6	0.407	0.4
Lab056	No results reported															
Lab057	0.053	-0.2	1.040	0.4	0.615	0.1	0.100	-0.7	1.370	0.5	1.120	0.4	0.045	-0.3	0.317	-0.5
Lab058	0.065	0.7	0.942	0.0	0.648	0.3	0.137	0.5	1.321	0.3	1.126	0.4	0.053	0.4	0.382	0.2
Lab059	0.064	0.6	1.770	3.6	0.925	2.1	0.146	0.8	1.400	0.6	1.440	1.6	0.043	-0.4	0.400	0.4
Lab060	0.044	-0.9	0.869	-0.3	0.746	0.9	0.059	-2.1	1.140	-0.3	0.863	-0.6	0.040	-0.7	0.400	0.4
Lab061	0.058	0.1	0.830	-0.4	0.590	-0.1	0.094	-0.9	1.280	0.2	1.010	-0.1	0.046	-0.2	0.330	-0.4
Lab062	0.062	0.4	1.100	0.7	0.598	0.0	0.122	0.0	1.290	0.2	1.170	0.5	0.055	0.6	0.353	-0.2
Lab063	0.061	0.3	1.240	1.3	NA	NA	0.099	-0.7	1.110	-0.4	1.120	0.4	0.049	0.1	0.320	-0.5
Lab064	0.065	0.7	0.872	-0.3	0.733	0.9	0.193	2.3	1.160	-0.2	0.988	-0.2	0.037	-1.0	0.333	-0.4
Lab065	0.066	0.7	0.989	0.2	0.607	0.0	0.132	0.3	1.301	0.3	1.067	0.1	0.113	5.0	0.359	-0.1
Lab066	0.054	-0.1	0.790	-0.6	0.500	-0.7	0.144	0.7	1.243	0.1	1.070	0.2	0.050	0.2	0.469	1.1
Lab067	No results reported															
Lab068	0.041	-1.1	1.550	2.6	0.730	0.8	0.105	-0.6	1.150	-0.2	1.100	0.3	0.024	-2.0	0.350	-0.2
Lab069	0.059	0.2	0.812	-0.5	0.686	0.6	0.138	0.5	1.440	0.7	1.070	0.2	0.053	0.4	0.376	0.1

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

Lab Code	Iprodione	z score (FFP RSD 25 %)		Pyridaben	z score (FFP RSD 25 %)		Spiromesifen	z score (FFP RSD 25 %)		Tau-fluvalinate	z score (FFP RSD 25 %)		Tebuconazole	z score (FFP RSD 25 %)		Tebufenpyrad	z score (FFP RSD 25 %)		Thiabendazole	z score (FFP RSD 25 %)		Thiamethoxam	z score (FFP RSD 25 %)			
		0.01			0.01			0.01			0.01			0.01			0.01			0.01			0.01		0.01	
		MRRL (mg/kg)			0.01			0.01			0.01			0.01			0.01			0.01			0.01		0.01	
Robust mean (mg/kg)	0.056		0.935		0.603		0.122		1.220		1.029		0.048		0.367											
Lab070	0.046	-0.7	1.030	0.4	0.708	0.7	0.126	0.1	1.260	0.1	1.060	0.1	0.059	0.9	0.388	0.2										
Lab071	0.052	-0.3	0.580	-1.5	0.450	-1.0	0.074	-1.6	1.310	0.3	0.751	-1.1	0.059	0.9	0.379	0.1										
Lab072	0.066	0.7	0.898	-0.2	0.624	0.1	0.140	0.6	1.205	0.0	0.765	-1.0	0.043	-0.4	0.418	0.6										
Lab073	0.074	1.3	1.130	0.8	0.727	0.8	0.131	0.3	1.370	0.5	1.220	0.7	0.056	0.7	0.409	0.5										
Lab074	NA	NA	0.949	0.1	0.563	-0.3	0.085	-1.2	1.300	0.3	1.090	0.2	0.047	-0.1	NA	NA										
Lab075	0.041	-1.1	1.050	0.5	0.727	0.8	0.193	2.3	1.200	-0.1	1.110	0.3	0.052	0.3	0.373	0.1										
Lab076	0.042	-1.0	0.941	0.0	0.060	-3.6	0.108	-0.5	0.925	-1.0	0.860	-0.7	0.029	-1.6	0.311	-0.6										
Lab077	0.067	0.8	1.290	1.5	0.867	1.8	0.167	1.5	1.620	1.3	1.380	1.4	0.054	0.5	0.434	0.7										
Lab078	0.033	-1.6	0.669	-1.1	0.529	-0.5	0.077	-1.5	0.775	-1.5	0.665	-1.4	NA	NA	0.224	-1.6										
Lab079	0.053	-0.3	0.864	-0.3	0.522	-0.5	0.116	-0.2	1.131	-0.3	0.959	-0.3	0.056	0.6	0.408	0.4										
Lab080	0.044	-0.9	1.150	0.9	0.822	1.5	0.128	0.2	1.570	1.1	1.180	0.6	0.043	-0.4	0.402	0.4										
Lab081	0.079	1.6	1.033	0.4	0.512	-0.6	0.150	0.9	1.340	0.4	1.080	0.2	0.050	0.1	0.403	0.4										
Lab082	ND	-3.3	0.710	-1.0	NA	NA	0.120	-0.1	0.890	-1.1	0.770	-1.0	0.014	-2.8	NA	NA										
Lab083	0.050	-0.4	0.910	-0.1	0.600	0.0	0.099	-0.8	1.200	-0.1	1.000	-0.1	0.073	2.1	0.390	0.3										
Lab084	0.041	-1.1	1.100	0.7	0.600	0.0	0.110	-0.4	1.400	0.6	1.100	0.3	0.039	-0.8	NA	NA										
Lab085	0.039	-1.2	1.020	0.4	0.561	-0.3	0.157	1.1	1.410	0.6	1.150	0.5	0.042	-0.5	0.384	0.2										
Lab086	0.080	1.7	0.720	-0.9	0.565	-0.3	0.090	-1.0	1.434	0.7	0.955	-0.3	0.061	1.1	0.462	1.0										
Lab087	0.060	0.3	1.170	1.0	0.760	1.0	0.160	1.2	1.300	0.3	1.210	0.7	0.043	-0.4	0.351	-0.2										
Lab088	NA	NA	NA	NA	NA	NA	NA	NA	0.861	-1.2	NA	NA	NA	NA	NA	NA										
Lab089	0.045	-0.8	0.947	0.1	0.540	-0.4	0.100	-0.7	1.210	0.0	1.059	0.1	0.046	-0.2	0.352	-0.2										
Lab090	0.046	-0.7	0.680	-1.1	NA	NA	0.127	0.2	0.965	-0.8	NA	NA	0.061	1.1	0.425	0.6										
Lab091	0.071	1.1	0.926	0.0	0.612	0.1	0.158	1.2	1.360	0.5	0.885	-0.6	0.034	-1.2	0.394	0.3										
Lab092	0.051	-0.4	0.990	0.2	0.610	0.0	0.140	0.6	1.200	-0.1	1.100	0.3	0.052	0.3	0.380	0.1										
Lab093	0.057	0.1	1.459	2.2	0.539	-0.4	0.122	0.0	1.264	0.1	0.962	-0.3	0.045	-0.3	0.336	-0.3										
Lab094	0.064	0.6	1.062	0.5	0.669	0.4	0.107	-0.5	1.370	0.5	1.050	0.1	0.155	5.0	0.473	1.2										
Lab095	0.070	1.0	0.769	-0.7	0.727	0.8	0.137	0.5	1.386	0.5	1.153	0.5	0.064	1.3	0.395	0.3										
Lab096	NA	NA	0.857	-0.3	NA	NA	0.106	-0.5	NA	NA	NA	NA	0.075	2.2	NA	NA										
Lab097	NA	NA	0.610	-1.4	NA	NA	ND	-3.7	1.300	0.3	0.630	-1.6	0.033	-1.3	0.330	-0.4										
Lab098	0.023	-2.4	0.560	-1.6	0.490	-0.7	0.095	-0.9	1.260	0.1	0.910	-0.5	0.031	-1.4	0.320	-0.5										
Lab099	0.045	-0.8	0.986	0.2	0.560	-0.3	0.187	2.1	1.110	-0.4	0.974	-0.2	0.048	0.0	0.289	-0.9										
Lab100	0.061	0.4	0.742	-0.8	0.433	-1.1	0.142	0.7	0.941	-0.9	0.848	-0.7	0.055	0.6	0.360	-0.1										
Lab101	0.069	0.9	1.040	0.4	NA	NA	0.095	-0.9	1.120	-0.3	0.952	-0.3	0.049	0.1	0.332	-0.4										
Lab102	0.110	3.8	0.812	-0.5	0.469	-0.9	ND	-4.0	1.224	0.0	0.985	-0.2	0.051	0.2	0.516	1.6										
Lab103	0.074	1.3	0.902	-0.1	0.432	-1.1	ND	-4.0	1.238	0.1	0.941	-0.3	0.046	-0.2	0.575	2.3										
Lab104	ND	-3.3	0.929	0.0	0.384	-1.5	0.137	0.5	1.094	-0.4	0.957	-0.3	0.047	-0.1	0.365	0.0										
Lab105	0.048	-0.6	1.150	0.9	0.481	-0.8	0.086	-1.2	1.160	-0.2	1.340	1.2	0.045	-0.3	0.315	-0.6										
Lab106	0.046	-0.7	0.824	-0.5	0.535	-0.5	0.093	-0.9	1.160	-0.2	0.955	-0.3	0.078	2.5	0.264	-1.1										
Lab107	0.069	0.9	0.998	0.3	0.576	-0.2	0.112	-0.3	1.170	-0.2	1.250	0.9	0.051	0.3	0.387	0.2										
Lab108	0.050	-0.4	0.610	-1.4	0.750	1.0	0.180	1.9	0.850	-1.2	0.920	-0.4	0.320	5.0	0.160	-2.3										
Lab109	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA										
Lab110	0.041	-1.1	NA	NA	NA	NA	0.166	1.4	0.903	-1.0	NA	NA	0.033	-1.3	0.300	-0.7										
Lab111	0.068	0.9	1.180	1.0	0.434	-1.1	0.190	2.2	1.070	-0.5	0.807	-0.9	0.054	0.5	0.440	0.8										
Lab112	ND	-3.3	NA	NA	NA	NA	NA	NA	3.170	5.0	NA	NA	0.048	0.0	NA	NA										
Lab113	0.057	0.1	0.829	-0.5	NA	NA	0.064	-1.9	0.887	-1.1	NA	NA	0.049	0.1	0.356	-0.1										
Lab114	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	1.220	0.7	NA	NA	NA	NA										
Lab115	0.071	1.1	0.642	-1.3	0.417	-1.2	0.171	1.6	1.190	-0.1	0.939	-0.3	0.049	0.1	0.356	-0.1										
Lab116	0.057	0.0	0.979	0.2	0.569	-0.2	0.121	0.0	1.160	-0.2	0.935	-0.4	0.035	-1.1	0.322	-0.5										
Lab117	0.039	-1.2	0.963	0.1	0.568	-0.2	0.116	-0.2	1.450	0.8	1.100	0.3	0.043	-0.4	0.375	0.1										

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

Lab Code	Iprodione	z score (FFP RSD 25 %)		Pyridaben	z score (FFP RSD 25 %)		Spiromesifen	z score (FFP RSD 25 %)		Tau-fluvalinate	z score (FFP RSD 25 %)		Tebuconazole	z score (FFP RSD 25 %)		Tebufenpyrad	z score (FFP RSD 25 %)		Thiabendazole	z score (FFP RSD 25 %)		Thiamethoxam	z score (FFP RSD 25 %)			
		0.01			0.01			0.01			0.01			0.01			0.01			0.01			0.01		0.01	
		MRRL (mg/kg)			0.01			0.01			0.01			0.01			0.01			0.01			0.01		0.01	
Robust mean (mg/kg)	0.056		0.935		0.603		0.122		1.220		1.029		0.048		0.367											
Lab118	0.014	-3.0	0.747	-0.8	0.416	-1.2	0.057	-2.1	0.871	-1.1	0.604	-1.7	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA			
Lab119	0.071	1.1	0.735	-0.9	0.559	-0.3	0.202	2.6	0.895	-1.1	0.993	-0.1	0.052	0.3	0.302	-0.7										
Lab120	0.067	0.8	1.150	0.9	NA	NA	NA	NA	1.210	0.0	1.090	0.2	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA			
Lab121	0.056	0.0	0.914	-0.1	0.546	-0.4	0.110	-0.4	1.330	0.4	0.979	-0.2	0.045	-0.2	0.366	0.0										
Lab122	0.050	-0.4	1.023	0.4	0.642	0.3	0.133	0.4	1.276	0.2	1.111	0.3	0.057	0.8	0.391	0.3										
Lab123	0.060	0.3	0.738	-0.8	0.653	0.3	0.145	0.8	1.140	-0.3	0.767	-1.0	0.043	-0.4	0.350	-0.2										
Lab124	Participation cancelled																									
Lab125	0.061	0.4	0.931	0.0	0.568	-0.2	0.132	0.3	1.270	0.2	1.020	0.0	0.043	-0.4	0.395	0.3										
Lab126	0.062	0.4	1.070	0.6	0.599	0.0	0.260	4.5	1.240	0.1	1.250	0.9	0.063	1.3	0.387	0.2										
Lab127	0.040	-1.1	1.090	0.7	0.650	0.3	0.141	0.6	1.310	0.3	1.050	0.1	0.056	0.6	0.391	0.3										
Lab128	0.050	-0.4	1.170	1.0	0.706	0.7	0.178	1.8	1.260	0.1	1.080	0.2	0.055	0.6	0.365	0.0										
Lab129	0.056	0.0	1.310	1.6	NA	NA	0.129	0.2	1.200	-0.1	1.110	0.3	NA	NA	0.486	1.3										
Lab130	0.047	-0.6	0.882	-0.2	0.433	-1.1	0.123	0.0	1.000	-0.7	1.190	0.6	0.039	-0.7	0.417	0.5										
Lab131	NA	NA	0.871	-0.3	NA	NA	NA	NA	1.090	-0.4	1.010	-0.1	0.040	-0.7	0.281	-0.9										
Lab132	NA	NA	1.090	0.7	0.570	-0.2	NA	NA	1.180	-0.1	1.150	0.5	0.029	-1.6	0.341	-0.3										
Lab133	0.040	-1.1	0.730	-0.9	0.240	-2.4	0.064	-1.9	0.940	-0.9	0.710	-1.2	0.047	-0.1	0.320	-0.5										
Lab134	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.726	-1.2	0.058	0.8	0.435	0.7										
Lab135	0.050	-0.4	0.920	-0.1	NA	NA	0.130	0.3	1.210	0.0	1.030	0.0	0.040	-0.7	0.330	-0.4										
Lab136	0.063	0.5	1.060	0.5	0.737	0.9	0.114	-0.3	1.490	0.9	1.240	0.8	0.042	-0.5	0.377	0.1										
Lab137	0.071	1.1	0.757	-0.8	0.884	1.9	0.146	0.8	1.341	0.4	0.969	-0.2	0.043	-0.4	0.336	-0.3										
Lab138	0.050	-0.4	0.850	-0.4	0.530	-0.5	0.110	-0.4	1.080	-0.5	1.080	0.2	0.040	-0.7	0.340	-0.3										
Lab139	0.017	-2.8	0.482	-1.9	0.277	-2.2	0.073	-1.6	0.863	-1.2	0.443	-2.3	0.042	-0.5	0.266	-1.1										
Lab140	0.050	-0.5	0.991	0.2	0.594	-0.1	0.129	0.2	1.070	-0.5	1.010	-0.1	0.045	-0.3	0.314	-0.6										
Lab141	0.059	0.2	1.210	1.2	0.780	1.2	0.100	-0.7	1.420	0.7	1.240	0.8	0.047	-0.1	0.310	-0.6										
Lab142	0.064	0.5	0.845	-0.4	0.621	0.1	0.164	1.4	1.110	-0.4	1.040	0.0	NA	NA	0.252	-1.3										
Lab143	0.051	-0.4	0.814	-0.5	0.474	-0.9	0.077	-1.5	1.285	0.2	1.134	0.4	0.050	0.2	0.376	0.1										
Lab144	0.062	0.4	1.098	0.7	0.681	0.5	0.117	-0.2	1.689	1.5	1.295	1.0	0.045	-0.2	0.419	0.6										
Lab145	0.038	-1.3	0.900	-0.1	0.580	-0.2	0.140	0.6	1.390	0.6	1.330	1.2	0.046	-0.2	0.390	0.3										
Lab146	0.042	-1.0	1.101	0.7	0.738	0.9	0.060	-2.0	1.528	1.0	1.264	0.9	0.063	1.3	0.344	-0.3										
Lab147	0.082	1.9	1.052	0.5	0.669	0.4	NA	NA	1.154	-0.2	0.982	-0.2	0.043	-0.4	NA	NA										
Lab148	0.073	1.2	0.980	0.2	0.790	1.2	0.150	0.9	1.600	1.2	1.200	0.7	0.053	0.4	0.360	-0.1										
Lab149	Participation cancelled																									
Lab150	0.046	-0.7	0.855	-0.3	0.578	-0.2	0.143	0.7	1.040	-0.6	0.959	-0.3	0.039	-0.8	NA	NA										
Lab151	NA	NA	0.833	-0.4	NA	NA	0.113	-0.3	1.592	1.2	0.802	-0.9	NA	NA	NA	NA										
Lab152	0.117	4.4	0.919	-0.1	0.524	-0.5	0.172	1.6	1.401	0.6	1.192	0.6	0.059	0.9	0.431	0.7										
Lab153	0.021	-2.5	0.981	0.2	0.851	1.6	0.071	-1.7	0.576	-2.1	0.962	-0.3	0.049	0.1	0.256	-1.2										
Lab154	0.033	-1.7	1.050	0.5	0.422	-1.2	NA	NA	1.830	2.0	1.270	0.9	0.294	5.0	0.316	-0.6										
Lab155	0.085	2.1	1.060	0.5	0.656	0.4	0.213	3.0	1.600	1.2	1.280	1.0	0.038	-0.9	0.275	-1.0										
Lab156	0.079	1.6	1.222	1.2	NA	NA	0.131	0.3	1.141	-0.3	0.861	-0.7	0.051	0.3	NA	NA										
Lab157	0.063	0.5	0.985	0.2	NA	NA	0.130	0.3	1.280	0.2	1.020	0.0	0.042	-0.5	NA	NA										
Lab158	0.054	-0.1	1.040	0.4	0.641	0.3	0.104	-0.6	1.220	0.0	1.040	0.0	0.047	-0.1	0.336	-0.3										
Lab159	0.091	2.5	0.729	-0.9	0.486	-0.8	NA	NA	1.160	-0.2	1.020	0.0	0.056	0.7	0.285	-0.9										
Lab160	NA	NA	1.280	1.5	0.583	-0.1	0.194	2.4	1.070	-0.5	NA	NA	NA	NA	NA	NA										
Lab161	0.047	-0.6	0.958	0.1	NA	NA	0.076	-1.5	1.060	-0.5	1.420	1.5	0.053	0.4	NA	NA										
Lab162	0.055	-0.1	0.974	0.2	0.292	-2.1	0.098	-0.8	1.001	-0.7	0.687	-1.3	0.049	0.1	0.354	-0.1										
Lab163	0.082	1.9	1.087	0.7	0.698	0.6	0.152	1.0	1.302	0.3	1.106	0.3	0.046	-0.2	0.373	0.1										
Lab164	0.038	-1.3	0.650	-1.2	0.190	-2.7	0.081	-1.3	0.840	-1.2	0.660	-1.4	0.081	2.8	0.370	0.0										
Lab165	0.046	-0.7	NA	NA	0.677	0.5	0.124	0.1	1.520	1.0	NA	NA	0.361	5.0	0.391	0.3										

