

**EUPT-FV23 v2 update:**

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

The only change with respect to version 1 is in APPENDIX 3. Results (mg/Kg) and z scores for FFP-RSD (25 %), where some z scores have changed (although not their classification as acceptable, questionable or unacceptable).



# EURL-PROFICIENCY TEST-FV-23

## Pesticide Residues in Aubergine

### Homogenate

Final Report - August 2021

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**EURL-EUROPEAN UNION PROFICIENCY TEST 23**  
**FOR THE DETERMINATION OF PESTICIDES IN FRUITS AND VEGETABLES USING**  
**MULTIRESIDUE METHODS**  
**2021**

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 (EUTs) 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 FV-23 has been organised by the EURL in Fruits and Vegetables at the University of Almería, Spain<sup>4</sup>.

Participation in European Proficiency Test FV-23 was mandatory for all National Reference Laboratories (NRLs), as well as all other EU official laboratories, involved in the determination of pesticide residues in fruits and vegetables for the EU multi-annual coordinated control programme or for their own national monitoring programmes. Additionally, laboratories from China, Costa Rica, Kenya, Peru, Serbia, Singapore and United Kingdom participated in this test.

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

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

<sup>2</sup> Regulation (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-two laboratories agreed to participate in EUPT-FV23.

The proficiency test was performed in 2021 using aubergine homogenate. The aubergines were cultivated in a greenhouse in Almería, Spain, and were treated before harvest using commercial formulations applied by spraying with conventional diffusers. After harvest, they were also treated with analytical standards. Eighteen mandatory pesticides and two voluntary ones were used for the treatment. In EUPT-FV23, participating laboratories were not provided with a 'blank' sample.

The test item, 200 g of aubergine homogenate containing pesticide residues, was shipped to participants on 1<sup>st</sup> February 2021. The deadline for results submission to the Organiser was 2<sup>nd</sup> March 2021. 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. The compulsory list contained 215 target pesticides. The pesticide target list is detailed in Annex A together with the voluntary target list, which contained 41 pesticides. The lists 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, or for which EFSA requested lower LOQs: aldrin (0.005 mg/kg), azinphos-methyl (0.005 mg/kg), cadusafos (0.005 mg/kg), carbaryl (0.005 mg/kg), carbofuran (0.005 mg/kg), carbofuran-3-hydroxy (0.005 mg/kg), chlorpyrifos (0.005 mg/kg), demeton-S-methylsulfone (0.005 mg/kg), diazinon (0.005 mg/kg), dichlorvos (0.005 mg/kg), dieldrin (0.005 mg/kg), dimethoate (0.003 mg/kg), ethoprophos (0.005 mg/kg), fenbuconazole (0.005 mg/kg), fipronil (0.004 mg/kg), fipronil sulfone (0.004 mg/kg), imazalil (0.005 mg/kg), monocrotophos (0.005 mg/kg), omethoate (0.003 mg/kg), oxydemeton-methyl (0.005 mg/kg) and triazophos (0.005 mg/kg).

Participants were asked to analyse and report results for any of the pesticides they found which were included in the target lists.

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

For the assessment of overall laboratory performance, the Average of the squared z scores ( $AZ^2$ ) was 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 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. For laboratories in Category B, individual z scores were calculated but the overall accuracy of their results was not assessed.

Laboratories that did not report results have not been classified into any category and are listed in Annex B with the remainder of laboratories that participated in EUPT-FV-23.

## 2. TEST ITEMS

### 2.1 Preparation of the treated test item

The aubergines were cultivated in a greenhouse in Almería, Spain, and were treated before harvest using commercial formulations applied by spraying with conventional diffusors. Additionally, they were post-harvest treated using analytical standards. The pesticides used as commercial formulations were acetamiprid, dimethoate, imazalil, methomyl, quinoxifen, spinosad, tau-fluvalinate and thiabendazole. The pesticides spiked as analytical standards were chlorfenapyr, chlorpyrifos, clofentezine, diazinon, enfosulfan-sulfate, fenarimol, flonicamid, flutianil, isofetamid, tetraconazole, triazophos and zoxamide.

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 EUPT-FV-23. Approximately 480 m<sup>2</sup> of aubergine plants were treated with commercial formulations, which were dissolved in water. Four days after the application, a representative sample of the treated aubergine was collected and analysed to check if the residue levels present were close to the target levels. As the residue levels in the aubergine were low for seven pesticides, a second treatment in the field was applied. Three days after the second treatment, the pesticide residue levels were checked, and as they were close to the target levels, 125 kg of aubergines were harvested (11 days after the first treatment). Subsequently, they were spiked 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 aubergine was mixed in a constantly spinning container until a homogeneous material was obtained. 200 g portions of the well-mixed homogenate were weighed out into screw-capped polyethylene plastic bottles, sealed and stored in a freezer at about - 20 °C prior to distribution to participants.

### 2.2 Homogeneity test

The homogeneity and stability tests were performed by the EURL-FV laboratory at the University of Almería (accredited under ISO/IEC 17025 by the Spanish accreditation body, ENAC). Ten bottles of the treated test item were randomly chosen from those stored in the freezer and analyses were performed on duplicate portions taken from each bottle. The injection sequence of the 20 extracts that were analysed by GC and LC was also randomly chosen. Quantification by GC-MS/MS and LC-MS/MS was performed using matrix matched calibration curves prepared with blank aubergine.

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 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  $\sigma_{all}^2 = (0.3 \times \text{FFP-RSD}(25\%) \times \text{mean concentration})^2$ . This was used to demonstrate that the between-bottle variance was not higher than the within-bottle variance.

**Table 1.** Statistical evaluation of the homogeneity test data (n = 20 analyses)

Pesticide	Mean Conc. (mg/Kg)	$S_s^2$	c	$S_s^2 < c$ Pass/Fail
Acetamiprid	0.176	0.000E+00	4.30E-04	Pass
Chlorfenapyr	0.296	0.000E+00	1.33E-03	Pass
Chlorpyrifos	0.065	0.000E+00	7.00E-05	Pass
Clofentezine	0.115	0.000E+00	4.20E-04	Pass
Diazinon	0.746	0.000E+00	8.52E-03	Pass
Dimethoate	0.077	9.889E-07	7.00E-05	Pass
Endosulfan sulfate	0.306	0.000E+00	1.34E-03	Pass
Fenarimol	0.266	0.000E+00	1.07E-03	Pass
Flonicamid	0.098	2.117E-06	1.00E-04	Pass
Imazalil	0.163	0.000E+00	3.50E-04	Pass
Methomyl	0.24	2.333E-05	6.30E-04	Pass
Quinoxifen	0.188	0.000E+00	4.70E-04	Pass
Spinosad	0.28	3.444E-05	1.05E-03	Pass
Tau-Fluvalinate	0.119	0.000E+00	5.90E-04	Pass
Tetraconazole	0.126	0.000E+00	3.40E-04	Pass
Thiabendazole	0.18	1.667E-06	3.80E-04	Pass
Triazophos	0.189	1.778E-05	5.20E-04	Pass
Zoxamide	0.178	0.000E+00	6.60E-04	Pass
<b>Voluntary Pesticides</b>				
Flutianil	0.038	0.000E+00	5.00E-05	Pass
Isofetamid	0.057	0.000E+00	1.20E-04	Pass

$S_s$ : Between-Sampling Standard Deviation

As can be seen from **Table 1**, all the pesticides evaluated in the aubergine test item passed the homogeneity test.

## 2.3 Stability tests

Stability tests were also carried out by the EURL-FV laboratory at the University of Almería (accredited under ISO/IEC 17025 by the Spanish accreditation body, ENAC). The tests were performed according to ISO 13528:2015, Annex B [2]. Shortly before the test item shipment, three bottles that were stored in the freezer at -20 °C were chosen randomly and stored in a -80 °C freezer (Day 1). After the deadline for reporting results, those three bottles stored at -80 °C, together with three other bottles that were stored in the freezer at -20 °C and were chosen randomly (Day 2) were analysed by duplicate.

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 Day 1 stability test,  $y_1$  the mean value of the Day 2 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**. 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.

**Table 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 2							(M2 – M1)	M2-M1 ≤ 0.3*σ
	Sample 2_A	Sample 2_B	Sample 15_A	Sample 15_B	Sample 225_A	Sample 225_B	Mean 1	Sample 5_A	Sample 5_B	Sample 114_A	Sample 114_B	Sample 158_A	Sample 158_B	Mean2		
Acetamidprid	0.180	0.180	0.170	0.180	0.180	0.190	0.180	0.170	0.180	0.170	0.180	0.180	0.180	0.177	-0.003	Pass
Chlorfenapyr	0.240	0.270	0.240	0.260	0.250	0.280	0.257	0.260	0.260	0.280	0.260	0.250	0.240	0.258	0.002	Pass
Chlorpyrifos	0.062	0.065	0.059	0.064	0.062	0.062	0.062	0.068	0.062	0.070	0.063	0.061	0.061	0.064	0.002	Pass
Clofentezine	0.120	0.140	0.120	0.130	0.130	0.150	0.132	0.120	0.120	0.120	0.120	0.150	0.140	0.128	-0.003	Pass
Diazinon	0.910	0.870	0.900	0.850	0.860	0.850	0.873	1.000	0.970	0.880	0.930	0.880	0.900	0.927	0.053	Pass
Dimethoate	0.077	0.078	0.076	0.076	0.076	0.077	0.077	0.072	0.076	0.079	0.078	0.076	0.080	0.077	0.000	Pass
Endosulfan sulfate	0.270	0.290	0.270	0.270	0.280	0.300	0.280	0.290	0.290	0.280	0.260	0.260	0.250	0.272	-0.008	Pass
Fenarimol	0.290	0.320	0.280	0.300	0.290	0.330	0.302	0.300	0.320	0.330	0.290	0.300	0.280	0.303	0.002	Pass
Flonicamid	0.100	0.110	0.100	0.100	0.110	0.100	0.103	0.100	0.110	0.110	0.100	0.100	0.110	0.105	0.002	Pass
Fluvalinate-tau	0.150	0.160	0.140	0.150	0.150	0.140	0.148	0.150	0.150	0.170	0.140	0.140	0.140	0.148	0.000	Pass
Imazalil	0.200	0.200	0.200	0.190	0.200	0.200	0.198	0.190	0.200	0.200	0.200	0.200	0.200	0.198	0.000	Pass
Methomyl	0.250	0.240	0.240	0.250	0.260	0.250	0.248	0.260	0.250	0.250	0.260	0.260	0.260	0.257	0.008	Pass
Quinoxifen	0.170	0.170	0.170	0.180	0.170	0.170	0.172	0.180	0.170	0.180	0.160	0.160	0.160	0.168	-0.003	Pass
Spinosad (sum)	0.320	0.310	0.300	0.330	0.310	0.330	0.317	0.330	0.300	0.310	0.300	0.290	0.330	0.310	-0.007	Pass
Tetraconazole	0.170	0.170	0.160	0.170	0.170	0.180	0.170	0.160	0.170	0.170	0.160	0.170	0.170	0.167	-0.003	Pass
Thiabendazol	0.180	0.190	0.180	0.180	0.190	0.190	0.185	0.180	0.190	0.190	0.190	0.180	0.190	0.187	0.002	Pass
Triazophos	0.250	0.250	0.240	0.250	0.240	0.230	0.243	0.240	0.250	0.240	0.250	0.250	0.230	0.243	0.000	Pass
Zoxamide	0.160	0.170	0.150	0.170	0.160	0.180	0.165	0.160	0.150	0.150	0.150	0.170	0.160	0.157	-0.008	Pass
<b>Voluntary Pesticides</b>																
Flutianil	0.038	0.040	0.036	0.039	0.038	0.037	0.038	0.040	0.040	0.043	0.036	0.035	0.037	0.039	0.001	Pass
Isofetamid	0.057	0.061	0.054	0.057	0.057	0.056	0.057	0.059	0.058	0.065	0.053	0.053	0.055	0.057	0.000	Pass

Moreover, regarding the stability of the sample arriving not completely frozen, a duplicate analysis of three bottles reproducing the delivery conditions that the samples experienced for 48 hours was performed (Day 3). Laboratories could therefore be sufficiently confident in accepting the treated test item even if it was not completely frozen. All the pesticides passed this second stability test. Results for this 48-hour stability test are indicated in **Table 3**.

As one of the parcels sent to an EU Member State arrived after 72 hours of the shipment, an additional stability test reproducing the delivery conditions that the samples experienced for 72 hours was performed (Day 4). All the pesticides passed this third stability test. Results for this 72-hour stability test are indicated in **Table 4**.

**Table 3.** Statistical test for analytical precision and to demonstrate stability for the 48-hour time-elapse interval.

(mg/Kg)	Day 1							Day 3							(M3 – M1)	M3-M1 ≤ 0.3*σ
	Sample 2_A	Sample 2_B	Sample 15_A	Sample 15_B	Sample 225_A	Sample 225_B	Mean 1	Sample 86_A	Sample 86_B	Sample 204_A	Sample 204_B	Sample 134_A	Sample 134_B	Mean3		
Acetamiprid	0.180	0.180	0.170	0.180	0.180	0.190	0.180	0.180	0.180	0.170	0.170	0.180	0.180	0.177	-0.003	Pass
Chlorfenapyr	0.240	0.270	0.240	0.260	0.250	0.280	0.257	0.250	0.250	0.270	0.250	0.270	0.250	0.257	0.000	Pass
Chlorpyrifos	0.062	0.065	0.059	0.064	0.062	0.062	0.062	0.062	0.061	0.068	0.053	0.058	0.060	0.060	-0.002	Pass
Clofentezine	0.120	0.140	0.120	0.130	0.130	0.150	0.132	0.140	0.120	0.140	0.120	0.130	0.120	0.128	-0.003	Pass
Diazinon	0.910	0.870	0.900	0.850	0.860	0.850	0.873	0.880	0.930	0.890	0.890	0.890	0.850	0.888	0.015	Pass
Dimethoate	0.077	0.078	0.076	0.076	0.076	0.077	0.077	0.079	0.077	0.075	0.075	0.078	0.076	0.077	0.000	Pass
Endosulfan sulfate	0.270	0.290	0.270	0.270	0.280	0.300	0.280	0.270	0.280	0.290	0.260	0.290	0.270	0.277	-0.003	Pass
Fenarimol	0.290	0.320	0.280	0.300	0.290	0.330	0.302	0.290	0.280	0.320	0.290	0.310	0.290	0.297	-0.005	Pass
Flonicamid	0.100	0.110	0.100	0.100	0.110	0.100	0.103	0.100	0.100	0.100	0.100	0.100	0.100	0.100	-0.003	Pass
Fluvalinate-tau	0.150	0.160	0.140	0.150	0.150	0.140	0.148	0.150	0.140	0.170	0.120	0.140	0.140	0.143	-0.005	Pass
Imazalil	0.200	0.200	0.200	0.190	0.200	0.200	0.198	0.200	0.200	0.190	0.190	0.200	0.200	0.197	-0.002	Pass
Methomyl	0.250	0.240	0.240	0.250	0.260	0.250	0.248	0.250	0.250	0.250	0.250	0.250	0.260	0.252	0.003	Pass
Quinoxifen	0.170	0.170	0.170	0.180	0.170	0.170	0.172	0.170	0.180	0.180	0.150	0.160	0.160	0.167	-0.005	Pass
Spinosad (sum)	0.320	0.310	0.300	0.330	0.310	0.330	0.317	0.330	0.310	0.300	0.320	0.290	0.300	0.308	-0.008	Pass
Tetraconazole	0.170	0.170	0.160	0.170	0.170	0.180	0.170	0.170	0.170	0.180	0.160	0.170	0.160	0.168	-0.002	Pass
Thiabendazol	0.180	0.190	0.180	0.180	0.190	0.190	0.185	0.190	0.190	0.180	0.180	0.180	0.180	0.183	-0.002	Pass
Triazophos	0.250	0.250	0.240	0.250	0.240	0.230	0.243	0.230	0.240	0.240	0.240	0.250	0.280	0.247	0.003	Pass
Zoxamide	0.160	0.170	0.150	0.170	0.160	0.180	0.165	0.170	0.170	0.160	0.150	0.160	0.140	0.158	-0.007	Pass
Voluntary Pesticides																
Flutianil	0.038	0.040	0.036	0.039	0.038	0.037	0.038	0.039	0.036	0.043	0.033	0.036	0.036	0.037	-0.001	Pass
Isofetamid	0.057	0.061	0.054	0.057	0.057	0.056	0.057	0.057	0.055	0.064	0.050	0.053	0.054	0.056	-0.002	Pass

**Table 4.** Statistical test for analytical precision and to demonstrate stability for the 72-hour time-elapse interval.

(mg/Kg)	Day 1							Day 4							(M4 – M1)	M4-M1 ≤ 0.3*σ
	Sample 2_A	Sample 2_B	Sample 15_A	Sample 15_B	Sample 225_A	Sample 225_B	Mean 1	Sample 74_A	Sample 74_B	Sample 216_A	Sample 216_B	Sample 144_A	Sample 144_B	Mean 4		
Acetamiprid	0.180	0.180	0.170	0.180	0.180	0.190	0.180	0.180	0.180	0.180	0.180	0.190	0.182	0.002	Pass	
Chlorfenapyr	0.240	0.270	0.240	0.260	0.250	0.280	0.257	0.250	0.280	0.260	0.260	0.260	0.280	0.265	0.008	Pass
Chlorpyrifos	0.062	0.065	0.059	0.064	0.062	0.062	0.062	0.062	0.057	0.063	0.061	0.063	0.068	0.062	0.000	Pass
Clofentezine	0.120	0.140	0.120	0.130	0.130	0.150	0.132	0.120	0.130	0.130	0.120	0.130	0.140	0.128	-0.003	Pass
Diazinon	0.910	0.870	0.900	0.850	0.860	0.850	0.873	0.830	0.930	0.930	0.990	0.880	0.870	0.905	0.032	Pass
Dimethoate	0.077	0.078	0.076	0.076	0.076	0.077	0.077	0.079	0.079	0.078	0.076	0.078	0.079	0.078	0.002	Pass
Endosulfan sulfate	0.270	0.290	0.270	0.270	0.280	0.300	0.280	0.270	0.310	0.270	0.290	0.270	0.290	0.283	0.003	Pass
Fenarimol	0.290	0.320	0.280	0.300	0.290	0.330	0.302	0.290	0.320	0.300	0.300	0.300	0.320	0.305	0.003	Pass
Flonicamid	0.100	0.110	0.100	0.100	0.110	0.100	0.103	0.100	0.110	0.100	0.110	0.100	0.110	0.105	0.002	Pass
Fluvalinate-tau	0.150	0.160	0.140	0.150	0.150	0.140	0.148	0.140	0.140	0.150	0.150	0.150	0.140	0.145	-0.003	Pass
Imazalil	0.200	0.200	0.200	0.190	0.200	0.200	0.198	0.200	0.200	0.200	0.200	0.200	0.200	0.200	0.002	Pass
Methomyl	0.250	0.240	0.240	0.250	0.260	0.250	0.248	0.250	0.250	0.250	0.260	0.250	0.250	0.252	0.003	Pass
Quinoxifen	0.170	0.170	0.170	0.180	0.170	0.170	0.172	0.160	0.150	0.180	0.170	0.180	0.170	0.168	-0.003	Pass
Spinosad (sum)	0.320	0.310	0.300	0.330	0.310	0.330	0.317	0.290	0.340	0.300	0.320	0.310	0.300	0.310	-0.007	Pass
Tetraconazole	0.170	0.170	0.160	0.170	0.170	0.180	0.170	0.170	0.170	0.160	0.170	0.180	0.190	0.173	0.003	Pass
Thiabendazol	0.180	0.190	0.180	0.180	0.190	0.190	0.185	0.190	0.190	0.180	0.180	0.190	0.190	0.187	0.002	Pass
Triazophos	0.250	0.250	0.240	0.250	0.240	0.230	0.243	0.240	0.250	0.240	0.220	0.230	0.220	0.233	-0.010	Pass
Zoxamide	0.160	0.170	0.150	0.170	0.160	0.180	0.165	0.160	0.160	0.160	0.170	0.150	0.160	0.160	-0.005	Pass
Voluntary Pesticides																
Flutianil	0.038	0.040	0.036	0.039	0.038	0.037	0.038	0.039	0.035	0.040	0.039	0.040	0.039	0.039	0.001	Pass
Isofetamid	0.057	0.061	0.054	0.057	0.057	0.056	0.057	0.056	0.052	0.059	0.056	0.059	0.057	0.057	-0.001	Pass

## **2.4 Distribution of test items and protocol to participants**

One bottle of frozen treated test item was shipped to each participant in boxes containing dry ice. The test items were sent out on 1<sup>st</sup> February 2021. All the shipments to EU/EFTA countries arrived within the first 72 hours.

Before sample shipment, the laboratories received full instructions (Annex A) for the receipt and storage of the test item, 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. After applying for the test, each participant laboratory received their Lab Code and password, thus allowing them to participate. This ensured that confidentiality was maintained throughout the duration of Proficiency Test 23. The Target Pesticide List and the Minimum Required Reporting Levels (MRRLs), as established by the Advisory Group, 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 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, considering 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. Results that were  $\geq 10$  times above or below the assigned value were excluded for the calculation of the assigned value. 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 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 EUPs. 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 7**.

### 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 limit (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 value of  $|z| > 5$  has 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 evaluated 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 B. 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-two laboratories agreed to participate in this proficiency test. Nine did not report results, so the total number of laboratories submitting results was 173. The results reported by all the laboratories are presented in this report. However, only results reported by laboratories from EU-countries and EFTA-countries (Iceland, Norway, and Switzerland) have been included in the statistical treatment. The results from the laboratories in China, Costa Rica, Kenya, Peru, Serbia, Singapore and United Kingdom have not been included. This last group totals 15 laboratories that reported results.

Eighteen pesticides from the compulsory pesticide target list and two voluntary pesticides were used to treat the sample and were present in the test item at concentrations above the MRRL. Additionally, one pesticide, omethoate, was present in the test item, but at concentrations below the MRRL. The presence of omethoate is due to the degradation of dimethoate, which was used for the treatment of the aubergine plants.

A summary of the reported results for the pesticides evaluated can be seen below in **Table 5**.

**Table 5.** Summary of Reported Results

Pesticides	No. of Reported Results	No. of False Negative Results	No. of Not Analysed Results	Percentage of Reported Results <sup>a</sup> (out of 159)
Acetamiprid	148	3	8	93
Chlorfenapyr	147	5	7	92
Chlorpyrifos	156	3	0	98
Clofentezine	138	4	17	87
Diazinon	158	0	1	99
Dimethoate	155	1	3	97
Endosulfan sulfate	150	6	3	94
Fenarimol	152	2	5	96
Flonicamid	130	5	24	82
Imazalil	156	1	2	98
Methomyl	145	3	11	91
Quinoxifen	156	1	2	98
Spinosad	141	5	13	89
Tau-Fluvalinate	147	3	9	92
Tetraconazole	153	3	3	96
Thiabendazole	152	2	5	96
Triazophos	154	1	4	97
Zoxamide	143	3	13	90
<b>Voluntary Pesticides</b>				
Flutianil	34	4	121	21
Isofetamid	44	5	110	28

<sup>a</sup> The percentage of Reported Results comes from 159 laboratories. It does not take into account the fifteen laboratories from China, Costa Rica, Kenya, Peru, Serbia, Singapore and United Kingdom.

#### 4.1.1 False positives

Thirty-six laboratories (including non-EU countries) reported results for 16 additional pesticides that were not present in the test item. These pesticides and the residue levels reported are presented in **Table 6**, together with the MRRLs and reporting limits (RLs). Where the reported concentrations of the erroneously detected pesticide were higher than the assigned MRRL value in the Target Pesticide List (Annex A), the result has been considered as a false positive. If the concentrations reported were below the MRRLs, or if the pesticides did not appear in the pesticide list included in Annex A, then they were not considered to be false positives.

Nine laboratories reported omethoate (present in the test item at concentrations below the MRRL). Four of those labs reported omethoate at concentrations above the MRRL. However, the Scientific Committee decided that it would not be considered as a false positive result.

**Table 6.** Laboratories that reported as quantitative results for pesticides that were not present in the treated test item

Laboratory Code	Pesticide	Concentration (mg/kg)	Determination Technique	RL (mg/kg)	MRRL (mg/kg)
5§	Dicofol (sum of p, p' and o,p' isomers)	0.0449	GC-TOF	0.01	0.01
7§	Dicofol (sum of p, p' and o,p' isomers)	0.0442	GC-Orbitrap	0.01	0.01
25§	Dicofol (sum of p, p' and o,p' isomers)	0.047	GC-MS/MS (QQQ)	0.01	0.01
39	Spinetoram	0.0269	LC-MS/MS QQQ	0.01	0.01



Laboratory Code	Pesticide	Concentration (mg/kg)	Determination Technique	RL (mg/kg)	MRRL (mg/kg)
47	Flutolanil	0.053	LC - MS/MS	0.01	0.01
69s	Chlorothalonil	0.018	GC- (μ) ECD	0.01	0.01
79	Aldicarb	0.154	LC-MS/MS QQQ	0.01	0.01
79	Thiacloprid	0.155	LC-MS/MS QQQ	0.01	0.01
81	Dicofol (sum of p, p' and o,p' isomers)	0.035	GC-MS/MS (QQQ)	0.01	0.01
91	Dicofol (sum of p, p' and o,p' isomers)	0.034	GC-MS/MS (QQQ)	0.02	0.01
101	Dieldrin	0.34	GC-MS/MS (QQQ)	0.005	0.005
103	Dicofol (sum of p, p' and o,p' isomers)	0.0217	GC-MS/MS (QQQ)	0.01	0.01
105	Azoxystrobin	0.019	GC-MS/MS (QQQ)	0.01	0.01
105	Dicofol (sum of p, p' and o,p' isomers)	0.038	GC-MS/MS (QQQ)	0.01	0.01
105	Endosulfan beta	0.010	GC-MS/MS (QQQ)	0.01	0.01
105	Fenamiphos	0.026	GC-MS/MS (QQQ)	0.01	0.01
105	Lambda-cyhalothrin (sum of isomers)	0.038	GC-MS/MS (QQQ)	0.01	0.01
105	Tebuconazole	0.017	GC-MS/MS (QQQ)	0.01	0.01
107	Dicofol (sum of p, p' and o,p' isomers)	0.020	GC-MS/MS (QQQ)	0.01	0.01
115	Chlorpyrifos-methyl	0.098	GC-MS	0.01	0.01
123	Azoxystrobin	0.01	LC - MS/MS	0.01	0.01
123	Fenpropathrin	0.0395	LC - MS/MS	0.01	0.01
123	Phoxim	0.0950	LC - MS/MS	0.02	0.01
135	Dicofol (sum of p, p' and o,p' isomers)	0.013	GC-MS/MS (QQQ)	0.01	0.01
137s	Dicofol (sum of p, p' and o,p' isomers)	0.033	GC-TOF	0.01	0.01
143	Spinetoram	0.035	LC-MS/MS QQQ	0.01	0.01
171	Dicofol (sum of p, p' and o,p' isomers)	0.024	GC-MS/MS (QQQ)	0.01	0.01
177	Dicofol (sum of p, p' and o,p' isomers)	0.027	GC-MS/MS (QQQ)	0.02	0.01
191	Dicofol (sum of p, p' and o,p' isomers)	0.0350	GC- (μ) ECD	0.01	0.01
197	Dicofol (sum of p, p' and o,p' isomers)	0.023	GC-MS/MS (QQQ)	0.005	0.01
219	Dicofol (sum of p, p' and o,p' isomers)	0.015	GC-MS/MS (QQQ)	0.01	0.01
223	Dicofol (sum of p, p' and o,p' isomers)	0.025	GC-MS/MS (QQQ)	0.01	0.01
227s	Endosulfan beta	0.27	GC-MS/MS (QQQ)	0.01	0.01
237	Dicofol (sum of p, p' and o,p' isomers)	0.011	GC-MS/MS (QQQ)	0.01	0.01
251	Dicofol (sum of p, p' and o,p' isomers)	0.022	GC-MS/MS (QQQ)	0.005	0.01
251	Tricyclazole	0.18	GC-MS/MS (QQQ)	0.005	0.01
263	Spinetoram	0.010	LC-MS/MS QQQ	0.01	0.01
293	Tebuconazole	0.184	LC-MS/MS QQQ	0.01	0.01
295	Dicofol (sum of p, p' and o,p' isomers)	0.585	GC-MS/MS (QQQ)	0.01	0.01
297	Dicofol (sum of p, p' and o,p' isomers)	0.022	GC-MS/MS (QQQ)	0.01	0.01
305	Dicofol (sum of p, p' and o,p' isomers)	0.027	GC-MS/MS (QQQ)	0.01	0.01
325	Dicofol (sum of p, p' and o,p' isomers)	0.031	GC-MS/MS (QQQ)	0.01	0.01
327	Dicofol (sum of p, p' and o,p' isomers)	0.038	GC-MS/MS (QQQ)	0.01	0.01
329	Dicofol (sum of p, p' and o,p' isomers)	0.0291	GC-MS/MS (QQQ)	0.01	0.01

Laboratory Code	Pesticide	Concentration (mg/kg)	Determination Technique	RL (mg/kg)	MRRL (mg/kg)
339§	Dicofol (sum of <i>p</i> , <i>p'</i> and <i>o,p'</i> isomers)	0.045	GC-MS/MS (QQQ)	0.01	0.01

§ Non-EU/EFTA laboratories

Twenty-five laboratories reported dicofol (sum of *p*, *p'* and *o,p'* isomers). Some of them specified in the comments box that they had only detected *o,p'*-dicofol. The EURL-FV could not detect it, neither by GC-MS/MS (triple quadrupole) nor by GC-MS (Q-TOF), employing high resolution mass spectrometry. The study of this high number of false positive results led to two possible hypotheses. The first one was based on the common transitions shared by *o,p'*-dicofol and fenarimol, one of the compounds present in the test item. Those two pesticides have similar structures and some of their fragment ions are the same (Figure 1).