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

Lab Code	Iprodione	z score (FFP RSD 25 %)		Pyridaben	z score (FFP RSD 25 %)		Spiromesifen	z score (FFP RSD 25 %)		Tau-fluvalinate	z score (FFP RSD 25 %)		Tebuconazole	z score (FFP RSD 25 %)		Tebufenpyrad	z score (FFP RSD 25 %)		Thiabendazole	z score (FFP RSD 25 %)		Thiamethoxam	z score (FFP RSD 25 %)		
		MRRL (mg/kg)	0.01		0.01	0.01		0.01	0.01		0.01	0.01		0.01	0.01		0.01	0.01		0.01	0.01		0.01	0.01	0.01
Robust mean (mg/kg)	0.056		0.935		0.603		0.122		1.220		1.029		0.048		0.367										
Lab166	0.040	-1.1	0.890	-0.2	0.520	-0.6	0.140	0.6	0.940	-0.9	0.800	-0.9	0.040	-0.7	0.360	-0.1									
Lab167	0.060	0.3	1.030	0.4	0.797	1.3	0.029	-3.1	1.370	0.5	1.220	0.7	0.033	-1.2	0.166	-2.2									
Lab168	0.070	1.0	0.756	-0.8	0.910	2.0	0.152	1.0	0.922	-1.0	0.778	-1.0	0.063	1.3	0.402	0.4									
Lab169	0.044	-0.9	NA	NA	NA	NA	0.112	-0.3	0.960	-0.9	NA	NA	0.046	-0.2	NA	NA									
Lab170	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.049	0.1	0.535	1.8									
Lab171	0.056	0.0	0.982	0.2	0.576	-0.2	0.118	-0.1	1.140	-0.3	0.996	-0.1	0.042	-0.5	0.348	-0.2									
Lab172	0.050	-0.4	0.880	-0.2	0.490	-0.7	0.158	1.2	1.140	-0.3	1.190	0.6	0.050	0.2	0.350	-0.2									
Lab173	0.072	1.2	ND	-4.0	0.202	-2.7	ND	-3.7	1.050	-0.6	0.215	-3.2	0.022	-2.1	0.227	-1.5									
Lab174	0.070	1.0	0.753	-0.8	0.256	-2.3	0.131	0.3	0.993	-0.7	0.922	-0.4	0.050	0.2	0.545	1.9									
Lab175	0.021	-2.5	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA									
Lab176	0.054	-0.1	1.280	1.5	0.528	-0.5	0.122	0.0	1.540	1.0	1.230	0.8	0.044	-0.3	0.483	1.3									
Lab177	0.056	0.0	0.954	0.1	NA	NA	0.096	-0.9	1.440	0.7	1.130	0.4	0.059	0.9	0.365	0.0									
Lab178	0.053	-0.2	0.944	0.0	NA	NA	0.112	-0.3	1.017	-0.7	0.915	-0.4	0.051	0.3	0.319	-0.5									
Lab179	0.045	-0.8	1.000	0.3	0.600	0.0	0.130	0.3	1.300	0.3	0.980	-0.2	0.050	0.2	0.370	0.0									
Lab180	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA									
Lab181	0.108	3.7	NA	NA	NA	NA	NA	NA	1.280	0.2	NA	NA	NA	NA	NA	NA									
Lab182	ND	-3.3	0.051	-3.8	0.970	2.4	0.016	-3.5	0.061	-3.8	0.034	-3.9	NA	NA	NA	NA									
Lab183	0.034	-1.6	0.811	-0.5	0.281	-2.1	NA	NA	0.762	-1.5	0.773	-1.0	0.037	-0.9	0.269	-1.1									
Lab184	0.062	0.4	0.860	-0.3	0.470	-0.9	0.120	-0.1	1.300	0.3	1.000	-0.1	0.048	0.0	0.330	-0.4									
Lab185	0.077	1.5	NA	NA	0.496	-0.7	0.114	-0.3	1.380	0.5	0.487	-2.1	0.027	-1.8	0.488	1.3									
Lab186	0.054	-0.1	0.956	0.1	0.624	0.1	0.127	0.2	1.338	0.4	1.081	0.2	0.038	-0.9	0.503	1.5									

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 fenpyrazamine and penthiopyrad (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	Fenpyrazamine	z score (FFP RSD 25 %)	Penthiopyrad	z score (FFP RSD 25 %)
	MRRL (mg/kg)		0.01	
Robust mean (mg/kg)	0.131		0.058	
Lab001	0.160	0.9	NA	NA
Lab002	0.144	0.4	0.063	0.3
Lab003	0.120	-0.3	0.042	-1.1
Lab004	NA	NA	NA	NA
Lab005	0.138	0.2	0.053	-0.3
Lab006	0.125	-0.2	0.052	-0.4
Lab007	0.100	-0.9	0.040	-1.2
Lab008	0.160	0.9	0.061	0.2
Lab009	NA	NA	0.053	-0.3
Lab010	0.095	-1.1	0.036	-1.5
Lab011	NA	NA	NA	NA
Lab012	0.133	0.1	0.054	-0.3
Lab013	NA	NA	NA	NA
Lab014	0.130	0.0	0.109	3.5
Lab015	0.120	-0.3	0.047	-0.8
Lab016	0.112	-0.6	0.050	-0.6
Lab017	NA	NA	NA	NA
Lab018	0.127	-0.1	0.055	-0.2
Lab019	NA	NA	NA	NA
Lab020	NA	NA	NA	NA
Lab021	NA	NA	NA	NA
Lab022	NA	NA	NA	NA
Lab023	0.142	0.3	0.065	0.5
Lab024	0.135	0.1	0.071	0.9
Lab025	NA	NA	NA	NA
Lab026	0.108	-0.7	0.055	-0.2
Lab027	NA	NA	NA	NA
Lab028	NA	NA	NA	NA
Lab029	0.112	-0.6	0.054	-0.3
Lab030	NA	NA	NA	NA
Lab031	0.114	-0.5	0.059	0.1
Lab032	NA	NA	NA	NA
Lab033	0.121	-0.3	NA	NA
Lab034	NA	NA	NA	NA
Lab035	NA	NA	NA	NA
Lab036	NA	NA	NA	NA
Lab037	0.200	2.1	0.080	1.5
Lab038	NA	NA	0.062	0.3
Lab039	NA	NA	NA	NA
Lab040	0.140	0.3	0.069	0.8
Lab041	NA	NA	NA	NA
Lab042	NA	NA	NA	NA
Lab043	NA	NA	NA	NA
Lab044	0.130	0.0	0.071	0.9
Lab045	NA	NA	NA	NA
Lab046	0.101	-0.9	0.043	-1.0

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

Lab Code	Fenpyrazamine	z score (FFP RSD 25 %)	Penthiopyrad	z score (FFP RSD 25 %)
	MRRL (mg/kg)		0.01	
	Robust mean (mg/kg)		0.058	
Lab047	0.122	-0.3	0.043	-1.0
Lab048	0.131	0.0	0.068	0.7
Lab049	0.101	-0.9	0.055	-0.2
Lab050	0.123	-0.2	0.065	0.5
Lab051	0.126	-0.2	0.049	-0.6
Lab052	0.118	-0.4	0.055	-0.2
Lab053	0.119	-0.4	0.053	-0.3
Lab054	NA	NA	NA	NA
Lab055	0.135	0.1	0.072	0.9
Lab056	No results reported			
Lab057	NA	NA	NA	NA
Lab058	NA	NA	NA	NA
Lab059	0.152	0.6	NA	NA
Lab060	NA	NA	NA	NA
Lab061	0.100	-0.9	0.051	-0.5
Lab062	0.142	0.3	0.055	-0.2
Lab063	NA	NA	NA	NA
Lab064	0.129	-0.1	ND	-3.3
Lab065	NA	NA	NA	NA
Lab066	0.129	-0.1	0.052	-0.4
Lab067	No results reported			
Lab068	0.150	0.6	0.067	0.6
Lab069	0.132	0.0	0.053	-0.4
Lab070	0.214	2.5	0.057	-0.1
Lab071	NA	NA	NA	NA
Lab072	NA	NA	NA	NA
Lab073	0.147	0.5	0.071	0.9
Lab074	NA	NA	NA	NA
Lab075	NA	NA	NA	NA
Lab076	NA	NA	0.016	-2.9
Lab077	0.169	1.2	NA	NA
Lab078	NA	NA	NA	NA
Lab079	0.123	-0.2	0.053	-0.4
Lab080	0.144	0.4	0.066	0.5
Lab081	0.109	-0.7	0.051	-0.5
Lab082	NA	NA	NA	NA
Lab083	NA	NA	NA	NA
Lab084	NA	NA	NA	NA
Lab085	0.141	0.3	0.060	0.1
Lab086	NA	NA	NA	NA
Lab087	0.137	0.2	0.063	0.3
Lab088	NA	NA	NA	NA
Lab089	NA	NA	NA	NA
Lab090	NA	NA	NA	NA
Lab091	NA	NA	NA	NA
Lab092	0.130	0.0	0.062	0.3
Lab093	NA	NA	NA	NA
Lab094	0.132	0.0	0.055	-0.2
Lab095	NA	NA	NA	NA
Lab096	NA	NA	NA	NA



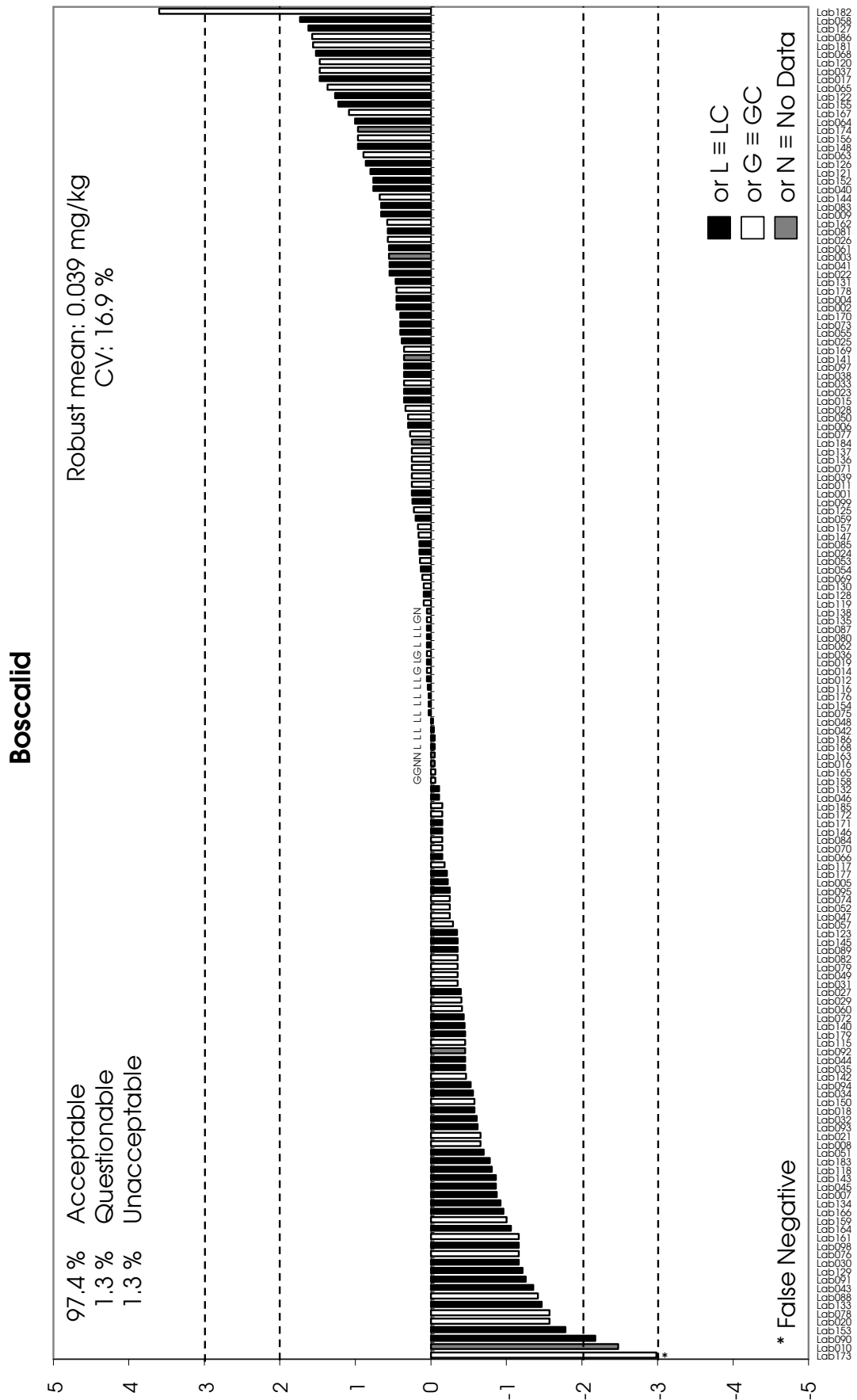
**APPENDIX 3. Results (mg/Kg) and z scores for FFP RSD (25 %).**

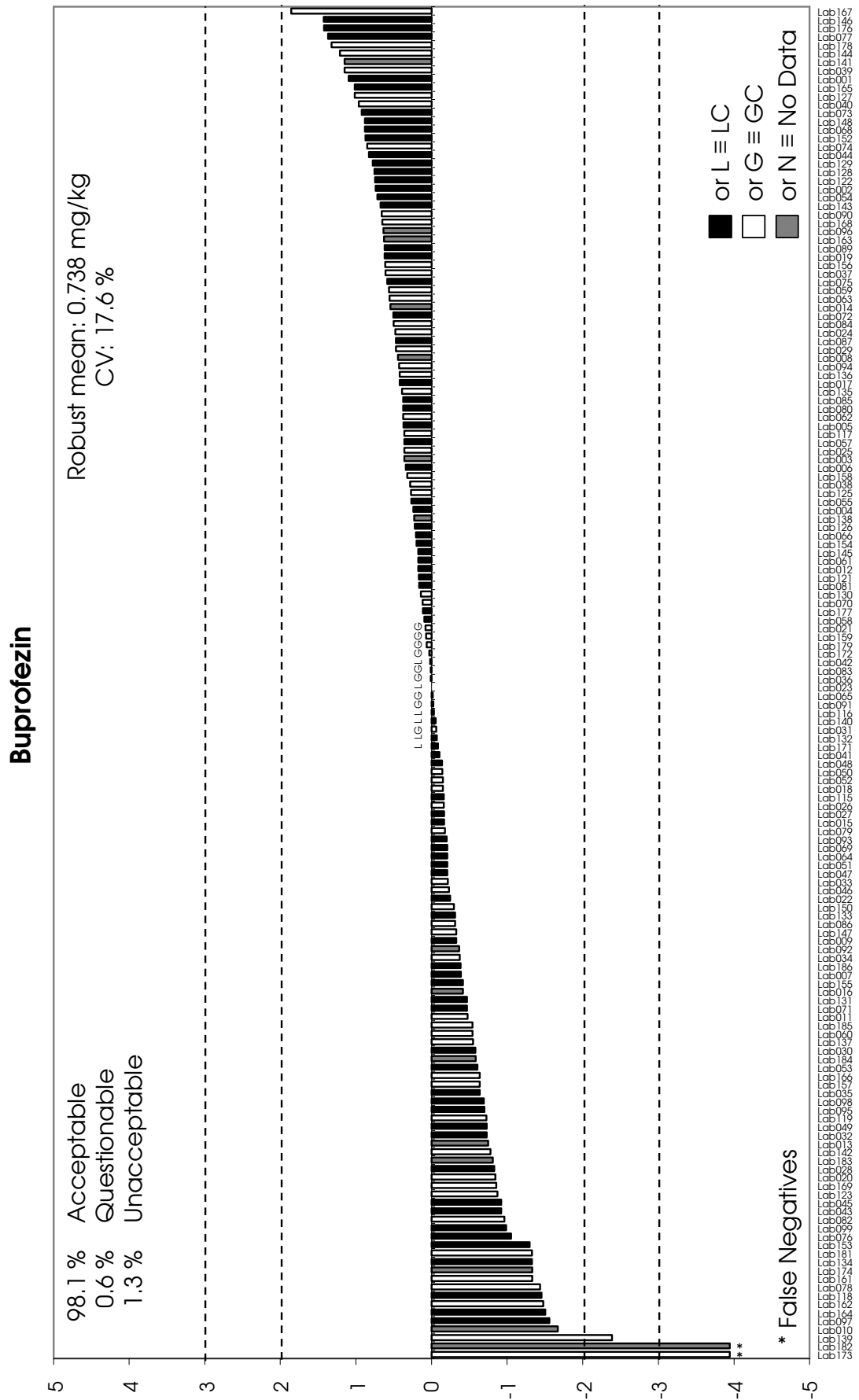
Lab Code	Fenpyrazamine	z score (FFP RSD 25 %)	Penthiopyrad	z score (FFP RSD 25 %)
MRRL (mg/kg)	0.01		0.01	
Robust mean (mg/kg)	0.131		0.058	
Lab097	NA	NA	NA	NA
Lab098	0.130	0.0	0.037	-1.4
Lab099	0.102	-0.9	0.059	0.0
Lab100	NA	NA	0.050	-0.6
Lab101	NA	NA	NA	NA
Lab102	NA	NA	NA	NA
Lab103	NA	NA	NA	NA
Lab104	NA	NA	NA	NA
Lab105	NA	NA	NA	NA
Lab106	0.109	-0.7	0.043	-1.1
Lab107	NA	NA	NA	NA
Lab108	NA	NA	NA	NA
Lab109	NA	NA	NA	NA
Lab110	NA	NA	NA	NA
Lab111	NA	NA	NA	NA
Lab112	NA	NA	NA	NA
Lab113	NA	NA	NA	NA
Lab114	NA	NA	NA	NA
Lab115	0.148	0.5	0.078	1.4
Lab116	NA	NA	NA	NA
Lab117	NA	NA	NA	NA
Lab118	NA	NA	NA	NA
Lab119	0.140	0.3	0.080	1.5
Lab120	NA	NA	NA	NA
Lab121	0.133	0.1	0.053	-0.3
Lab122	0.141	0.3	NA	NA
Lab123	NA	NA	NA	NA
Lab124	Participation cancelled			
Lab125	0.148	0.5	0.074	1.1
Lab126	NA	NA	NA	NA
Lab127	NA	NA	NA	NA
Lab128	0.134	0.1	0.062	0.3
Lab129	NA	NA	NA	NA
Lab130	NA	NA	NA	NA
Lab131	NA	NA	NA	NA
Lab132	NA	NA	NA	NA
Lab133	0.100	-0.9	NA	NA
Lab134	NA	NA	NA	NA
Lab135	NA	NA	NA	NA
Lab136	0.155	0.7	0.062	0.3
Lab137	NA	NA	NA	NA
Lab138	NA	NA	NA	NA
Lab139	NA	NA	NA	NA
Lab140	0.114	-0.5	0.054	-0.3
Lab141	0.150	0.6	0.061	0.2
Lab142	NA	NA	0.053	-0.3
Lab143	NA	NA	NA	NA
Lab144	0.135	0.1	0.085	1.9
Lab145	0.140	0.3	0.058	0.0
Lab146	0.183	1.6	0.062	0.3

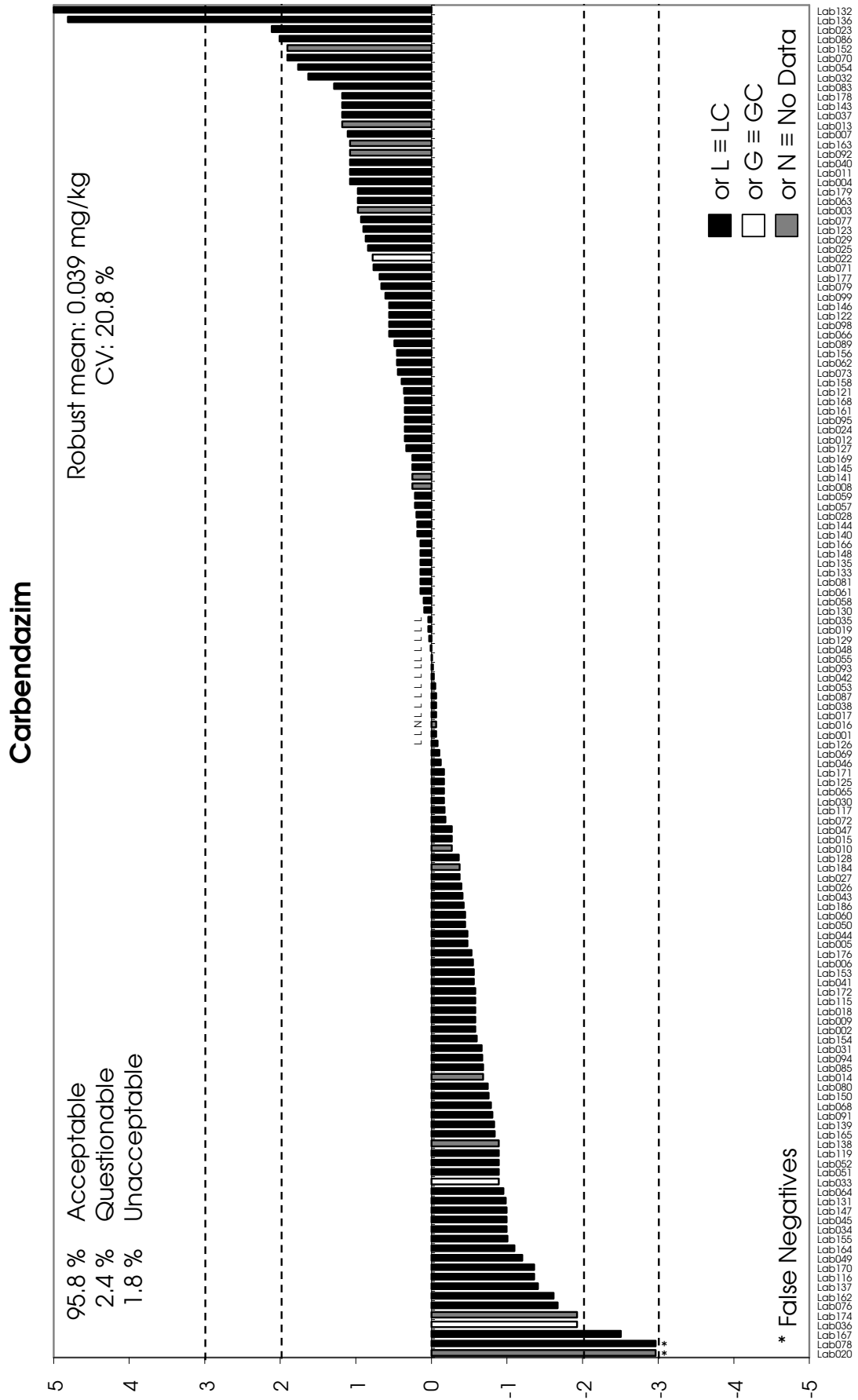
APPENDIX 3. Results (mg/Kg) and z scores for FFP RSD (25 %).

Lab Code	Fenpyrazamine	z score (FFP RSD 25 %)	Penthiopyrad	z score (FFP RSD 25 %)
MRRL (mg/kg)	0.01		0.01	
Robust mean (mg/kg)	0.131		0.058	
Lab147	NA	NA	0.066	0.5
Lab148	0.170	1.2	0.052	-0.4
Lab149	Participation cancelled			
Lab150	0.119	-0.4	0.053	-0.4
Lab151	NA	NA	NA	NA
Lab152	0.152	0.6	NA	NA
Lab153	NA	NA	NA	NA
Lab154	NA	NA	NA	NA
Lab155	0.142	0.3	0.047	-0.8
Lab156	NA	NA	NA	NA
Lab157	NA	NA	NA	NA
Lab158	0.122	-0.3	0.053	-0.4
Lab159	0.099	-1.0	NA	NA
Lab160	NA	NA	NA	NA
Lab161	NA	NA	NA	NA
Lab162	NA	NA	NA	NA
Lab163	NA	NA	NA	NA
Lab164	NA	NA	NA	NA
Lab165	0.220	2.7	NA	NA
Lab166	NA	NA	NA	NA
Lab167	NA	NA	NA	NA
Lab168	NA	NA	NA	NA
Lab169	NA	NA	NA	NA
Lab170	NA	NA	NA	NA
Lab171	0.147	0.5	0.058	0.0
Lab172	0.118	-0.4	0.057	-0.1
Lab173	NA	NA	NA	NA
Lab174	0.100	-0.9	0.050	-0.6
Lab175	NA	NA	NA	NA
Lab176	NA	NA	ND	-3.3
Lab177	NA	NA	NA	NA
Lab178	NA	NA	NA	NA
Lab179	ND	-3.7	0.060	0.1
Lab180	NA	NA	NA	NA
Lab181	NA	NA	NA	NA
Lab182	NA	NA	NA	NA
Lab183	NA	NA	NA	NA
Lab184	0.130	0.0	0.060	0.1
Lab185	NA	NA	NA	NA
Lab186	NA	NA	NA	NA

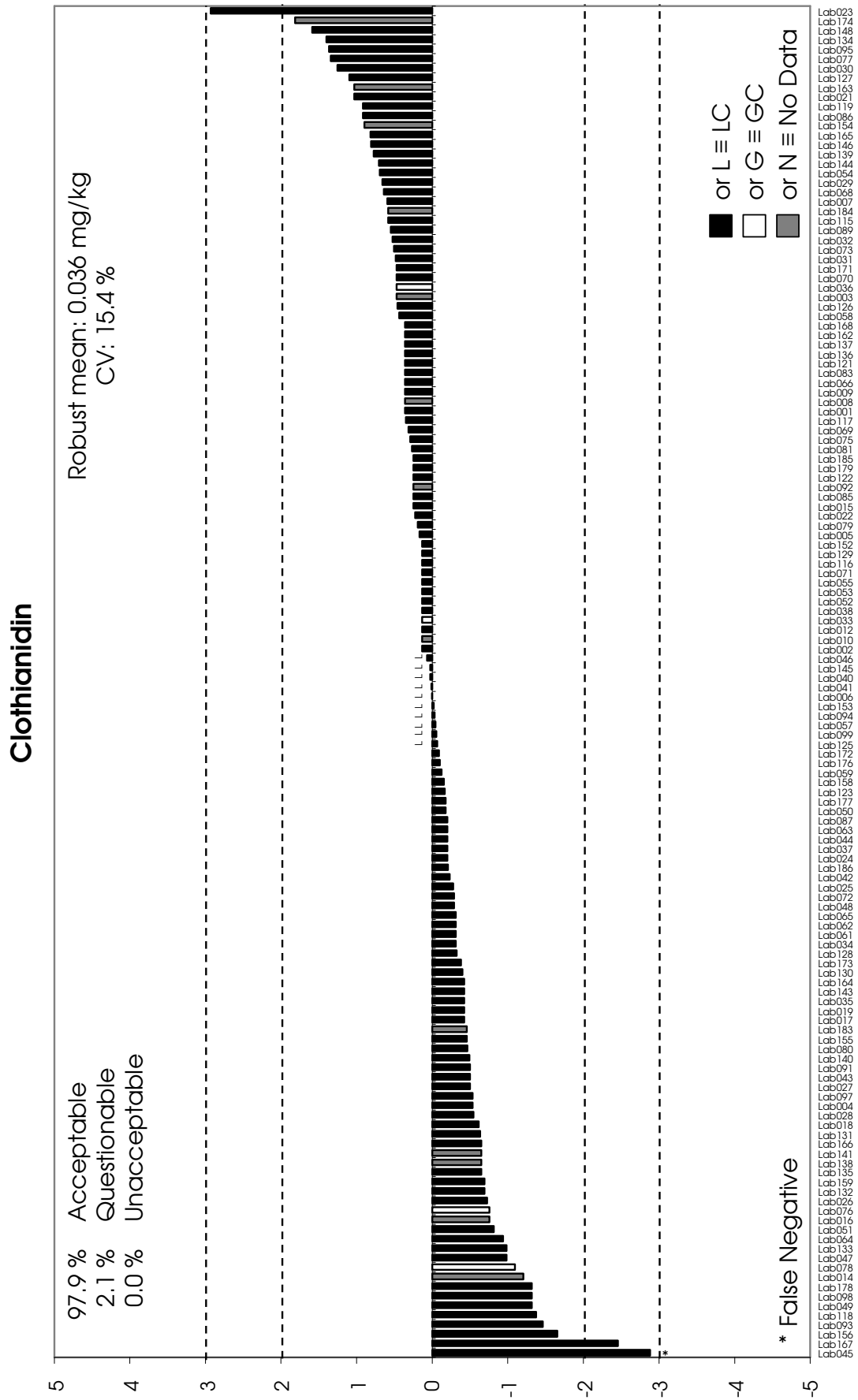
NA: Not analysed  
 ND: Not detected (False negative)

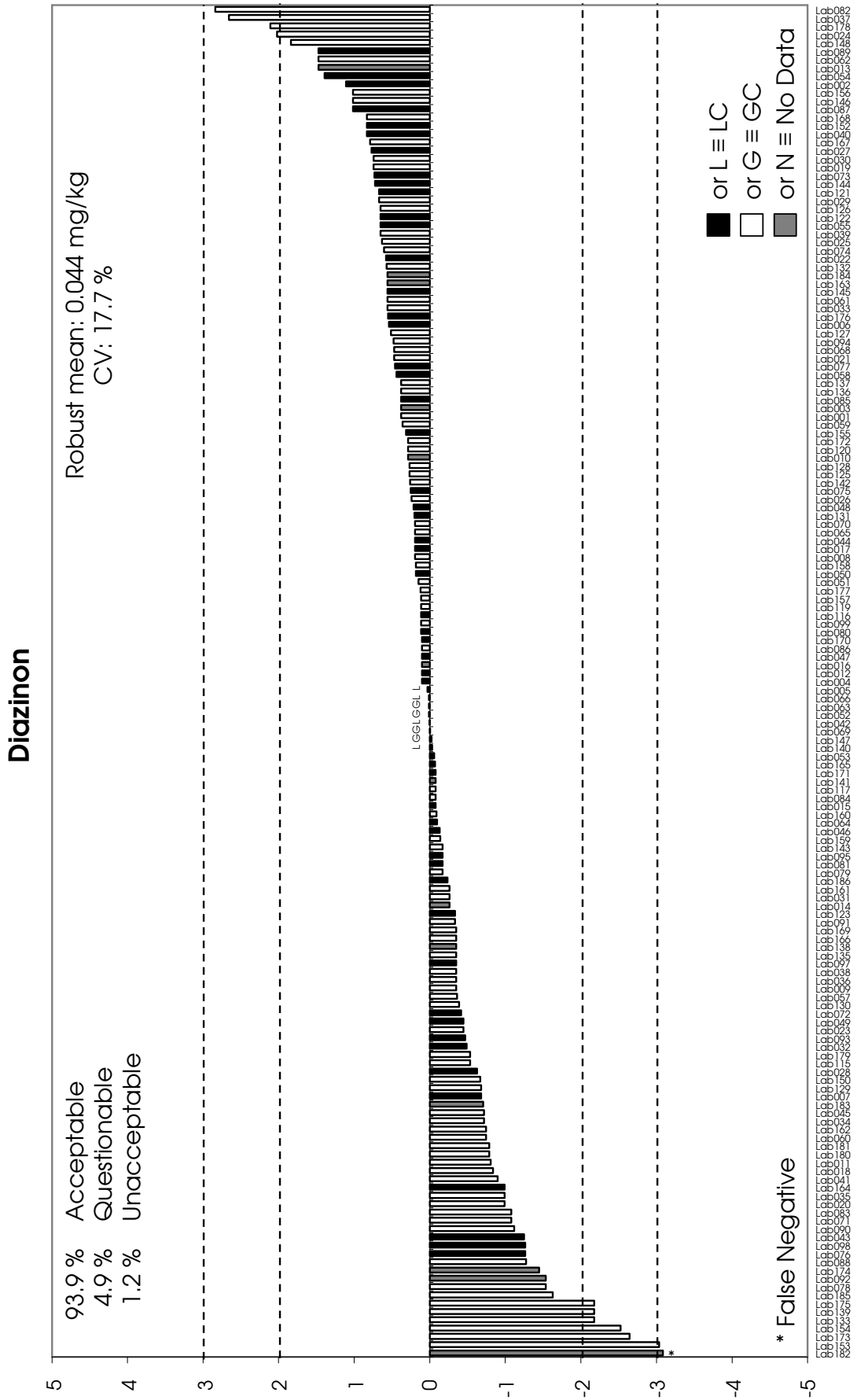




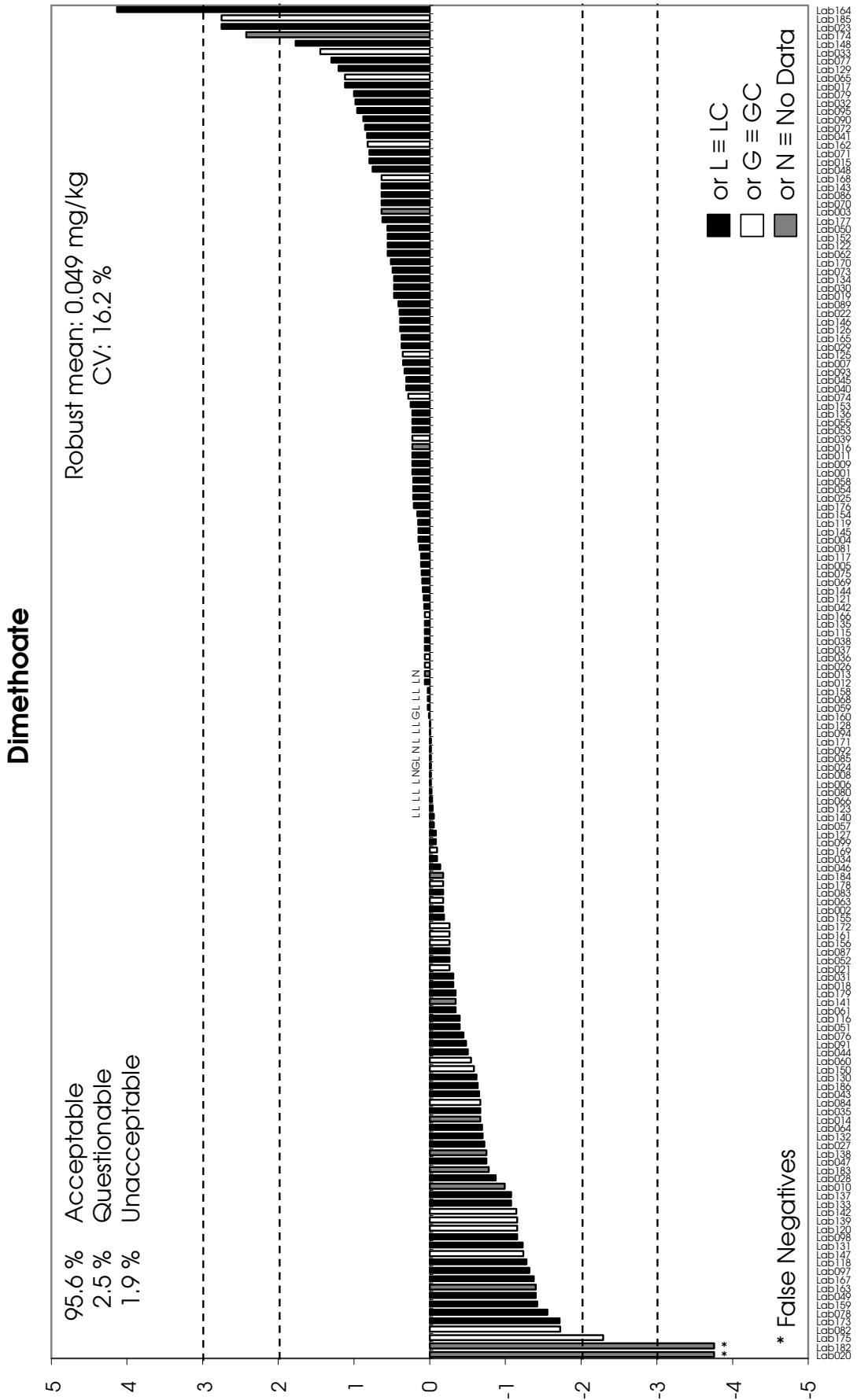


APPENDIX 4. Graphical representation of z scores for FFP RSD (25 %).