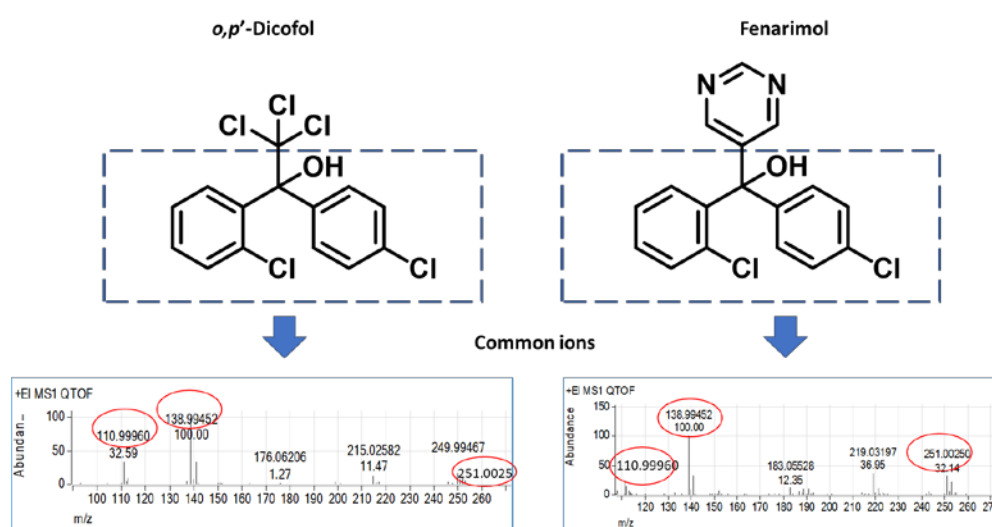
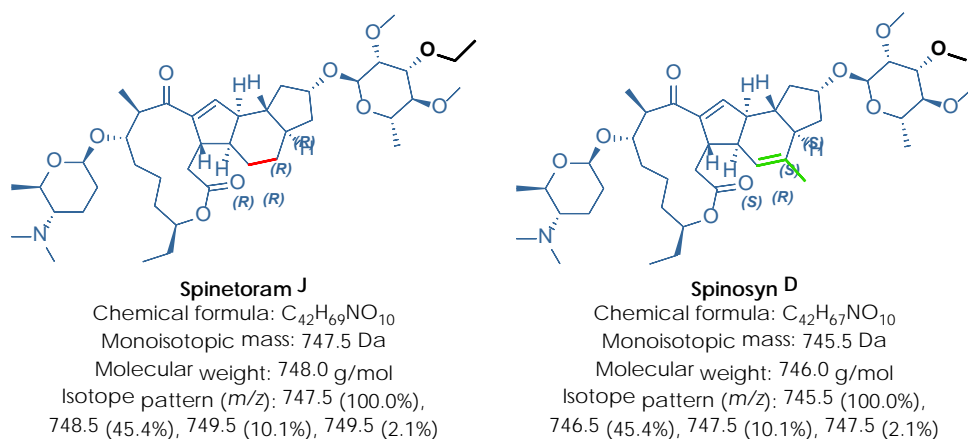


Figure 1. Chemical structures and common ions of *o,p'*-dicofol and fenarimol.

However, their retention times are not so close (1 minute of difference with a total run time of 20 minutes). For this reason, a second hypothesis was evaluated: the possibility that natural components of the aubergine could share ions with *o,p'*-dicofol. With the purpose to check whether this hypothesis could be correct, blank aubergine (the same as the test item, but without pesticides) was analysed by GC-MS (Q-TOF), working with an extraction window of 1 Da. Two chromatographic peaks corresponding to three ions of *o,p'*-dicofol (139, 250 and 251) were found at retention times close to the one of *o,p'*-dicofol. However, the same aubergine was used for the preparation of the test item of the screening proficiency test, EUPT-SM13, and no laboratories reported it. Accordingly, it is not clear which one of the hypotheses was the correct one, or maybe it was a combination of both of them, depending on the conditions of each laboratory.

Another false positive that was reported by multiple participants was spinetoram (three laboratories reported it). A detailed study on this issue revealed that spinetoram J shares, at least, two mass transitions with spinosyn D. Furthermore, their retention times are very similar or even identical, depending on the chromatographic conditions. In fact, while the monoisotopic masses of these two analytes are different, 2 isotopologues of spinosyn D have identical molecular mass to the first

decimal unit as the monoisotopic mass of spinetoram J (747.5 Da) (**Figure 2**). This fact, combined with fragment ions with identical nominal masses shared by spinetoram J and spinosyn D ( $m/z$  98 and 142), means that these two analytes share (at least) two mass transitions,  $748 > 98$  and  $748 > 142$ . Thus, the common mass transitions can possibly lead to false positive reporting, particularly when analysing samples with low resolution mass spectrometry.



**Figure 2.** Chemical structures of spinetoram J and spinosyn D.

#### 4.1.2 False negatives

**Tables 7 a and b** summarise the results from laboratories (including non-EU laboratories, indicated with S) that reported false negatives, presented as 'Not Detected' (ND).

**Table 7.a** Laboratories that failed to report pesticides that were present in the treated test item.

Laboratory Code	Acetamiprid	Chlorfenapyr	Chlorpyrifos	Clofentezine	Dimethoate	Endosulfan sulfate	Fenarimol	Flonicamid	Imazail	Methomyl	Quinoxifen	Spinosad	Tau-Fluvalinate	Tetraconazole	Thiabendazole	Triazophos	Zoxamide
25 <sup>S</sup>						ND											
45		ND	ND			ND						ND	ND	ND		ND	
49						ND											
75					ND												
81													ND				
101		ND															
105	ND			ND			ND	ND		ND	ND	ND		ND	ND		ND
115			ND														
127						ND											
165												ND					
183		ND	ND			ND	ND	ND									
211								ND									
227 <sup>S</sup>									ND								
229																	ND
243								ND									
271						ND											
287	ND			ND						ND		ND			ND		
293														ND			

Laboratory Code	Acetamiprid	Chlorfenapyr	Chlorpyrifos	Clofentezine	Dimethoate	Endosulfan sulfate	Fenarimol	Flonicamid	Imazalil	Methomyl	Quinoxifen	Spinosad	Tau-Fluvalinate	Tetraconazole	Thiabendazole	Triazophos	Zoxamide
295																	ND
313									ND								
321 <sup>§</sup>								ND									
339 <sup>§</sup>														ND			
343	ND			ND				ND		ND		ND					
351		ND															
359				ND													
367		ND				ND							ND				

<sup>§</sup> Non-EU/EFTA laboratories

**Table 7.b**

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

Voluntary Pesticides		
Lab Code	Flutianil	Isofetamid
47	ND	
197		ND
209	ND	
237		ND
263		ND
363	ND	ND
367	ND	ND

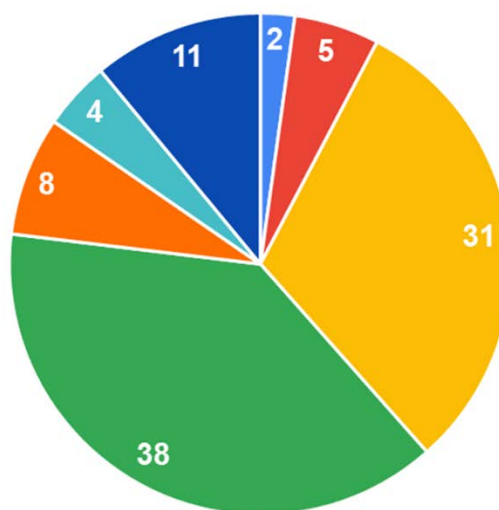
ND: Not detected

#### 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)). The histograms of both the compulsory and voluntary pesticides present in the test item are presented in **Appendix 2**.

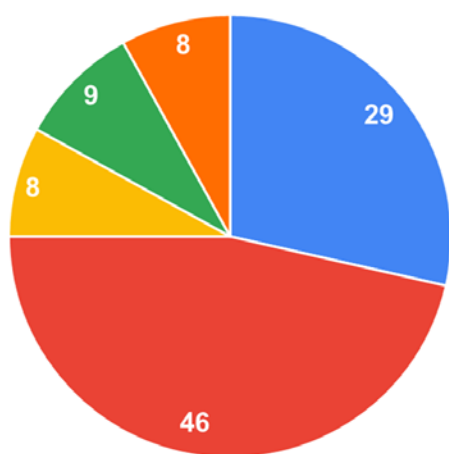
The statistical analysis of spinosad revealed multimodality in the distribution of the results. The reason for this multimodality is that spinosad is a mixture of spinosyn A and spinosyn D, and the differences in the analytical standards used and in the quantification procedures led to different populations of results. With the aim to learn about the different quantification procedures, a questionnaire was sent to the participants. One hundred and thirteen laboratories answered it. The first question referred to the analytical standard used for quantification of spinosad. 92 % of the labs used spinosad analytical standard (mixture of spinosyn A and spinosyn D), and 8 % employed separate analytical standards of spinosyn A and spinosyn D. Those ones that used the spinosad technical mixture reported different proportions of spinosyns A and D in their standards, that ranged from 50/50 to 90/10. 4 % of the labs declared that the proportion of the different components was not indicated in the analytical certificate, and 11 % didn't know the proportion. **Figure 3** shows the different ratios of spinosyn A/spinosyn D in the spinosad analytical standards employed by the participants.

- approx. 50/50
- approx. 60/40
- approx. 70/30
- approx. 80/20
- approx. 90/10
- Not indicated on the analytical certificate
- Unknown



**Figure 3.** Percentage of answers to the question: *If you used the spinosad analytical standard, could you please specify the proportion of spinosyn A and spinosyn D in your mix?*

As regards quantification of the chromatographic peaks, Figure 4 summarises the different answers. Only 8 % of the labs quantified with the individual analytical standards, as was learned from the question about the analytical standard used. 46 % of the participants quantified spinosyn A with the component spinosyn A of the spinosad analytical standard considering its proportion in the mix, doing the same for spinosyn D, and summing both concentrations. Those two approaches are the correct ones for calculating the concentration of spinosad.



- Quantifying spinosyn A with the individual analytical std. of spinosyn A, spinosyn D with the individual analytical std. of spinosyn D, and summing both concentrations.
- By summing chromatographic areas of spinosyn A and D and comparing with the sum of areas of spinosyn A and D in the analytical standard.
- Quantifying spinosyn A with the component spinosyn A of the spinosad analytical standard CONSIDERING its proportion in the mix, doing the same for spinosyn D, and summing both concentrations.
- Quantifying spinosyn A with the component spinosyn A of the spinosad analytical standard NOT CONSIDERING its proportion in the mix, doing the same for spinosyn D, and SUMMING both concentrations.
- Quantifying spinosyn A with the component spinosyn A of the spinosad analytical standard NOT CONSIDERING its proportion in the mix, doing the same for spinosyn D, and reporting the AVERAGE of both concentrations.

**Figure 4.** Percentage of answers to the question: *As regards quantification of the chromatographic peaks, how did you quantify spinosad?*

29 % of the laboratories quantified spinosad by summing chromatographic areas of spinosyn A and D and comparing with the sum of areas of spinosyn A and D in the analytical standard. This approach assumes that the instrumental responses of spinosyn A and D are the same, but they are

not, as they have significant differences in their fragmentation mechanisms.<sup>5</sup> Finally, 9 % and 8 % of the participants quantified each spinosyn with its corresponding component of the spinosad analytical standard not considering its proportion in the mix, and summing or reporting the average of both concentrations, respectively. In these cases, as the proportion is not considered, quantification is not correct. Therefore, based on the answers of the questionnaire, 46 % of the spinosad results were not correctly quantified. The z scores classification of those labs that quantified following one of the correct approaches was 95 % acceptable, 2 % questionable and 3 % unacceptable. For those labs that followed inaccurate approaches, the classification of z scores was: 76 % acceptable, 6 % questionable and 18 % unacceptable.

A re-evaluation of the assigned value was performed considering only those EU/EFTA laboratories that quantified spinosad following a correct approach. This way, spinosad robust mean was 0.197 mg/kg, whereas the assigned value considered for the evaluation of the results after removing the second mode (see section 4.2) was 0.196 mg/kg. In the same way, the robust mean of spinosyns A and D was calculated: Spinosyn A: 0.139 mg/kg (CV\* 18.2 %) and Spinosyn D: 0.057 mg/kg (CV\* 34.4 %). The high dispersion of the results of spinosyn D might be explained by its low proportion in some of the technical mixtures of spinosad, or even by the fact that the proportions certified by the suppliers might not be accurate.

#### 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, after exclusion of gross errors (those results  $\geq 10$  times above or below the assigned value). Only one result reported for chlorpyrifos (0.98 mg/kg) was excluded for the calculation of its assigned value as, following the previously mentioned criterion, it was considered a gross error.

In the case of spinosad, the distribution of the results presented bimodality, as explained in section 4.1. The second mode corresponded to 12 results with an overestimation of their concentration. For that reason, the Advisory Group agreed to exclude the second mode for the calculation of the assigned value. The robust mean considering all the EU/EFTA results was 0.203 mg/kg, whereas the robust mean of the EU/EFTA results of the first mode (assigned value) was 0.196 mg/kg.

The assigned values for the eighteen compulsory and the two voluntary pesticides and their uncertainties are presented in **Table 8**.

Assigned values and robust standard deviations differ from those published in the Preliminary Report: after the submission of results, a preliminary evaluation was performed and sent to the participants in March 2021. For the calculation of those assigned values, UK was considered as an EU Member State. During the EUPT Scientific Committee meeting, it was noted that UK should be treated as a non-EU/EFTA country (from 2021 onwards). For that reason, the results of the four UK participants were removed for the calculation of the new assigned values. The comparison of the robust mean before and after removing UK results is shown in **Table 9**.

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<sup>5</sup> Technical report "Comparison of the instrumental response of different constituents of specific pesticides" ([https://www.eurl-pesticides.eu/userfiles/file/EURL-FV%20\(2020-M41\)%20Comparison\\_instrumental\\_response\\_isomers.pdf](https://www.eurl-pesticides.eu/userfiles/file/EURL-FV%20(2020-M41)%20Comparison_instrumental_response_isomers.pdf))

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, also employing this value for the calculation of the uncertainty. These RSDs can be seen in **Table 8**.

**Table 8.** 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* (%)
Acetamiprid	0.01	0.175	0.002	148	25	13.4
Chlorfenapyr	0.01	0.299	0.006	147	25	18.3
Chlorpyrifos	0.005	0.070	0.001	156	25	19.1
Clofentezine	0.01	0.096	0.002	138	25	22.8
Diazinon	0.005	0.759	0.013	158	25	17.3
Dimethoate	0.003	0.079	0.001	155	25	14.8
Endosulfan sulfate	0.01	0.283	0.007	150	25	24.6
Fenarimol	0.01	0.320	0.007	152	25	22.2
Flonicamid	0.01	0.102	0.002	130	25	16.0
Imazalil	0.005	0.188	0.004	156	25	20.5
Methomyl	0.01	0.226	0.003	145	25	14.5
Quinoxifen	0.01	0.194	0.003	156	25	17.3
Spinosad	0.01	0.196	0.004	141/129^	25	17.2
Tau-Fluvalinate	0.01	0.130	0.003	147	25	25.3
Tetraconazole	0.01	0.156	0.003	153	25	17.5
Thiabendazole	0.01	0.197	0.003	152	25	14.8
Triazophos	0.005	0.211	0.005	154	25	22.0
Zoxamide	0.01	0.157	0.003	143	25	18.0
<b>Voluntary Pesticides</b>						
Flutianil	0.01	0.060	0.002	34	25	18.9
Isofetamid	0.01	0.059	0.002	44	25	13.4

^ 129 for the calculation of the assigned value

**Table 9.** Comparison of Robust mean values before and after removing UK results.

Pesticides	Robust Mean Preliminary Report (with UK results (mg/kg))	Robust Mean (Assigned Value, mg/kg)
Acetamiprid	0.175	0.175
Chlorfenapyr	0.299	0.299
Chlorpyrifos	0.070	0.070
Clofentezine	0.096	0.096
Diazinon	0.755	0.759
Dimethoate	0.079	0.079
Endosulfan sulfate	0.283	0.283
Fenarimol	0.319	0.320
Flonicamid	0.102	0.102
Imazalil	0.187	0.188
Methomyl	0.226	0.226
Quinoxifen	0.193	0.194
Spinosad	0.196	0.196
Tau-Fluvalinate	0.130	0.130
Tetraconazole	0.156	0.156
Thiabendazole	0.196	0.197

Pesticides	Robust Mean Preliminary Report (with UK results (mg/kg))	Robust Mean (Assigned Value, mg/kg)
Triazophos	0.210	0.211
Zoxamide	0.156	0.157
Voluntary Pesticides		
Flutianil	0.059	0.060
Isofetamid	0.059	0.059

### 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 concentrations reported for each pesticide. The z scores of laboratories from non-EU countries have been included in **Appendix 3**, but have not been considered in **Table 10**, where the classification of z scores reported by EU/EFTA laboratories is shown.

**Table 10.** Classification of z scores for the pesticides reported (only EU/EFTA participants)

Pesticides	Acceptable (%)	Questionable (%)	Unacceptable (%)
Acetamiprid	96.8	1.3	1.9
Chlorfenapyr	91.7	3.8	4.5
Chlorpyrifos	95.1	1.8	3.1
Clofentezine	89.7	4.8	5.5
Diazinon	98.1	0.6	1.2
Dimethoate	96.9	1.3	1.9
Endosulfan sulfate	88.8	5.0	6.3
Fenarimol	94.3	3.2	2.5
Flonicamid	93.5	1.4	5.1
Imazalil	96.1	1.3	2.6
Methomyl	86.7	2.0	11.3
Quinoxifen	89.0	5.2	5.8
Spinosad	95.0	1.9	3.1
Tau-Fluvalinate	94.9	1.3	3.8
Tetraconazole	95.0	4.4	0.6
Thiabendazole	95.3	2.0	2.7
Triazophos	84.6	2.6	12.8
Zoxamide	90.0	0.0	10.0
Voluntary Pesticides			
Flutianil	95.0	3.1	1.9
Isofetamid	96.9	2.5	0.6

z scores for false negative results have been calculated using the MRRL value given in the Target Pesticide List (Annex A) 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 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 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 (193), 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**.

Ninety-eight of the 159 EU and EFTA laboratories that submitted results were classified into Category A (62 %).

From the  $AZ^2$ , 93 % were classed as 'good', 5 % as 'satisfactory' and 0 % as 'unsatisfactory' (Only considering EU and EFTA laboratories).

Of the 61 EU and EFTA laboratories in Category B, 29 had reported a false positive result. Twenty-one of them would have been classified into Category A if not for that false positive result.

**Table 11** shows all the laboratories in Category A (including non-EU laboratories, indicated with \$), the number of pesticides reported, the percentage of pesticides analysed from the compulsory target list, the  $AZ^2$  values and their subclassifications. Laboratories that reported false negative results in Category A are marked with the symbol  $\emptyset$ .

**Table 11.** Performance and Classification of laboratories in Category A using the  $AZ^2$  formula

Lab Code	No. of pesticides detected (max.18)	% of pesticides analysed from target list	$AZ^2$	Classification
9	18	99	0.4	Good
11	18	100	0.1	Good
13	18	100	1.3	Good
15	18	97	1.6	Good
19	18	95	0.7	Good
21	18	99	1.2	Good
31	18	100	0.2	Good
36	18	97	0.5	Good
41	18	97	0.3	Good
43	18	100	0.2	Good
51	18	100	1.4	Good
53	18	100	0.1	Good

Lab Code	No. of pesticides detected (max.18)	% of pesticides analysed from target list	AZ <sup>2</sup>	Classification
55	18	97	0.5	Good
57	18	100	0.4	Good
59	18	97	1.7	Good
61	18	96	1.0	Good
63	18	97	2.2	Satisfactory
67	18	100	0.4	Good
71	18	100	0.6	Good
73	18	96	0.7	Good
77	18	96	0.1	Good
83	18	94	0.6	Good
85	18	100	0.3	Good
87	18	96	0.1	Good
89	18	99	1.1	Good
93	18	93	0.2	Good
95	18	91	1.4	Good
97	18	100	1.0	Good
99	18	100	0.3	Good
109 <sup>§</sup>	18	92	0.8	Good
111	18	91	0.7	Good
113 <sup>§</sup>	18	95	0.2	Good
117 <sup>§</sup>	18	97	0.8	Good
119	17	90	1.7	Good
121	18	93	0.9	Good
125	18	100	0.3	Good
127 $\Theta$	17	97	1.2	Good
129	18	99	0.6	Good
131	18	97	0.3	Good
133	17	90	0.8	Good
147	18	100	0.4	Good
149	18	100	0.2	Good
151	18	96	0.4	Good
157	18	96	0.1	Good
159	18	100	0.6	Good
161	18	99	0.4	Good
163	18	92	0.9	Good
167	18	100	0.2	Good
169	18	99	0.2	Good
179	18	100	0.2	Good
181	18	100	2.1	Satisfactory
187	18	96	0.5	Good
193	18	100	0.3	Good
195	16	91	0.8	Good
199	18	98	0.8	Good
201	18	97	0.3	Good
203	18	99	0.9	Good
205 <sup>§</sup>	18	98	0.3	Good
207	18	100	0.2	Good
209	18	100	0.3	Good

Lab Code	No. of pesticides detected (max.18)	% of pesticides analysed from target list	AZ <sup>2</sup>	Classification
211 ⊖	17	99	1.3	Good
215	18	100	0.6	Good
217	18	92	1.0	Good
221	18	100	0.6	Good
225	18	95	0.2	Good
233	18	99	0.3	Good
235	18	98	1.3	Good
239	18	94	0.8	Good
241	18	100	1.2	Good
243 ⊖	17	100	2.2	Satisfactory
245	18	100	1.3	Good
249	18	99	0.2	Good
255	18	99	0.9	Good
259	18	91	0.4	Good
261	18	99	0.3	Good
265	18	97	1.6	Good
267	18	93	0.3	Good
271 ⊖	17	100	2.1	Satisfactory
275	18	100	0.6	Good
279	18	93	0.5	Good
281	18	99	0.4	Good
285	18	100	0.1	Good
289	18	100	1.6	Good
303	18	100	0.3	Good
309	18	100	1.6	Good
311	18	99	0.6	Good
315	18	100	1.0	Good
317	18	100	1.1	Good
319	18	100	0.1	Good
321 <sup>§</sup> ⊖	17	91	1.3	Good
323 <sup>§</sup>	18	94	1.0	Good
331	18	99	2.6	Satisfactory
333	18	94	1.6	Good
335	18	96	1.9	Good
337	18	99	0.2	Good
341	18	94	1.2	Good
349	18	100	0.2	Good
353	18	100	0.2	Good
355	18	100	0.3	Good
357	18	95	0.9	Good
359 ⊖	17	93	2.0	Good
361	18	98	0.2	Good
363	18	100	0.2	Good
368	18	100	0.1	Good

⊖ Laboratories reporting a false negative result  
<sup>§</sup> Non-EU/EFTA laboratories

**Table 12** shows all the laboratories in Category B (including non-EU laboratories, indicated with §), 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 the symbol ⊖ and laboratories reporting a false positive are marked with a '+’.

**Table 12.** Performance of laboratories in Category B

Lab Code	No. of pesticides detected	% of pesticides detected	% of pesticides analysed from target list	No. of total z scores	No. of acceptable z scores (z score ≤ 2.0)
5§+	18	100	100	18	17
7§+	18	100	100	18	18
17	17	94	65	17	16
25§ ⊖+	17	94	100	18	17
27	13	72	61	13	13
29	17	94	82	17	17
33	17	94	78	17	16
35	15	83	63	15	13
39+	18	100	99	18	18
45 ⊖	9	50	87	16	8
47+	18	100	100	18	16
49 ⊖	17	94	85	18	17
69§+	17	94	89	17	17
75 ⊖	13	72	80	14	12
79+	18	100	92	18	18
81 ⊖+	17	94	100	18	17
91+	17	94	81	17	17
101 ⊖+	17	94	92	18	17
103+	18	100	100	18	18
105 ⊖+	6	33	73	16	5
107+	6	33	33	6	6
115 ⊖+	13	72	53	14	7
123+	18	100	79	18	3
135+	18	100	97	18	18
137§+	18	100	99	18	18
139	12	67	60	12	12
141	15	83	57	15	15
143+	18	100	99	18	16
153	17	94	89	17	16
165 ⊖	11	61	56	12	11
171+	18	100	100	18	18
173	17	94	74	17	15
175§	12	67	62	12	10
177+	17	94	93	17	17
183 ⊖	13	72	81	18	7
185	18	100	84	18	18
188	17	94	86	17	17
191+	7	39	27	7	7
197+	18	100	99	18	18
213	17	94	58	17	15

Lab Code	No. of pesticides detected	% of pesticides detected	% of pesticides analysed from target list	No. of total z scores	No. of acceptable z scores (z score $\leq$ 2.0)
219+	18	100	98	18	18
223+	18	100	97	18	18
227 <sup>§</sup> $\ominus$ +	16	89	92	17	15
229 $\ominus$	11	61	48	12	11
237+	18	100	100	18	17
247	6	33	26	6	5
251+	18	100	98	18	18
253	14	78	40	14	12
257	17	94	77	17	17
263+	18	100	100	18	18
273	12	67	53	12	12
287 $\ominus$	12	67	86	17	11
291	13	72	57	13	13
293 $\ominus$ +	17	94	87	18	13
295 $\ominus$ +	12	67	57	13	9
297+	18	100	100	18	16
299	17	94	88	17	17
301	13	72	61	13	12
305+	18	100	96	18	18
307	15	83	60	15	11
313 $\ominus$	14	78	71	15	9
325+	18	100	100	18	18
327+	18	100	94	18	17
329+	18	100	100	18	17
339 <sup>§</sup> $\ominus$ +	17	94	100	18	17
343 $\ominus$	13	72	93	18	8
347	13	72	65	13	10
351 $\ominus$	15	83	71	16	13
367 $\ominus$	15	83	100	18	15

$\ominus$  Laboratories reporting a false negative result  
 + Laboratories reporting a false positive result  
<sup>§</sup> Non-EU/EFTA laboratories

The AZ<sup>2</sup> graphical representation for EU/EFTA laboratories classified into Category A can be seen in **Appendix 6**. The EU National Reference Laboratories (NRLs) for Fruits and Vegetables have been plotted using a different 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-21, out of 172 laboratories (EU and EFTA), 112 were in Category A with the following classes: 0 'unsatisfactory', 4 'satisfactory' and 108 'good'.
- For EUPT-FV-22, out of 155 laboratories (EU and EFTA), 104 were in Category A with the following classes: 4 'unsatisfactory', 6 'satisfactory' and 94 'good'.

## 5. CONCLUSIONS

One hundred and eighty-two laboratories agreed to participate in EUPT-FV-23. Nine of them did not submit results. From the remaining 173 laboratories that submitted results, 14 did not belong to EU nor EFTA countries, so their results were not considered for the estimation of the assigned value.

From the total 20 pesticides used to treat the test item, 18 mandatory and two voluntary pesticides were evaluated in EUPT-FV-23, based on the analysis of aubergine homogenate.

The statistical analysis of spinosad revealed multimodality in the distribution of the results. The differences in the analytical standards used and in the quantification procedures led to different populations of results. The evaluation of a questionnaire sent to the participants revealed that 46 % of the labs that answered the survey did not quantify spinosad correctly. The calculation of the robust mean of spinosad considering only those EU/EFTA laboratories that quantified it following a correct approach led to a value of 0.197 mg/kg, almost the same as the assigned value considered for the evaluation of the results, 0.196 mg/kg. Spinosyn D presented a high dispersion of the results which might be explained by its low proportion in some of the technical mixtures of spinosad, or even by the fact that the proportion of the different components certified by the suppliers might not be accurate. The EURL-FV will undertake further study of this matter.

Of a total number of 2862 possible determinations from EU/EFTA laboratories (159 laboratories by 18 evaluated pesticides), 93.7 % were reported, 4.5 % were not analysed and 1.8 % were not detected (false negative results). The number of false positive results was particularly large: 29 EU/EFTA laboratories (36 considering the whole population) reported 15 mandatory pesticides (16 considering the whole population) as false positives. This exceptionally high number of false positives could be partly, though not wholly, explained by the reporting of dicofol (sum of *p*, *p'* and *o,p'* isomers) by 25 participants. The reason for this false positive could be common ion transitions shared by dicofol and fenarimol (one of the compounds present in the test item) or the presence of aubergine natural components with the same ions as *o,p'*-dicofol.