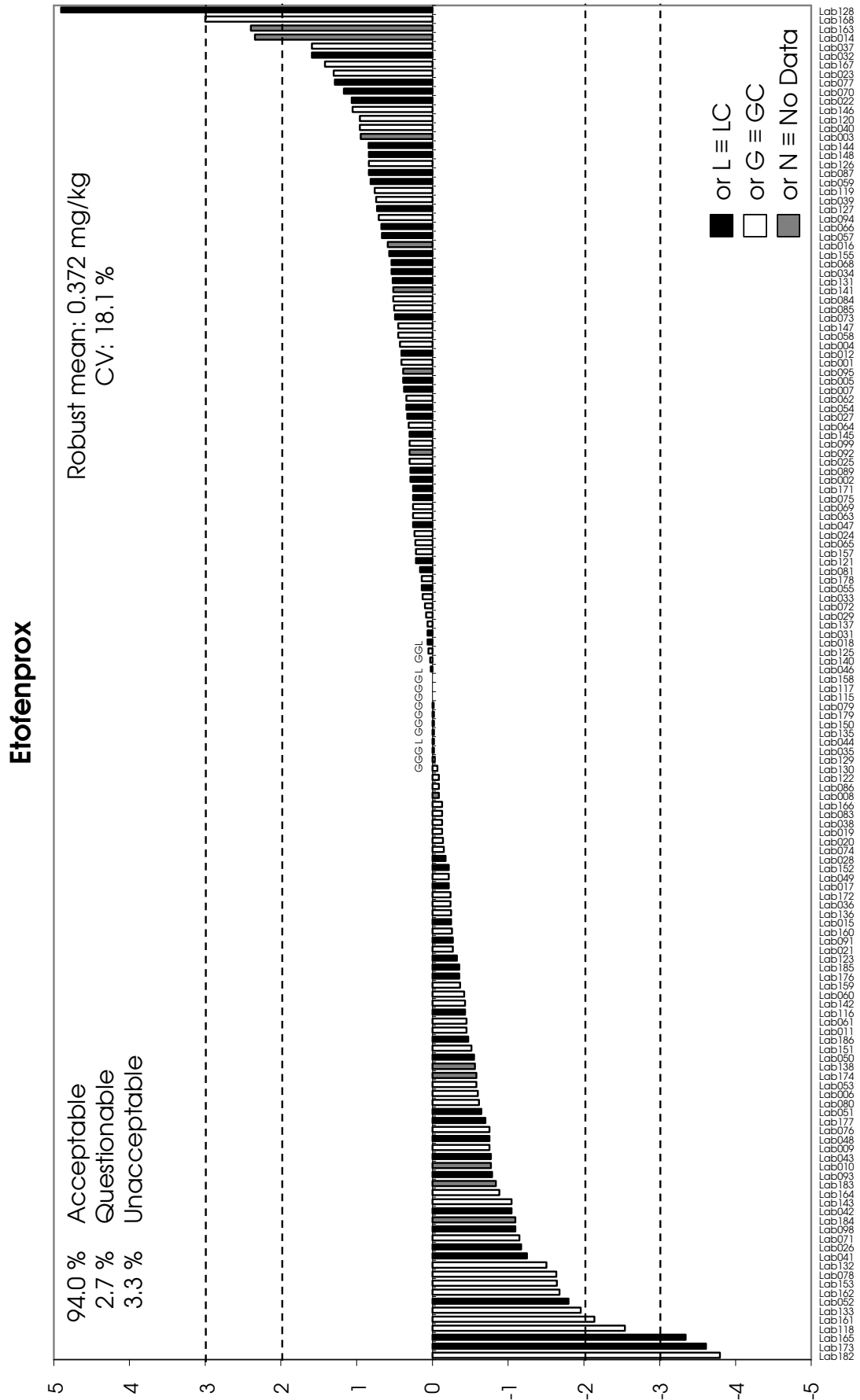




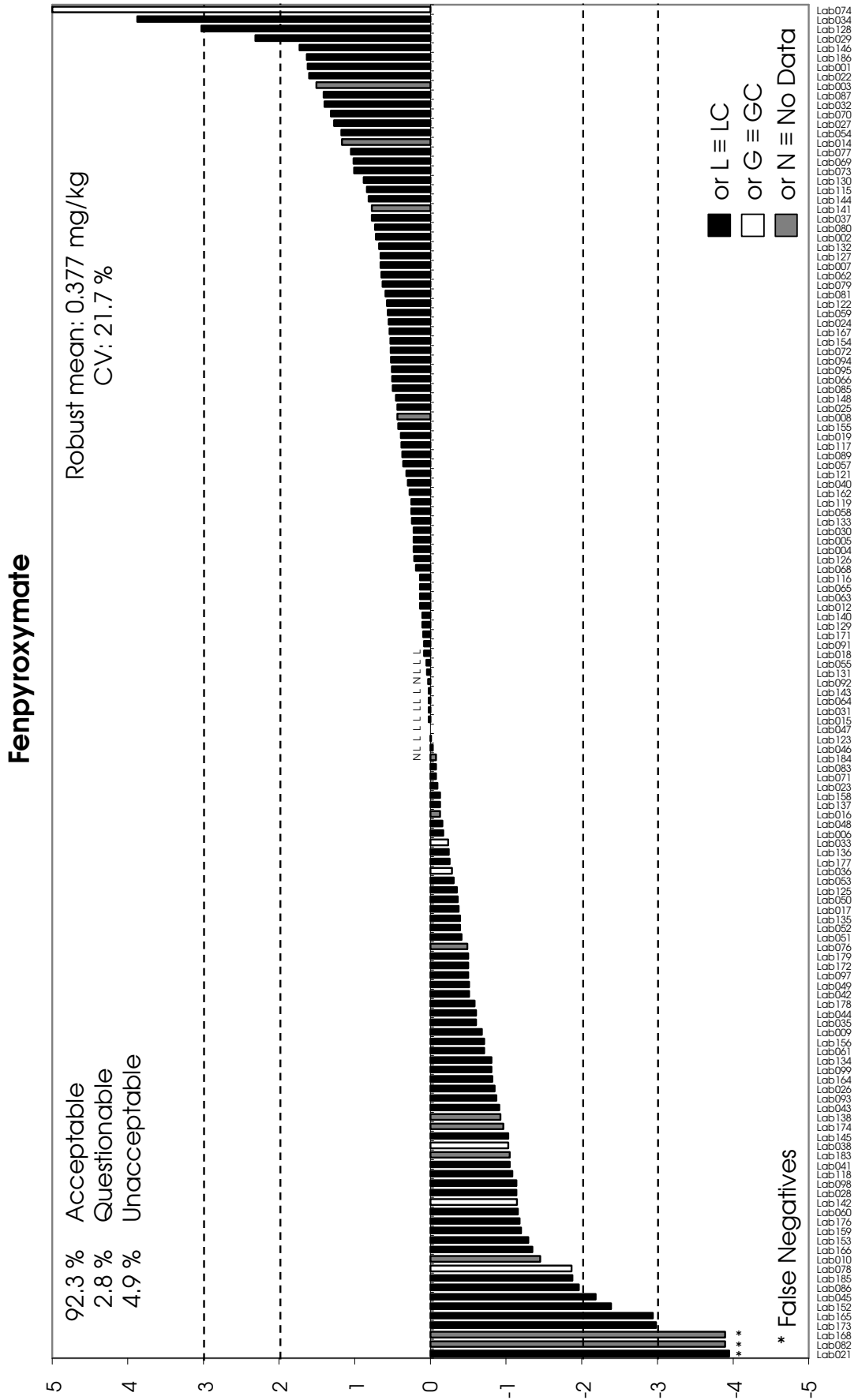
APPENDIX 4. Graphical representation of z scores for FFP RSD (25 %).





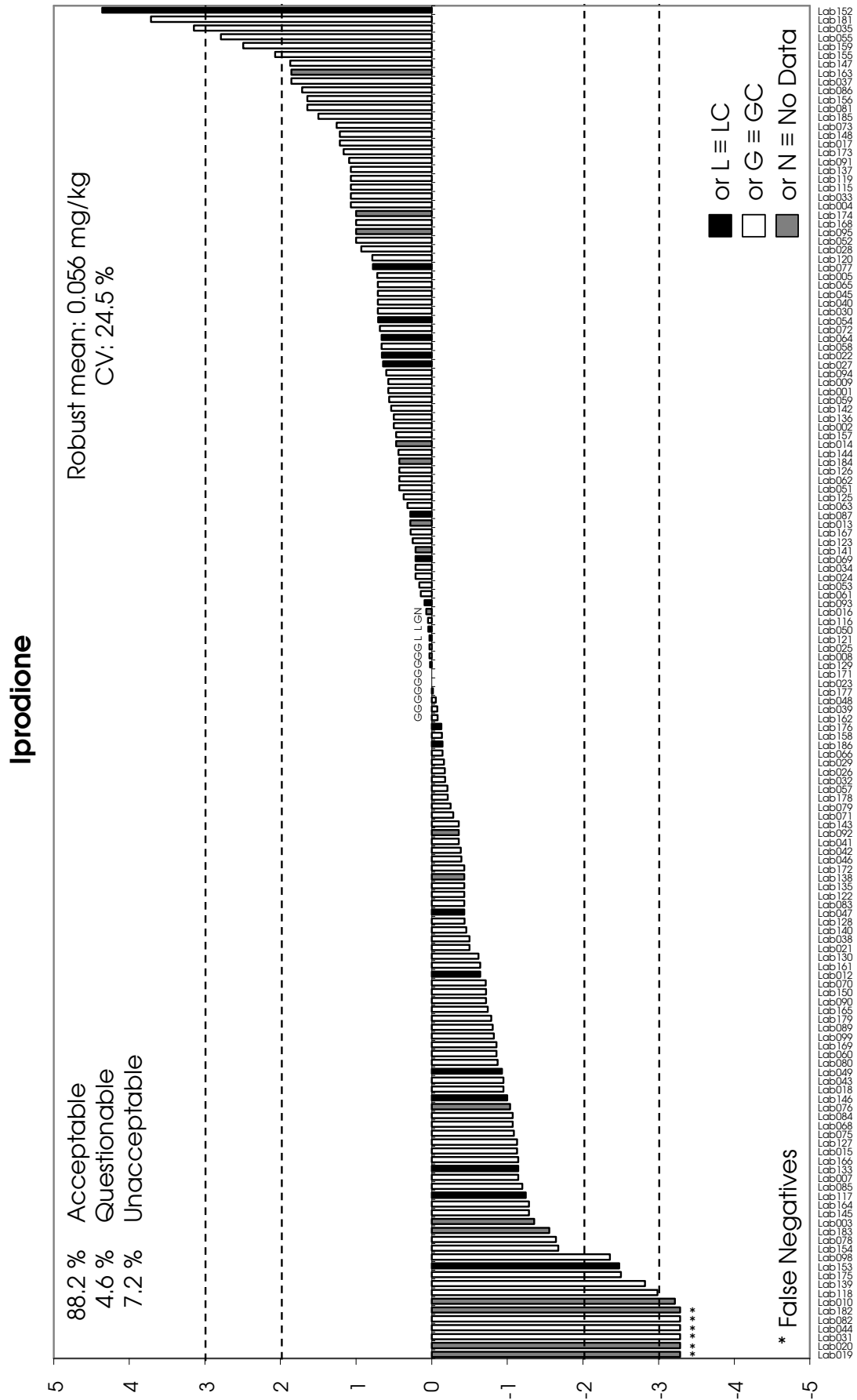


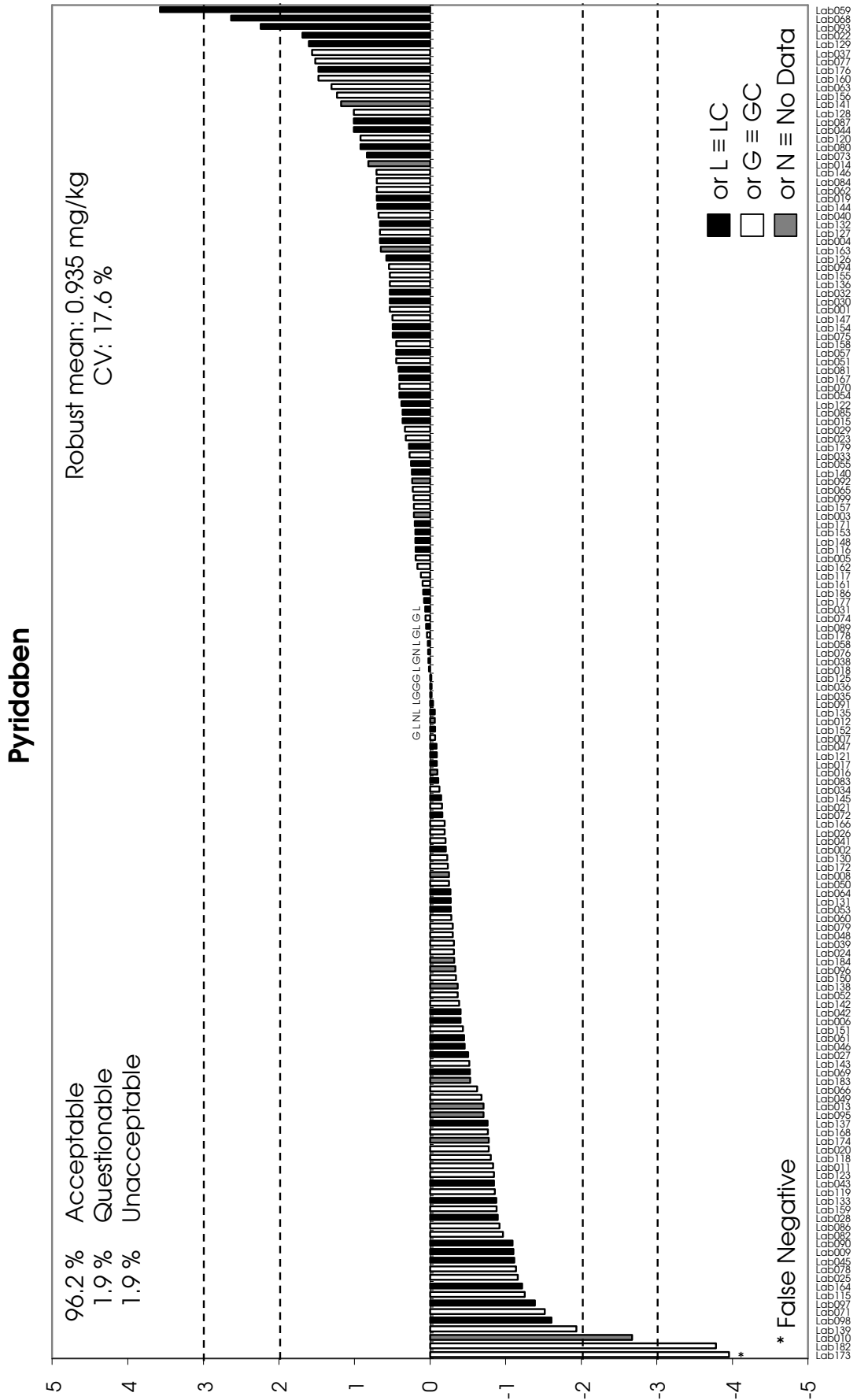
APPENDIX 4. Graphical representation of z scores for FFP RSD (25 %).



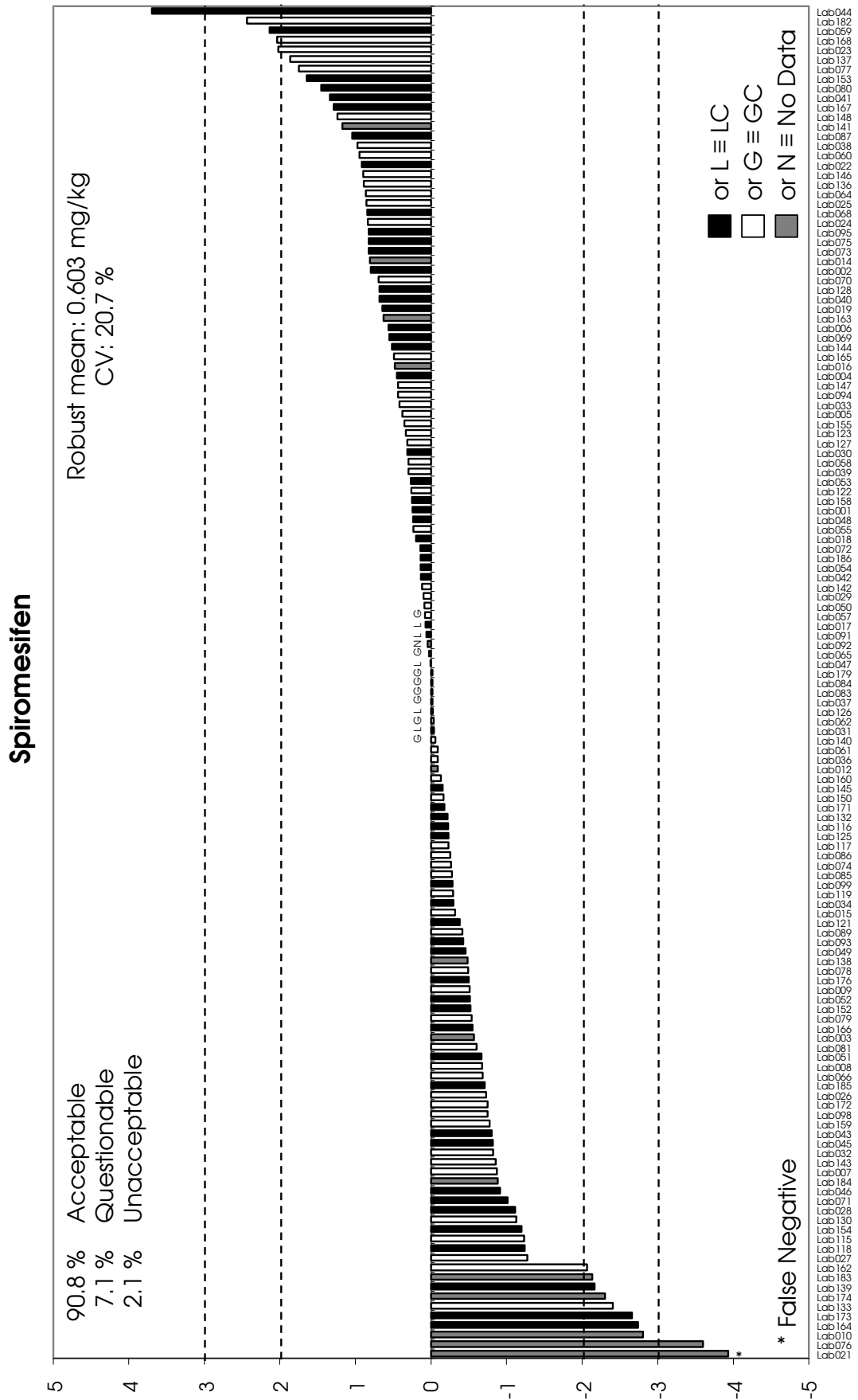


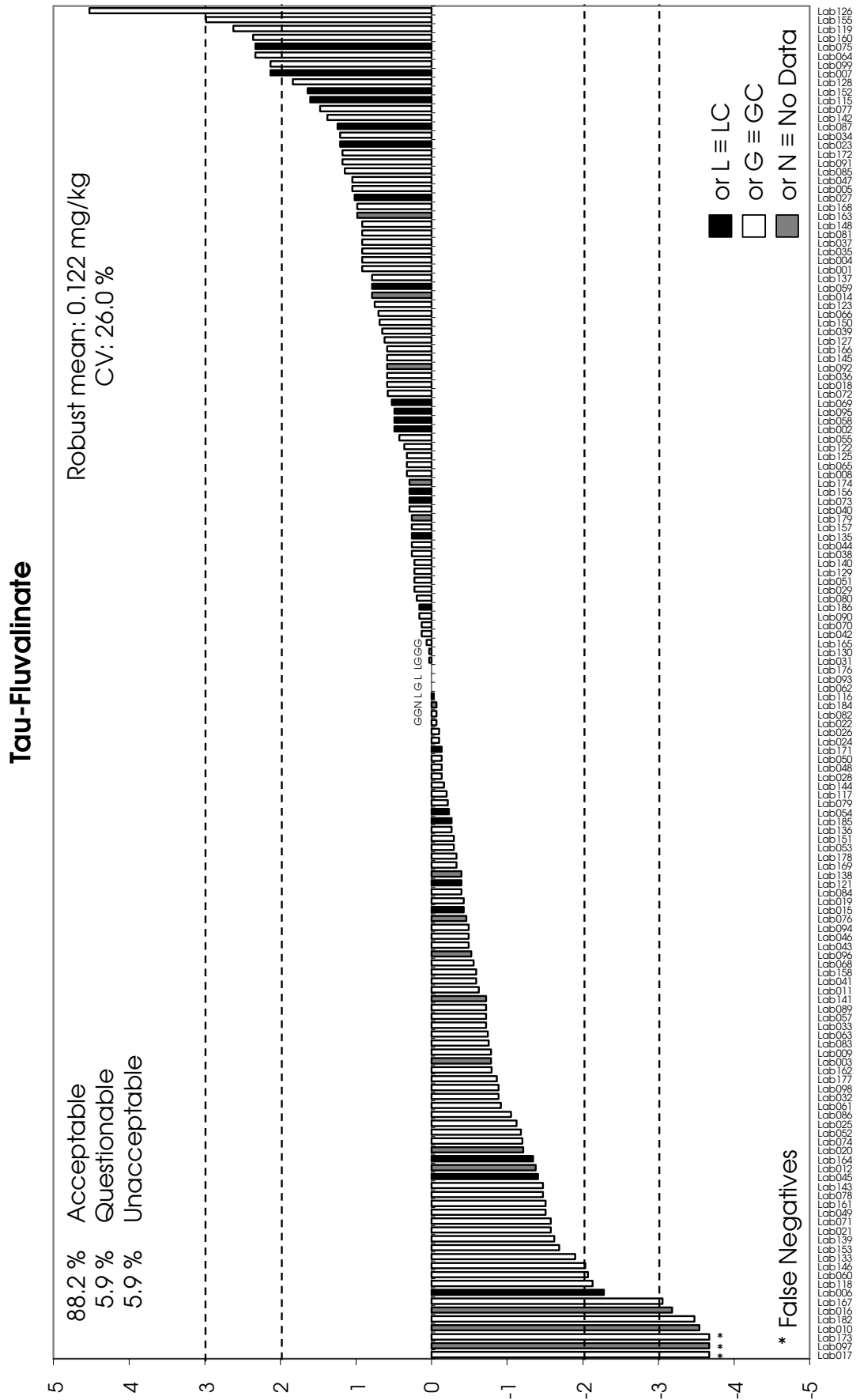
APPENDIX 4. Graphical representation of z scores for FFP RSD (25 %).