The total number of z scores of laboratories from EU/EFTA countries was 2732, with 93.8 % of them acceptable, 2.6 % questionable and 3.6 % unacceptable.

62 % of the EU and EFTA laboratories that submitted results were classified into Category A. Of them, 95 % were classed as 'good', 5 % as 'satisfactory' and 0 % as 'unsatisfactory'.

The robust standard deviation (CV\*) was in all cases below 25.3 %, with an average value of 18.7 % for the 18 pesticides evaluated.

Participation in this year's European Proficiency Test 23 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, Costa Rica, Kenya, Peru, Serbia, Singapore and United Kingdom participated in EUPT-FV-23.

## 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. Rodríguez-Torreblanca, A. Andersson, A. R. Fernández-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.

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Acetamiprid (mg/kg)		Chlorfenapyr (mg/kg)		Chlorpyrifos (mg/kg)		Clofentezine (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.180	0.170	0.290	0.300	0.064	0.063	0.130	0.110
0.180	0.180	0.300	0.300	0.064	0.074	0.110	0.140
0.180	0.170	0.300	0.310	0.070	0.061	0.130	0.100
0.190	0.170	0.310	0.310	0.059	0.069	0.130	0.100
0.180	0.160	0.300	0.280	0.067	0.058	0.120	0.110
0.170	0.180	0.270	0.350	0.066	0.068	0.120	0.100
0.170	0.180	0.290	0.280	0.069	0.064	0.120	0.090
0.160	0.180	0.290	0.310	0.061	0.060	0.120	0.110
0.170	0.180	0.260	0.280	0.064	0.070	0.120	0.110
0.170	0.190	0.300	0.290	0.059	0.069	0.100	0.130

Diazinon (mg/kg)		Dimethoate (mg/kg)		Endosulfan sulfate (mg/kg)		Fenarimol (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.810	0.720	0.079	0.077	0.300	0.310	0.270	0.270
0.790	0.740	0.079	0.081	0.300	0.300	0.260	0.270
0.770	0.790	0.078	0.076	0.310	0.320	0.270	0.280
0.760	0.720	0.080	0.080	0.320	0.310	0.280	0.270
0.760	0.700	0.079	0.073	0.310	0.290	0.270	0.250
0.750	0.690	0.075	0.080	0.290	0.360	0.250	0.320
0.700	0.750	0.072	0.077	0.300	0.300	0.260	0.260
0.800	0.700	0.075	0.078	0.290	0.320	0.250	0.270
0.740	0.760	0.073	0.076	0.280	0.280	0.240	0.240
0.800	0.660	0.075	0.076	0.320	0.300	0.280	0.260

Flonicamid (mg/kg)		Imazalil (mg/kg)		Methomyl (mg/kg)		Quinoxifen (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.100	0.097	0.170	0.160	0.240	0.240	0.190	0.200
0.100	0.099	0.170	0.170	0.250	0.250	0.180	0.200
0.100	0.095	0.170	0.150	0.240	0.250	0.190	0.190
0.100	0.100	0.170	0.160	0.240	0.230	0.180	0.190
0.098	0.095	0.170	0.150	0.250	0.240	0.190	0.170
0.100	0.098	0.170	0.170	0.240	0.230	0.190	0.190
0.096	0.099	0.150	0.160	0.230	0.240	0.200	0.190
0.094	0.093	0.160	0.170	0.240	0.240	0.190	0.170
0.093	0.097	0.150	0.160	0.240	0.240	0.190	0.200
0.097	0.100	0.160	0.170	0.230	0.230	0.170	0.190

The sample numbers used for this test were: 13, 53, 92, 113, 173, 180, 182, 199, 227 and 233.



## APPENDIX 1. Homogeneity.

Spinosad (mg/kg)		Tau-Fluvalinate (mg/kg)		Tetraconazole (mg/kg)		Thiabendazole (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.270	0.290	0.110	0.110	0.130	0.130	0.190	0.180
0.280	0.280	0.120	0.150	0.130	0.130	0.190	0.180
0.250	0.290	0.130	0.100	0.120	0.110	0.180	0.180
0.250	0.280	0.100	0.130	0.140	0.120	0.190	0.180
0.260	0.280	0.130	0.090	0.140	0.100	0.180	0.170
0.300	0.300	0.110	0.140	0.140	0.140	0.180	0.180
0.280	0.290	0.140	0.110	0.110	0.130	0.170	0.180
0.300	0.290	0.100	0.110	0.130	0.130	0.170	0.180
0.270	0.300	0.110	0.150	0.110	0.120	0.170	0.180
0.270	0.260	0.100	0.130	0.110	0.140	0.180	0.180

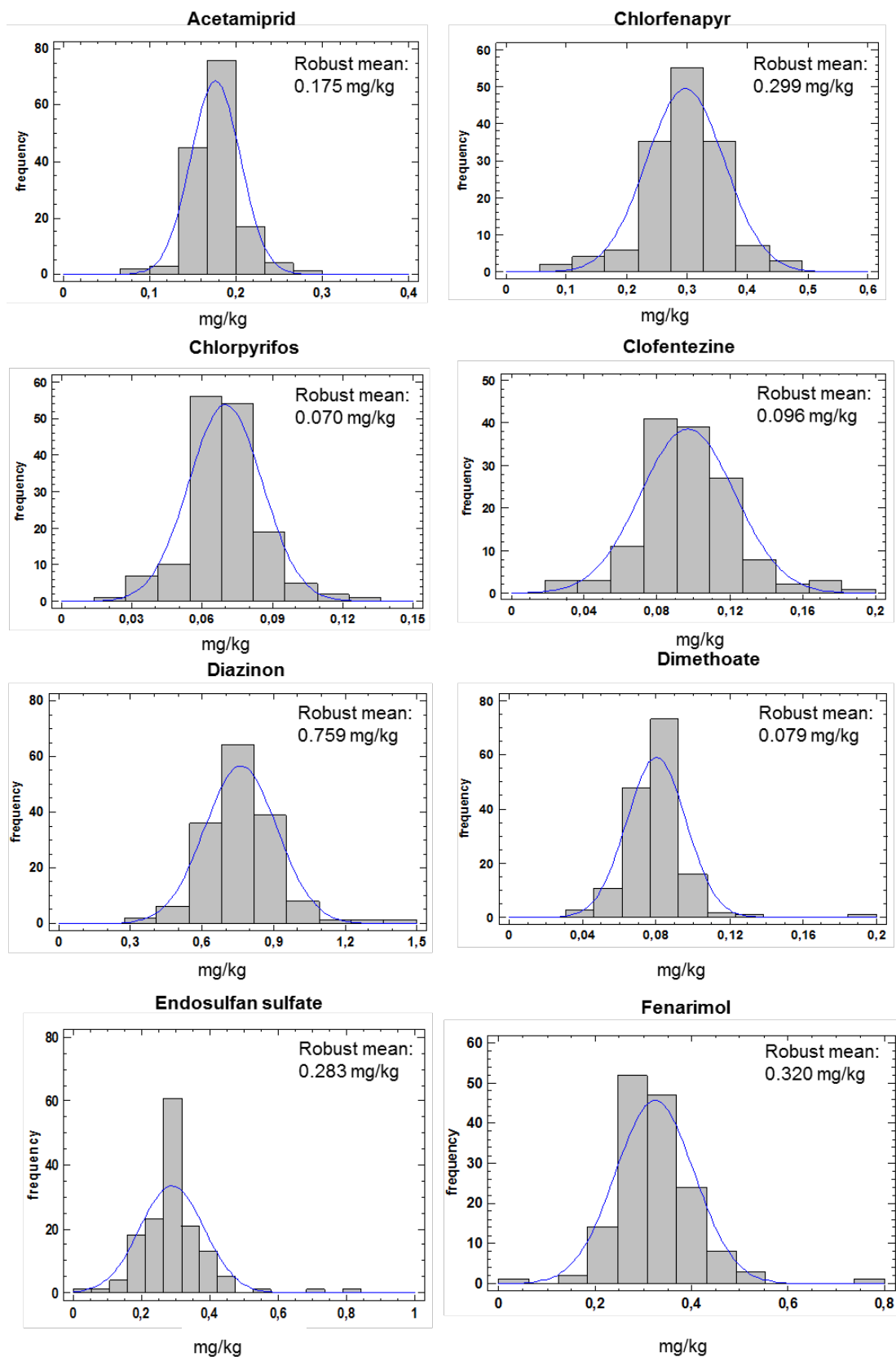
Triazophos (mg/kg)		Zoxamide (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.200	0.190	0.200	0.160
0.200	0.200	0.190	0.190
0.200	0.190	0.180	0.180
0.200	0.190	0.200	0.170
0.190	0.160	0.190	0.140
0.190	0.200	0.180	0.190
0.170	0.190	0.160	0.180
0.190	0.200	0.190	0.190
0.180	0.170	0.150	0.160
0.170	0.200	0.160	0.190

### Voluntary Pesticides

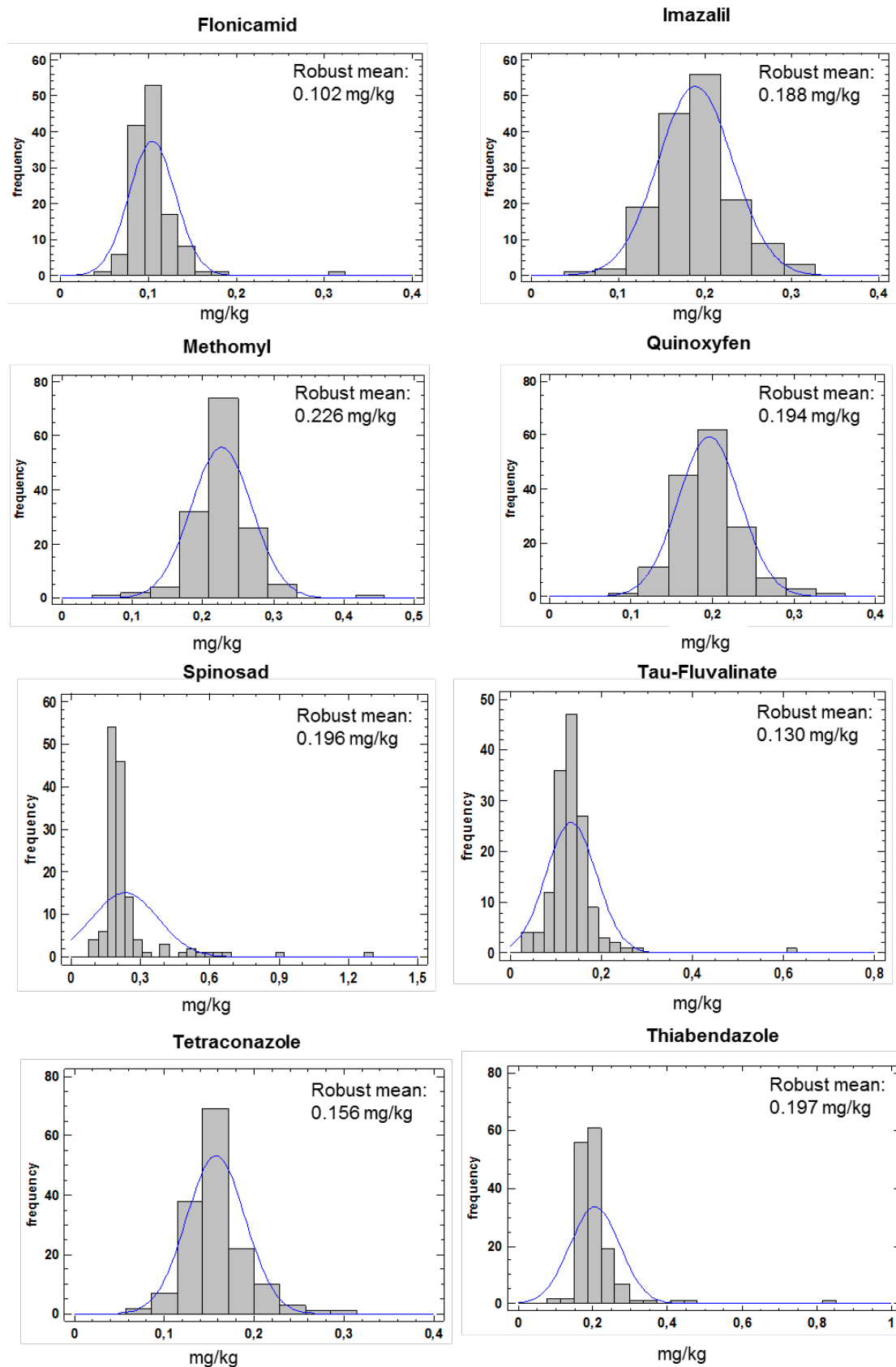
Flutianil (mg/kg)		Isofetamid (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.035	0.036	0.053	0.056
0.038	0.047	0.058	0.071
0.044	0.033	0.065	0.052
0.034	0.042	0.050	0.065
0.043	0.030	0.065	0.045
0.034	0.044	0.054	0.066
0.043	0.036	0.066	0.054
0.033	0.034	0.051	0.052
0.034	0.045	0.052	0.068
0.031	0.040	0.046	0.060

The sample numbers used for this test were: 13, 53, 92, 113, 173, 180, 182, 199, 227 and 233.

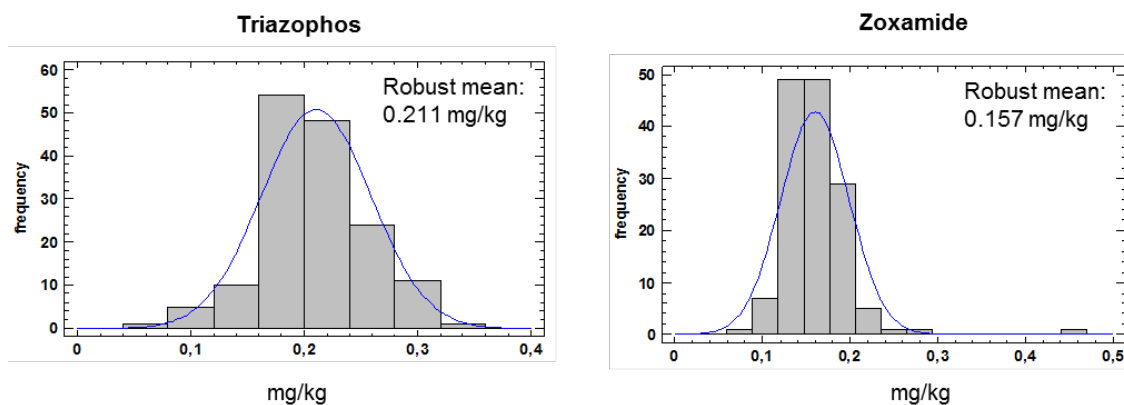
EU/EFTA results presented as histograms. The robust mean of spinosad corresponds to the assigned value, after removing the second mode.



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



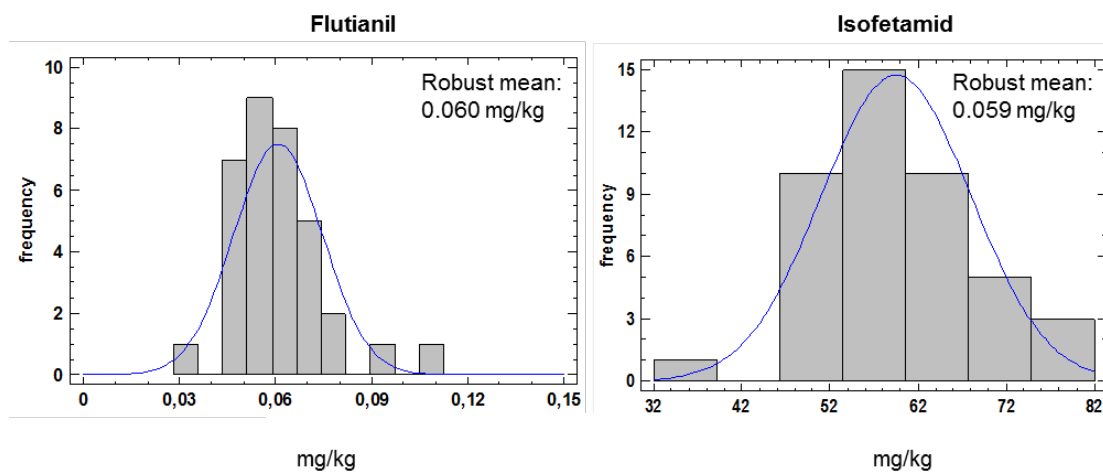
APPENDIX 2. Histograms of residue data for each pesticide from EU/EFTA laboratories.




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**Voluntary pesticides**

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

Results reported by the laboratories for the mandatory pesticides acetamiprid, chlorfenapyr, chlorpyrifos, clofentezine, diazinon, dimethoate, endosulfan sulfate, fenarimol, flonicamid, imazalil, methomyl, quinoxifen, spinosad, tau-fluvalinate, tetraconazole, thiabendazole, triazophos and zoxamide (mg/kg) and their calculated z score value using FFP-RSD 25 %

Lab Code	Acetamiprid	z score (FFP-RSD 25 %)			Chlorfenapyr	z score (FFP-RSD 25 %)			Chlorpyrifos	z score (FFP-RSD 25 %)			Clofentezine	z score (FFP-RSD 25 %)			Diazinon	z score (FFP-RSD 25 %)			Dimethoate	z score (FFP-RSD 25 %)			Endosulfan sulfate	z score (FFP-RSD 25 %)			Fenarimol	z score (FFP-RSD 25 %)			Flonicamid	z score (FFP-RSD 25 %)		
MRRL (mg/kg)	0.010				0.010				0.005				0.010				0.005				0.003				0.010				0.010				0.010			
Robust mean (mg/kg)	0.175				0.299				0.070				0.096				0.759				0.079				0.283				0.320				0.102			
5	0.194	0.4	0.274	-0.3	0.061	-0.5	0.045	-2.1	0.626	-0.7	0.074	-0.3	0.295	0.2	0.226	-1.2	0.106	0.2																		
7	0.184	0.2	0.275	-0.3	0.065	-0.3	0.070	-1.1	0.608	-0.8	0.078	0.0	0.311	0.4	0.250	-0.9	0.096	-0.2																		
9	0.137	-0.9	0.328	0.4	0.071	0.1	0.073	-1.0	0.714	-0.2	0.061	-0.9	0.222	-0.9	0.326	0.1	0.083	-0.7																		
11	0.173	0.0	0.315	0.2	0.072	0.1	0.095	0.0	0.764	0.0	0.073	-0.3	0.192	-1.3	0.322	0.0	0.094	-0.3																		
13	0.150	-0.6	0.316	0.2	0.075	0.3	0.044	-2.2	0.800	0.2	0.073	-0.3	0.270	-0.2	0.330	0.1	0.117	0.6																		
15	0.189	0.3	0.427	1.7	0.114	2.5	0.093	-0.1	1.410	3.4	0.090	0.5	0.292	0.1	0.369	0.6	0.107	0.2																		
17	0.200	0.6	0.242	-0.8	0.049	-1.2	0.104	0.3	0.683	-0.4	0.083	0.2	0.281	0.0	0.353	0.4	0.116	0.6																		
19	0.179	0.1	0.270	-0.4	0.061	-0.5	0.131	1.4	0.980	1.2	0.086	0.3	0.265	-0.3	0.464	1.8	0.107	0.2																		
21	0.165	-0.2	0.340	0.5	0.091	1.2	0.165	2.8	0.851	0.5	0.075	-0.2	0.275	-0.1	0.419	1.2	0.100	-0.1																		
23	No results reported																																			
25	0.174	0.0	0.338	0.5	0.059	-0.6	0.079	-0.7	0.693	-0.4	0.087	0.4	ND	-3.9	0.280	-0.5	0.113	0.4																		
27	0.166	-0.2	NA	NA	0.090	1.2	0.113	0.7	0.995	1.2	0.081	0.1	NA	NA	0.300	-0.2	NA	NA																		
29	0.181	0.1	0.287	-0.2	0.077	0.4	0.136	1.6	0.653	-0.6	0.071	-0.4	0.298	0.2	0.311	-0.1	0.092	-0.4																		
31	0.200	0.6	0.340	0.5	0.071	0.1	0.110	0.6	0.910	0.8	0.081	0.1	0.310	0.4	0.330	0.1	0.104	0.1																		
33	0.174	0.0	0.256	-0.6	0.058	-0.7	0.081	-0.6	0.762	0.0	0.068	-0.6	0.147	-1.9	0.298	-0.3	0.107	0.2																		
35	0.129	-1.1	0.189	-1.5	0.045	-1.4	NA	NA	0.510	-1.3	0.059	-1.0	0.201	-1.2	0.213	-1.3	NA	NA																		
36	0.174	0.0	0.344	0.6	0.082	0.7	0.122	1.1	0.841	0.4	0.082	0.2	0.333	0.7	0.446	1.6	0.097	-0.2																		
39	0.184	0.2	0.276	-0.3	0.051	-1.1	0.132	1.5	0.790	0.2	0.086	0.3	0.281	0.0	0.422	1.3	0.100	-0.1																		
41	0.140	-0.8	0.340	0.5	0.075	0.3	0.084	-0.5	0.580	-0.9	0.080	0.0	0.280	0.0	0.260	-0.7	0.095	-0.3																		
43	0.193	0.4	0.311	0.2	0.061	-0.5	0.090	-0.3	0.679	-0.4	0.090	0.5	0.286	0.0	0.337	0.2	0.103	0.0																		
45	0.170	-0.1	ND	-3.9	ND	-3.7	NA	NA	0.750	-0.1	0.110	1.5	ND	-3.9	0.390	0.9	NA	NA																		
47	0.162	-0.3	0.366	0.9	0.078	0.5	0.081	-0.6	1.078	1.7	0.075	-0.2	0.432	2.1	0.523	2.5	0.090	-0.5																		
49	0.230	1.3	0.311	0.2	0.062	-0.4	0.052	-1.8	0.680	-0.4	0.095	0.8	ND	-3.9	0.256	-0.8	0.138	1.4																		
51	0.185	0.2	0.244	-0.7	0.060	-0.6	0.082	-0.6	0.760	0.0	0.068	-0.6	0.295	0.2	0.244	-0.9	0.108	0.2																		
53	0.179	0.1	0.304	0.1	0.069	0.0	0.101	0.2	0.797	0.2	0.085	0.3	0.296	0.2	0.313	-0.1	0.097	-0.2																		
55	0.216	0.9	0.231	-0.9	0.056	-0.8	0.089	-0.3	0.637	-0.6	0.074	-0.3	0.222	-0.9	0.253	-0.8	0.125	0.9																		
57	0.197	0.5	0.340	0.5	0.092	1.3	0.113	0.7	0.902	0.8	0.087	0.4	0.302	0.3	0.360	0.5	0.121	0.8																		
59	0.172	-0.1	0.310	0.1	0.076	0.4	0.095	-0.1	0.705	-0.3	0.089	0.5	0.282	0.0	0.275	-0.6	0.107	0.2																		

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

Lab Code	Acetamiprid	z score (FFP-RSD 25 %)		Chlorfenvinphos	z score (FFP-RSD 25 %)		Chlorpyrifos	z score (FFP-RSD 25 %)		Clofentezine	z score (FFP-RSD 25 %)		Diazinon	z score (FFP-RSD 25 %)		Dimethoate	z score (FFP-RSD 25 %)		Endosulfan sulfate	z score (FFP-RSD 25 %)		Fenarimol	z score (FFP-RSD 25 %)		Flonicamid	z score (FFP-RSD 25 %)	
MRRL (mg/kg)	0.010			0.010			0.005			0.010			0.005			0.003			0.010			0.010			0.010		
Robust mean (mg/kg)	0.175			0.299			0.070			0.096			0.759			0.079			0.283			0.320			0.102		
61	0.200	0.6	0.394	1.3	0.105	2.0	0.123	1.1	1.040	1.5	0.086	0.3	0.315	0.4	0.421	1.3	0.118	0.6									
63	0.251	1.7	0.344	0.6	0.077	0.4	0.089	-0.3	0.924	0.9	0.085	0.3	0.821	5.0	0.435	1.4	0.121	0.8									
65	No results reported																										
67	0.153	-0.5	0.346	0.6	0.075	0.3	0.082	-0.6	0.723	-0.2	0.069	-0.5	0.356	1.0	0.427	1.3	0.095	-0.3									
69	0.150	-0.6	0.270	-0.4	0.054	-0.9	0.085	-0.5	0.480	-1.5	0.066	-0.7	NA	NA	0.230	-1.1	0.086	-0.6									
71	0.187	0.3	0.340	0.5	0.080	0.6	0.095	-0.1	0.939	0.9	0.091	0.6	0.183	-1.4	0.384	0.8	0.111	0.4									
73	0.174	0.0	0.260	-0.5	0.057	-0.7	0.079	-0.7	0.580	-0.9	0.072	-0.4	0.160	-1.7	0.259	-0.8	0.097	-0.2									
75	NA	NA	0.394	1.3	0.089	1.1	0.100	0.1	0.886	0.7	ND	-3.8	0.394	1.6	0.398	1.0	NA	NA									
77	0.172	-0.1	0.265	-0.5	0.070	0.0	0.085	-0.5	0.766	0.0	0.079	0.0	0.283	0.0	0.287	-0.4	0.101	0.0									
79	0.156	-0.4	0.311	0.2	0.077	0.4	0.125	1.2	0.933	0.9	0.072	-0.4	0.163	-1.7	0.357	0.5	0.073	-1.1									
81	0.158	-0.4	0.272	-0.4	0.067	-0.2	0.127	1.3	0.593	-0.9	0.075	-0.2	0.216	-1.0	0.268	-0.6	0.081	-0.8									
83	0.176	0.0	0.253	-0.6	0.052	-1.0	0.076	-0.8	0.659	-0.5	0.081	0.1	0.241	-0.6	0.250	-0.9	0.075	-1.1									
85	0.174	0.0	0.334	0.5	0.068	-0.1	0.105	0.4	0.709	-0.3	0.086	0.3	0.316	0.5	0.295	-0.3	0.108	0.2									
87	0.170	-0.1	0.281	-0.2	0.066	-0.2	0.117	0.9	0.721	-0.2	0.077	-0.1	0.314	0.4	0.315	-0.1	0.095	-0.3									
89	0.162	-0.3	0.285	-0.2	0.070	0.0	0.085	-0.5	1.328	3.0	0.078	-0.1	0.276	-0.1	0.490	2.1	0.106	0.2									
91	0.177	0.0	0.310	0.1	0.074	0.2	0.091	-0.2	0.763	0.0	0.074	-0.3	0.286	0.0	0.318	0.0	NA	NA									
93	0.210	0.8	0.350	0.7	0.072	0.1	0.090	-0.3	0.820	0.3	0.078	-0.1	0.230	-0.8	0.300	-0.2	0.114	0.5									
95	0.170	-0.1	0.350	0.7	0.094	1.4	0.110	0.6	0.660	-0.5	0.087	0.4	0.270	-0.2	0.270	-0.6	0.100	-0.1									
97	0.290	2.6	0.276	-0.3	0.076	0.4	0.072	-1.0	0.851	0.5	0.083	0.2	0.264	-0.3	0.308	-0.1	0.114	0.5									
99	0.152	-0.5	0.367	0.9	0.064	-0.3	0.084	-0.5	0.676	-0.4	0.084	0.2	0.322	0.5	0.278	-0.5	0.092	-0.4									
101	0.170	-0.1	ND	-3.9	0.077	0.4	0.068	-1.2	0.740	-0.1	0.083	0.2	0.320	0.5	0.290	-0.4	0.088	-0.5									
103	0.174	0.0	0.181	-1.6	0.065	-0.3	0.068	-1.2	0.631	-0.7	0.092	0.6	0.301	0.2	0.232	-1.1	0.086	-0.6									
105	ND	-3.8	0.243	-0.8	0.042	-1.6	ND	-3.6	0.581	-0.9	0.088	0.4	0.529	3.5	ND	-3.9	ND	-3.6									
107	NA	NA	0.314	0.2	0.070	0.0	NA	NA	0.887	0.7	NA	NA	0.314	0.4	NA	NA	NA	NA									
109	0.174	0.0	0.415	1.5	0.089	1.1	0.104	0.3	0.723	-0.2	0.076	-0.2	0.362	1.1	0.323	0.0	0.078	-0.9									
111	0.151	-0.5	0.327	0.4	0.038	-1.8	0.067	-1.2	0.902	0.8	0.076	-0.2	0.282	0.0	0.354	0.4	0.136	1.3									
113	0.177	0.0	0.266	-0.4	0.058	-0.7	0.104	0.3	0.552	-1.1	0.078	-0.1	0.264	-0.3	0.259	-0.8	0.090	-0.5									
115	0.096	-1.8	NA	NA	ND	-3.7	0.099	0.1	0.954	1.0	0.091	0.6	0.208	-1.1	NA	NA	NA	NA									
117	0.176	0.0	0.235	-0.9	0.054	-0.9	0.076	-0.8	0.553	-1.1	0.070	-0.5	0.243	-0.6	0.193	-1.6	0.088	-0.5									
119	0.159	-0.4	0.257	-0.6	0.068	-0.1	0.084	-0.5	0.451	-1.6	0.061	-0.9	0.239	-0.6	0.223	-1.2	0.087	-0.6									
121	0.145	-0.7	0.251	-0.6	0.065	-0.3	0.182	3.5	0.740	-0.1	0.080	0.0	0.250	-0.5	0.280	-0.5	0.120	0.7									

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

Lab Code	Acetamidrid	z score (FFP-RSD 25 %)			Chlorfenapyr			Chlorpyrifos			Clofentezine			Diazihon			Dimethoate			z score (FFP-RSD 25 %)			Endosulfan sulfate			z score (FFP-RSD 25 %)			Fenarimol			z score (FFP-RSD 25 %)			Flonicamid			z score (FFP-RSD 25 %)		
MRRL (mg/kg)	0.010	z score (FFP-RSD 25 %)			0.010			0.005			0.010			0.005			0.003			z score (FFP-RSD 25 %)			0.010			z score (FFP-RSD 25 %)			0.010			z score (FFP-RSD 25 %)			0.010			z score (FFP-RSD 25 %)		
Robust mean (mg/kg)	0.175	0.299			0.070			0.096			0.759			0.079			0.283			0.320			0.102			0.102			0.102			0.102			0.102					
123	0.071	-2.4	0.069	-3.1	0.038	-1.8	0.036	-2.5	0.287	-2.5	0.039	-2.0	0.048	-3.3	0.057	-3.3	0.060	-1.6																						
125	0.170	-0.1	0.310	0.1	0.067	-0.2	0.087	-0.4	0.930	0.9	0.076	-0.2	0.330	0.7	0.320	0.0	0.088	-0.5																						
127	0.191	0.4	0.292	-0.1	0.078	0.4	0.102	0.2	0.879	0.6	0.090	0.5	ND	-3.9	0.391	0.9	0.106	0.2																						
129	0.210	0.8	0.302	0.0	0.067	-0.2	0.037	-2.5	0.683	-0.4	0.076	-0.2	0.288	0.1	0.315	-0.1	0.095	-0.3																						
131	0.175	0.0	0.254	-0.6	0.047	-1.3	0.091	-0.2	0.686	-0.4	0.074	-0.3	0.256	-0.4	0.290	-0.4	0.103	0.0																						
133	0.174	0.0	0.132	-2.2	0.090	1.2	0.087	-0.4	0.742	-0.1	0.067	-0.6	0.374	1.3	0.409	1.1	NA	NA																						
135	0.170	-0.1	0.329	0.4	0.086	0.9	0.126	1.2	0.889	0.7	0.084	0.2	0.357	1.0	0.474	1.9	0.102	0.0																						
137	0.149	-0.6	0.233	-0.9	0.050	-1.1	0.060	-1.5	0.438	-1.7	0.070	-0.5	0.309	0.4	0.214	-1.3	0.090	-0.5																						
139	NA	NA	0.336	0.5	0.077	0.4	NA	NA	0.808	0.3	0.064	-0.8	0.330	0.7	0.408	1.1	NA	NA																						
141	0.134	-0.9	0.208	-1.2	0.053	-1.0	NA	NA	0.648	-0.6	0.080	0.0	0.197	-1.2	0.283	-0.5	NA	NA																						
143	0.148	-0.6	0.368	0.9	0.980	5.0	0.105	0.4	1.090	1.7	0.055	-1.2	0.186	-1.4	0.349	0.4	0.090	-0.5																						
145	No results reported																																							
147	0.183	0.2	0.335	0.5	0.067	-0.2	0.113	0.7	0.881	0.6	0.088	0.4	0.307	0.3	0.324	0.1	0.098	-0.2																						
149	0.183	0.2	0.253	-0.6	0.079	0.5	0.107	0.4	0.854	0.5	0.090	0.5	0.297	0.2	0.326	0.1	0.114	0.5																						
151	0.170	-0.1	0.300	0.0	0.059	-0.6	0.079	-0.7	0.640	-0.6	0.082	0.1	0.200	-1.2	0.250	-0.9	0.096	-0.2																						
153	0.240	1.5	0.370	0.9	0.067	-0.2	NA	NA	0.470	-1.5	0.083	0.2	0.370	1.2	0.310	-0.1	0.140	1.5																						
157	0.163	-0.3	0.272	-0.4	0.067	-0.2	0.096	0.0	0.798	0.2	0.074	-0.3	0.314	0.4	0.293	-0.3	0.095	-0.3																						
159	0.162	-0.3	0.299	0.0	0.066	-0.2	0.105	0.4	0.744	-0.1	0.077	-0.1	0.305	0.3	0.313	-0.1	0.178	3.0																						
161	0.144	-0.7	0.269	-0.4	0.058	-0.7	0.072	-1.0	0.710	-0.3	0.071	-0.4	0.277	-0.1	0.203	-1.5	0.093	-0.3																						
163	0.193	0.4	0.310	0.1	0.070	0.0	0.025	-3.0	0.663	-0.5	0.084	0.2	0.324	0.6	0.297	-0.3	0.101	0.0																						
165	0.167	-0.2	NA	NA	0.076	0.4	0.114	0.7	0.980	1.2	0.072	-0.4	NA	NA	0.346	0.3	NA	NA																						
167	0.202	0.6	0.275	-0.3	0.065	-0.3	0.085	-0.5	0.667	-0.5	0.083	0.2	0.285	0.0	0.261	-0.7	0.116	0.6																						
169	0.184	0.2	0.317	0.2	0.070	0.0	0.098	0.1	0.742	-0.1	0.087	0.4	0.367	1.2	0.292	-0.3	0.115	0.5																						
171	0.170	-0.1	0.247	-0.7	0.063	-0.4	0.075	-0.9	0.713	-0.2	0.062	-0.9	0.291	0.1	0.317	0.0	0.094	-0.3																						
173	0.160	-0.3	NA	NA	0.070	0.0	0.120	1.0	0.780	0.1	0.090	0.5	0.220	-0.9	0.340	0.3	0.110	0.3																						
175	0.160	-0.3	0.240	-0.8	0.050	-1.1	NA	NA	0.330	-2.3	0.090	0.5	0.220	-0.9	0.210	-1.4	NA	NA																						
177	0.160	-0.3	0.360	0.8	0.078	0.5	0.093	-0.1	0.700	-0.3	0.088	0.4	0.270	-0.2	0.310	-0.1	NA	NA																						
179	0.153	-0.5	0.265	-0.5	0.062	-0.4	0.083	-0.6	0.682	-0.4	0.077	-0.1	0.247	-0.5	0.281	-0.5	0.098	-0.2																						
181	0.175	0.0	0.352	0.7	0.086	0.9	0.116	0.8	0.813	0.3	0.088	0.4	0.284	0.0	0.519	2.5	0.313	5.0																						
183	0.173	0.0	ND	-3.9	ND	-3.7	0.166	2.9	0.796	0.2	0.044	-1.8	ND	-3.9	ND	-3.9	ND	-3.6																						
185	0.190	0.3	0.220	-1.1	0.079	0.5	0.110	0.6	0.840	0.4	0.085	0.3	0.240	-0.6	0.410	1.1	0.097	-0.2																						

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

Lab Code	Acetamidiprid	z score (FFP-RSD 25 %)	Chlorfenapyr	z score (FFP-RSD 25 %)	Chlorpyrifos	z score (FFP-RSD 25 %)	Clofentezine	z score (FFP-RSD 25 %)	Diazinon	z score (FFP-RSD 25 %)	Dimethoate	z score (FFP-RSD 25 %)	Endosulfan sulfate	z score (FFP-RSD 25 %)	Fenarimol	z score (FFP-RSD 25 %)	Flonicamid	z score (FFP-RSD 25 %)
	MRRL (mg/kg)		0.010		0.010		0.005		0.010		0.005		0.003		0.010		0.010	
	Robust mean (mg/kg)		0.175		0.299		0.070		0.096		0.759		0.079		0.283		0.320	
187	0.160	-0.3	0.284	-0.2	0.058	-0.7	0.093	-0.1	0.562	-1.0	0.081	0.1	0.184	-1.4	0.257	-0.8	0.090	-0.5
188	0.187	0.3	0.250	-0.7	0.062	-0.4	NA	NA	0.690	-0.4	0.072	-0.4	0.230	-0.8	0.260	-0.7	0.093	-0.3
191	NA	NA	0.257	-0.6	0.064	-0.3	NA	NA	0.744	-0.1	0.104	1.2	0.296	0.2	NA	NA	NA	NA
193	0.179	0.1	0.281	-0.2	0.080	0.6	0.116	0.8	0.719	-0.2	0.072	-0.4	0.336	0.7	0.279	-0.5	0.098	-0.2
195	0.171	-0.1	0.201	-1.3	0.057	-0.7	0.095	-0.1	0.623	-0.7	0.079	0.0	0.291	0.1	0.243	-1.0	NA	NA
197	0.140	-0.8	0.310	0.1	0.078	0.5	0.090	-0.3	0.940	1.0	0.089	0.5	0.370	1.2	0.430	1.4	0.076	-1.0
199	0.170	-0.1	0.340	0.5	0.064	-0.3	0.130	1.4	0.920	0.8	0.094	0.7	0.320	0.5	0.300	-0.2	0.110	0.3
201	0.129	-1.1	0.259	-0.5	0.061	-0.5	0.082	-0.6	0.636	-0.7	0.076	-0.2	0.253	-0.4	0.223	-1.2	0.106	0.2
203	0.140	-0.8	0.230	-0.9	0.061	-0.5	0.091	-0.2	0.520	-1.3	0.054	-1.3	0.230	-0.8	0.200	-1.5	0.082	-0.8
205	0.200	0.6	0.370	0.9	0.066	-0.2	0.096	0.0	0.680	-0.4	0.077	-0.1	0.260	-0.3	0.460	1.8	0.098	-0.2
207	0.149	-0.6	0.306	0.1	0.067	-0.1	0.084	-0.5	0.708	-0.3	0.070	-0.5	0.299	0.2	0.283	-0.5	0.091	-0.4
209	0.146	-0.7	0.301	0.0	0.062	-0.5	0.078	-0.8	0.667	-0.5	0.076	-0.1	0.324	0.6	0.317	0.0	0.087	-0.6
211	0.154	-0.5	0.282	-0.2	0.067	-0.2	0.078	-0.8	0.708	-0.3	0.093	0.7	0.301	0.2	0.290	-0.4	ND	-3.6
213	0.205	0.7	0.454	2.1	0.088	1.0	0.079	-0.7	0.801	0.2	0.080	0.0	0.437	2.2	0.277	-0.5	0.080	-0.9
215	0.140	-0.8	0.408	1.5	0.091	1.2	0.074	-0.9	0.775	0.1	0.063	-0.8	0.399	1.6	0.362	0.5	0.083	-0.7
217	0.179	0.1	0.304	0.1	0.104	2.0	0.128	1.3	0.730	-0.2	0.100	1.0	0.373	1.3	0.364	0.6	0.117	0.6
219	0.190	0.3	0.295	-0.1	0.065	-0.3	0.072	-1.0	0.808	0.3	0.107	1.4	0.298	0.2	0.349	0.4	0.115	0.5
221	0.156	-0.4	0.290	-0.1	0.066	-0.2	0.100	0.1	0.706	-0.3	0.105	1.3	0.301	0.2	0.223	-1.2	0.107	0.2
223	0.216	0.9	0.293	-0.1	0.083	0.8	0.104	0.3	0.752	0.0	0.089	0.5	0.290	0.1	0.381	0.8	0.101	0.0
225	0.170	-0.1	0.346	0.6	0.073	0.2	0.104	0.3	0.752	0.0	0.075	-0.2	0.353	1.0	0.267	-0.7	0.093	-0.3
227	0.140	-0.8	0.260	-0.5	0.060	-0.6	0.090	-0.3	0.590	-0.9	0.030	-2.5	0.270	-0.2	0.310	-0.1	NA	NA
229	NA	NA	0.330	0.4	0.078	0.5	NA	NA	0.742	-0.1	NA	NA	0.314	0.4	0.326	0.1	NA	NA
231	No results reported																	
233	0.160	-0.3	0.267	-0.4	0.067	-0.2	0.107	0.4	0.652	-0.6	0.069	-0.5	0.302	0.3	0.419	1.2	0.079	-0.9
235	0.157	-0.4	0.209	-1.2	0.057	-0.7	0.105	0.4	0.481	-1.5	0.085	0.3	0.192	-1.3	0.181	-1.7	0.102	0.0
237	0.174	0.0	0.377	1.0	0.084	0.8	0.110	0.6	0.891	0.7	0.076	-0.2	0.411	1.8	0.354	0.4	0.044	-2.3
239	0.166	-0.2	0.356	0.8	0.083	0.8	0.130	1.4	0.847	0.5	0.068	-0.6	0.394	1.6	0.391	0.9	0.090	-0.5
241	0.214	0.9	0.324	0.3	0.076	0.4	0.102	0.2	0.784	0.1	0.122	2.1	0.378	1.3	0.408	1.1	0.142	1.6
243	0.178	0.1	0.313	0.2	0.079	0.5	0.075	-0.9	0.778	0.1	0.078	-0.1	0.089	-2.7	0.251	-0.9	ND	-3.6
245	0.182	0.2	0.337	0.5	0.081	0.6	0.055	-1.7	0.758	0.0	0.080	0.0	0.272	-0.2	0.295	-0.3	0.102	0.0
247	NA	NA	NA	NA	0.060	-0.6	NA	NA	0.635	-0.7	NA	NA	0.468	2.6	NA	NA	NA	NA



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

Lab Code	Acetamiprid	z score (FFP-RSD 25 %)			Chlorfenapyr	z score (FFP-RSD 25 %)			Chlorpyrifos	z score (FFP-RSD 25 %)			Clofentezine	z score (FFP-RSD 25 %)			Diazinon	z score (FFP-RSD 25 %)			Dimethoate	z score (FFP-RSD 25 %)			Endosulfan sulfate	z score (FFP-RSD 25 %)			Fenarimol	z score (FFP-RSD 25 %)			Flonicamid	z score (FFP-RSD 25 %)		
MRRL (mg/kg)	0.010				0.010				0.005				0.010				0.005				0.003				0.010				0.010				0.010			
Robust mean (mg/kg)	0.175				0.299				0.070				0.096				0.759				0.079				0.283				0.320				0.102			
249	0.166	-0.2	0.313	0.2	0.071	0.1	0.095	-0.1	0.850	0.5	0.061	-0.9	0.273	-0.1	0.311	-0.1	0.097	-0.2																		
251	0.180	0.1	0.360	0.8	0.080	0.6	0.088	-0.4	0.860	0.5	0.085	0.3	0.280	0.0	0.310	-0.1	0.100	-0.1																		
253	0.210	0.8	0.301	0.0	0.058	-0.7	NA	NA	0.724	-0.2	0.101	1.1	0.733	5.0	0.258	-0.8	NA	NA																		
255	0.169	-0.1	0.318	0.2	0.058	-0.7	0.025	-3.0	0.607	-0.8	0.077	-0.1	0.293	0.1	0.199	-1.5	0.094	-0.3																		
257	0.150	-0.6	0.300	0.0	0.080	0.6	NA	NA	0.750	-0.1	0.060	-1.0	0.320	0.5	0.330	0.1	0.080	-0.9																		
259	0.155	-0.5	0.256	-0.6	0.062	-0.4	0.067	-1.2	0.837	0.4	0.051	-1.4	0.240	-0.6	0.354	0.4	0.095	-0.3																		
261	0.182	0.2	0.384	1.1	0.072	0.1	0.086	-0.4	0.709	-0.3	0.071	-0.4	0.280	0.0	0.255	-0.8	0.091	-0.4																		
263	0.187	0.3	0.312	0.2	0.088	1.0	0.110	0.6	0.796	0.2	0.079	0.0	0.217	-0.9	0.285	-0.4	0.090	-0.5																		
265	0.233	1.3	0.264	-0.5	0.055	-0.9	0.116	0.8	0.707	-0.3	0.087	0.4	0.115	-2.4	0.276	-0.5	0.134	1.3																		
267	0.156	-0.4	0.314	0.2	0.071	0.0	0.089	-0.3	0.667	-0.5	0.080	0.0	0.241	-0.6	0.339	0.2	0.090	-0.5																		
269	No results reported																																			
271	0.189	0.3	0.312	0.2	0.073	0.2	0.115	0.8	0.670	-0.5	0.083	0.2	ND	-3.9	0.281	-0.5	0.109	0.3																		
273	NA	NA	0.306	0.1	0.056	-0.8	NA	NA	0.646	-0.6	0.070	-0.5	0.210	-1.0	0.281	-0.5	NA	NA																		
275	0.160	-0.3	0.320	0.3	0.070	0.0	0.100	0.1	1.010	1.3	0.070	-0.5	0.340	0.8	0.260	-0.7	0.080	-0.9																		
277	No results reported																																			
279	0.170	-0.1	0.350	0.7	0.096	1.5	0.084	-0.5	0.650	-0.6	0.070	-0.5	0.300	0.2	0.360	0.5	0.110	0.3																		
281	0.198	0.5	0.329	0.4	0.078	0.5	0.116	0.8	0.855	0.5	0.095	0.8	0.317	0.5	0.373	0.7	0.119	0.7																		
283	No results reported																																			
285	0.176	0.0	0.255	-0.6	0.065	-0.3	0.087	-0.4	0.748	-0.1	0.075	-0.2	0.253	-0.4	0.293	-0.3	0.097	-0.2																		
287	ND	-3.8	0.254	-0.6	0.072	0.1	ND	-3.6	0.909	0.8	0.078	-0.1	0.269	-0.2	0.266	-0.7	NA	NA																		
289	0.124	-1.2	0.236	-0.8	0.050	-1.1	0.092	-0.2	0.560	-1.1	0.041	-1.9	0.180	-1.5	0.328	0.1	0.067	-1.4																		
291	0.162	-0.3	0.382	1.1	0.083	0.8	0.113	0.7	0.912	0.8	0.065	-0.7	0.388	1.5	NA	NA	NA	NA																		
293	0.264	2.0	0.245	-0.7	0.105	2.0	0.149	2.2	0.915	0.8	0.138	3.0	0.370	1.2	0.411	1.1	0.110	0.3																		
295	NA	NA	0.305	0.1	0.073	0.2	NA	NA	0.448	-1.6	0.186	5.0	0.251	-0.5	0.378	0.7	NA	NA																		
297	0.213	0.9	0.377	1.0	0.087	1.0	0.170	3.0	0.825	0.3	0.107	1.4	0.395	1.6	0.484	2.1	0.122	0.8																		
299	0.148	-0.6	0.297	0.0	0.077	0.4	0.099	0.1	0.740	-0.1	0.059	-1.0	0.268	-0.2	0.449	1.6	0.076	-1.0																		
301	0.197	0.5	NA	NA	0.038	-1.8	NA	NA	0.675	-0.4	0.101	1.1	0.275	-0.1	0.246	-0.9	NA	NA																		
303	0.170	-0.1	0.287	-0.2	0.080	0.6	0.092	-0.2	0.919	0.8	0.088	0.4	0.282	0.0	0.385	0.8	0.106	0.2																		
305	0.170	-0.1	0.320	0.3	0.066	-0.2	0.085	-0.5	0.680	-0.4	0.077	-0.1	0.230	-0.8	0.290	-0.4	0.094	-0.3																		
307	0.163	-0.3	0.075	-3.0	0.035	-2.0	NA	NA	0.554	-1.1	0.070	-0.5	0.110	-2.4	0.225	-1.2	NA	NA																		
309	0.163	-0.3	0.490	2.5	0.127	3.3	0.109	0.5	0.744	-0.1	0.067	-0.6	0.453	2.4	0.304	-0.2	0.091	-0.4																		

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

Lab Code	Acetamiprid	z score (FFP-RSD 25 %)		Chlorfenapyr	z score (FFP-RSD 25 %)		Chlorpyrifos	z score (FFP-RSD 25 %)		Clofentezine	z score (FFP-RSD 25 %)		Diazinon	z score (FFP-RSD 25 %)		Dimethoate	z score (FFP-RSD 25 %)		Endosulfan sulfate	z score (FFP-RSD 25 %)		Fenarimol	z score (FFP-RSD 25 %)		Flonicamid	z score (FFP-RSD 25 %)	
MRRL (mg/kg)	0.010			0.010			0.005			0.010			0.005			0.003			0.010			0.010			0.010		
Robust mean (mg/kg)	0.175			0.299			0.070			0.096			0.759			0.079			0.283			0.320			0.102		
311	0.170	-0.1	0.280	-0.3	0.061	-0.5	0.074	-0.9	0.680	-0.4	0.073	-0.3	0.170	-1.6	0.270	-0.6	0.079	-0.9									
313	0.175	0.0	0.141	-2.1	0.038	-1.8	NA	NA	0.654	-0.6	0.056	-1.2	0.155	-1.8	0.159	-2.0	0.134	1.3									
315	0.229	1.2	0.385	1.1	0.086	0.9	0.123	1.1	0.893	0.7	0.095	0.8	0.251	-0.5	0.476	2.0	0.127	1.0									
317	0.172	-0.1	0.145	-2.1	0.037	-1.9	0.061	-1.5	0.880	0.6	0.079	0.0	0.165	-1.7	0.226	-1.2	0.105	0.1									
319	0.158	-0.4	0.283	-0.2	0.076	0.4	0.104	0.3	0.767	0.0	0.078	-0.1	0.318	0.5	0.352	0.4	0.096	-0.2									
321	0.198	0.5	0.309	0.1	0.038	-1.8	0.079	-0.7	0.495	-1.4	0.106	1.3	0.211	-1.0	0.261	-0.7	ND	-3.6									
323	0.205	0.7	0.230	-0.9	0.050	-1.1	0.048	-2.0	0.620	-0.7	0.085	0.3	0.224	-0.8	0.200	-1.5	0.115	0.5									
325	0.183	0.2	0.381	1.1	0.066	-0.2	0.077	-0.8	0.860	0.5	0.090	0.5	0.266	-0.2	0.263	-0.7	0.089	-0.5									
327	0.199	0.5	0.273	-0.4	0.067	-0.2	0.066	-1.3	0.705	-0.3	0.069	-0.5	0.230	-0.8	0.268	-0.6	0.113	0.4									
329	0.220	1.0	0.208	-1.2	0.060	-0.6	0.098	0.1	0.609	-0.8	0.095	0.8	0.220	-0.9	0.306	-0.2	0.103	0.1									
331	0.140	-0.8	0.330	0.4	0.100	1.7	0.160	2.6	1.100	1.8	0.077	-0.1	0.270	-0.2	0.750	5.0	0.090	-0.5									
333	0.245	1.6	0.228	-1.0	0.054	-0.9	0.135	1.6	0.584	-0.9	0.070	-0.5	0.304	0.3	0.371	0.6	0.171	2.7									
335	0.192	0.4	0.273	-0.4	0.070	0.0	0.078	-0.8	0.853	0.5	0.067	-0.6	0.373	1.3	0.410	1.1	0.104	0.1									
337	0.198	0.5	0.370	0.9	0.078	0.5	0.088	-0.4	0.742	-0.1	0.078	-0.1	0.327	0.6	0.345	0.3	0.107	0.2									
339	0.193	0.4	0.235	-0.9	0.058	-0.7	0.095	-0.1	0.500	-1.4	0.080	0.0	0.197	-1.2	0.234	-1.1	0.092	-0.4									
341	0.172	-0.1	0.151	-2.0	0.042	-1.6	0.104	0.3	0.376	-2.0	0.079	0.0	0.194	-1.3	0.261	-0.7	0.080	-0.9									
343	ND	-3.8	0.470	2.3	0.110	2.3	ND	-3.6	0.960	1.1	0.061	-0.9	0.460	2.5	0.500	2.3	ND	-3.6									
345	No results reported																										
347	0.169	-0.1	NA	NA	0.024	-2.6	0.069	-1.1	NA	NA	0.100	1.0	0.179	-1.5	0.230	-1.1	NA	NA									
349	0.179	0.1	0.284	-0.2	0.057	-0.7	0.081	-0.6	0.703	-0.3	0.083	0.2	0.287	0.1	0.285	-0.4	0.095	-0.3									
351	0.220	1.0	ND	-3.9	0.074	0.2	0.134	1.6	0.909	0.8	0.098	0.9	NA	NA	0.402	1.0	0.135	1.3									
353	0.205	0.7	0.255	-0.6	0.060	-0.6	0.110	0.6	0.740	-0.1	0.080	0.0	0.270	-0.2	0.270	-0.6	0.130	1.1									
355	0.184	0.2	0.262	-0.5	0.062	-0.4	0.101	0.2	0.710	-0.3	0.071	-0.4	0.279	-0.1	0.356	0.5	0.134	1.3									
357	0.206	0.7	0.250	-0.7	0.064	-0.3	0.111	0.6	0.890	0.7	0.094	0.7	0.160	-1.7	0.340	0.3	0.130	1.1									
359	0.224	1.1	0.246	-0.7	0.039	-1.8	ND	-3.6	0.803	0.2	0.077	-0.1	0.365	1.2	0.381	0.8	0.129	1.1									
361	0.185	0.2	0.380	1.1	0.083	0.8	0.095	-0.1	0.750	-0.1	0.070	-0.5	0.320	0.5	0.330	0.1	0.106	0.2									
363	0.180	0.1	0.290	-0.1	0.077	0.4	0.110	0.6	0.890	0.7	0.087	0.4	0.300	0.2	0.320	0.0	0.110	0.3									
365	No results reported																										
367	0.160	-0.3	ND	-3.9	0.072	0.1	0.100	0.1	0.630	-0.7	0.090	0.5	ND	-3.9	0.330	0.1	0.110	0.3									
368	0.168	-0.2	0.300	0.0	0.064	-0.3	0.114	0.7	0.695	-0.3	0.072	-0.4	0.276	-0.1	0.320	0.0	0.098	-0.2									

NA: Not analysed

ND: Not detected (False negative)