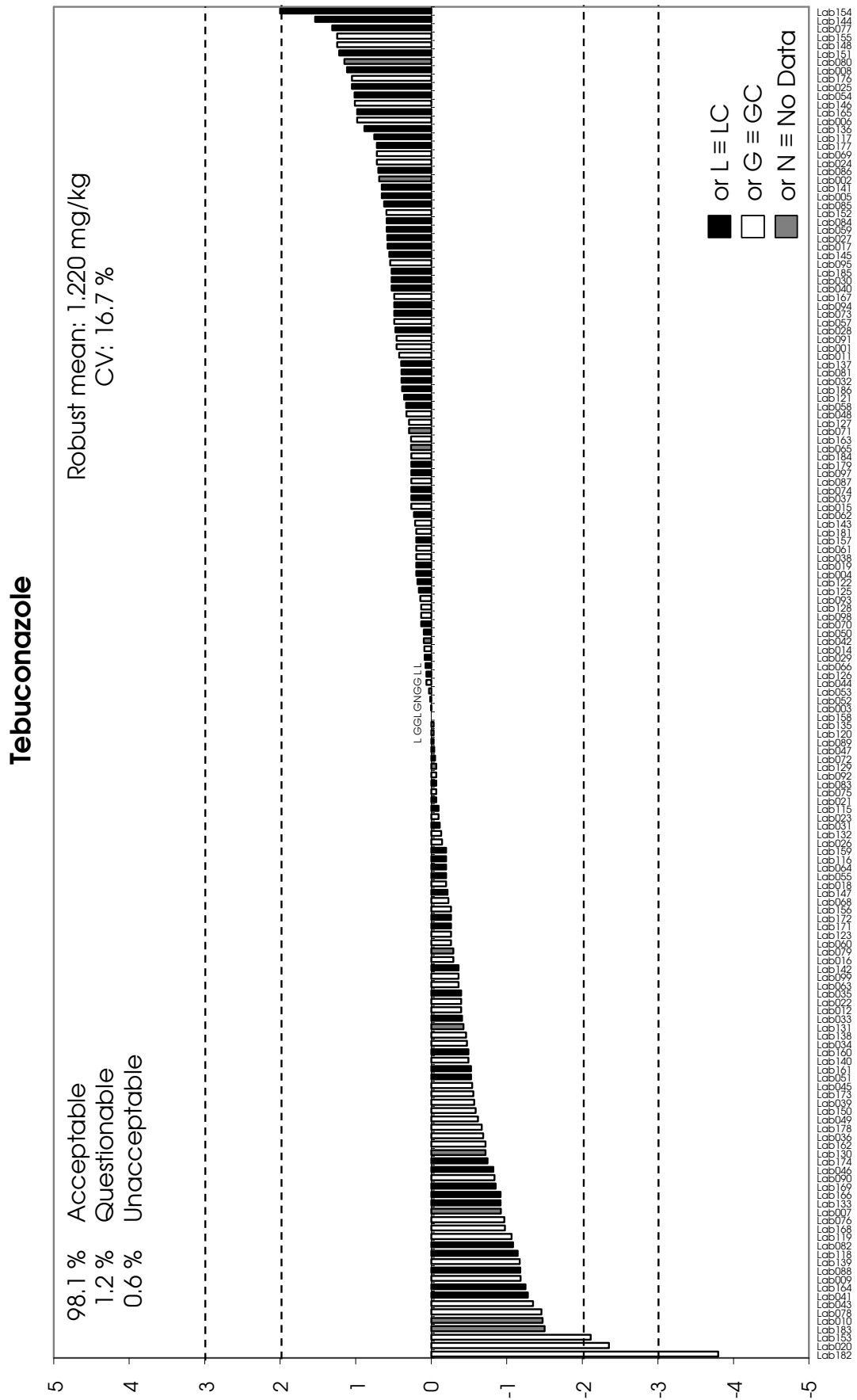


APPENDIX 4. Graphical representation of z scores for FFP RSD (25 %).





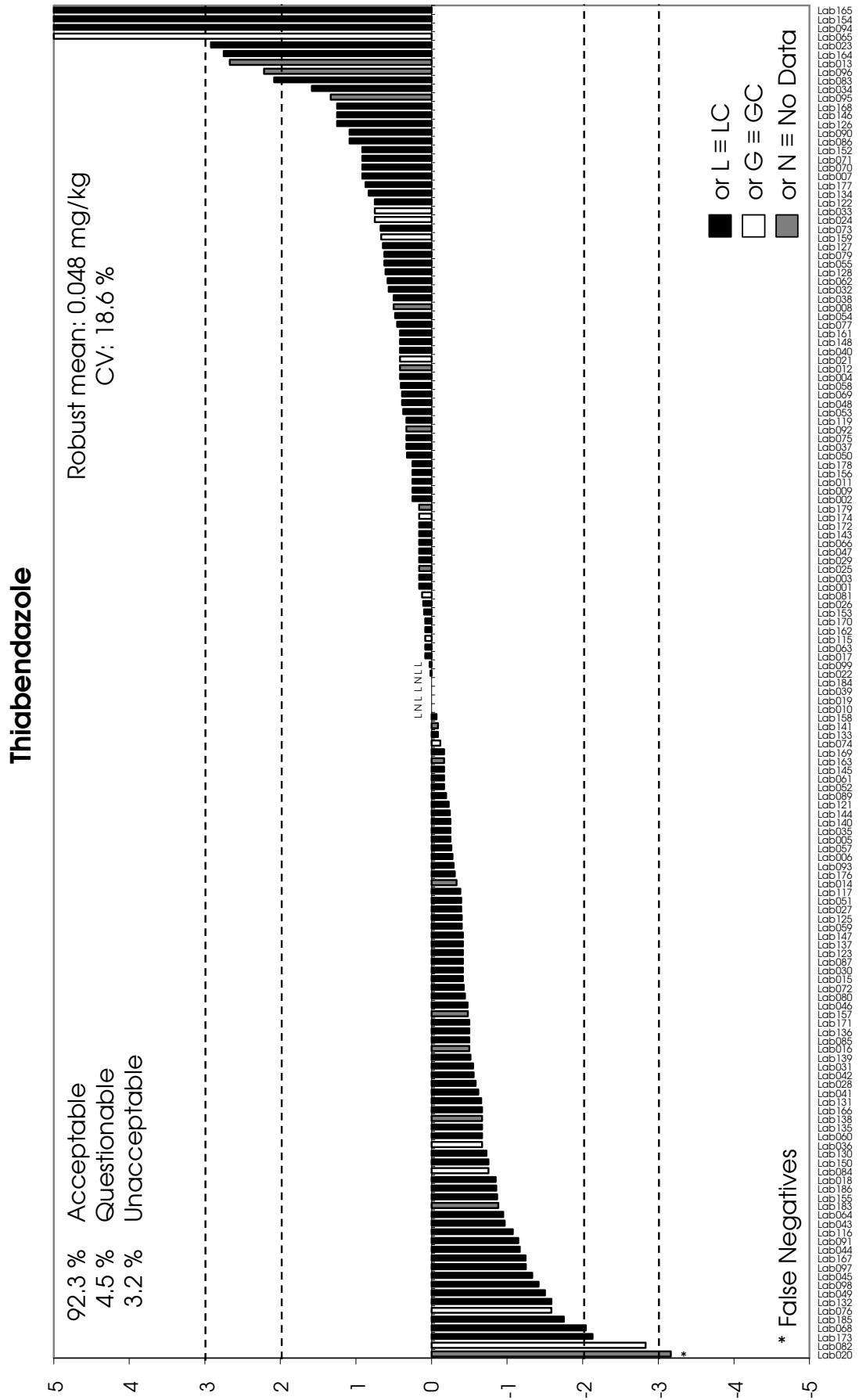
APPENDIX 4. Graphical representation of z scores for FFP RSD (25 %).

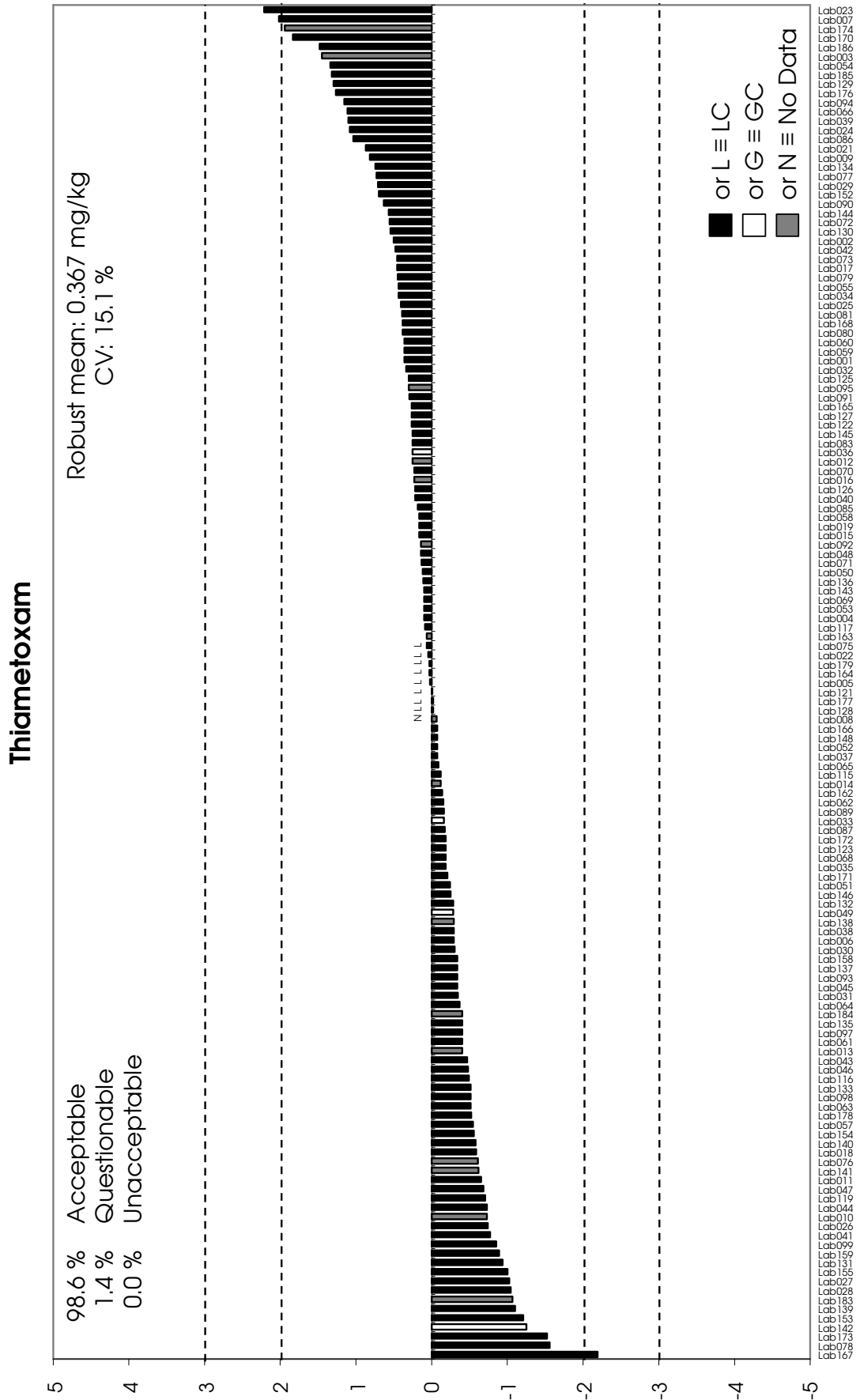




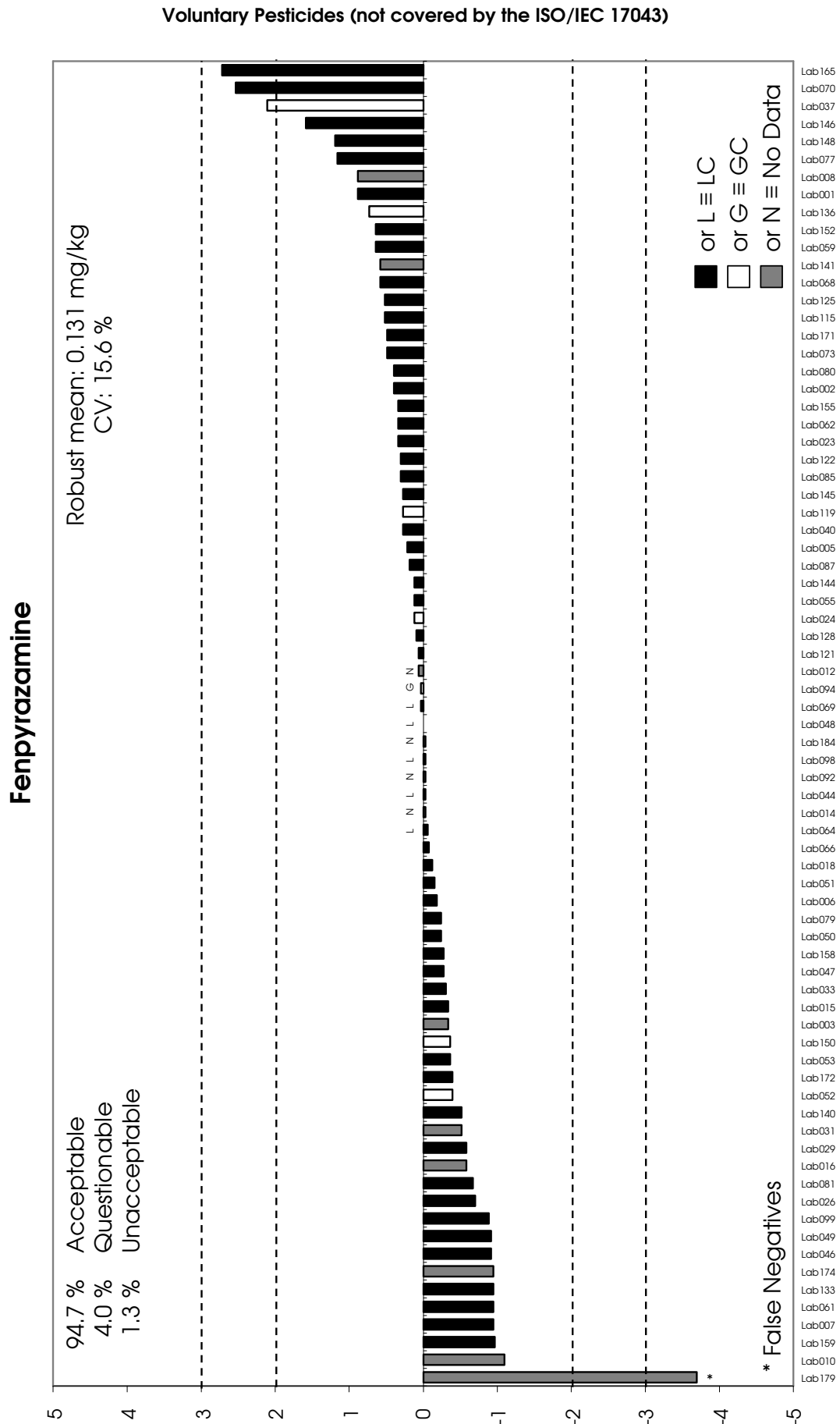


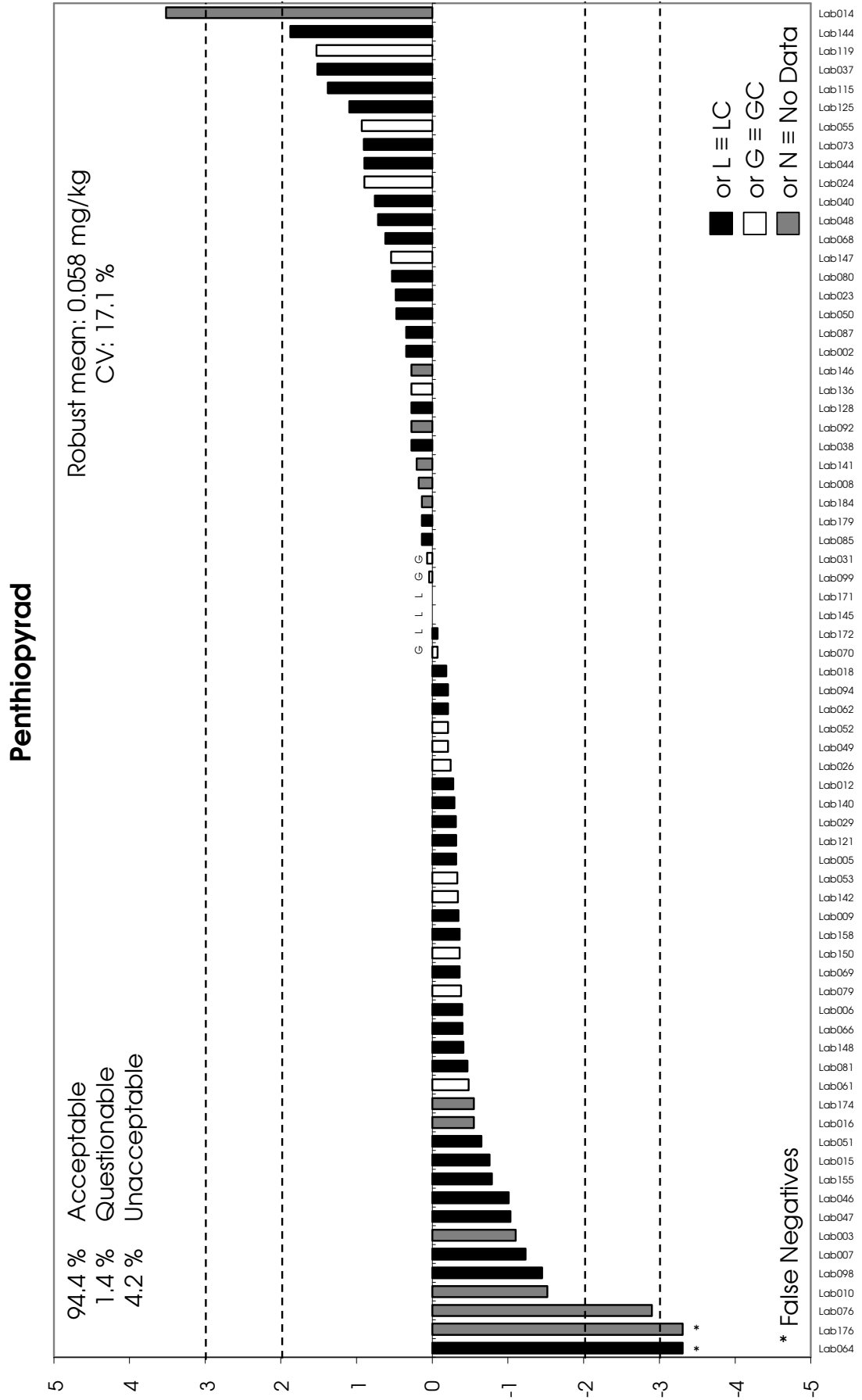
APPENDIX 4. Graphical representation of z scores for FFP RSD (25 %).