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

Lab Code	Imazali	z score (FFP-RSD 25 %)			Methomyl			Quinoxifen			Spinosad			Tau-Fluvalinate			Tetraconazole			Thiabendazole			Triazophos			Zoxamide		
		MRRL (mg/kg)	0.005	z score (FFP-RSD 25 %)	0.01	z score (FFP-RSD 25 %)	0.010	z score (FFP-RSD 25 %)	0.010	z score (FFP-RSD 25 %)	0.010	z score (FFP-RSD 25 %)	0.010	z score (FFP-RSD 25 %)	0.010	z score (FFP-RSD 25 %)	0.010	z score (FFP-RSD 25 %)	0.005	z score (FFP-RSD 25 %)	0.010	z score (FFP-RSD 25 %)	0.010					
		Robust mean (mg/kg)	0.188		0.226		0.194		0.196		0.130		0.156		0.197		0.211		0.157									
5	0.172	-0.3	0.231	0.1	0.173	-0.4	0.17	-0.5	0.159	0.9	0.13	-0.7	0.148	-1.0	0.218	0.1	0.158	0.0										
7	0.172	-0.3	0.254	0.5	0.183	-0.2	0.206	0.2	0.157	0.8	0.144	-0.3	0.146	-1.0	0.217	0.1	0.161	0.1										
9	0.164	-0.5	0.198	-0.5	0.198	0.1	0.22	0.5	0.167	1.1	0.164	0.2	0.181	-0.3	0.203	-0.1	0.174	0.4										
11	0.171	-0.4	0.228	0.0	0.196	0.0	0.203	0.1	0.147	0.5	0.153	-0.1	0.183	-0.3	0.233	0.4	0.154	-0.1										
13	0.23	0.9	0.451	4.0	0.198	0.1	0.173	-0.5	0.1	-0.9	0.15	-0.1	0.2	0.1	0.2	-0.2	0.181	0.6										
15	0.22	0.7	0.238	0.2	0.29	2.0	0.24	0.9	0.128	-0.1	0.157	0.0	0.239	0.9	0.265	1.0	0.126	-0.8										
17	0.175	-0.3	0.222	-0.1	0.149	-0.9	0.084	-2.3	0.112	-0.5	0.195	1.0	0.246	1.0	0.182	-0.5	NA	NA										
19	0.203	0.3	0.248	0.4	0.172	-0.4	0.222	0.5	0.143	0.4	0.207	1.3	0.205	0.2	0.269	1.1	0.186	0.7										
21	0.256	1.5	0.226	0.0	0.251	1.2	0.2	0.1	0.163	1.0	0.202	1.2	0.195	0.0	0.296	1.6	0.19	0.8										
23	No results reported																											
25	0.177	-0.2	0.183	-0.8	0.177	-0.3	0.202	0.1	0.151	0.7	0.15	-0.1	0.191	-0.1	0.222	0.2	0.167	0.3										
27	0.175	-0.3	0.195	-0.6	0.275	1.7	0.207	0.2	NA	NA	0.15	-0.1	0.22	0.5	0.21	0.0	NA	NA										
29	0.265	1.7	0.202	-0.4	0.211	0.4	NA	NA	0.126	-0.1	0.145	-0.3	0.177	-0.4	0.189	-0.4	0.172	0.4										
31	0.2	0.3	0.25	0.4	0.18	-0.3	0.21	0.3	0.107	-0.7	0.178	0.6	0.2	0.1	0.19	-0.4	0.18	0.6										
33	0.213	0.5	0.197	-0.5	0.173	-0.4	0.184	-0.2	0.064	-2.0	0.161	0.1	0.223	0.5	0.178	-0.6	NA	NA										
35	0.14	-1.0	0.169	-1.0	0.13	-1.3	1.294	5.0	0.078	-1.6	0.128	-0.7	0.414	4.4	0.143	-1.3	NA	NA										
36	0.17	-0.4	0.229	0.0	0.221	0.6	0.25	1.1	0.149	0.6	0.194	1.0	0.196	0.0	0.236	0.5	0.175	0.5										
39	0.177	-0.2	0.223	-0.1	0.207	0.3	0.184	-0.2	0.1	-0.9	0.146	-0.3	0.185	-0.2	0.185	-0.5	0.103	-1.4										
41	0.22	0.7	0.21	-0.3	0.16	-0.7	0.18	-0.3	0.14	0.3	0.13	-0.7	0.2	0.1	0.18	-0.6	0.13	-0.7										
43	0.222	0.7	0.245	0.3	0.183	-0.2	0.154	-0.9	0.14	0.3	0.152	-0.1	0.226	0.6	0.214	0.1	0.162	0.1										
45	0.21	0.5	0.24	0.2	0.21	0.3	ND	-3.8	ND	-3.7	ND	-3.7	0.23	0.7	ND	-3.9	0.47	5.0										
47	0.147	-0.9	0.256	0.5	0.248	1.1	0.172	-0.5	0.147	0.5	0.188	0.8	0.186	-0.2	0.249	0.7	0.139	-0.5										
49	0.235	1.0	0.288	1.1	0.197	0.1	0.174	-0.5	0.136	0.2	0.14	-0.4	0.193	-0.1	0.251	0.8	0.182	0.6										
51	0.206	0.4	0.242	0.3	0.175	-0.4	0.214	0.4	0.279	4.6	0.141	-0.4	0.222	0.5	0.208	-0.1	0.149	-0.2										
53	0.185	-0.1	0.216	-0.2	0.189	-0.1	0.205	0.2	0.127	-0.1	0.147	-0.2	0.188	-0.2	0.24	0.6	0.123	-0.9										
55	0.212	0.5	0.266	0.7	0.144	-1.0	0.211	0.3	0.102	-0.9	0.125	-0.8	0.218	0.4	0.173	-0.7	0.132	-0.6										
57	0.207	0.4	0.26	0.6	0.193	0.0	0.218	0.4	0.097	-1.0	0.174	0.5	0.216	0.4	0.212	0.0	0.172	0.4										
59	0.275	1.9	0.249	0.4	0.193	0.0	0.529	5.0	0.118	-0.4	0.173	0.4	0.194	-0.1	0.177	-0.6	0.132	-0.6										
61	0.197	0.2	0.235	0.2	0.252	1.2	0.24	0.9	0.149	0.6	0.206	1.3	0.201	0.1	0.24	0.6	0.213	1.4										
63	0.173	-0.3	0.306	1.4	0.246	1.1	0.236	0.8	0.179	1.5	0.158	0.1	0.185	-0.2	0.21	0.0	0.118	-1.0										
65	No results reported																											
67	0.221	0.7	0.18	-0.8	0.202	0.2	0.177	-0.4	0.16	0.9	0.162	0.2	0.169	-0.6	0.19	-0.4	0.156	0.0										

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

Lab Code	Imazali	z score (FFP-RSD 25 %)		Methomyl	z score (FFP-RSD 25 %)		Quinoxifen	z score (FFP-RSD 25 %)		Spinosad	z score (FFP-RSD 25 %)		Tau-Fluvalinate	z score (FFP-RSD 25 %)		Tetraconazole	z score (FFP-RSD 25 %)		Thiabendazole	z score (FFP-RSD 25 %)		Triazophos	z score (FFP-RSD 25 %)		Zoxamide	z score (FFP-RSD 25 %)	
MRRL (mg/kg)	0.005			0.01			0.010			0.010			0.010			0.010			0.010			0.005			0.010		
Robust mean (mg/kg)	0.188			0.226			0.194			0.196			0.130			0.156			0.197			0.211			0.157		
69	0.14	-1.0	0.18	-0.8	0.14	-1.1	0.17	-0.5	0.1	-0.9	0.098	-1.5	0.16	-0.8	0.12	-1.7	0.12	-0.9									
71	0.179	-0.2	0.283	1.0	0.187	-0.1	0.202	0.1	0.194	2.0	0.166	0.3	0.205	0.2	0.261	1.0	0.188	0.8									
73	0.13	-1.2	0.23	0.1	0.14	-1.1	0.159	-0.8	0.152	0.7	0.132	-0.6	0.16	-0.8	0.162	-0.9	0.12	-0.9									
75	0.171	-0.4	NA	NA	0.227	0.7	NA	NA	0.05	-2.5	0.156	0.0	0.17	-0.5	0.262	1.0	0.185	0.7									
77	0.202	0.3	0.223	-0.1	0.186	-0.2	0.172	-0.5	0.114	-0.5	0.15	-0.1	0.161	-0.7	0.234	0.4	0.16	0.1									
79	0.172	-0.3	0.177	-0.9	0.224	0.6	0.18	-0.3	0.131	0.0	0.149	-0.2	0.177	-0.4	0.278	1.3	0.202	1.2									
81	0.164	-0.5	0.203	-0.4	0.172	-0.4	0.168	-0.6	ND	-3.7	0.134	-0.6	0.151	-0.9	0.204	-0.1	0.145	-0.3									
83	0.226	0.8	0.231	0.1	0.155	-0.8	0.155	-0.8	0.077	-1.6	0.132	-0.6	0.203	0.1	0.201	-0.2	0.131	-0.7									
85	0.185	-0.1	0.283	1.0	0.234	0.8	0.228	0.7	0.159	0.9	0.148	-0.2	0.231	0.7	0.216	0.1	0.181	0.6									
87	0.172	-0.3	0.223	-0.1	0.186	-0.2	0.194	0.0	0.117	-0.4	0.147	-0.2	0.188	-0.2	0.232	0.4	0.145	-0.3									
89	0.196	0.2	0.214	-0.2	0.256	1.3	0.188	-0.2	0.132	0.1	0.164	0.2	0.174	-0.5	0.106	-2.0	0.151	-0.1									
91	0.188	0.0	0.214	-0.2	0.208	0.3	0.23	0.7	0.111	-0.6	0.168	0.3	0.183	-0.3	0.193	-0.3	0.144	-0.3									
93	0.21	0.5	0.198	-0.5	0.22	0.5	0.186	-0.2	0.11	-0.6	0.15	-0.1	0.175	-0.4	0.19	-0.4	0.18	0.6									
95	0.19	0.1	0.14	-1.5	0.33	2.8	0.19	-0.1	0.23	3.1	0.13	-0.7	0.18	-0.3	0.27	1.1	0.16	0.1									
97	0.122	-1.4	0.333	1.9	0.234	0.8	0.218	0.4	0.144	0.4	0.175	0.5	0.203	0.1	0.276	1.2	0.202	1.2									
99	0.151	-0.8	0.2	-0.5	0.183	-0.2	0.187	-0.2	0.177	1.5	0.152	-0.1	0.177	-0.4	0.187	-0.5	0.152	-0.1									
101	0.17	-0.4	0.22	-0.1	0.25	1.2	0.19	-0.1	0.14	0.3	0.18	0.6	0.22	0.5	0.25	0.7	0.16	0.1									
103	0.16	-0.6	0.219	-0.1	0.172	-0.4	0.183	-0.3	0.136	0.2	0.106	-1.3	0.177	-0.4	0.183	-0.5	0.134	-0.6									
105	0.2	0.3	ND	-3.8	ND	-3.8	ND	-3.8	NA	NA	ND	-3.7	ND	-3.8	NA	NA	ND	-3.7									
107	NA	NA	NA	NA	NA	NA	NA	NA	0.129	0.0	NA	NA	NA	NA	0.129	-1.6	NA	NA									
109	0.174	-0.3	0.182	-0.8	0.188	-0.1	0.131	-1.3	0.204	2.3	0.149	-0.2	0.192	-0.1	0.226	0.3	0.159	0.1									
111	0.157	-0.7	0.2	-0.5	0.145	-1.0	0.229	0.7	0.151	0.7	0.108	-1.2	0.2	0.1	0.197	-0.3	0.117	-1.0									
113	0.143	-1.0	0.217	-0.2	0.174	-0.4	0.19	-0.1	0.124	-0.2	0.148	-0.2	0.187	-0.2	0.187	-0.5	0.142	-0.4									
115	0.092	-2.0	0.251	0.4	0.3	2.2	NA	NA	0.031	-3.0	0.295	3.6	0.345	3.0	0.319	2.1	0.21	1.4									
117	0.156	-0.7	0.214	-0.2	0.141	-1.1	0.143	-1.1	0.096	-1.0	0.126	-0.8	0.164	-0.7	0.155	-1.1	0.107	-1.3									
119	0.042	-3.1	0.181	-0.8	0.147	-1.0	0.313	2.4	0.104	-0.8	0.117	-1.0	0.11	-1.8	0.175	-0.7	NA	NA									
121	0.2	0.3	0.238	0.2	0.175	-0.4	0.225	0.6	0.11	-0.6	0.17	0.4	0.222	0.5	0.175	-0.7	0.152	-0.1									
123	0.093	-2.0	0.1	-2.2	0.096	-2.0	0.078	-2.4	0.075	-1.7	0.068	-2.3	0.075	-2.5	0.092	-2.3	0.075	-2.1									
125	0.23	0.9	0.22	-0.1	0.19	-0.1	0.22	0.5	0.16	0.9	0.16	0.1	0.21	0.3	0.27	1.1	0.16	0.1									
127	0.224	0.8	0.231	0.1	0.204	0.2	0.273	1.6	0.152	0.7	0.17	0.4	0.211	0.3	0.239	0.5	0.164	0.2									
129	0.157	-0.7	0.194	-0.6	0.179	-0.3	0.172	-0.5	0.134	0.1	0.11	-1.2	0.162	-0.7	0.178	-0.6	0.135	-0.6									
131	0.13	-1.2	0.225	0.0	0.164	-0.6	0.194	0.0	0.138	0.3	0.144	-0.3	0.171	-0.5	0.172	-0.7	0.155	0.0									

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

Lab Code	Imazali	z score (FFP-RSD 25 %)			Methomyl			Quinoxifen			Spinosad			Tau-Fluvalinate			Tetraconazole			Thiabendazole			Triazophos			Zoxamide		
		MRRL (mg/kg)	0.005	0.188	0.01	0.226	0.194	0.196	0.130	0.156	0.197	0.211	0.157	z score (FFP-RSD 25 %)	0.010	0.010	z score (FFP-RSD 25 %)	0.005	0.010	z score (FFP-RSD 25 %)	0.010	z score (FFP-RSD 25 %)	0.005	0.010				
133	0.193	0.1	0.24	0.2	0.159	-0.7	0.213	0.3	0.17	1.2	0.133	-0.6	0.173	-0.5	0.191	-0.4	0.119	-1.0										
135	0.194	0.1	0.234	0.1	0.225	0.6	0.232	0.7	0.181	1.6	0.209	1.4	0.197	0.0	0.232	0.4	0.179	0.6										
137	0.15	-0.8	0.187	-0.7	0.15	-0.9	0.17	-0.5	0.108	-0.7	0.12	-0.9	0.135	-1.3	0.17	-0.8	0.129	-0.7										
139	0.149	-0.8	NA	NA	0.207	0.3	NA	NA	NA	NA	0.154	0.0	0.17	-0.5	0.169	-0.8	0.176	0.5										
141	0.128	-1.3	0.203	-0.4	0.144	-1.0	NA	NA	0.088	-1.3	0.165	0.2	0.195	0.0	0.142	-1.3	0.162	0.1										
143	0.207	0.4	0.112	-2.0	0.251	1.2	0.241	0.9	0.104	-0.8	0.196	1.0	0.223	0.5	0.274	1.2	0.18	0.6										
145	No results reported																											
147	0.222	0.7	0.141	-1.5	0.217	0.5	0.22	0.5	0.137	0.2	0.162	0.2	0.192	-0.1	0.281	1.3	0.173	0.4										
149	0.195	0.2	0.261	0.6	0.222	0.6	0.221	0.5	0.141	0.3	0.153	-0.1	0.231	0.7	0.222	0.2	0.179	0.6										
151	0.22	0.7	0.21	-0.3	0.19	-0.1	0.18	-0.3	0.09	-1.2	0.12	-0.9	0.2	0.1	0.18	-0.6	0.14	-0.4										
153	0.22	0.7	0.3	1.3	0.23	0.7	0.63	5.0	0.15	0.6	0.14	-0.4	0.26	1.3	0.23	0.4	0.18	0.6										
157	0.167	-0.4	0.238	0.2	0.184	-0.2	0.198	0.0	0.132	0.1	0.121	-0.9	0.184	-0.3	0.172	-0.7	0.143	-0.4										
159	0.18	-0.2	0.216	-0.2	0.187	-0.1	0.192	-0.1	0.13	0.0	0.139	-0.4	0.155	-0.9	0.202	-0.2	0.138	-0.5										
161	0.167	-0.4	0.24	0.2	0.19	-0.1	0.168	-0.6	0.099	-0.9	0.122	-0.9	0.211	0.3	0.173	-0.7	0.162	0.1										
163	0.209	0.5	0.189	-0.7	0.186	-0.2	0.159	-0.8	0.188	1.8	0.178	0.6	0.224	0.5	0.164	-0.9	0.197	1.0										
165	0.121	-1.4	0.225	0.0	0.245	1.1	ND	-3.8	NA	NA	0.159	0.1	NA	NA	NA	NA	0.188	0.8										
167	0.187	0.0	0.181	-0.8	0.191	-0.1	0.216	0.4	0.123	-0.2	0.125	-0.8	0.177	-0.4	0.188	-0.4	0.153	-0.1										
169	0.158	-0.6	0.229	0.0	0.176	-0.4	0.183	-0.3	0.125	-0.1	0.161	0.1	0.204	0.1	0.202	-0.2	0.149	-0.2										
171	0.142	-1.0	0.194	-0.6	0.201	0.2	0.154	-0.9	0.116	-0.4	0.138	-0.5	0.127	-1.4	0.198	-0.2	0.155	0.0										
173	0.19	0.1	0.18	-0.8	0.21	0.3	0.48	5.0	0.61	5.0	0.17	0.4	0.2	0.1	0.18	-0.6	0.18	0.6										
175	NA	NA	0.14	-1.5	NA	NA	0.3	2.1	0.18	1.5	NA	NA	0.19	-0.1	0.19	-0.4	NA	NA										
177	0.17	-0.4	0.21	-0.3	0.21	0.3	0.27	1.5	0.14	0.3	0.14	-0.4	0.19	-0.1	0.2	-0.2	0.16	0.1										
179	0.198	0.2	0.193	-0.6	0.168	-0.5	0.194	0.0	0.113	-0.5	0.135	-0.5	0.174	-0.5	0.207	-0.1	0.122	-0.9										
181	0.262	1.6	0.263	0.6	0.219	0.5	0.192	-0.1	0.137	0.2	0.18	0.6	0.217	0.4	0.249	0.7	0.171	0.4										
183	0.113	-1.6	0.221	-0.1	0.306	2.3	0.691	5.0	0.219	2.8	0.276	3.1	0.22	0.5	0.331	2.3	0.221	1.6										
185	0.21	0.5	0.22	-0.1	0.22	0.5	0.24	0.9	0.12	-0.3	0.18	0.6	0.19	-0.1	0.24	0.6	0.17	0.3										
187	0.151	-0.8	0.237	0.2	0.171	-0.5	0.183	-0.3	0.083	-1.4	0.139	-0.4	0.191	-0.1	0.191	-0.4	0.135	-0.6										
188	0.17	-0.4	0.204	-0.4	0.18	-0.3	0.26	1.3	0.147	0.5	0.15	-0.1	0.172	-0.5	0.17	-0.8	0.155	0.0										
191	NA	NA	NA	NA	0.205	0.2	NA	NA	NA	NA	0.186	0.8	NA	NA	NA	NA	NA	NA										
193	0.153	-0.7	0.226	0.0	0.221	0.6	0.176	-0.4	0.139	0.3	0.206	1.3	0.19	-0.1	0.19	-0.4	0.169	0.3										
195	0.212	0.5	0.231	0.1	0.2	0.1	NA	NA	0.096	-1.0	0.158	0.1	0.2	0.1	0.062	-2.8	0.156	0.0										
197	0.21	0.5	0.22	-0.1	0.21	0.3	0.16	-0.7	0.16	0.9	0.21	1.4	0.16	-0.8	0.23	0.4	0.14	-0.4										

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

Lab Code	Imazali	z score (FFP-RSD 25 %)	Methomyl	z score (FFP-RSD 25 %)	Quinoxifen	z score (FFP-RSD 25 %)	Spinosad	z score (FFP-RSD 25 %)	Tau-Fluvalinate	z score (FFP-RSD 25 %)	Tetraconazole	z score (FFP-RSD 25 %)	Thiabendazole	z score (FFP-RSD 25 %)	Triazophos	z score (FFP-RSD 25 %)	Zoxamide	z score (FFP-RSD 25 %)										
																			MRRL (mg/kg)	0.005	0.01	0.010	0.010	0.010	0.010	0.010	0.005	0.010
																			Robust mean (mg/kg)	0.188	0.226	0.194	0.196	0.130	0.156	0.197	0.211	0.157
199	0.23	0.9	0.23	0.1	0.22	0.5	0.18	-0.3	0.097	-1.0	0.15	-0.1	0.33	2.7	0.27	1.1	0.18	0.6										
201	0.198	0.2	0.222	-0.1	0.176	-0.4	0.17	-0.5	0.136	0.2	0.133	-0.6	0.179	-0.4	0.199	-0.2	0.138	-0.5										
203	0.13	-1.2	0.14	-1.5	0.16	-0.7	0.15	-0.9	0.11	-0.6	0.12	-0.9	0.18	-0.3	0.18	-0.6	0.13	-0.7										
205	0.18	-0.2	0.23	0.1	0.19	-0.1	0.22	0.5	0.12	-0.3	0.18	0.6	0.21	0.3	0.18	-0.6	0.16	0.1										
207	0.185	-0.1	0.182	-0.8	0.179	-0.3	0.185	-0.2	0.117	-0.4	0.141	-0.4	0.184	-0.3	0.197	-0.3	0.133	-0.6										
209	0.142	-1.0	0.212	-0.3	0.171	-0.5	0.188	-0.2	0.13	0.0	0.125	-0.8	0.172	-0.5	0.188	-0.4	0.128	-0.7										
211	0.192	0.1	0.069	-2.8	0.18	-0.3	0.191	-0.1	0.128	-0.1	0.134	-0.6	0.182	-0.3	0.191	-0.4	0.129	-0.7										
213	0.243	1.2	0.242	0.3	0.218	0.5	0.181	-0.3	NA	NA	0.158	0.1	0.188	-0.2	0.218	0.1	0.139	-0.5										
215	0.205	0.4	0.193	-0.6	0.22	0.5	0.209	0.3	0.163	1.0	0.166	0.3	0.184	-0.3	0.208	-0.1	0.178	0.5										
217	0.288	2.1	0.262	0.6	0.218	0.5	0.214	0.4	0.127	-0.1	0.17	0.4	0.232	0.7	0.305	1.8	0.189	0.8										
219	0.218	0.6	0.259	0.6	0.164	-0.6	0.17	-0.5	0.136	0.2	0.152	-0.1	0.217	0.4	0.22	0.2	0.128	-0.7										
221	0.155	-0.7	0.218	-0.1	0.2	0.1	0.217	0.4	0.2	2.2	0.152	-0.1	0.177	-0.4	0.283	1.4	0.15	-0.2										
223	0.167	-0.4	0.24	0.2	0.178	-0.3	0.214	0.4	0.165	1.1	0.168	0.3	0.235	0.8	0.232	0.4	0.167	0.3										
225	0.137	-1.1	0.217	-0.2	0.193	0.0	0.215	0.4	0.133	0.1	0.136	-0.5	0.187	-0.2	0.192	-0.4	0.178	0.5										
227	ND	-3.9	0.2	-0.5	0.15	-0.9	0.28	1.7	0.18	1.5	0.15	-0.1	0.17	-0.5	0.16	-1.0	0.13	-0.7										
229	0.187	0.0	NA	NA	0.198	0.1	NA	NA	0.16	0.9	0.159	0.1	0.189	-0.2	0.225	0.3	ND	-3.7										
231	No results reported																											
233	0.178	-0.2	0.195	-0.6	0.19	-0.1	0.229	0.7	0.157	0.8	0.143	-0.3	0.194	-0.1	0.175	-0.7	0.165	0.2										
235	0.146	-0.9	0.21	-0.3	0.16	-0.7	0.159	-0.8	0.034	-3.0	0.104	-1.3	0.167	-0.6	0.173	-0.7	0.127	-0.8										
237	0.197	0.2	0.222	-0.1	0.223	0.6	0.133	-1.3	0.118	-0.4	0.184	0.7	0.18	-0.3	0.253	0.8	0.162	0.1										
239	0.219	0.7	0.188	-0.7	0.199	0.1	0.175	-0.4	0.188	1.8	0.208	1.3	0.213	0.3	0.251	0.8	0.195	1.0										
241	0.313	2.7	0.314	1.6	0.179	-0.3	0.18	-0.3	0.138	0.3	0.164	0.2	0.239	0.9	0.171	-0.8	0.147	-0.3										
243	0.213	0.5	0.186	-0.7	0.186	-0.2	0.393	4.0	0.139	0.3	0.163	0.2	0.173	-0.5	0.219	0.2	0.169	0.3										
245	0.194	0.1	0.259	0.6	0.202	0.2	0.235	0.8	0.216	2.7	0.15	-0.1	0.284	1.8	0.191	-0.4	0.268	2.8										
247	0.198	0.2	NA	NA	0.281	1.8	NA	NA	NA	NA	NA	NA	NA	NA	0.298	1.7	NA	NA										
249	0.161	-0.6	0.228	0.0	0.192	0.0	0.184	-0.2	0.146	0.5	0.187	0.8	0.177	-0.4	0.234	0.4	0.156	0.0										
251	0.22	0.7	0.24	0.2	0.21	0.3	0.17	-0.5	0.1	-0.9	0.14	-0.4	0.18	-0.3	0.2	-0.2	0.14	-0.4										
253	0.124	-1.4	0.218	-0.1	0.137	-1.2	0.18	-0.3	NA	NA	0.186	0.8	0.823	5.0	0.246	0.7	NA	NA										
255	0.165	-0.5	0.277	0.9	0.153	-0.8	0.195	0.0	0.128	-0.1	0.117	-1.0	0.189	-0.2	0.181	-0.6	0.134	-0.6										
257	0.11	-1.7	0.17	-1.0	0.2	0.1	0.18	-0.3	0.16	0.9	0.16	0.1	0.15	-1.0	0.21	0.0	0.14	-0.4										
259	0.2	0.3	0.245	0.3	0.174	-0.4	0.26	1.3	0.134	0.1	0.147	-0.2	0.172	-0.5	0.187	-0.5	0.139	-0.5										
261	0.211	0.5	0.234	0.1	0.217	0.5	0.22	0.5	0.14	0.3	0.122	-0.9	0.182	-0.3	0.251	0.8	0.144	-0.3										

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

Lab Code	Imazali	z score (FFP-RSD 25 %)		Methomyl		z score (FFP-RSD 25 %)		Quinoxifen		z score (FFP-RSD 25 %)		Spinosad		z score (FFP-RSD 25 %)		Tau-Fluvalinate		z score (FFP-RSD 25 %)		Tetraconazole		z score (FFP-RSD 25 %)		Thiabendazole		z score (FFP-RSD 25 %)		Triazophos		z score (FFP-RSD 25 %)		Zoxamide		z score (FFP-RSD 25 %)	
		MRRL (mg/kg)	0.005	0.01	0.01	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010		
		Robust mean (mg/kg)	0.188																															0.226	0.194
263	0.168	-0.4	0.248	0.4	0.194	0.0	0.156	-0.8	0.174	1.4	0.156	0.0	0.193	-0.1	0.24	0.6	0.166	0.2																	
265	0.223	0.8	0.284	1.0	0.231	0.8	0.242	0.9	0.046	-2.6	0.207	1.3	0.231	0.7	0.095	-2.2	0.196	1.0																	
267	0.156	-0.7	0.186	-0.7	0.189	-0.1	0.165	-0.6	0.097	-1.0	0.155	0.0	0.171	-0.5	0.213	0.0	0.117	-1.0																	
269	No results reported																																		
271	0.211	0.5	0.259	0.6	0.184	-0.2	0.41	4.4	0.15	0.6	0.157	0.0	0.236	0.8	0.235	0.5	0.141	-0.4																	
273	0.207	0.4	NA	NA	0.178	-0.3	NA	NA	0.157	0.8	0.13	-0.7	0.197	0.0	0.21	0.0	NA	NA																	
275	0.12	-1.4	0.2	-0.5	0.15	-0.9	0.14	-1.1	0.17	1.2	0.16	0.1	0.15	-1.0	0.23	0.4	0.16	0.1																	
277	No results reported																																		
279	0.16	-0.6	0.29	1.1	0.18	-0.3	0.15	-0.9	0.11	-0.6	0.16	0.1	0.2	0.1	0.14	-1.3	0.14	-0.4																	
281	0.213	0.5	0.23	0.1	0.191	-0.1	0.275	1.6	0.138	0.3	0.178	0.6	0.212	0.3	0.214	0.1	0.176	0.5																	
283	No results reported																																		
285	0.198	0.2	0.227	0.0	0.158	-0.7	0.195	0.0	0.131	0.0	0.142	-0.4	0.194	-0.1	0.189	-0.4	0.135	-0.6																	
287	0.321	2.8	ND	-3.8	0.184	-0.2	ND	-3.8	0.131	0.0	0.101	-1.4	ND	-3.8	0.159	-1.0	0.115	-1.1																	
289	0.141	-1.0	0.133	-1.6	0.151	-0.9	0.115	-1.7	0.097	-1.0	0.094	-1.6	0.156	-0.8	0.112	-1.9	0.11	-1.2																	
291	0.176	-0.2	NA	NA	0.182	-0.2	0.138	-1.2	NA	NA	0.133	-0.6	0.13	-1.4	NA	NA	0.152	-0.1																	
293	0.206	0.4	0.272	0.8	0.271	1.6	0.268	1.5	0.19	1.9	ND	-3.7	0.266	1.4	0.28	1.3	0.203	1.2																	
295	0.259	1.5	NA	NA	0.195	0.0	NA	NA	0.109	-0.6	0.239	2.1	0.452	5.0	0.262	1.0	ND	-3.7																	
297	0.276	1.9	0.287	1.1	0.226	0.7	0.25	1.1	0.145	0.5	0.21	1.4	0.256	1.2	0.292	1.5	0.222	1.7																	
299	0.142	-1.0	0.236	0.2	NA	NA	0.156	-0.8	0.095	-1.1	0.15	-0.1	0.182	-0.3	0.187	-0.5	0.124	-0.8																	
301	0.206	0.4	0.258	0.6	0.31	2.4	0.17	-0.5	0.097	-1.0	NA	NA	0.275	1.6	0.164	-0.9	NA	NA																	
303	0.163	-0.5	0.249	0.4	0.214	0.4	0.223	0.5	0.16	0.9	0.184	0.7	0.2	0.1	0.259	0.9	0.175	0.5																	
305	0.2	0.3	0.2	-0.5	0.18	-0.3	0.18	-0.3	0.13	0.0	0.14	-0.4	0.18	-0.3	0.17	-0.8	0.15	-0.2																	
307	0.185	-0.1	NA	NA	0.172	-0.4	0.23	0.7	0.045	-2.6	0.24	2.2	0.271	1.5	0.138	-1.4	0.19	0.8																	
309	0.165	-0.5	0.189	-0.7	0.175	-0.4	0.165	-0.6	0.098	-1.0	0.118	-1.0	0.154	-0.9	0.144	-1.3	0.16	0.1																	
311	0.19	0.1	0.2	-0.5	0.19	-0.1	0.2	0.1	0.075	-1.7	0.16	0.1	0.17	-0.5	0.16	-1.0	0.11	-1.2																	
313	ND	-3.9	0.215	-0.2	0.139	-1.1	NA	NA	0.057	-2.2	0.077	-2.0	0.169	-0.6	0.103	-2.0	NA	NA																	
315	0.204	0.4	0.238	0.2	0.25	1.2	0.122	-1.5	0.121	-0.3	0.194	1.0	0.217	0.4	0.292	1.5	0.147	-0.3																	
317	0.164	-0.5	0.216	-0.2	0.166	-0.6	0.196	0.0	0.129	0.0	0.188	0.8	0.197	0.0	0.32	2.1	0.152	-0.1																	
319	0.234	1.0	0.215	-0.2	0.207	0.3	0.194	0.0	0.119	-0.3	0.163	0.2	0.205	0.2	0.208	-0.1	0.171	0.4																	
321	0.162	-0.5	0.219	-0.1	0.191	-0.1	0.195	0.0	0.122	-0.2	0.127	-0.7	0.199	0.0	0.17	-0.8	0.146	-0.3																	
323	0.166	-0.5	0.191	-0.6	0.136	-1.2	0.269	1.5	0.08	-1.5	0.125	-0.8	0.198	0.0	0.16	-1.0	0.153	-0.1																	
325	0.156	-0.7	0.23	0.1	0.155	-0.8	0.16	-0.7	0.079	-1.6	0.159	0.1	0.237	0.8	0.166	-0.8	0.146	-0.3																	