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





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

Lab Code	Boscalid	Buprofezin	Carbendazim	Clothianidin	Diazinon	Dimethoate	Ethofenprox	Fenpyroximate	Imazalil	Iprodione	Pyridaben	Spyromesifen	Tau-Fluvalinate	Tebuconazole	Tebufenpyrad	Thiabendazole	Thiamethoxam	No. of z scores	AZ <sup>2</sup>
Lab001	0,3	1,1	-0,1	0,4	0,4	0,2	0,4	1,6	-1,3	0,6	0,5	0,2	0,9	0,5	0,1	0,2	0,4	17	0,5
Lab002	0,5	0,7	-0,6	0,1	1,1	-0,2	0,3	0,7	0,9	0,5	-0,2	0,8	0,5	0,7	0,5	0,3	0,5	17	0,4
Lab003	0,6	0,4	1,0	0,5	0,4	0,6	0,9	1,5	0,8	-1,4	0,2	-0,6	-0,8	0,0	-0,1	0,2	1,4	17	0,6
Lab004	0,5	0,2	1,1	-0,5	0,1	0,1	0,4	0,2	1,4	1,1	0,7	0,5	0,9	0,2	1,0	0,4	0,1	17	0,5
Lab005	-0,2	0,4	-0,5	0,2	0,0	0,1	0,4	0,2	-0,4	0,7	0,2	0,4	1,0	0,7	0,3	-0,3	0,0	17	0,2
Lab006	0,3	0,3	-0,5	0,0	0,5	0,0	-0,6	-0,2	1,0	NA	-0,4	0,6	-2,3	1,0	0,4	-0,3	-0,3	16	0,6
Lab007	-0,9	-0,4	1,1	0,6	-0,7	0,3	0,4	0,7	1,0	-1,1	-0,1	-0,9	2,1	-0,9	-0,5	0,9	2,0	17	1,0
Lab008	-0,7	0,4	0,2	0,4	0,2	0,0	-0,1	0,4	-0,7	0,0	-0,2	-0,7	0,3	1,1	1,1	0,5	-0,1	17	0,3
Lab009	0,7	-0,3	-0,6	0,4	-0,4	0,2	-0,8	-0,7	0,1	0,6	-1,1	-0,5	-0,8	-1,2	-0,6	0,3	0,8	17	0,4
Lab010	-2,5	-1,7	-0,3	0,1	0,3	-1,0	-0,8	-1,5	-0,9	-3,2	-2,7	-2,8	-3,5	-1,5	-0,9	0,0	-0,7	17	3,2
Lab012	0,1	0,2	0,4	0,1	0,1	0,1	0,4	0,1	0,9	-0,6	-0,1	-0,1	-1,4	-0,4	-0,1	0,4	0,3	17	0,2
Lab014	0,1	0,5	-0,7	-1,2	-0,3	-0,7	2,3	1,2	-1,5	0,5	0,8	0,8	0,8	0,1	0,6	-0,3	-0,1	17	0,8
Lab015	0,4	-0,2	-0,3	0,2	-0,1	0,8	-0,2	0,0	-0,8	-1,1	0,4	-0,3	-0,4	0,3	-0,2	-0,4	0,2	17	0,2
Lab016	0,0	-0,4	-0,1	-0,8	0,1	0,2	0,6	-0,1	-0,4	0,1	-0,1	0,5	-3,2	-0,3	-0,3	-0,5	0,2	17	0,7
Lab017	1,5	0,4	-0,1	-0,4	0,2	1,1	-0,2	-0,4	0,8	1,2	-0,1	0,1	-3,7	0,6	0,5	0,1	0,5	17	1,2
Lab018	-0,6	-0,2	-0,6	-0,6	-0,8	-0,3	0,1	0,1	0,4	-1,0	0,0	0,2	0,6	-0,2	-0,3	-0,9	-0,6	17	0,3
Lab019	0,1	0,6	0,0	-0,4	0,7	0,5	-0,1	0,4	1,2	-3,3	0,7	0,6	-0,4	0,2	0,9	0,0	0,2	17	0,9
Lab023	0,4	0,0	2,1	2,9	-0,4	2,7	1,3	-0,1	1,3	0,0	0,3	2,0	1,2	-0,1	0,0	2,9	2,2	17	2,5
Lab024	0,2	0,5	0,4	-0,2	2,0	0,0	0,2	0,6	1,1	0,2	-0,3	0,8	-0,1	0,7	0,6	0,8	1,1	17	0,6
Lab025	0,4	0,4	0,8	-0,3	0,6	0,2	0,3	0,4	2,0	0,0	-1,2	0,9	-1,1	1,0	-0,6	0,2	0,4	17	0,6
Lab026	0,6	-0,2	-0,4	-0,7	0,2	0,1	-1,2	-0,8	-0,7	-0,2	-0,2	-0,7	-0,1	-0,1	-0,3	0,1	-0,7	17	0,3
Lab027	-0,4	-0,2	-0,4	-0,5	0,8	-0,7	0,3	1,3	-1,3	0,6	-0,5	-1,3	1,0	0,6	0,6	-0,4	-1,0	17	0,6
Lab028	0,3	-0,8	0,2	-0,5	-0,6	-0,9	-0,2	-1,1	0,3	0,9	-0,9	-1,1	-0,1	0,5	-0,5	-0,6	-1,0	17	0,5
Lab029	-0,4	0,5	0,9	0,7	0,7	0,4	0,1	2,3	-0,2	-0,2	0,3	0,1	0,2	0,1	0,2	0,2	0,7	17	0,5
Lab031	-0,4	-0,1	-0,7	0,5	-0,3	-0,3	0,1	0,0	0,3	-3,3	0,1	0,0	0,0	-0,1	-0,2	-0,6	-0,3	17	0,7
Lab032	-0,6	-0,7	1,6	0,5	-0,5	1,0	1,6	1,4	1,6	-0,2	0,5	-0,8	-0,9	0,4	1,3	0,6	0,3	17	0,9
Lab033	0,4	-0,2	-0,9	0,1	0,6	1,4	0,1	-0,2	-0,4	1,1	0,3	0,4	-0,7	-0,4	-0,6	0,8	-0,2	17	0,4
Lab034	-0,6	-0,4	-1,0	-0,3	-0,7	-0,1	0,5	3,9	-0,8	0,2	-0,1	-0,3	1,2	-0,5	-0,4	1,6	0,4	17	1,3
Lab037	1,5	0,6	1,2	-0,2	2,7	0,1	1,6	0,8	1,8	1,9	1,6	0,0	0,9	0,3	-0,1	0,3	-0,1	17	1,5
Lab038	0,4	0,3	-0,1	0,1	-0,4	0,1	-0,1	-1,0	1,1	-0,5	0,0	1,0	0,3	0,2	-0,3	0,5	-0,3	17	0,3
Lab040	0,8	1,0	1,1	0,0	0,8	0,3	1,0	0,3	2,3	0,7	0,7	0,7	0,3	0,5	0,7	0,4	0,2	17	0,7
Lab042	0,0	0,0	0,0	-0,2	0,0	0,1	-1,0	-0,5	0,8	-0,4	-0,4	0,1	0,1	0,1	0,6	-0,6	0,5	17	0,2
Lab043	-1,4	-0,9	-0,4	-0,5	-1,2	-0,7	-0,8	-0,9	0,0	-1,0	-0,8	-0,8	-0,5	-1,3	-1,1	-1,0	-0,5	17	0,8
Lab044	-0,5	0,8	-0,5	-0,2	0,2	-0,5	0,0	-0,6	-0,6	-3,3	1,0	3,7	0,3	0,1	0,6	-1,2	-0,7	17	1,8
Lab046	-0,1	-0,2	-0,1	0,1	-0,1	-0,1	0,0	0,0	-0,6	-0,4	-0,5	-0,9	-0,5	-0,8	-0,4	-0,5	-0,5	17	0,2
Lab047	-0,3	-0,2	-0,3	-1,0	0,1	-0,7	0,3	0,0	-0,5	-0,4	-0,1	0,0	1,0	0,0	-0,1	0,2	-0,7	17	0,2
Lab048	0,0	-0,1	0,0	-0,3	0,2	0,8	-0,8	-0,2	-0,1	-0,1	-0,3	0,2	-0,1	0,3	0,5	0,4	0,1	17	0,1
Lab049	-0,4	-0,7	-1,2	-1,3	-0,4	-1,4	-0,2	-0,5	0,7	-0,9	-0,7	-0,5	-1,5	-0,6	-0,6	-1,5	-0,3	17	0,8
Lab050	0,3	-0,1	-0,4	-0,2	0,2	0,6	-0,5	-0,4	0,1	0,0	-0,3	0,1	-0,1	0,1	0,5	0,3	0,1	17	0,1
Lab051	-0,7	-0,2	-0,9	-0,8	0,1	-0,4	-0,6	-0,4	-1,0	0,4	0,4	-0,7	0,2	-0,5	-0,5	-0,4	-0,2	17	0,3
Lab052	-0,3	-0,2	-0,9	0,1	0,0	-0,3	-1,8	-0,4	0,3	1,0	-0,4	-0,5	-1,2	0,0	-0,3	-0,2	-0,1	17	0,4
Lab053	0,1	-0,6	-0,1	0,1	-0,1	0,2	-0,6	-0,3	0,0	0,2	-0,3	0,3	-0,3	0,0	1,0	0,4	0,1	17	0,1
Lab055	0,4	0,3	0,0	0,1	0,7	0,2	0,1	0,1	0,9	2,8	0,3	0,2	0,4	-0,2	-0,2	0,6	0,4	17	0,6
Lab057	-0,3	0,4	0,2	0,0	-0,4	-0,1	0,7	0,4	0,3	-0,2	0,4	0,1	-0,7	0,5	0,4	-0,3	-0,5	17	0,2
Lab058	1,7	0,1	0,1	0,4	0,4	0,2	0,5	0,3	-0,1	0,7	0,0	0,3	0,5	0,3	0,4	0,4	0,2	17	0,3
Lab059	0,2	0,6	0,2	-0,1	0,4	0,0	0,8	0,6	-0,2	0,6	3,6	2,1	0,8	0,6	1,6	-0,4	0,4	17	1,4
Lab061	0,6	0,2	0,1	-0,3	0,6	-0,3	-0,5	-0,7	-0,1	0,1	-0,4	-0,1	-0,9	0,2	-0,1	-0,2	-0,4	17	0,2
Lab062	0,1	0,4	0,5	-0,3	1,5	0,6	0,3	0,6	0,4	0,4	0,7	0,0	0,0	0,2	0,5	0,6	-0,2	17	0,3
Lab064	1,0	-0,2	-1,0	-0,9	-0,1	-0,7	0,3	0,0	0,1	0,7	-0,3	0,9	2,3	-0,2	-0,2	-1,0	-0,4	17	0,7
Lab065	1,4	0,0	-0,2	-0,3	0,2	1,1	0,2	0,1	-0,6	0,7	0,2	0,0	0,3	0,3	0,1	5,0	-0,1	17	1,7
Lab066	-0,2	0,2	0,6	0,4	0,0	0,0	0,7	0,5	0,9	-0,1	-0,6	-0,7	0,7	0,1	0,2	0,2	1,1	17	0,3

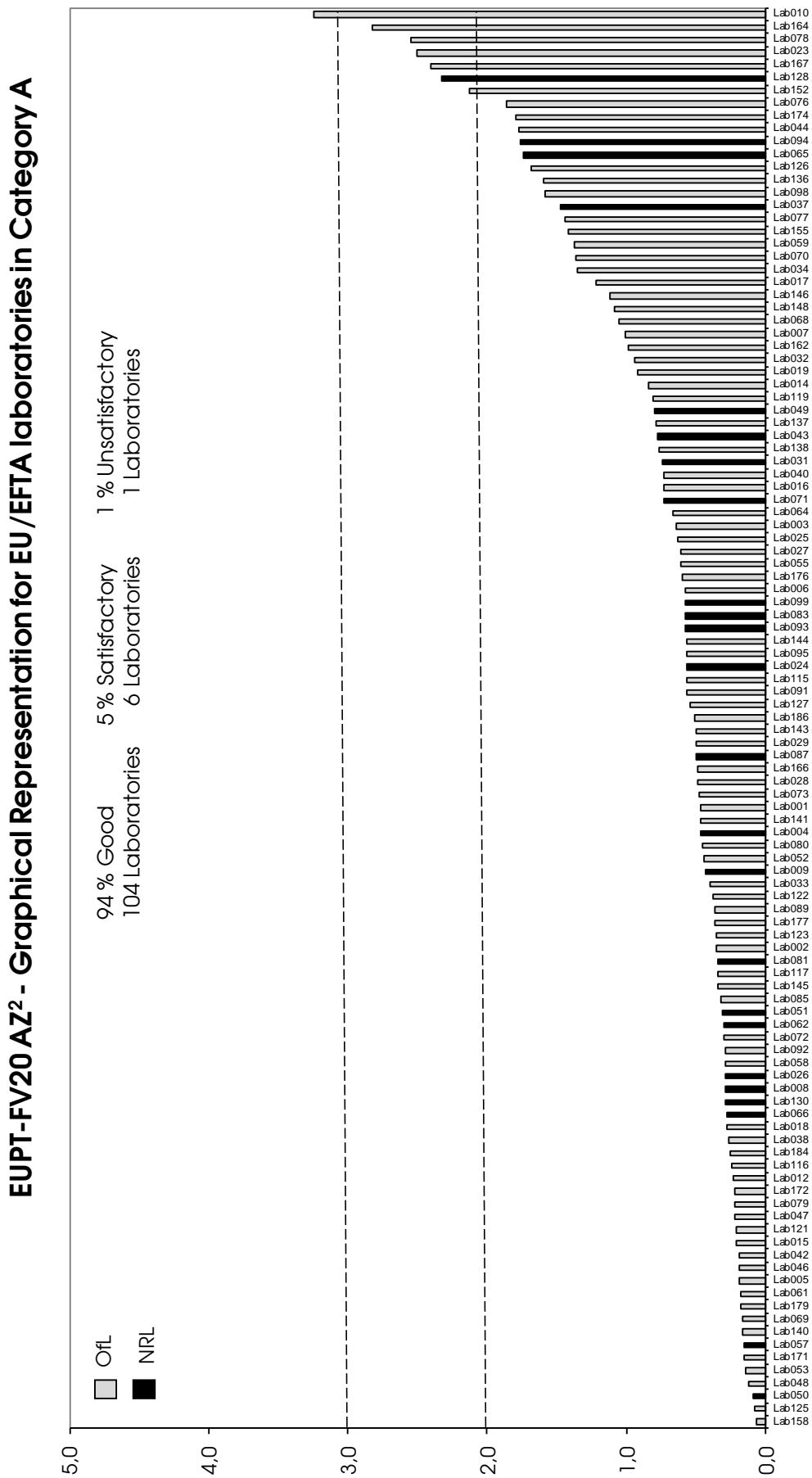
APPENDIX 5. Average of the Squared z scores (AZ<sup>2</sup>) for laboratories in Category A.

Lab Code	Boscalid	Buprofezin	Carbendazim	Clothianidin	Diazinon	Dimethoate	Ethofenprox	Fenpyroximate	Imazalil	Iprodione	Pyridaben	Spyromesifen	Tau-Fluvalinate	Tebuconazole	Tebufenpyrad	Thiabendazole	Thiamethoxam	No. of z scores	AZ <sup>2</sup>
Lab068	1,5	0,9	-0,8	0,6	0,5	0,0	0,5	0,2	0,3	-1,1	2,6	0,8	-0,6	-0,2	0,3	-2,0	-0,2	17	1,0
Lab069	0,1	-0,2	-0,1	0,3	0,0	0,1	0,3	1,0	-0,1	0,2	-0,5	0,6	0,5	0,7	0,2	0,4	0,1	17	0,2
Lab070	-0,2	0,1	1,9	0,5	0,2	0,6	1,2	1,3	3,7	-0,7	0,4	0,7	0,1	0,1	0,1	0,9	0,2	17	1,4
Lab071	0,3	-0,5	0,8	0,1	-1,1	0,8	-1,2	-0,1	0,2	-0,3	-1,5	-1,0	-1,6	0,3	-1,1	0,9	0,1	17	0,7
Lab072	-0,4	0,5	-0,2	-0,3	-0,4	0,9	0,1	0,5	-0,9	0,7	-0,2	0,1	0,6	0,0	-1,0	-0,4	0,6	17	0,3
Lab073	0,4	0,9	0,4	0,5	0,7	0,5	0,5	1,0	0,5	1,3	0,8	0,8	0,3	0,5	0,7	0,7	0,5	17	0,5
Lab076	-1,2	-1,1	-1,7	-0,8	-1,3	-0,4	-0,8	-0,5	-2,0	-1,0	0,0	-3,6	-0,5	-1,0	-0,7	-1,6	-0,6	17	1,9
Lab077	0,3	1,4	0,9	1,3	0,5	1,3	1,3	1,1	-1,5	0,8	1,5	1,8	1,5	1,3	1,4	0,5	0,7	17	1,4
Lab078	-1,6	-1,4	-3,0	-1,1	-1,5	-1,6	-1,6	-1,9	-1,4	-1,6	-1,1	-0,5	-1,5	-1,5	-1,4	NA	-1,6	16	2,5
Lab079	-0,4	-0,2	0,7	0,2	-0,2	1,0	0,0	0,6	0,7	-0,3	-0,3	-0,5	-0,2	-0,3	-0,3	0,6	0,4	17	0,2
Lab080	0,1	0,4	-0,7	-0,5	0,1	0,0	-0,6	0,7	-0,4	-0,9	0,9	1,5	0,2	1,1	0,6	-0,4	0,4	17	0,5
Lab081	0,6	0,2	0,1	0,3	-0,2	0,1	0,2	0,6	-0,8	1,6	0,4	-0,6	0,9	0,4	0,2	0,1	0,4	17	0,3
Lab083	0,7	0,0	1,3	0,4	-1,1	-0,2	-0,1	-0,1	0,9	-0,4	-0,1	0,0	-0,8	-0,1	-0,1	2,1	0,3	17	0,6
Lab085	0,2	0,4	-0,7	0,2	0,4	0,0	0,5	0,5	-0,5	-1,2	0,4	-0,3	1,1	0,6	0,5	-0,5	0,2	17	0,3
Lab087	0,1	0,5	-0,1	-0,2	1,0	-0,3	0,8	1,4	-0,3	0,3	1,0	1,0	1,2	0,3	0,7	-0,4	-0,2	17	0,5
Lab089	-0,4	0,6	0,5	0,5	1,5	0,4	0,3	0,4	1,1	-0,8	0,1	-0,4	-0,7	0,0	0,1	-0,2	-0,2	17	0,4
Lab091	-1,3	0,0	-0,8	-0,5	-0,3	-0,5	-0,3	0,1	-1,3	1,1	0,0	0,1	1,2	0,5	-0,6	-1,2	0,3	17	0,6
Lab092	-0,5	-0,4	1,1	0,2	-1,5	0,0	0,3	0,0	-0,4	-0,4	0,2	0,0	0,6	-0,1	0,3	0,3	0,1	17	0,3
Lab093	-0,6	-0,2	0,0	-1,5	-0,5	0,3	-0,8	-0,9	0,1	0,1	2,2	-0,4	0,0	0,1	-0,3	-0,3	-0,3	17	0,6
Lab094	-0,5	0,4	-0,7	0,0	0,5	0,0	0,7	0,5	0,6	0,6	0,5	0,4	-0,5	0,5	0,1	5,0	1,2	17	1,8
Lab095	-0,3	-0,7	0,4	1,4	-0,2	1,0	0,4	0,5	0,9	1,0	-0,7	0,8	0,5	0,5	0,5	1,3	0,3	17	0,6
Lab098	-1,2	-0,7	0,6	-1,3	-1,3	-1,2	-1,1	-1,1	-2,3	-2,4	-1,6	-0,7	-0,9	0,1	-0,5	-1,4	-0,5	17	1,6
Lab099	0,2	-1,0	0,6	-0,1	0,1	-0,1	0,3	-0,8	1,2	-0,8	0,2	-0,3	2,1	-0,4	-0,2	0,0	-0,9	17	0,6
Lab100	-0,8	-0,8	5,0	0,3	-0,4	-0,1	-0,8	-0,1	0,3	0,4	-0,8	-1,1	0,7	-0,9	-0,7	0,6	-0,1	17	1,9
Lab102	0,8	0,8	0,5	1,8	1,2	0,9	-0,6	-0,1	-1,4	3,8	-0,5	-0,9	-4,0	0,0	-0,2	0,2	1,6	17	2,6
Lab103	0,2	0,8	2,9	3,8	1,4	2,8	-0,2	-0,3	-1,3	1,3	-0,1	-1,1	-4,0	0,1	-0,3	-0,2	2,3	17	3,5
Lab104	0,6	-0,4	1,9	-0,2	0,1	-0,3	-0,4	0,6	-0,4	-3,3	0,0	-1,5	0,5	-0,4	-0,3	-0,1	0,0	17	1,1
Lab106	-1,0	-1,0	4,5	-0,7	0,9	-0,4	-0,7	-1,2	-1,5	-0,7	-0,5	-0,5	-0,9	-0,2	-0,3	2,5	-1,1	17	2,2
Lab115	-0,5	-0,2	-0,6	0,6	-0,5	0,1	0,0	0,8	-0,8	1,1	-1,3	-1,2	1,6	-0,1	-0,3	0,1	-0,1	17	0,6
Lab116	0,0	0,0	-1,4	0,1	0,1	-0,4	-0,4	0,1	0,2	0,0	0,2	-0,2	0,0	-0,2	-0,4	-1,1	-0,5	17	0,2
Lab117	-0,2	0,4	-0,2	0,3	-0,1	0,1	0,0	0,4	-1,7	-1,2	0,1	-0,2	-0,2	0,8	0,3	-0,4	0,1	17	0,3
Lab119	0,1	-0,7	-0,9	0,9	0,1	0,2	0,8	0,3	-0,3	1,1	-0,9	-0,3	2,6	-1,1	-0,1	0,3	-0,7	17	0,8
Lab121	0,8	0,2	0,4	0,4	0,7	0,1	0,2	0,3	-1,2	0,0	-0,1	-0,4	-0,4	0,4	-0,2	-0,2	0,0	17	0,2
Lab122	1,3	0,7	0,6	0,2	0,7	0,6	-0,1	0,6	1,1	-0,4	0,4	0,3	0,4	0,2	0,3	0,8	0,3	17	0,4
Lab123	-0,3	-0,9	0,9	-0,2	-0,3	0,0	-0,3	0,0	-1,2	0,3	-0,8	0,3	0,8	-0,3	-1,0	-0,4	-0,2	17	0,4
Lab125	0,2	0,3	-0,2	-0,1	0,3	0,4	0,1	-0,4	-0,4	0,4	0,0	-0,2	0,3	0,2	0,0	-0,4	0,3	17	0,1
Lab126	0,9	0,2	-0,1	0,5	0,7	0,4	0,8	0,2	1,7	0,4	0,6	0,0	4,5	0,1	0,9	1,3	0,2	17	1,7
Lab127	1,6	1,0	0,3	1,1	0,5	-0,1	0,7	0,7	-0,7	-1,1	0,7	0,3	0,6	0,3	0,1	0,6	0,3	17	0,5
Lab128	0,1	0,8	-0,4	-0,3	0,3	0,0	4,9	3,0	0,5	-0,4	1,0	0,7	1,8	0,1	0,2	0,6	0,0	17	2,3
Lab130	0,1	0,1	0,1	-0,4	-0,4	-0,6	-0,1	0,9	-0,3	-0,6	-0,2	-1,1	0,0	-0,7	0,6	-0,7	0,5	17	0,3
Lab136	0,3	0,4	4,8	0,4	0,4	0,2	-0,2	-0,2	0,5	0,5	0,5	0,9	-0,3	0,9	0,8	-0,5	0,1	17	1,6
Lab137	0,3	-0,5	-1,4	0,4	0,4	-1,1	0,1	-0,1	1,7	1,1	-0,8	1,9	0,8	0,4	-0,2	-0,4	-0,3	17	0,8
Lab138	0,1	0,2	-0,9	-0,6	-0,4	-0,7	-0,6	-0,9	-2,9	-0,4	-0,4	-0,5	-0,4	-0,5	0,2	-0,7	-0,3	17	0,8
Lab140	-0,4	-0,1	0,2	-0,5	0,0	-0,1	0,0	0,1	1,1	-0,5	0,2	-0,1	0,2	-0,5	-0,1	-0,3	-0,6	17	0,2
Lab141	0,4	1,2	0,2	-0,6	-0,1	-0,3	0,5	0,8	0,1	0,2	1,2	1,2	-0,7	0,7	0,8	-0,1	-0,6	17	0,5
Lab143	-0,9	0,7	1,2	-0,4	-0,2	0,6	-1,0	0,0	-0,7	-0,4	-0,5	-0,9	-1,5	0,2	0,4	0,2	0,1	17	0,5
Lab144	0,7	1,2	0,2	0,7	0,7	0,1	0,8	0,8	0,9	0,4	0,7	0,5	-0,2	1,5	1,0	-0,2	0,6	17	0,6
Lab145	-0,4	0,2	0,2	0,0	0,6	0,1	0,3	-1,0	0,3	-1,3	-0,1	-0,2	0,6	0,6	1,2	-0,2	0,3	17	0,3
Lab146	-0,2	1,4	0,6	0,8	1,0	0,4	1,1	1,7	0,9	-1,0	0,7	0,9	-2,0	1,0	0,9	1,3	-0,3	17	1,1
Lab148	1,0	0,9	0,1	1,6	1,8	1,8	0,8	0,5	0,9	1,2	0,2	1,2	0,9	1,2	0,7	0,4	-0,1	17	1,1
Lab152	0,8	0,9	1,9	0,1	0,8	0,6	-0,2	-2,4	0,2	4,4	-0,1	-0,5	1,6	0,6	0,6	0,9	0,7	17	2,1
Lab155	1,2	-0,4	-1,0	-0,5	0,3	-0,2	0,6	0,4	1,6	2,1	0,5	0,4	3,0	1,2	1,0	-0,9	-1,0	17	1,4

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

Lab Code	Boscalid	Buprofezin	Carbendazim	Clothianidin	Diazinon	Dimethoate	Ethofenprox	Fenpyroximate	Imazalil	Iprodione	Pyridaben	Spyromesifen	Tau-Fluvalinate	Tebuconazole	Tebufenpyrad	Thiabendazole	Thiamethoxam	No. of z scores	AZ <sup>2</sup>
Lab158	-0,1	0,3	0,4	-0,2	0,2	0,0	0,0	-0,1	0,2	-0,1	0,4	0,3	-0,6	0,0	0,0	-0,1	-0,3	17	0,1
Lab162	0,6	-1,5	-1,6	0,4	-0,7	0,8	-1,7	0,3	-0,1	-0,1	0,2	-2,1	-0,8	-0,7	-1,3	0,1	-0,1	17	1,0
Lab164	-1,1	-1,5	-1,1	-0,4	-1,0	4,1	-0,9	-0,8	-0,7	-1,3	-1,2	-2,7	-1,3	-1,2	-1,4	2,8	0,0	17	2,8
Lab166	-1,0	-0,6	0,1	-0,6	-0,4	0,1	-0,1	-1,3	-0,8	-1,1	-0,2	-0,6	0,6	-0,9	-0,9	-0,7	-0,1	17	0,5
Lab167	1,1	1,9	-2,5	-2,5	0,8	-1,4	1,4	0,5	-0,5	0,3	0,4	1,3	-3,1	0,5	0,7	-1,2	-2,2	17	2,4
Lab171	-0,2	-0,1	-0,2	0,5	-0,1	0,0	0,3	0,1	-1,3	0,0	0,2	-0,2	-0,1	-0,3	-0,1	-0,5	-0,2	17	0,2
Lab172	-0,2	0,0	-0,6	-0,1	0,3	-0,3	-0,2	-0,5	0,5	-0,4	-0,2	-0,7	1,2	-0,3	0,6	0,2	-0,2	17	0,2
Lab174	1,0	-1,3	-1,9	1,8	-1,4	2,4	-0,6	-1,0	-0,8	1,0	-0,8	-2,3	0,3	-0,7	-0,4	0,2	1,9	17	1,8
Lab176	0,0	1,4	-0,5	-0,1	0,6	0,2	-0,4	-1,2	-0,1	-0,1	1,5	-0,5	0,0	1,0	0,8	-0,3	1,3	17	0,6
Lab177	-0,2	0,1	0,7	-0,2	0,1	0,6	-0,7	-0,3	1,4	0,0	0,1	NA	-0,9	0,7	0,4	0,9	0,0	16	0,4
Lab179	-0,5	0,1	1,0	0,2	-0,5	-0,3	0,0	-0,5	0,2	-0,8	0,3	0,0	0,3	0,3	-0,2	0,2	0,0	17	0,2
Lab184	0,3	-0,6	-0,4	0,6	0,6	-0,2	-1,1	-0,1	-0,7	0,4	-0,3	-0,9	-0,1	0,3	-0,1	0,0	-0,4	17	0,3
Lab186	0,0	-0,4	-0,4	-0,2	-0,2	-0,6	-0,5	1,6	-1,3	-0,1	0,1	0,1	0,2	0,4	0,2	-0,9	1,5	17	0,5





## 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>8</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>9</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 (EC) 625/2017<sup>10</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>11</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>12</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.

---

<sup>8</sup> DG-SANTE = European Commission, Health and Food Safety Directorate-General

<sup>9</sup> For more information about the EURL/NRL/OfL-Network please refer to the EURL-Web-portal under:

<http://www.eurl-pesticides.eu>

<sup>10</sup> Regulation (EU) 2017/625 of the European Parliament and of the Council on official controls and other official activities performed to ensure the application of food and feed law, rules on animal health and welfare, plant health and plant protection products. Published at OJ of the EU L95 of 07.04.2017

<sup>11</sup> European Commission Proficiency Tests for Pesticide Residues in Fruits and Vegetables, Trends in Analytical Chemistry, 2010, 29 (1), 70 – 83.

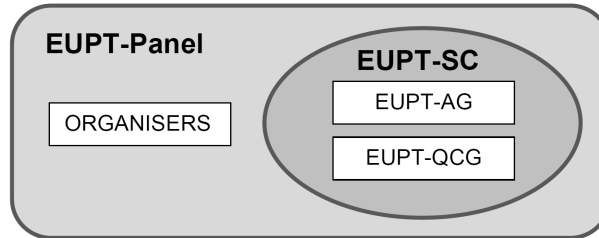
<sup>12</sup> Link to the List of current members of the EUPT Scientific Committee: <http://www.eurl-pesticides.eu/library/docs/allcrl/EUPT-SC.pdf>

## 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>13</sup> (for all OfLs analysing for pesticide residues within the framework of official controls<sup>14</sup> of food or feed)
- Art. 101 (1)(a) of Reg. (EC) 625/2017 (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 obliged 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.

Based on Reg. (EC) 625/2017, OfLs not paying the EUPT sample delivery fee will be initially warned that their participation in subsequent EUPTs could be denied. In case of a repetitive non-payment, the EUPT organisers will inform the competent authority to take action.

### **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.

<sup>13</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>14</sup> Official controls in the sense of Reg. (EC) 625/2017, 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.**

As laid down in Regulation 625/2017, NRLs are responsible for supporting and improving their own OfL-Network. On request from the NRLs, the EURLs will provide them with the PT-codes of the participating OfLs belonging to their OfL-Network. This will allow NRLs to follow the participation and performance of the laboratories within their network.

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

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

The official language used in all EUPTs is English.

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

At least 3 months before the 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  $|y_i - y| \leq 0.3 \times \sigma_{opt}$ , 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  $\sigma_{opt}$  the standard deviation used for proficiency assessment (typically 25% of the assigned value).

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

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

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 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 MRRRLs. 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>15</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 MRRRL 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 MRRRL will not be considered as false positives, even though these results should not have been reported.

---

<sup>15</sup> Document N° SANTE/11813/2017; Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed

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

### – **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 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.

### – **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>16</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:

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

---

<sup>16</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).

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

The percentage FFP-RSD is set at 25 % based on experience from results of previous EUPTs<sup>17</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.

### – z scores

This parameter is calculated using the following formula:

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

where  $x_i$  is the value reported by the laboratory,  $x_{pt}$  is the assigned value, and  $FFP - \sigma_{pt}$  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>18</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	N - 1
6	5.4	5	
7	6.3	6	
8	7.2	7	
9	8.1	8	
10	9.0	9	
11	9.9	10	
12	10.8	11	
13	11.7	12	N - 2
14	12.6	13	
15	13.5	13	
16	14.4	14	

<sup>17</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.

<sup>18</sup> ISO/IEC 17043:2010. Conformity assessment – General requirements for proficiency testing

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

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
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	
24	21.6	22	
25	22.5	22	
26	23.4	23	
			N - 3

### – 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>19,20</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.

<sup>19</sup> Formerly named "Sum of squared z scores (SZ<sup>2</sup>)"

<sup>20</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.



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

### **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.**

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 AZ<sup>2</sup> 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>21</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.

---

<sup>21</sup> Article 101 of the Regulation 625/2017



## **EUPF-FV20 SPECIFIC PROTOCOL**

### **European Union Proficiency Test for Pesticide Residues in Fruits and Vegetables (2018)**

#### **Introduction**

This protocol is complementary to the General Protocol of EU Proficiency Tests (EUPF) for Pesticide Residues in Food and Feed (8th 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 (23<sup>rd</sup> 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 (EUPFs) 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 pesticide residues in **green beans with pods**. The green beans were grown in a greenhouse in Almería. 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 using three bottles more and reproducing the sample shipment to see if there is any degradation of any of the pesticides present in the test item.

Together with the test item, a blank sample of green beans homogenate will be sent to the participants. The blank material should not contain any of the pesticides in the Target Pesticides List, at or above, the specified MRLs. If a pesticide is identified by the Organisers in the blank material and there is no possibility to find a different blank, that pesticide could be removed from the Target Pesticides List and it will be communicated to all the participants by e-mail.

#### **Amount of Test Item**

Participants will receive:

- Approximately 200 g of green beans test item treated with pesticides.
- Approximately 200 g of 'blank' green beans 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 5th March 2018. 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.

The Organisers will not take the responsibility for a parcel if it is retained at customs.

#### **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). **IMPORTANT: Due to the weather conditions, the green beans used for the test item contained less water amount than the green beans used as blank material as they were harvested three weeks later and the temperature was high. For that reason, it is necessary to add 0.5 ml of water per gram of test item before the extraction (e.g. if 10 g are used for the extraction, 5 ml of water should be added to the test item) Water should only be added to the test item material, NOT TO THE BLANK MATERIAL.**

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

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.

### Subcontracting

All analytical determinations concerning the test item treatment analysis will be performed in a laboratory which is ISO 17025 accredited, and which has been previously evaluated by the Organisers.

### 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 (AZ2) 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.

### Steps to follow

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

1. To participate, each laboratory must complete the Application Form on-line, whose link is 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 the Minimum Required Reporting Limits (MRRLs) are given. The MRRLs do not always correspond with the EU MRLs set for green beans.
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**.
3. The sample delivery will be **250 euros** for EU/EFTA participants and **350 euros** for participants 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 22<sup>nd</sup> February - 5<sup>th</sup> March 2018, prior to test item shipment. The aim is that laboratories provide information regarding their scope of analysis before receipt of the test item. 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 the **Test Item Receipt Form** to inform the Organiser that they have accepted the test item. This Form has a deadline: 9<sup>th</sup> March 2018, 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 the test item receipt form is not filled in, the Organiser will consider that the participant has accepted the test item.
7. Once the laboratory has analysed the test item and is ready to submit their data, they must enter their results at various steps by accessing the restricted area in the EURL-FV web site. The participant laboratories must respect the deadline for submitting their results - 26<sup>th</sup> March 2018- using the **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 MRRL and the participant's own RL will be used to help identify and calculate z scores for false negative results. This form

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

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/11813/2017. 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 will be recorded as 'ND'. If a pesticide was not sought, it will be recorded as 'NA' (Not Analysed). **If a laboratory fills in the scope form, but it does not report results neither fills in the methods form, all the pesticides will be considered as NA.**

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 26th March 2018. 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 green beans 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 2<sup>nd</sup>-6<sup>th</sup> April 2018. 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. When necessary, the Organiser will ask the participants by e-mail specific details about the methods of analysis used. 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 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.