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

Lab Code	Imazalil	z score (FFP-RSD 25 %)		Methomyl	z score (FFP-RSD 25 %)		Quinoxifen	z score (FFP-RSD 25 %)		Spinosad	z score (FFP-RSD 25 %)		Tau-Fluvalinate	z score (FFP-RSD 25 %)		Tetraconazole	z score (FFP-RSD 25 %)		Thiabendazole	z score (FFP-RSD 25 %)		Triazophos	z score (FFP-RSD 25 %)		Zoxamide	z score (FFP-RSD 25 %)	
	MRRL (mg/kg)	0.005	z score (FFP-RSD 25 %)	0.01	z score (FFP-RSD 25 %)	0.010	z score (FFP-RSD 25 %)	0.010	z score (FFP-RSD 25 %)	0.010	z score (FFP-RSD 25 %)	0.010	z score (FFP-RSD 25 %)	0.010	z score (FFP-RSD 25 %)	0.010	z score (FFP-RSD 25 %)	0.010	z score (FFP-RSD 25 %)	0.005	z score (FFP-RSD 25 %)	0.010	z score (FFP-RSD 25 %)	0.010	z score (FFP-RSD 25 %)		
	Robust mean (mg/kg)	0.188		0.226		0.194		0.196		0.130		0.156		0.197		0.211		0.157									
327	0.205	0.4	0.251	0.4	0.191	-0.1	0.89	5.0	0.131	0.0	0.136	-0.5	0.211	0.3	0.221	0.2	0.157	0.0									
329	0.189	0.0	0.212	-0.3	0.144	-1.0	0.419	4.5	0.129	0.0	0.16	0.1	0.268	1.4	0.194	-0.3	0.126	-0.8									
331	0.13	-1.2	0.21	-0.3	0.19	-0.1	0.1	-2.0	0.11	-0.6	0.17	0.4	0.18	-0.3	0.26	0.9	0.2	1.1									
333	0.261	1.6	0.251	0.4	0.126	-1.4	0.201	0.1	0.087	-1.3	0.101	-1.4	0.276	1.6	0.272	1.2	0.128	-0.7									
335	0.233	1.0	0.249	0.4	0.18	-0.3	0.61	5.0	0.144	0.4	0.147	-0.2	0.171	-0.5	0.232	0.4	0.231	1.9									
337	0.185	-0.1	0.255	0.5	0.215	0.4	0.191	-0.1	0.15	0.6	0.165	0.2	0.196	0.0	0.224	0.3	0.172	0.4									
339	0.186	0.0	0.22	-0.1	0.176	-0.4	0.17	-0.5	0.158	0.9	ND	-3.7	0.214	0.3	0.2	-0.2	0.172	0.4									
341	0.199	0.2	0.267	0.7	0.129	-1.3	0.221	0.5	0.081	-1.5	0.155	0.0	0.213	0.3	0.134	-1.5	0.129	-0.7									
343	0.25	1.3	ND	-3.8	0.26	1.4	ND	-3.8	0.14	0.3	0.23	1.9	0.23	0.7	0.29	1.5	0.24	2.1									
345	No results reported																										
347	0.155	-0.7	NA	NA	0.16	-0.7	0.545	5.0	0.255	3.9	0.117	-1.0	NA	NA	0.142	-1.3	0.11	-1.2									
349	0.14	-1.0	0.255	0.5	0.19	-0.1	0.208	0.2	0.13	0.0	0.154	0.0	0.189	-0.2	0.173	-0.7	0.122	-0.9									
351	0.284	2.1	0.296	1.2	0.258	1.3	0.532	5.0	0.15	0.6	0.223	1.7	0.23	0.7	0.268	1.1	NA	NA									
353	0.195	0.2	0.245	0.3	0.195	0.0	0.205	0.2	0.117	-0.4	0.145	-0.3	0.182	-0.3	0.19	-0.4	0.125	-0.8									
355	0.144	-0.9	0.2	-0.5	0.19	-0.1	0.208	0.2	0.105	-0.8	0.156	0.0	0.179	-0.4	0.235	0.5	0.145	-0.3									
357	0.223	0.8	0.27	0.8	0.16	-0.7	0.275	1.6	0.062	-2.1	0.18	0.6	0.173	-0.5	0.21	0.0	0.191	0.9									
359	0.304	2.5	0.253	0.5	0.13	-1.3	0.245	1.0	0.116	-0.4	0.132	-0.6	0.248	1.0	0.32	2.1	0.182	0.6									
361	0.165	-0.5	0.241	0.3	0.22	0.5	0.197	0.0	0.155	0.8	0.185	0.8	0.208	0.2	0.21	0.0	0.161	0.1									
363	0.18	-0.2	0.22	-0.1	0.18	-0.3	0.23	0.7	0.1	-0.9	0.17	0.4	0.17	-0.5	0.22	0.2	0.14	-0.4									
365	No results reported																										
367	0.21	0.5	0.25	0.4	0.16	-0.7	0.18	-0.3	ND	-3.7	0.16	0.1	0.2	0.1	0.23	0.4	0.16	0.1									
368	0.15	-0.8	0.226	0.0	0.181	-0.3	0.19	-0.1	0.133	0.1	0.149	-0.2	0.215	0.4	0.214	0.1	0.141	-0.4									

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 flutianil and isofetamid (mg/kg) and their calculated z score value using FFP-RSD 25 %.

Lab Code	Flutianil	z score (FFP-RSD 25 %)	Isofetamid	z score (FFP-RSD 25 %)
MRRL (mg/kg)	0.010		0.010	
Robust mean (mg/kg)	0.060		0.059	
5	0.056	-0.3	0.056	-0.2
7	0.057	-0.2	0.057	-0.1
9	NA	NA	NA	NA
11	NA	NA	NA	NA
13	0.05	-0.7	0.06	0.0
15	NA	NA	NA	NA
17	NA	NA	NA	NA
19	0.048	-0.8	NA	NA
21	NA	NA	NA	NA
23	No results reported			
25	NA	NA	NA	NA
27	NA	NA	NA	NA
29	NA	NA	NA	NA
31	NA	NA	NA	NA
33	NA	NA	NA	NA
35	NA	NA	NA	NA
36	0.073	0.9	0.062	0.2
39	NA	NA	NA	NA
41	NA	NA	NA	NA
43	0.062	0.2	0.05	-0.6
45	NA	NA	NA	NA
47	ND	-3.3	0.069	0.6
49	NA	NA	NA	NA
51	NA	NA	NA	NA
53	NA	NA	NA	NA
55	0.054	-0.4	0.058	-0.1
57	0.075	1.0	0.078	1.3
59	NA	NA	NA	NA
61	NA	NA	NA	NA
63	NA	NA	NA	NA
65	No results reported			
67	0.058	-0.1	NA	NA

Lab Code	Flutianil	z score (FFP-RSD 25 %)	Isofetamid	z score (FFP-RSD 25 %)
MRRL (mg/kg)	0.010		0.010	
Robust mean (mg/kg)	0.060		0.059	
NA	NA	NA	NA	NA
0.067	0.5	NA	NA	0.067
NA	NA	NA	NA	NA
0.062	0.2	0.07	0.7	0.062
NA	NA	NA	NA	NA
NA	NA	NA	NA	NA
NA	NA	0.05	-0.6	NA
0.047	-0.9	0.051	-0.6	0.047
NA	NA	NA	NA	NA
NA	NA	NA	NA	NA
0.044	-1.1	0.061	0.1	0.044
NA	NA	NA	NA	NA
NA	NA	NA	NA	NA
NA	NA	NA	NA	NA
NA	NA	0.07	0.7	NA
0.059	0.0	0.057	-0.2	0.059
NA	NA	NA	NA	NA
NA	NA	0.054	-0.3	NA
NA	NA	NA	NA	NA
NA	NA	NA	NA	NA
NA	NA	NA	NA	NA
NA	NA	NA	NA	NA
NA	NA	NA	NA	NA
NA	NA	NA	NA	NA
0.037	-1.5	0.047	-0.8	0.037
NA	NA	NA	NA	NA
NA	NA	NA	NA	NA
NA	NA	NA	NA	NA
0.056	-0.2	0.065	0.4	0.056
NA	NA	NA	NA	NA
NA	NA	NA	NA	NA
NA	NA	NA	NA	NA

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

Lab Code	Flutianil	z score (FFP-RSD 25 %)	Isofetamid	z score (FFP-RSD 25 %)
MRRL (mg/kg)	0.010		0.010	
Robust mean (mg/kg)	0.060		0.059	
133	NA	NA	NA	NA
135	0.073	0.9	0.076	1.1
137	0.048	-0.8	0.049	-0.7
139	NA	NA	NA	NA
141	NA	NA	NA	NA
143	0.07	0.7	0.063	0.2
145	No results reported			
147	0.062	0.2	0.063	0.2
149	0.105	3.0	0.065	0.4
151	NA	NA	NA	NA
153	NA	NA	NA	NA
157	NA	NA	NA	NA
159	NA	NA	NA	NA
161	0.063	0.2	0.057	-0.2
163	NA	NA	NA	NA
165	NA	NA	NA	NA
167	NA	NA	NA	NA
169	NA	NA	0.054	-0.4
171	NA	NA	0.062	0.2
173	NA	NA	NA	NA
175	NA	NA	NA	NA
177	NA	NA	NA	NA
179	NA	NA	NA	NA
181	0.055	-0.3	NA	NA
183	NA	NA	NA	NA
185	NA	NA	NA	NA
187	NA	NA	0.066	0.4
188	NA	NA	NA	NA
191	NA	NA	NA	NA
193	0.047	-0.9	0.053	-0.4
195	NA	NA	NA	NA
197	NA	NA	ND	-3.3
199	NA	NA	NA	NA
201	NA	NA	NA	NA

Lab Code	Flutianil	z score (FFP-RSD 25 %)	Isofetamid	z score (FFP-RSD 25 %)
MRRL (mg/kg)	0.010		0.010	
Robust mean (mg/kg)	0.060		0.059	
203	NA	NA	NA	NA
205	NA	NA	NA	NA
207	NA	NA	NA	NA
209	ND	-3.3	NA	NA
211	NA	NA	NA	NA
213	NA	NA	NA	NA
215	0.072	0.8	0.078	1.3
217	NA	NA	NA	NA
219	NA	NA	NA	NA
221	0.065	0.4	0.065	0.4
223	NA	NA	0.068	0.6
225	NA	NA	NA	NA
227	NA	NA	NA	NA
229	NA	NA	NA	NA
231	No results reported			
233	NA	NA	NA	NA
235	0.052	-0.5	NA	NA
237	0.056	-0.2	ND	-3.3
239	NA	NA	NA	NA
241	0.058	-0.1	0.057	-0.2
243	NA	NA	NA	NA
245	0.075	1.0	0.053	-0.4
247	NA	NA	NA	NA
249	NA	NA	0.059	0.0
251	0.061	0.1	NA	NA
253	NA	NA	NA	NA
255	NA	NA	0.057	-0.2
257	NA	NA	NA	NA
259	NA	NA	NA	NA
261	0.06	0.0	0.057	-0.2
263	NA	NA	ND	-3.3
265	NA	NA	NA	NA
267	NA	NA	NA	NA
269	No results reported			

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

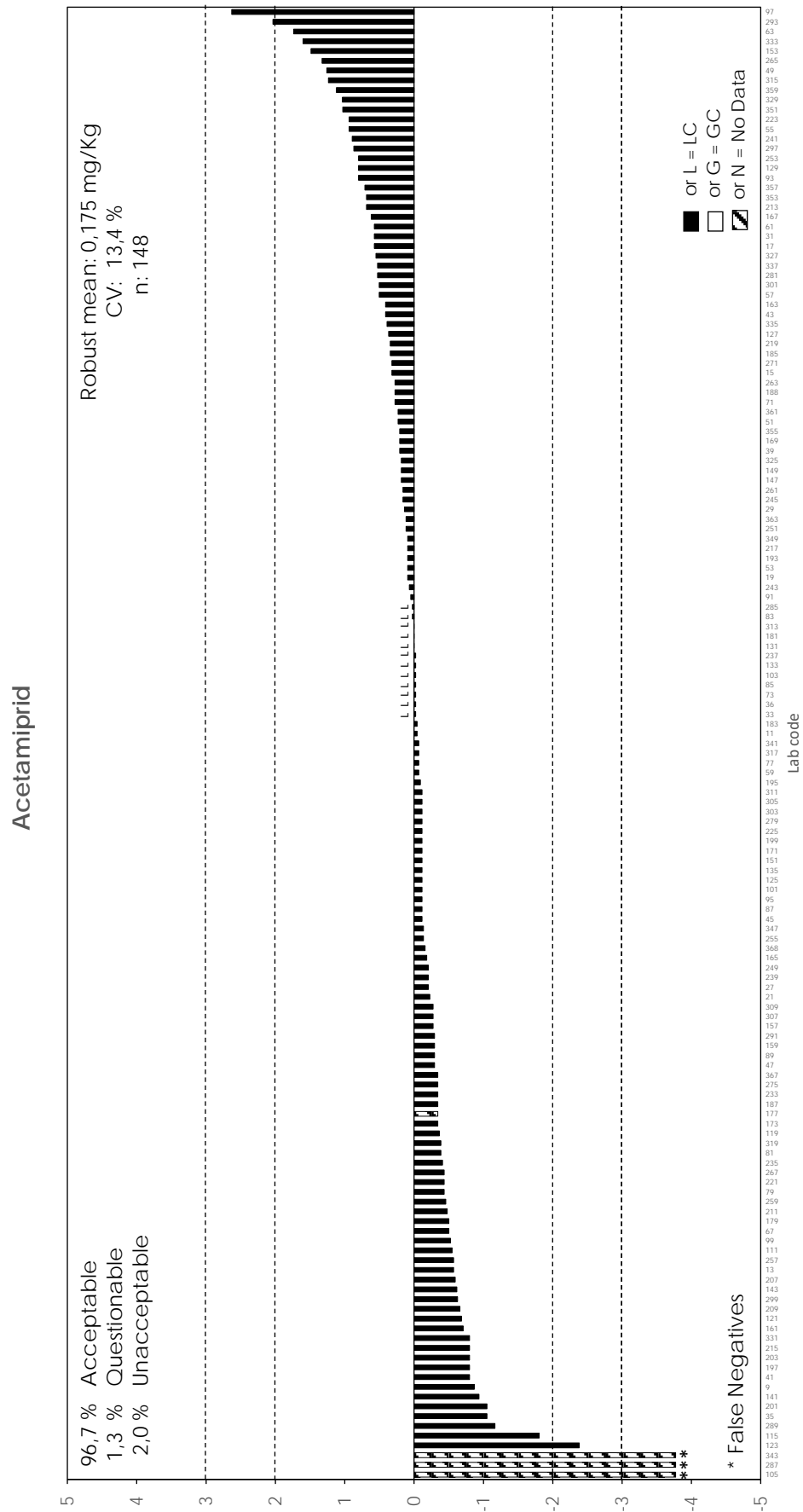
Lab Code	Flutianil	z score (FFP-RSD 25 %)	Isofetamid	z score (FFP-RSD 25 %)
MRRL (mg/kg)	0.010		0.010	
Robust mean (mg/kg)	0.060		0.059	
271	NA	NA	NA	NA
273	NA	NA	NA	NA
275	NA	NA	0.05	-0.6
277	No results reported			
279	NA	NA	NA	NA
281	NA	NA	NA	NA
283	No results reported			
285	NA	NA	0.06	0.0
287	NA	NA	NA	NA
289	NA	NA	0.035	-1.6
291	NA	NA	NA	NA
293	NA	NA	NA	NA
295	NA	NA	NA	NA
297	NA	NA	0.067	0.5
299	NA	NA	NA	NA
301	NA	NA	NA	NA
303	0.056	-0.2	0.068	0.6
305	NA	NA	0.053	-0.4
307	NA	NA	NA	NA
309	0.094	2.3	0.057	-0.2
311	NA	NA	0.051	-0.6
313	NA	NA	NA	NA
315	0.032	-1.9	0.053	-0.4
317	0.046	-0.9	0.054	-0.4
319	NA	NA	NA	NA

Lab Code	Flutianil	z score (FFP-RSD 25 %)	Isofetamid	z score (FFP-RSD 25 %)
MRRL (mg/kg)	0.010		0.010	
Robust mean (mg/kg)	0.060		0.059	
321	NA	NA	NA	NA
323	NA	NA	NA	NA
325	NA	NA	NA	NA
327	NA	NA	NA	NA
329	NA	NA	NA	NA
331	NA	NA	NA	NA
333	NA	NA	NA	NA
335	NA	NA	NA	NA
337	NA	NA	NA	NA
339	NA	NA	NA	NA
341	NA	NA	NA	NA
343	NA	NA	NA	NA
345	No results reported			
347	NA	NA	NA	NA
349	0.057	-0.2	0.048	-0.8
351	NA	NA	NA	NA
353	NA	NA	NA	NA
355	NA	NA	NA	NA
357	NA	NA	NA	NA
359	NA	NA	NA	NA
361	NA	NA	0.06	0.0
363	ND	-3.3	ND	-3.3
365	No results reported			
367	ND	-3.3	ND	-3.3
368	0.051	-0.6	0.054	-0.4

NA: Not analysed

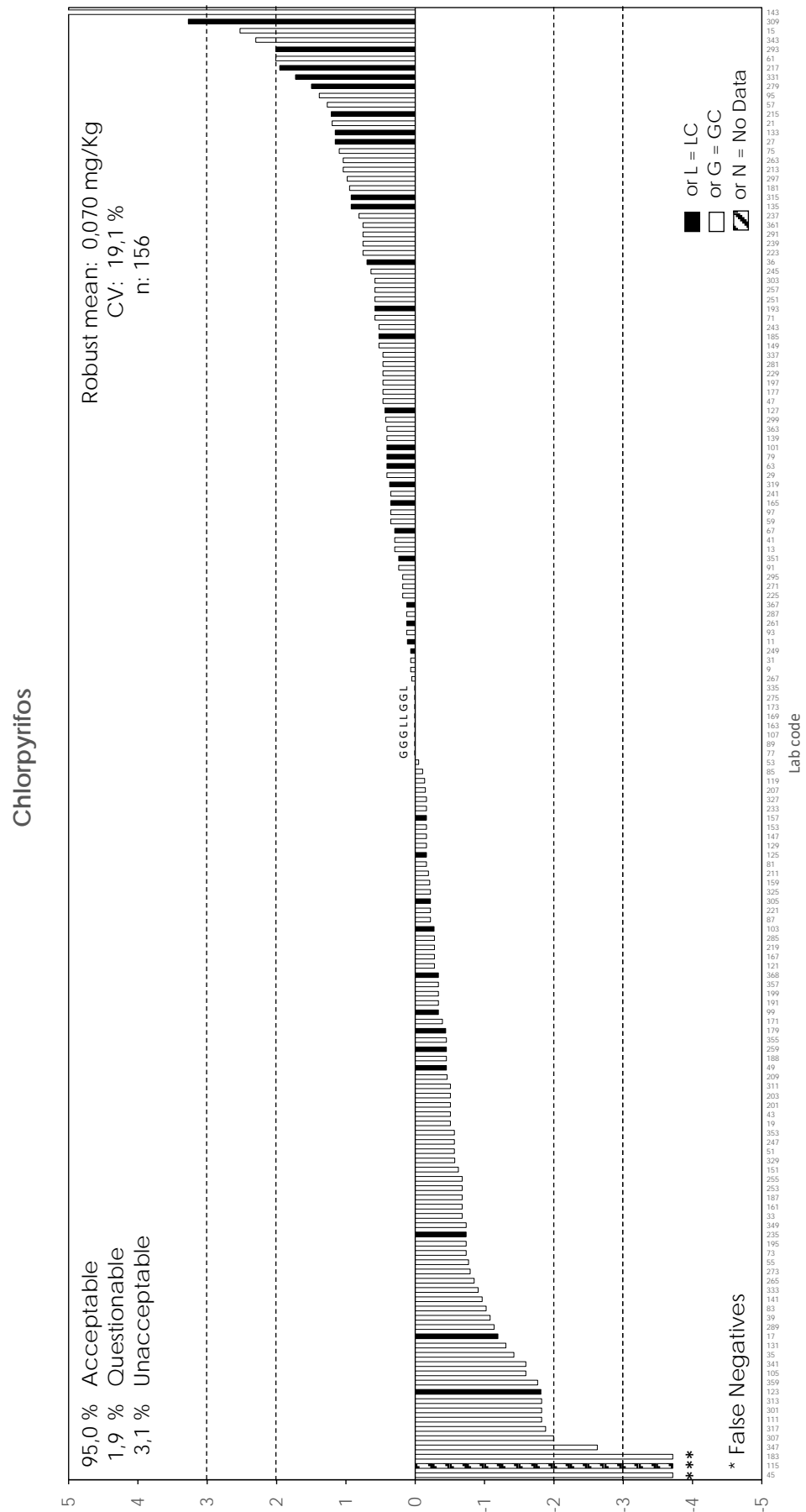
ND: Not detected (False negative)

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

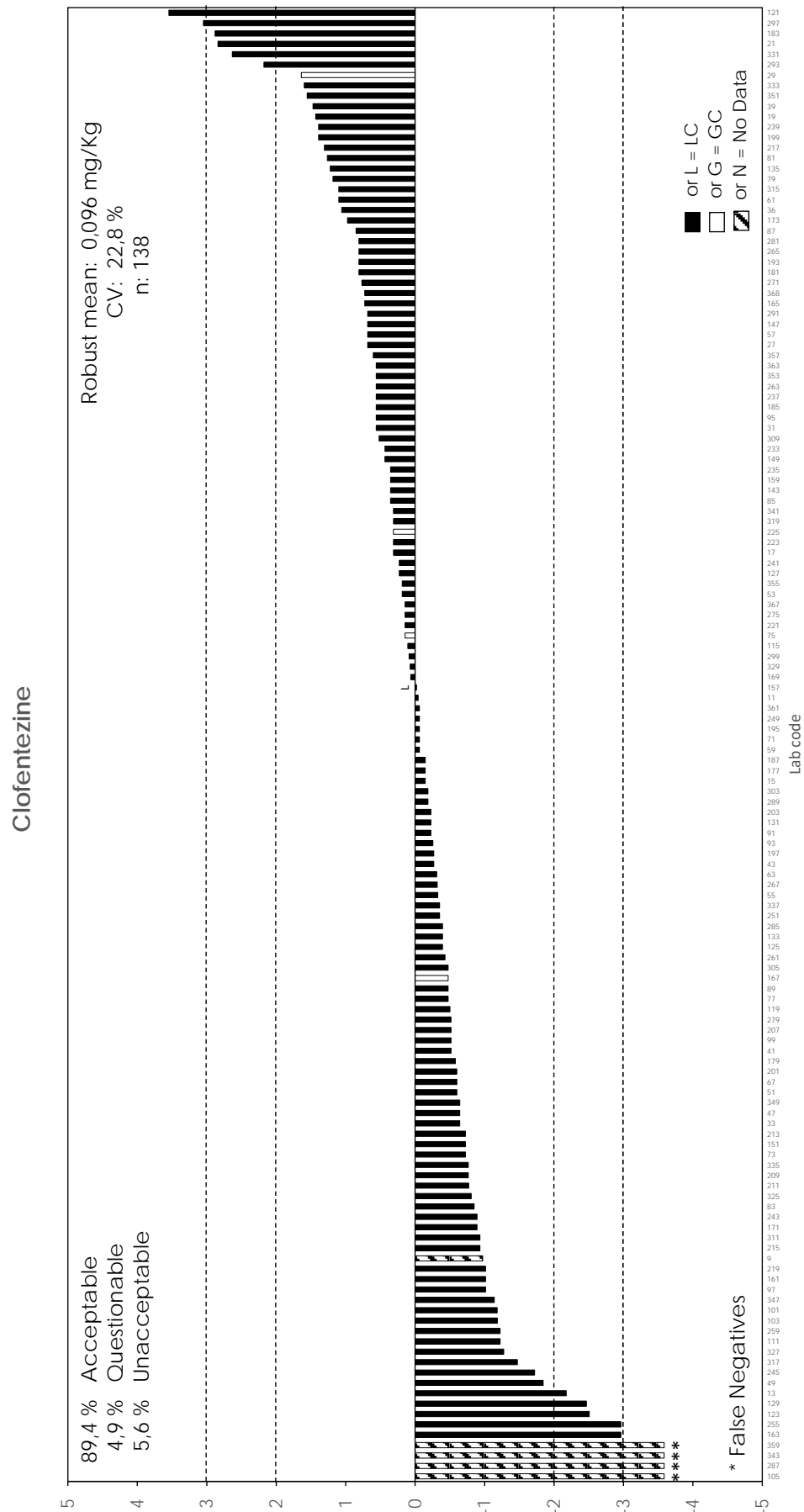


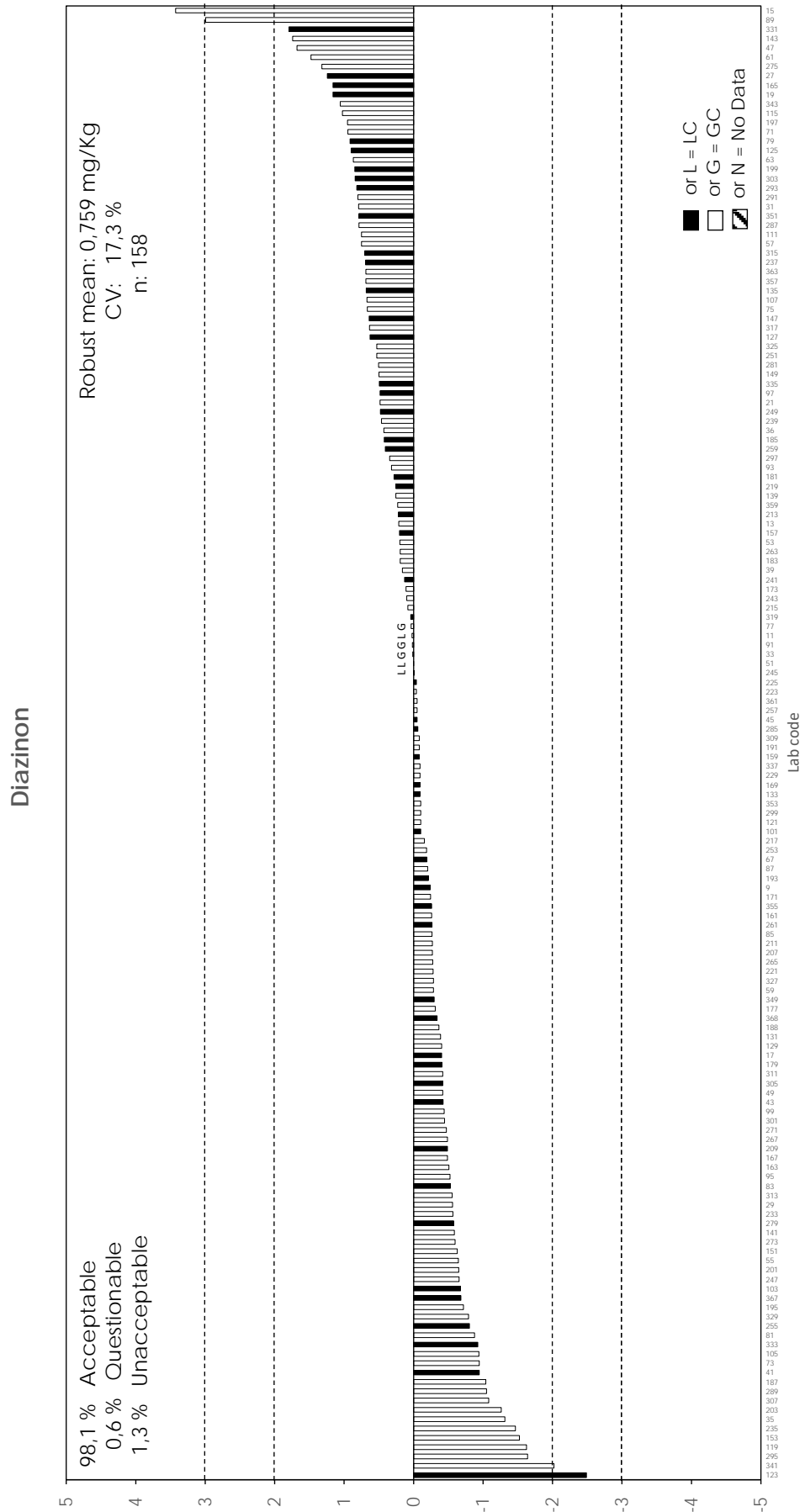
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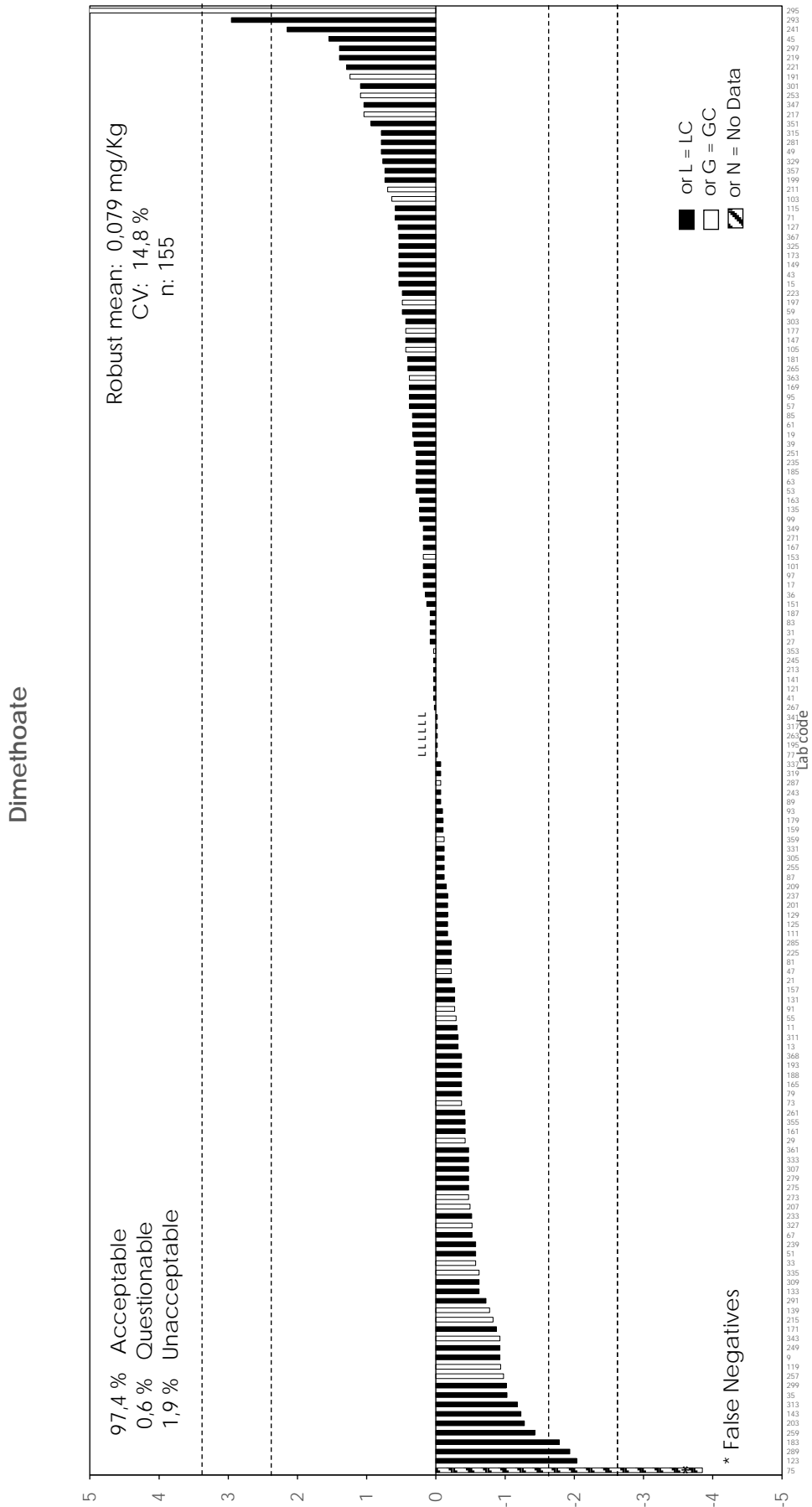


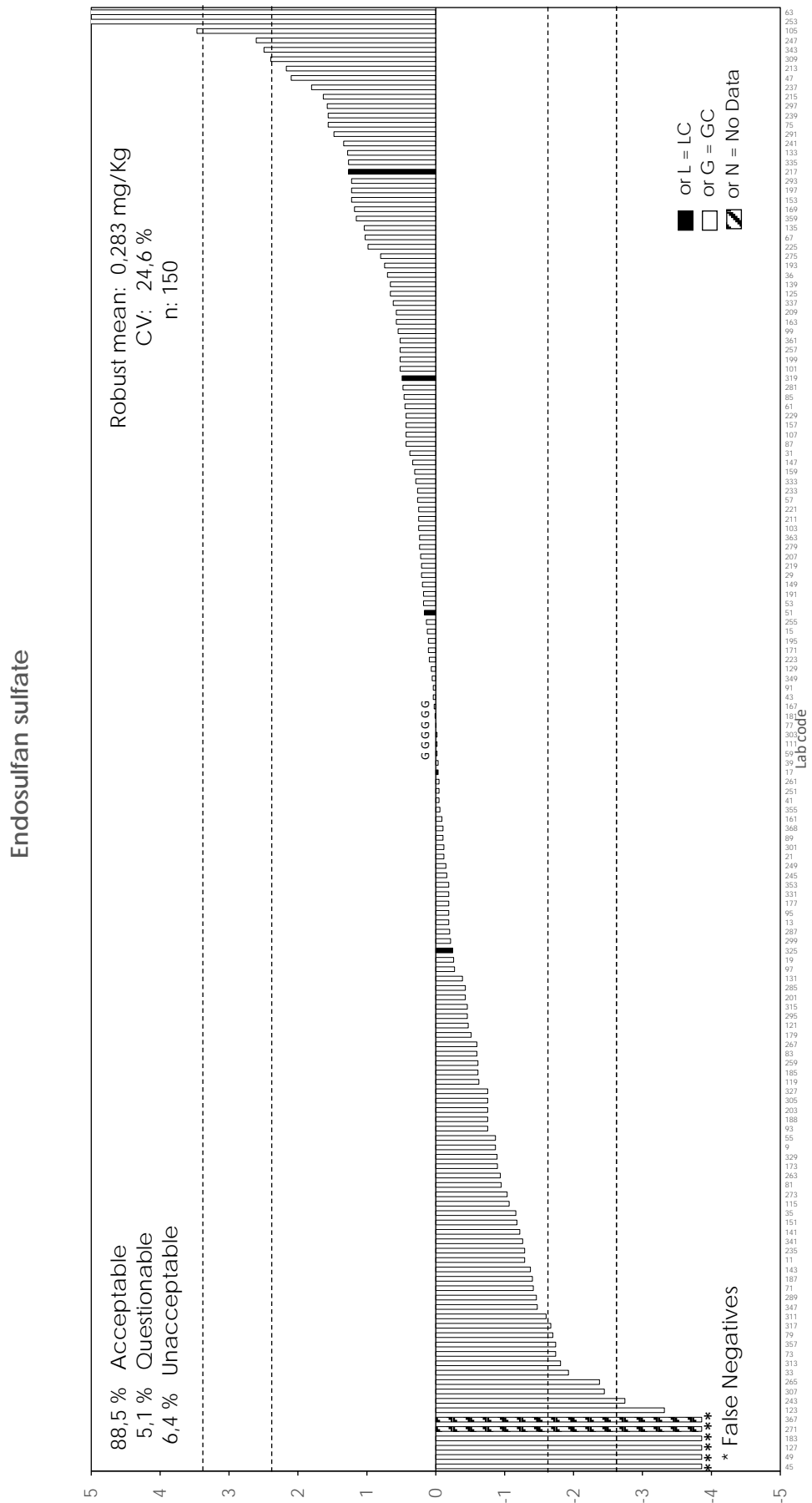
APPENDIX 4. Graphical representation of z scores for FFP-RSD (25 %).



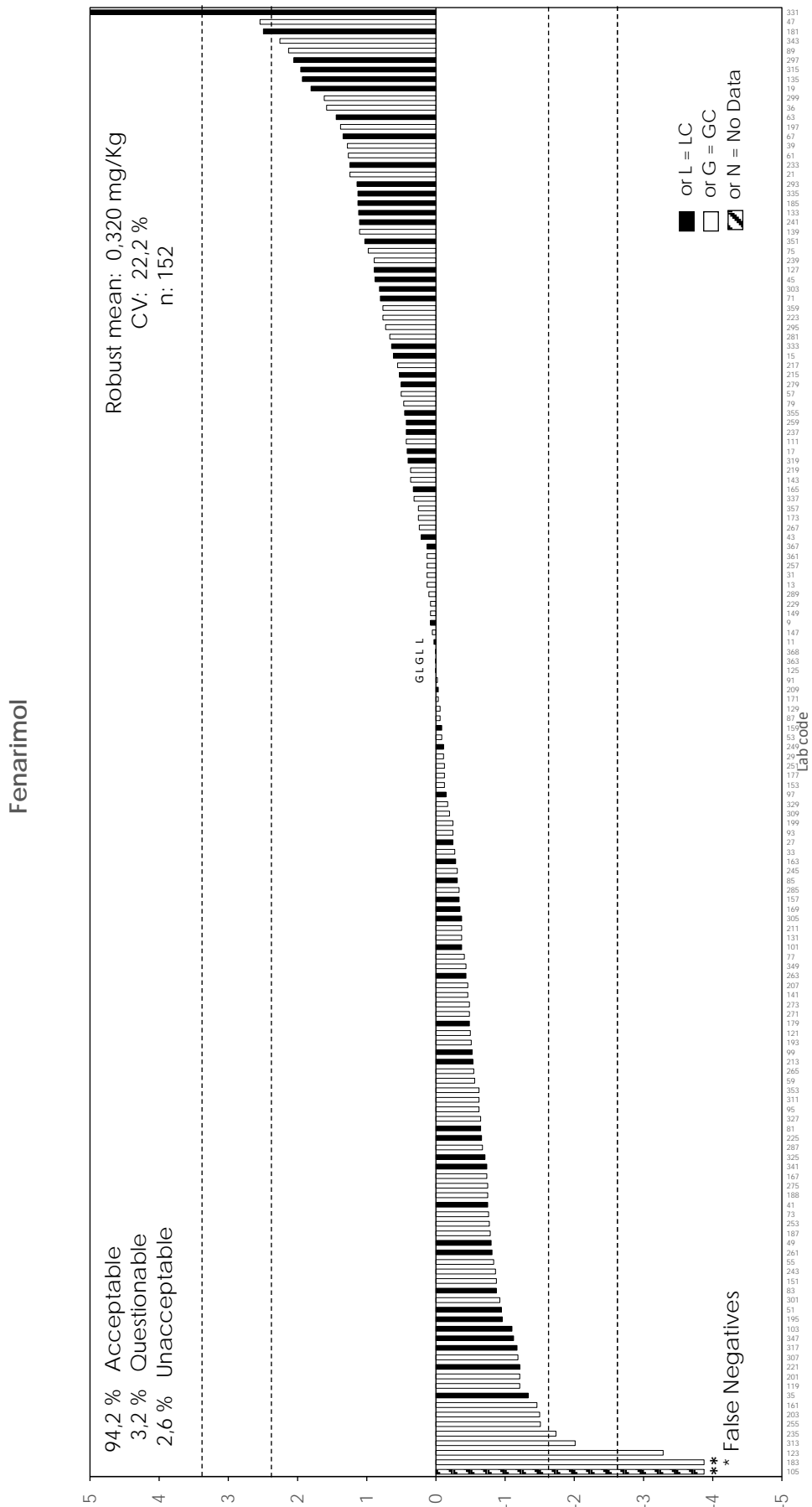


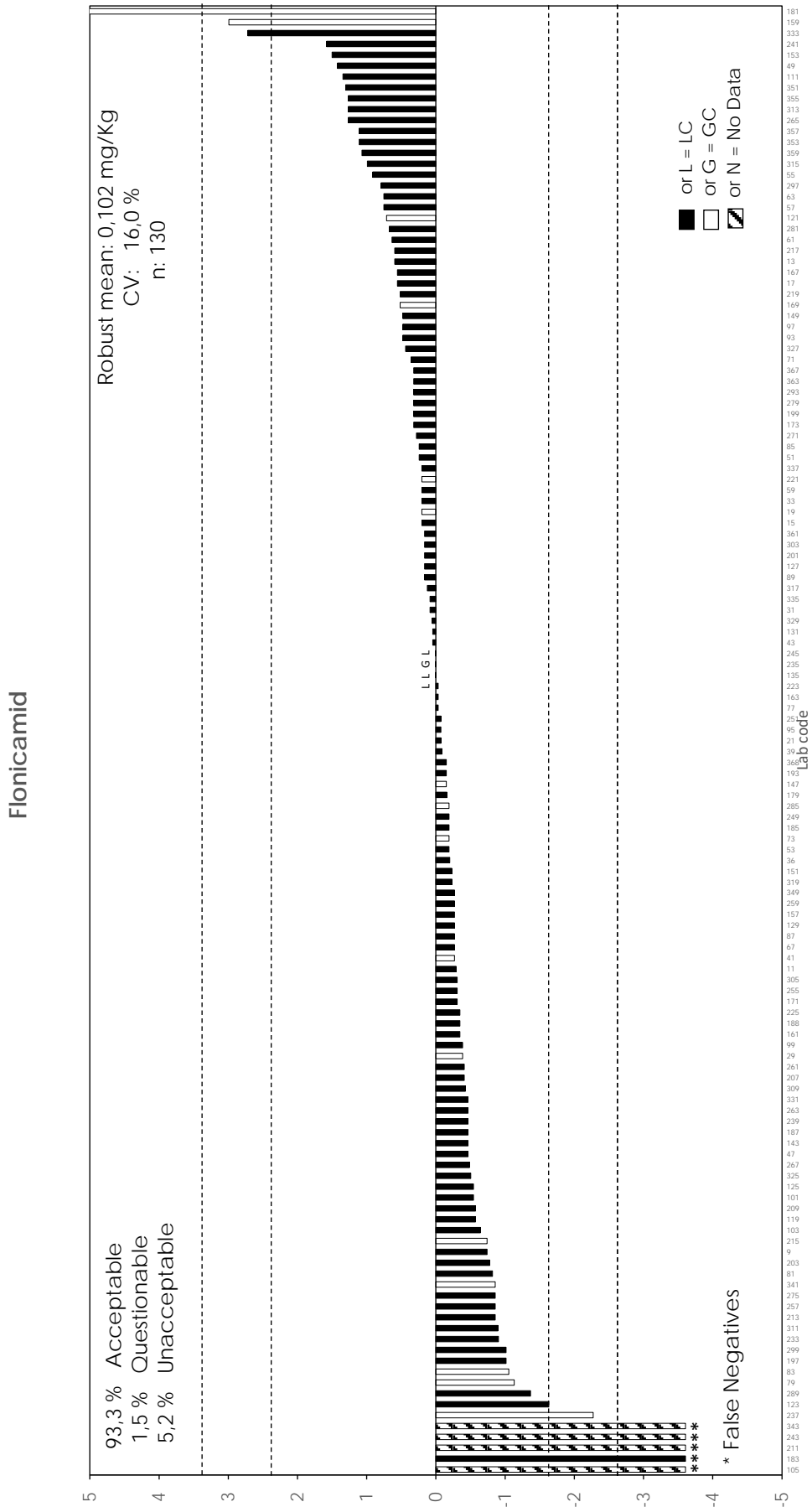




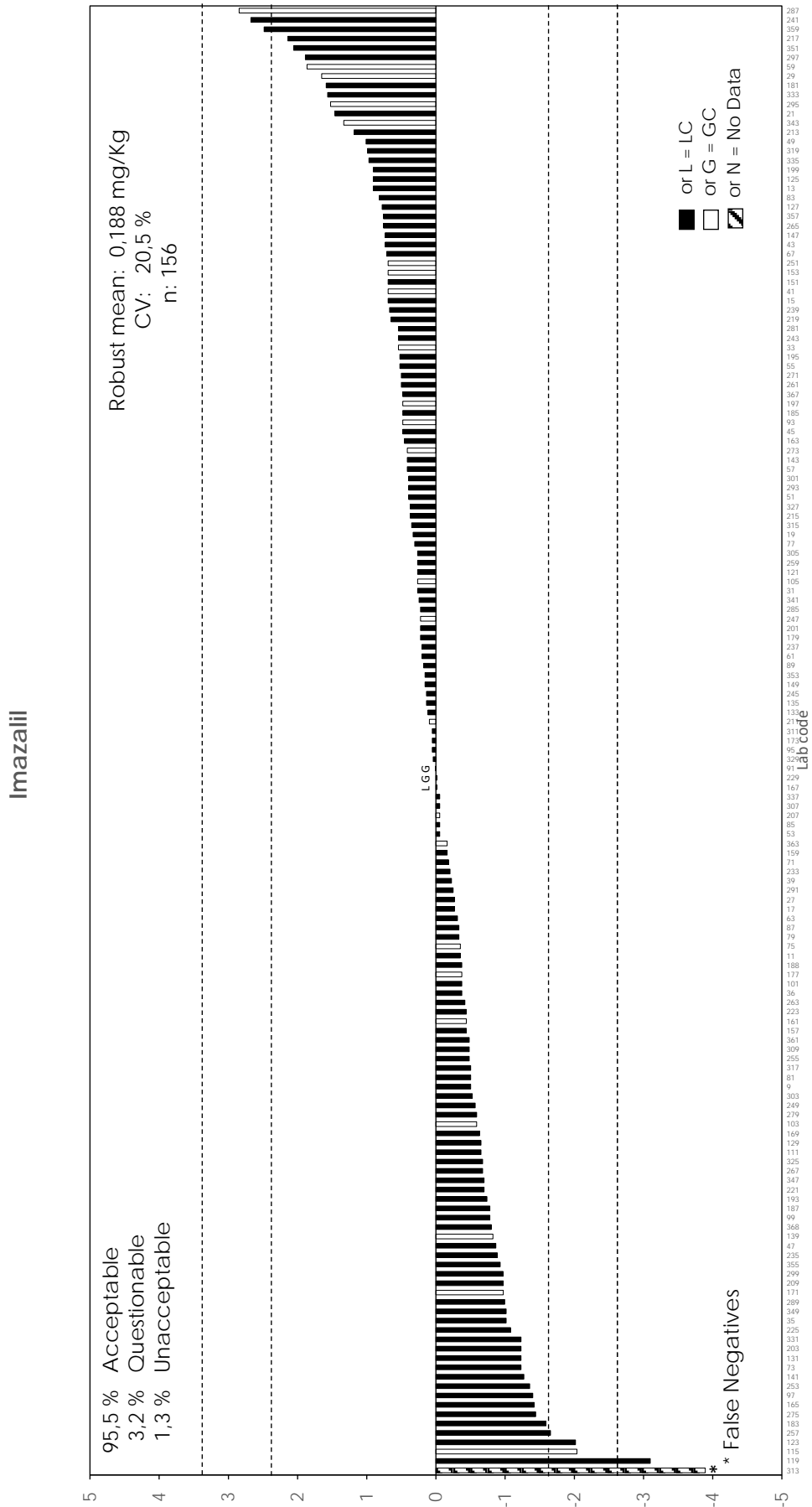


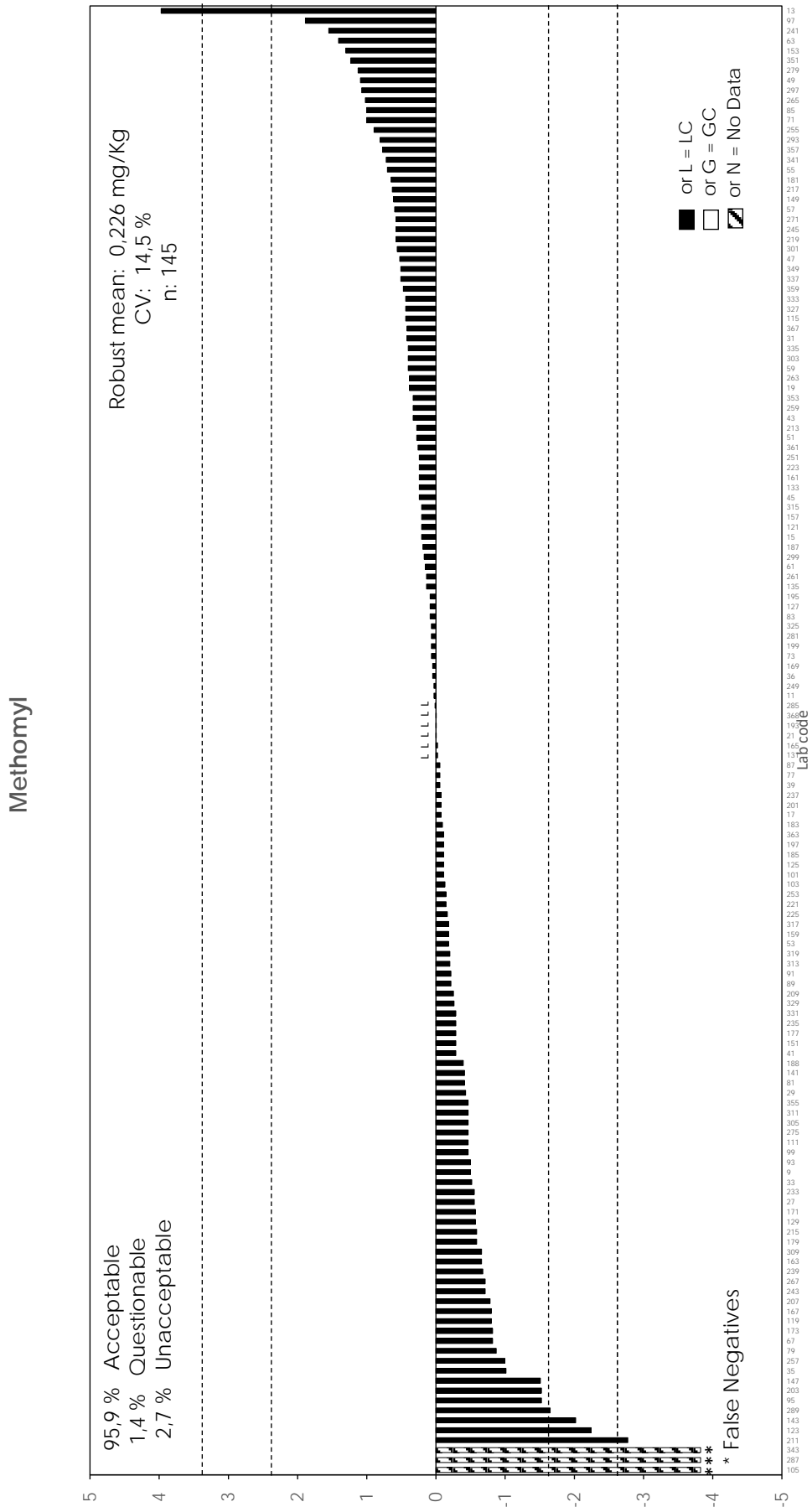
APPENDIX 4. Graphical representation of z scores for FFP-RSD (25 %).



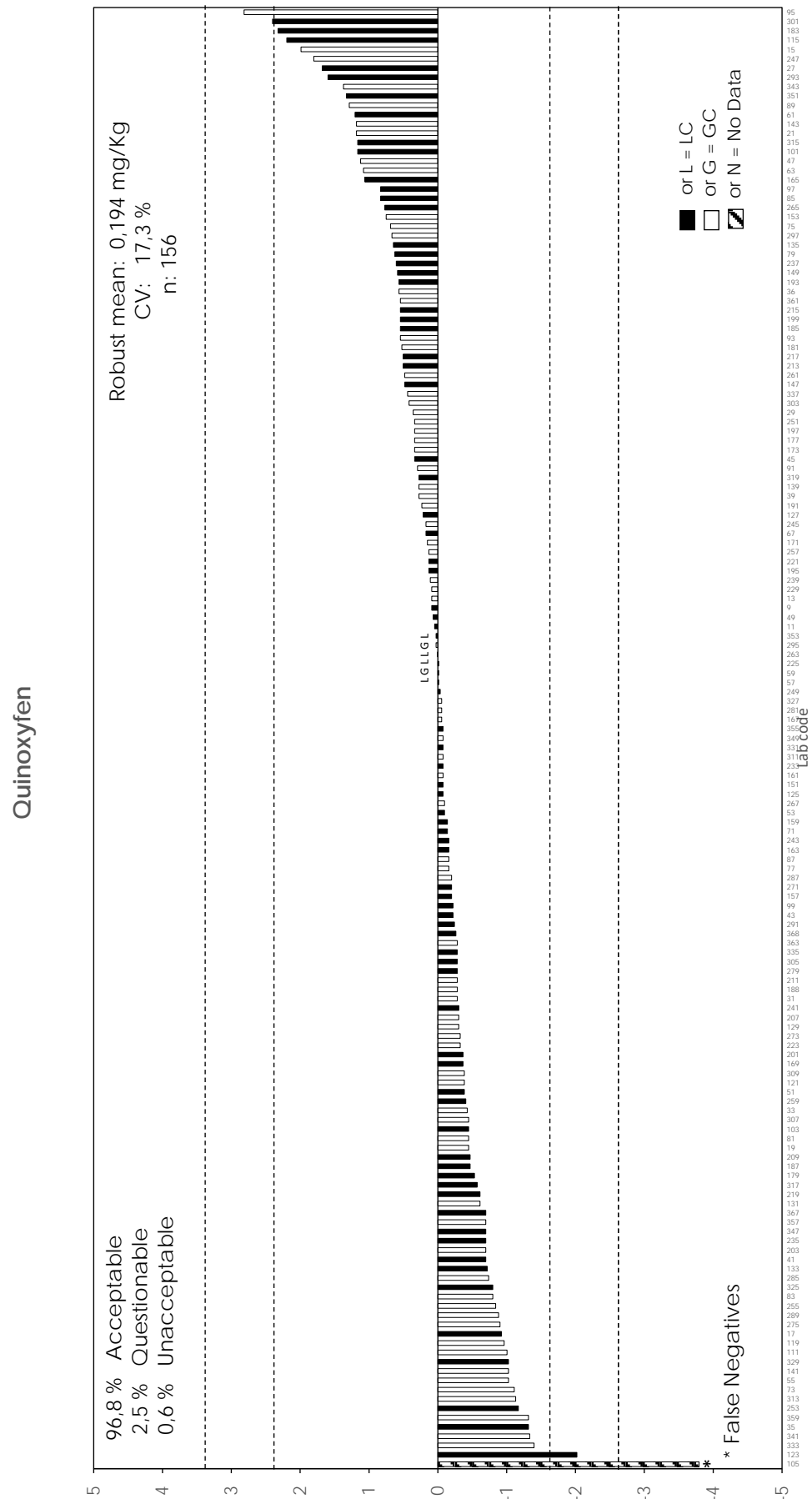


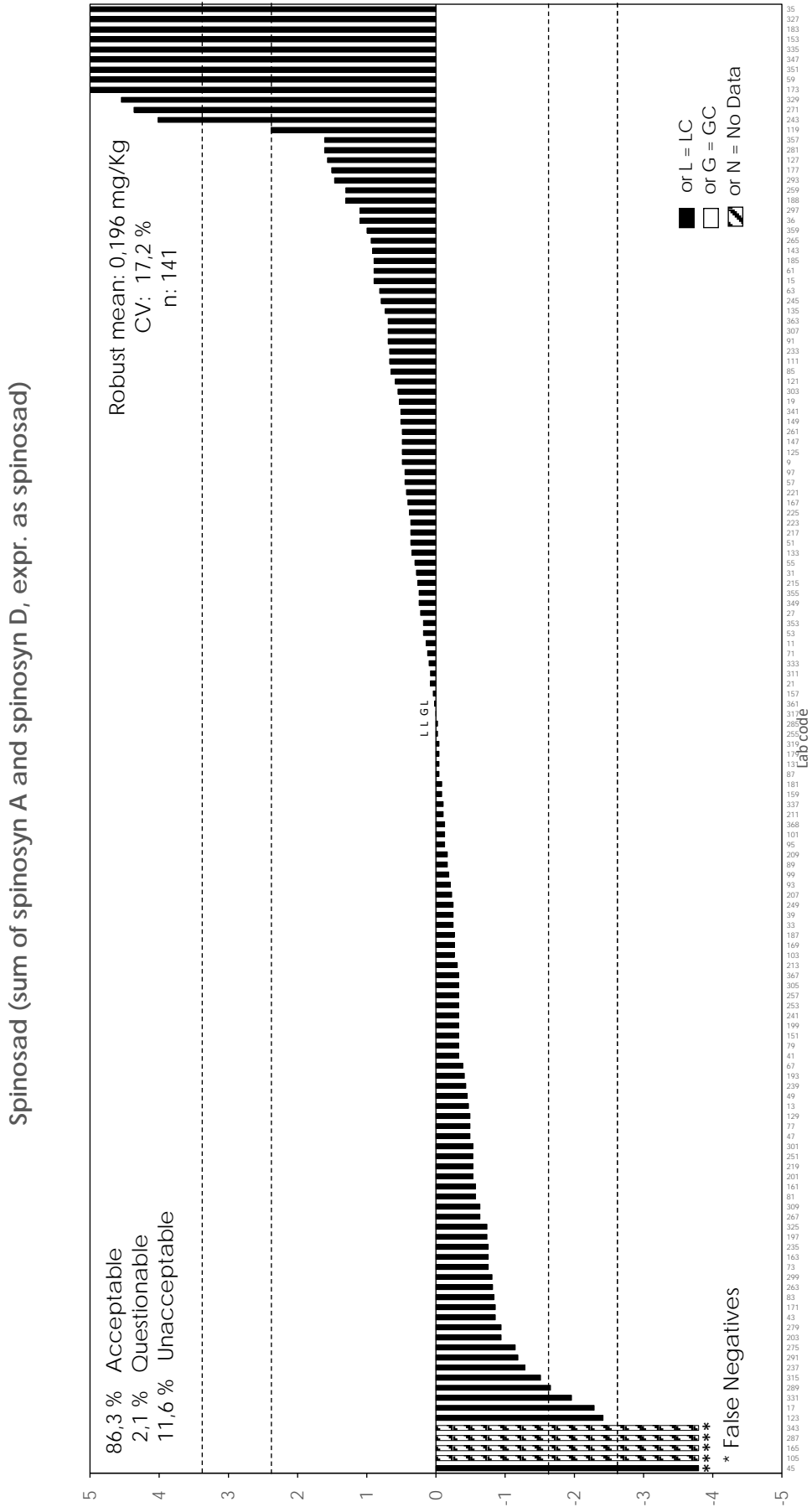
APPENDIX 4. Graphical representation of z scores for FFP-RSD (25 %).