### Calendar

ACTIVITY	DATE
Registration period in EURL DataPool	20 <sup>th</sup> December-5 <sup>th</sup> February
Specific Protocol published on the Web site.	19 <sup>th</sup> February 2018 at the latest
Sample distribution.	5 <sup>th</sup> March 2018
Deadline for receiving sample acceptance	9 <sup>th</sup> March 2018
Deadline for receiving results	26 <sup>th</sup> March 2018
Filling in additional information, if necessary.	2 <sup>nd</sup> -6 <sup>th</sup> April 2018
Preliminary Results: only results, no statistical treatment.	April 2018
Preliminary Report (containing preliminary assigned values and z scores)	May 2018
Final Report distributed to the Laboratories.	August 2018

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

### 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**

### 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  
Fax No.: +34 950015008

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

Dr. Amadeo R. Fernández-Alba	EURL-FV	amadeo@ual.es	+34 950015034
Dr. Carmen Ferrer Amate	EURL-FV	cferrer@ual.es	+34 950014102
Dr. Ana Lozano Fernández	EURL-FV	analozano@ual.es	+34 950015645
Mr. Octavio Malato Rodríguez	EURL-FV	omalato@ual.es	+34 950214423

### 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-20**

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

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

Pesticide	MRRL (mg/Kg)
Etofenprox	0.01
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>Flonicamid</b>	<b>0.01</b>
Flubendiamide	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
<b>Formetanate</b>	<b>0.01</b>
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
Isfenphos-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

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

Pesticide	MRRL (mg/Kg)
Myclobutanyl	0.01
Omethoate	0.003
Orthophenylphenol	0.01
Oxadixyl	0.01
Oxamyl	0.01
Oxydemeton-methyl	0.006
Paclobutrazole	0.01
Paraoxon-methyl	0.01
Parathion-ethyl	0.01
Parathion-methyl	0.01
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
<b>Prosulfocarb</b>	<b>0.01</b>
Prothioconazole (Prothioconazole-desthio)	0.01
Prothiofos	0.01
Pyraclostrobin	0.01
Pyridaben	0.01
Pyrimethanil	0.01
Pyriproxyfen	0.01
Quinoxifen	0.01
Spinosad (sum of spinosyn A and spinosyn D, expr. as spinosad)	0.01
Spirodiclofen	0.01
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
Terbuthylazine	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



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

Pesticide	MRRL (mg/Kg)
Vinclozolin (only parent compound)	0.01
Zoxamide	0.01

**In bold: new pesticides this year**

This list is based on Commission Implementing Regulation (EU) 2017/660 of 6 April 2017  
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-20**

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
Chlorfluazurone	0.01
Clomazone	0.01
Cyazofamid	0.01
Cyflufenamid	0.01
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
Metrafenone	0.01
Novaluron	0.01
Penflufen	0.01
Penthiopyrad	0.01
Pyrethrins	0.01
Quintozene	0.01
Pentachloro-aniline	0.01
Proquinazid	0.01
Pyridalil	0.01
Pyriofenone	0.01
Rotenone	0.01
Spinetoram	0.01
Spirotetramat	0.01
Spirotetramat metabolite BY108330-enol	0.01
Spirotetramat metabolite BY108330-ketohydroxy	0.01
Spirotetramat metabolite BY108330-monohydroxy	0.01
Spirotetramat metabolite BY108330 enol-glucoside	0.01
Sulfoxaflor	0.01
Tetramethrin	0.01
Tricyclazole	0.01

**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-20.**

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
Austria	Department for Pesticide and Food Analytics (PLMA)	Innsbruck	Yes
Belgium	Belgian Scientific Institute of Public Health	Brussels	Yes
Belgium	LOVAP NV - Pesticide Residues Lab	Geel	Yes
Belgium	Pesticide Lab (Cluj-Napoca)	Cluj-Napoca	Yes
France	Phytocontrol (Nimes) - Pesticide Lab	Nimes	Yes
Germany	LUFÄ Kiel - Pesticide Lab	Kiel	Yes
The Netherlands	Groen Agro Control - Pesticide Lab	Delfgauw	Yes
The Netherlands	Eurofins Lab Zeeuws-Vlaanderen B.V. - Pesticiden	Graauw	Yes
Belgium	PRIMORIS (Phytolab) - Belgium	Gent - Zwijnaarde	Yes
Bulgaria	Primoris Bulgaria AD - Pesticide Lab	Plovdiv	Yes
Bulgaria	CLCTC - Sofia I Pesticide Lab	Sofia	Yes
Bulgaria	Euro Lab Kapitan Andreevo (border check point)	Svilengrad	Yes
China	Beijing Uni-Star Inspection - Pesticide Lab	Beijing	Yes
China	Agro-product Safety Research Center - Guofang Pang	Beijing	Yes
China	SCDC - Pesticide Lab (Shanghai)	Shanghai	Yes
China	BGI-Shenzhen - Pesticide Lab	Shenzhen	Yes
Colombia	Universidad Nacional - LARP	Bogotá D.C.	Yes
Colombia	Agropecuaria Colombian Inst. - Pesticide Lab	Mosquera, Cundinamarca	Yes
Costa Rica	Pesticide Lab (San José)	San José	Yes
Croatia	Croatiakontrola - Pesticide Lab	Zagreb	Yes
Croatia	Center for Food Control - PBF, Zagreb	Zagreb	Yes
Croatia	Dr. Andrija Štampar - Pesticide Lab	Zagreb	Yes
Croatia	Bioinstitut d.o.o. - Pesticide Lab	Cakovec	Yes
Croatia	INSPECTO d.o.o. Laboratorij (Osijek)	Osijek	Yes
Croatia	Sample Control - Pesticide Lab	Zagreb	Yes
Croatia	Primorsko-goranska County, Rijeka - Pesticide Lab	Kotar County, Rijeka	Yes
Croatia	Croatian National Institute of Public Health- HZJZ	Zagreb	Yes
Cyprus	Pesticide Lab (Nicosia)	Nicosia	Yes
Czech Republic	VSCHT (Praha) - Pesticide Lab	Praha	Yes

**ANNEX 2. List of laboratories that agreed to participate in EUPT-FV20**

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
Czech Republic	CAFIA - Pesticide Lab (Praha)	Praha	Yes
Czech Republic	Pesticide Lab (Brno)	Brno	Yes
Denmark	Laboratoriet Ringsted - Pesticide Lab	Ringsted	Yes
Denmark	DTU, National Food Institute	Lyngby	Yes
Estonia	Pesticide Lab (Saku)	Saku	Yes
Estonia	Pesticide Lab (Tartu)	Tartu	Yes
Finland	Finnish Customs Laboratory	Espoo	Yes
Finland	MetropoliLab - Pesticide Lab	Helsinki	Yes
France	GIRPA-FREDON Pays de la Loire - Pesticide Lab	Beaucouzé	Yes
France	CAMP Méditerranée (Perpignan)	PERPIGNAN	Yes
France	SCL - Massy Cedex	Massy Cedex	Yes
France	INOVALYS Le Mans - Pesticide Lab	Le Mans	Yes
France	CAPINOV (Landerneau)	Landerneau	Yes
France	CERECO (GARONS)	GARONS	Yes
France	SCL (Montpellier)	Montpellier	Yes
Germany	CVUA Stuttgart - Pesticide Lab	Fellbach	Yes
Germany	IHU - Pesticide Lab (Hamburg)	Hamburg	Yes
Germany	LALLF - Pesticide Lab (Rostock)	Rostock	Yes
Germany	Pesticide Lab (Frankfurt (Oder))	Frankfurt (Oder)	Yes
Germany	LAVES - Pesticide Lab (Oldenburg)	Oldenburg	Yes
Germany	Pesticide Lab (Neumünster)	Neumünster	Yes
Germany	Institut für Lebensmittelchemie Speyer	Speyer	Yes
Germany	CVUA RRW - Pesticide Lab (Krefeld)	Krefeld	Yes
Germany	LUA Saarland - Pesticide Lab	Saarbrücken	Yes
Germany	BVL-NRL for Pesticides	Berlin	Yes
Germany	LUA Sachsen - Pesticide Lab	Dresden	Yes
Germany	Pesticide Lab (Tegernheim)	Tegernheim	Yes
Germany	Pesticide Lab (Kassel)	Kassel	Yes
Germany	ZInstSanBw Kiel - Pesticide Lab	Kronshagen	Yes
Germany	LTZ Augustenberg - Organic Chemistry	Karlsruhe	Yes

**ANNEX 2. List of laboratories that agreed to participate in EUPT-FV-20.**

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
Germany	Pesticide Lab (Bremen)	Bremen	Yes
Germany	KWALIS Fulda - Pesticide Lab	Dipperz	Yes
Germany	Pesticide Lab (Halle/Saale)	Halle/Saale	Yes
Germany	Pesticide Lab (Bad Langensalza)	Bad Langensalza	Yes
Germany	CVUA-MEL - Pesticide Lab (Münster)	Münster	Yes
Germany	Pesticide Lab (Erlangen)	Erlangen	Yes
Germany	Labor Mang - Pesticide Lab	Frankfurt	Yes
Germany	ILAU GmbH - Pesticide Lab	Anzing	Yes
Germany	Pesticide Lab (Garching-Hochbrück)	Garching-Hochbrück	Yes
Germany	Amt für Verbraucherschutz - PSM (Düsseldorf)	Düsseldorf	Yes
Germany	Eurofins SOFIA GmbH - Pesticide Lab	Berlin	Yes
Greece	GCSL - Pesticide Lab (Athens)	Athens	Yes
Greece	BPI - Pesticide Lab (Kifissia)	Kifissia	Yes
Greece	Agrolab S.A., Pesticide Lab - Thessaloniki	Thessaloniki - GR 570 22 , Sindos	Yes
Greece	Pesticide Lab (Thessaloniki)	Thessaloniki	Yes
Hungary	NFCSO Pesticide Lab (Hódmezovásárhely)	Hódmezovásárhely	Yes
Hungary	NFCSO Pesticide Lab (Szolnok)	Szolnok	Yes
Hungary	NFCSO - Pesticide Lab (Velence)	Velence	Yes
Hungary	NFCSO Pesticide Lab (Miskolc)	Miskolc	Yes
Iceland	Mafís - Pesticide Lab	Reykjavík	Yes
Ireland	Pesticide Lab (Co. Kildare)	Co. Kildare	Yes
Italy	ARPAL Sez. di La Spezia	La Spezia	Yes
Italy	ARPA VDA - Pesticide Lab	Saint Christophe	Yes
Italy	IZS LT - Pesticide Lab	Roma	Yes
Italy	ARPA-ER - Pesticide Lab	Ferrara	Yes
Italy	APPA-Puglia I Polo Alimenti Bari - Pesticide Lab	Bari	Yes
Italy	IZS PLV (sez. Cuneo) - Pesticide Lab	CUNEO	Yes
Italy	IZS Sardegna - Pesticide Lab	Sassari	No
Italy	ATS Milano - Laboratorio di Prevenzione	Milano	Yes
Italy	APPA Bolzano - Pesticide Lab	Bolzano	Yes

**ANNEX 2. List of laboratories that agreed to participate in EUPT-FV20**

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
Italy	IZSAM - Pesticide Lab	Teramo	Yes
Italy	IZS LT (sezione Firenze) - Pesticide Lab	San Martino alla Palma Scandicci (FI)	Yes
Italy	ISS - Pesticide Lab	Roma	Yes
Italy	IZS PB - Pesticide Lab	Foggia	Yes
Italy	IZSUM - Pesticide Lab	Perugia	Yes
Italy	ARPA Veneto (Laboratorio di Verona)	Verona	Yes
Italy	IZS Sicilia - Pesticide Lab	Palermo	Yes
Italy	ARPA FVG - Pesticide Lab (Udine)	Udine	Yes
Italy	IZSLER - Pesticide Lab	Brescia	Yes
Italy	ASF - Pesticide Lab	Firenze	Yes
Italy	APPA-SL Trento - Pesticide Lab	Trento	Yes
Italy	ARPAM - Pesticide Lab	Macerata	Yes
Italy	ARPA Lazio (sez. Latina) - Pesticide Lab	Latina	Yes
Italy	Pesticide Lab (Entratico)	Entratico	Yes
Italy	ARPAC - Pesticide Lab	Napoli	Yes
Italy	Laboratorio di Prevenzione (Bergamo)	Bergamo	Yes
Kenya	KEPHIS - Pesticide Lab (Nairobi)	Nairobi	Yes
Latvia	BIOR (Riga) - Pesticide Lab	Riga	Yes
Germany	GALAB Laboratories GmbH - Hamburg	Hamburg	Yes
Lithuania	NMVRVI - Pesticide Lab (Vilnius)	Vilnius	Yes
Luxembourg	LNS Food lab	Dudelange	Yes
Germany	Eurofins (Hamburg) - Pesticide Lab	Hamburg	Yes
Norway	NIBIO - Department of Pesticide Chemistry	ÅS	Yes
Peru	Pesticide Lab (Lima)	Lima	Yes
Poland	VSES Warszawa - Pesticide Lab	Warszaw	Yes
Poland	InHort (Skierniewice) - Pesticide Lab	Skierniewice	Yes
Poland	Inst. of Plant Protection - Pesticide Lab Poznan	Poznan	Yes
Poland	IPP-NRL - Pesticide Lab (Bialystok)	Bialystok	Yes
Poland	Pesticide Lab (Torun)	Torun	Yes
Poland	VSES Opole - Pesticide Lab	Opole	Yes

**ANNEX 2. List of laboratories that agreed to participate in EUPT-FV-20.**

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
Poland	VSES Wroclaw - Pesticide Lab	Wroclaw	Yes
Poland	VSES Lodz - Pesticide Lab	Lodz	Yes
Poland	J.S. Hamilton Poland - Pesticide Lab	Gdynia	Yes
Poland	Pesticide Lab (Sosnicowice)	Sosnicowice	Yes
Poland	UO-Technology - Pesticide Lab	Slomczyn	Yes
Portugal	Pesticide Lab (Funchal - Madeira Island)	Funchal - Madeira Island	Yes
Portugal	INIAV - Pesticide Lab (Oeiras)	Oeiras	Yes
Portugal	Vairão - Pesticide Lab (Plant Origin Products)	Vairão - Vila do Conde	Yes
Romania	LRCRPPPV (Tirgu Mures) - Pesticide Lab	Tirgu Mures	Yes
Romania	DSVSA Bucuresti - Pesticide Lab	Bucharest	Yes
Romania	LCCRPPP (Bucharest) - Pesticide Lab	Bucharest	Yes
Romania	DSVSA Ialomita - Lab	Slobozia	Yes
Romania	DSVSA OLT - Lab	Slatina	Yes
Serbia	Center for Food Analysis (Belgrade)	Belgrade	Yes
Serbia	Inst. of Public Health of Belgrade - Pesticide Lab	Belgrade	Yes
Serbia	SP Laboratorija - Pesticide Lab	BECEJ	Yes
Singapore	Agr-food Authority of Singapore - Pesticide Lab	Singapore	Yes
Slovakia	State Veterinary and Food Institute (Bratislava)	Bratislava	Yes
Slovakia	Pesticide Lab of PHA SR - Bratislava	Bratislava	Yes
Slovenija	Pesticide Lab - Maribor	Maribor	Yes
Slovenija	Pesticide Lab - Maribor (Location Ljubljana)	Ljubljana	Yes
Spain	Ecosur - Pesticide Lab	Lorquí (Murcia)	Yes
Spain	Laboratorio Arbitral Agroalimentario	Madrid	Yes
Spain	LABORATORIO KUDAM, S.L.	Pilar de la Horadada (Alicante)	Yes
Spain	Laboratorio Agroalimentario de Granada	Granada	Yes
Spain	Laboratorio Agroalimentario de Extremadura	Cáceres	Yes
Spain	Analytica Alimentaria GmbH - Pesticide Lab	Almeria	Yes
Spain	Laboratorio de Salud Pública de Galicia	Lugo	Yes
Spain	Laboratorio Agroalimentario	Valencia	Yes

**ANNEX 2. List of laboratories that agreed to participate in EUPT-FV20**

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
Spain	Labs & Technological Services AGQ - Burguillos	Burguillos	Yes
Spain	Nasertic	Villava	Yes
Spain	Laboratorio Regional de la CCAA de La Rioja	Logroño	Yes
Spain	Laboratorio Agroambiental de Zaragoza	Zaragoza	Yes
Spain	Laboratorio de Residuos, Inst. Tecnol. de Canarias	Agüimes, Gran Canaria	Yes
Spain	Agricultural and Phytopathological Lab. of Galicia	Abegondo. A Coruña	No
Spain	National Center for Technology and Food Safety	San Adrián (Navarra)	Yes
Spain	LARAGA - Pesticide Lab (Toledo)	Toledo	Yes
Spain	Lab. Agrario Regional - Junta de Castilla y Leon	Burgos	Yes
Spain	Laboratori Agroalimentari de Cabriils - LAC	Cabriils	Yes
Spain	Eurofins AgriQ Spain - Pesticide Lab	Almeria	Yes
Spain	Laboratorio Salud Pública de Badajoz	Badajoz	Yes
Spain	Laboratorio de Producción y Sanidad Vegetal (Jaén)	Mengibar (Jaén)	Yes
Spain	Pesticide Lab (Majadahonda)	Majadahonda	Yes
Spain	Ainia (Valencia)	Valencia	Yes
Spain	SALUD PUBLICA (Almería) - Pesticide Lab	Almería	Yes
Spain	Laboratorio Agroalimentario y de Sanidad Animal	Murcia	Yes
Spain	Soivre Tenerife - Pesticide Lab	Santa Cruz de Tenerife	Yes
Spain	Laboratori Agència Salut Pública Barcelona	Barcelona	Yes
Spain	Laboratorio de Produccion y Sanidad Vegetal.	La Mojonera (Almeria)	Yes
Spain	SALUD PUBLICA (MADRID SALUD)	Madrid	Yes
Spain	Laboratorio de Salud Pública de Cuenca	Cuenca	Yes
Spain	Soivre Valencia - Pesticide Lab	Valencia	Yes
Spain	Laboratorio del SOIVRE	Almería	Yes
Sweden	Eurofins Food & Feed - Pesticide Lab (Lidköping)	Lidköping	Yes
Sweden	Science Department - Chemistry Division 1	Uppsala	Yes
Switzerland	Pesticide Lab (Aargau)	Aargau	Yes
Switzerland	SCAV - Pesticide Lab (GENEVE)	GENEVE	Yes
Switzerland	Pesticide Lab (Zürich)	Zürich	Yes



**ANNEX 2. List of laboratories that agreed to participate in EUPT-FV-20.**

<b>COUNTRY</b>	<b>LABORATORY NAME</b>	<b>CITY</b>	<b>REPORTED RESULTS</b>
Thailand	Central Laboratory - Pesticide Lab (Bangkok)	Bangkok	Yes
The Netherlands	NVWA - Pesticide Lab	Wageningen	Yes
United Kingdom	FERA - Pesticide Lab	York	Yes
United Kingdom	SASA - Pesticide Lab	Edinburgh	Yes
United Kingdom	Scientific Analysis Laboratories - Pesticide Lab	Bar Hill	Yes
United Kingdom	ALS - Contract Testing Laboratory	Cambridgeshire	Yes
Uruguay	UdelaR - Faculty of Chemistry (Montevideo)	Montevideo	Yes