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

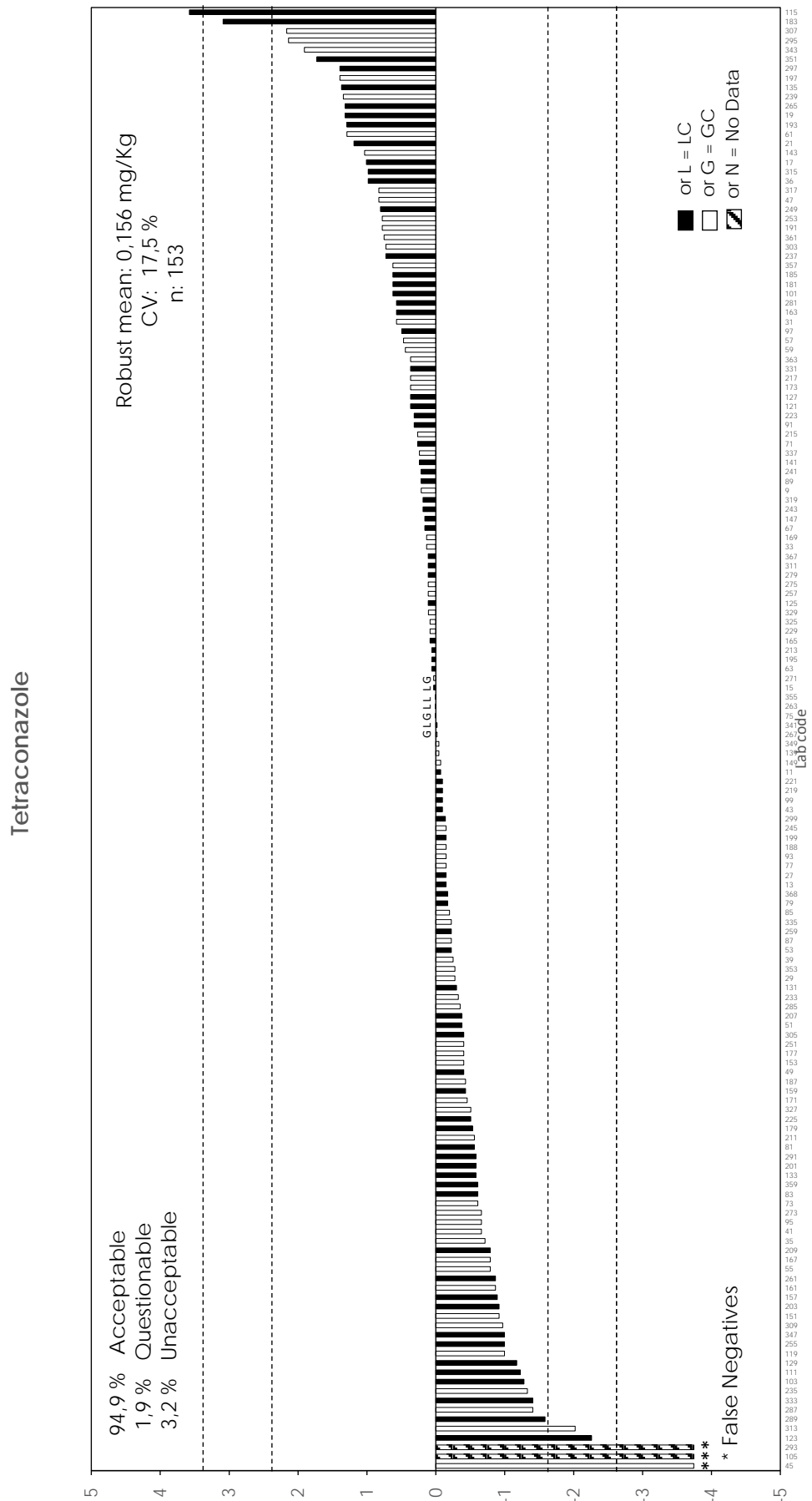




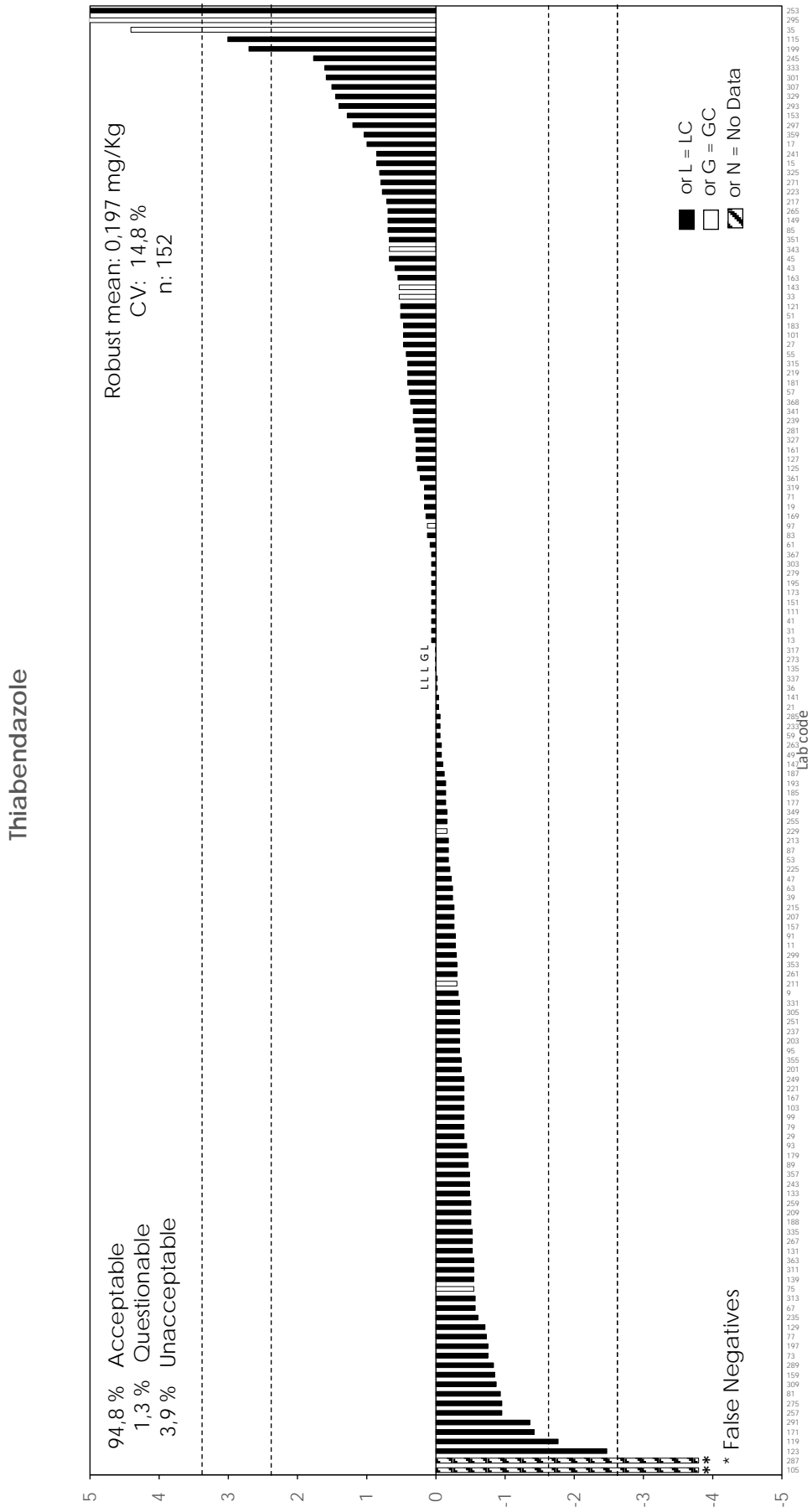


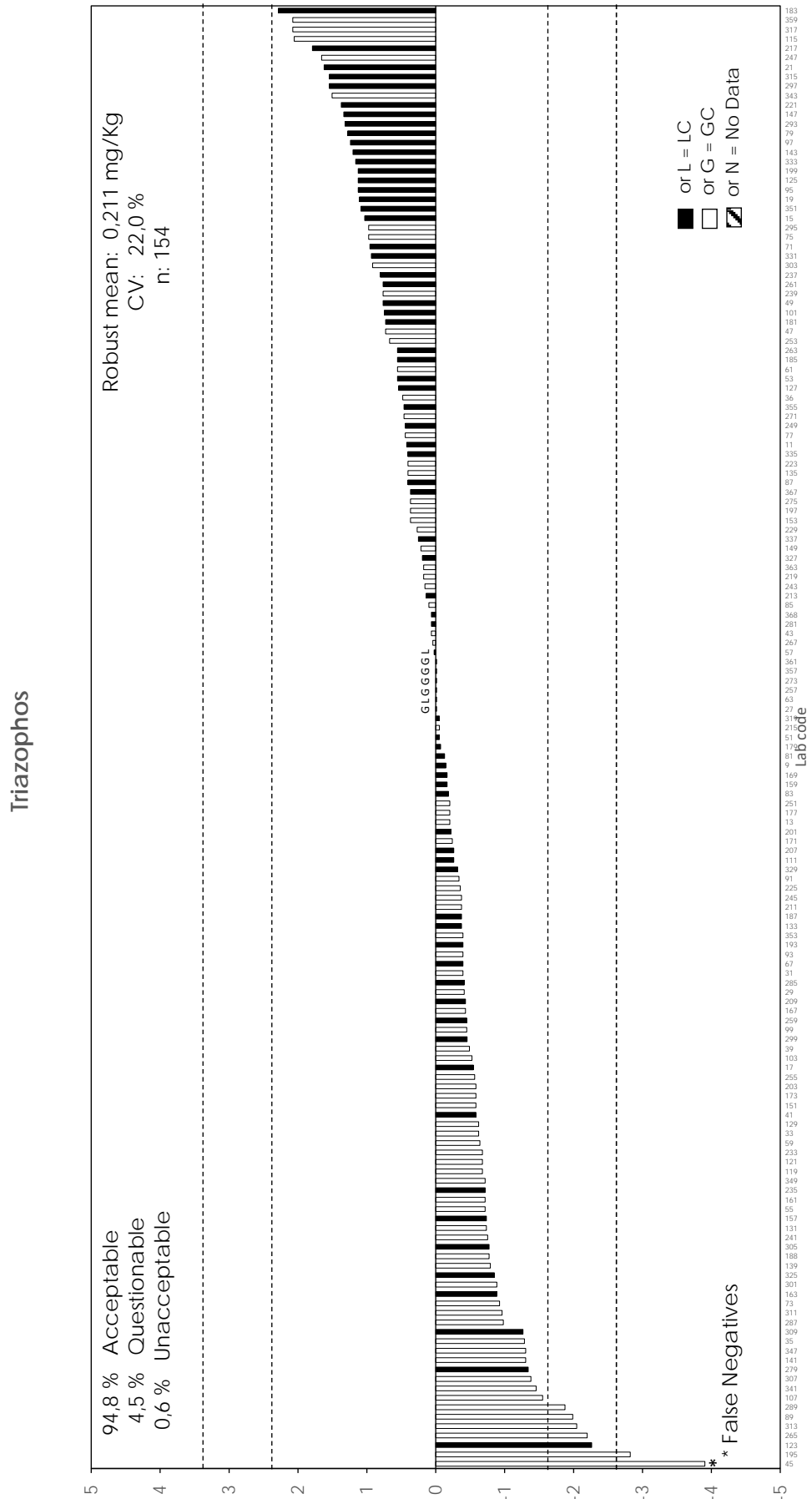
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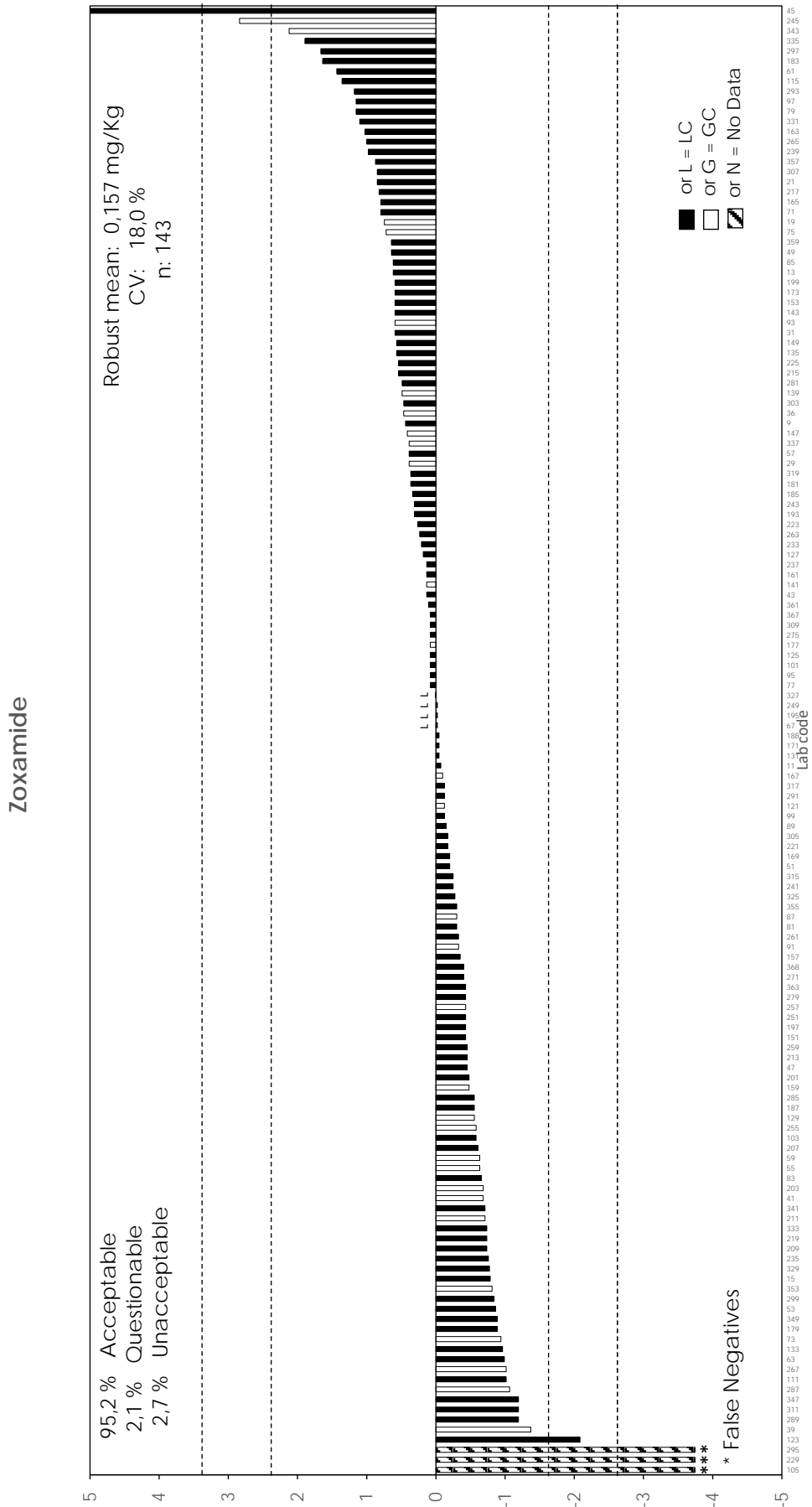


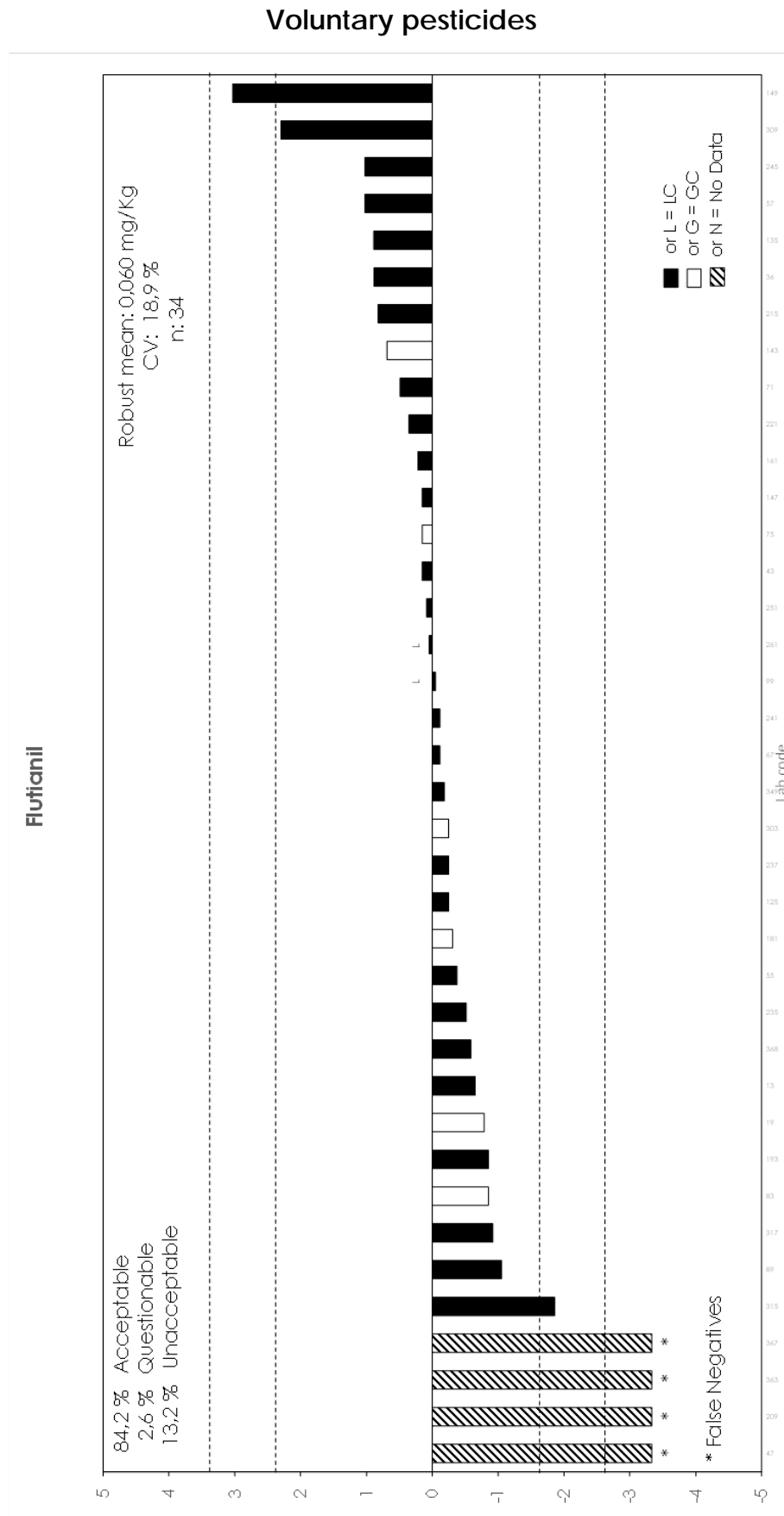


APPENDIX 4. Graphical representation of z scores for FFP-RSD (25 %).

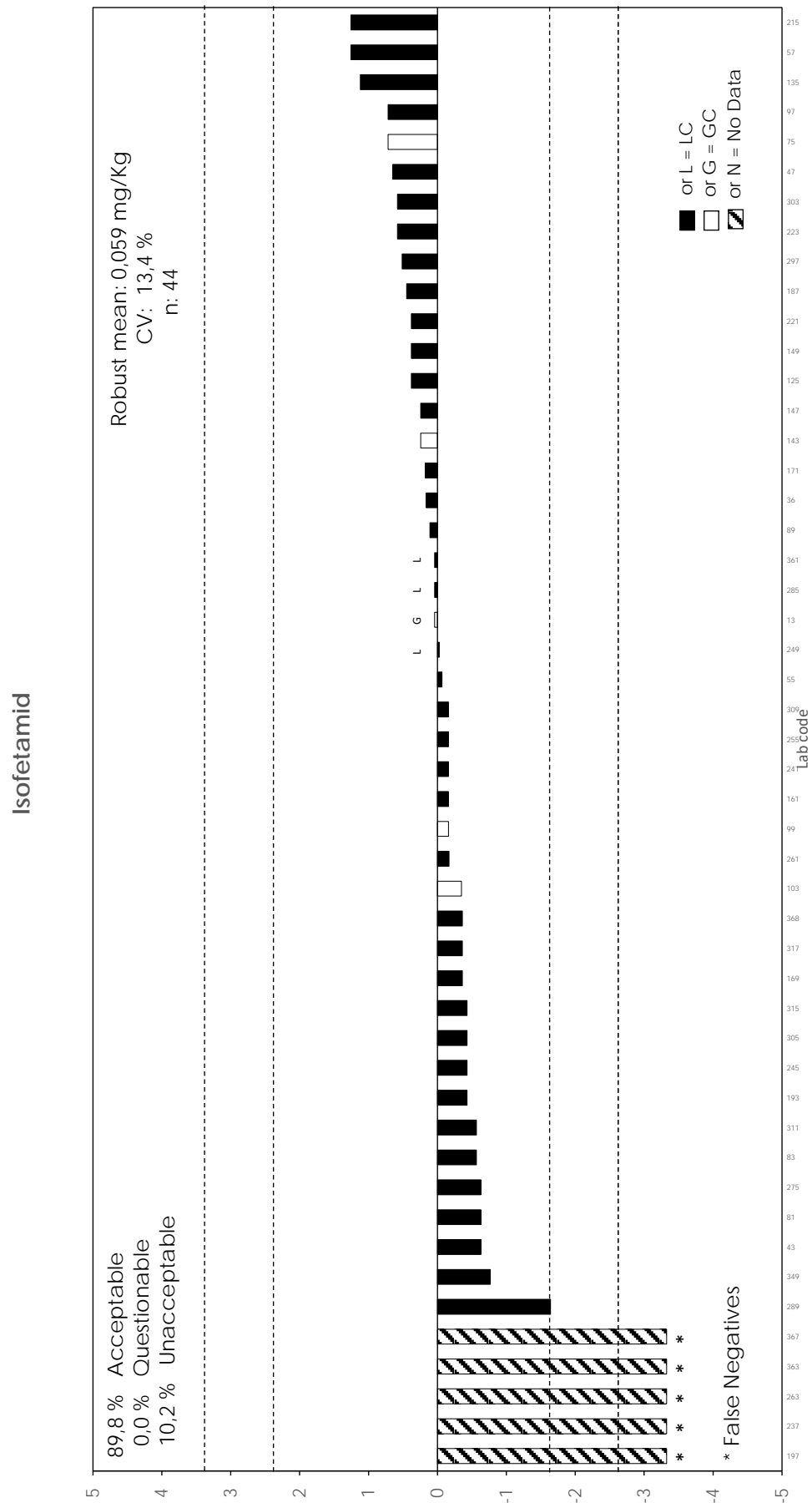








APPENDIX 4. Graphical representation of z scores for FFP-RSD (25 %).



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

Lab Code	Acetamidrid	Chlorfenapyr	Chlorpyrifos	Clofentezine	Diazinon	Dimethoate	Endosulfan sulfate	Fenarimol	Flonicamid	Imazalil	Methomyl	Quinoxifen	Spinosad	Tau-Fluvalinate	Tetraconazole	Thiabendazole	Triazophos	Zoxamide	No. of z scores	AZ <sup>2</sup>
	z score																			
9	-0.9	0.4	0.1	-1.0	-0.2	-0.9	-0.9	0.1	-0.7	-0.5	-0.5	0.1	0.5	1.1	0.2	-0.3	-0.1	0.4	18	0.4
11	0.0	0.2	0.1	0.0	0.0	-0.3	-1.3	0.0	-0.3	-0.4	0.0	0.0	0.1	0.5	-0.1	-0.3	0.4	-0.1	18	0.1
13	-0.6	0.2	0.3	-2.2	0.2	-0.3	-0.2	0.1	0.6	0.9	4.0	0.1	-0.5	-0.9	-0.1	0.1	-0.2	0.6	18	1.3
15	0.3	1.7	2.5	-0.1	3.4	0.5	0.1	0.6	0.2	0.7	0.2	2.0	0.9	-0.1	0.0	0.9	1.0	-0.8	18	1.6
19	0.1	-0.4	-0.5	1.4	1.2	0.3	-0.3	1.8	0.2	0.3	0.4	-0.4	0.5	0.4	1.3	0.2	1.1	0.7	18	0.7
21	-0.2	0.5	1.2	2.8	0.5	-0.2	-0.1	1.2	-0.1	1.5	0.0	1.2	0.1	1.0	1.2	0.0	1.6	0.8	18	1.2
31	0.6	0.5	0.1	0.6	0.8	0.1	0.4	0.1	0.1	0.3	0.4	-0.3	0.3	-0.7	0.6	0.1	-0.4	0.6	18	0.2
36	0.0	0.6	0.7	1.1	0.4	0.2	0.7	1.6	-0.2	-0.4	0.0	0.6	1.1	0.6	1.0	0.0	0.5	0.5	18	0.5
41	-0.8	0.5	0.3	-0.5	-0.9	0.0	0.0	-0.7	-0.3	0.7	-0.3	-0.7	-0.3	0.3	-0.7	0.1	-0.6	-0.7	18	0.3
43	0.4	0.2	-0.5	-0.3	-0.4	0.5	0.0	0.2	0.0	0.7	0.3	-0.2	-0.9	0.3	-0.1	0.6	0.1	0.1	18	0.2
51	0.2	-0.7	-0.6	-0.6	0.0	-0.6	0.2	-0.9	0.2	0.4	0.3	-0.4	0.4	4.6	-0.4	0.5	-0.1	-0.2	18	1.4
53	0.1	0.1	0.0	0.2	0.2	0.3	0.2	-0.1	-0.2	-0.1	-0.2	-0.1	0.2	-0.1	-0.2	-0.2	0.6	-0.9	18	0.1
55	0.9	-0.9	-0.8	-0.3	-0.6	-0.3	-0.9	-0.8	0.9	0.5	0.7	-1.0	0.3	-0.9	-0.8	0.4	-0.7	-0.6	18	0.5
57	0.5	0.5	1.3	0.7	0.8	0.4	0.3	0.5	0.8	0.4	0.6	0.0	0.4	-1.0	0.5	0.4	0.0	0.4	18	0.4
59	-0.1	0.1	0.4	-0.1	-0.3	0.5	0.0	-0.6	0.2	1.9	0.4	0.0	5.0	-0.4	0.4	-0.1	-0.6	-0.6	18	1.7
61	0.6	1.3	2.0	1.1	1.5	0.3	0.4	1.3	0.6	0.2	0.2	1.2	0.9	0.6	1.3	0.1	0.6	1.4	18	1.0
63	1.7	0.6	0.4	-0.3	0.9	0.3	5.0	1.4	0.8	-0.3	1.4	1.1	0.8	1.5	0.1	-0.2	0.0	-1.0	18	2.2
67	-0.5	0.6	0.3	-0.6	-0.2	-0.5	1.0	1.3	-0.3	0.7	-0.8	0.2	-0.4	0.9	0.2	-0.6	-0.4	0.0	18	0.4
71	0.3	0.5	0.6	-0.1	0.9	0.6	-1.4	0.8	0.4	-0.2	1.0	-0.1	0.1	2.0	0.3	0.2	1.0	0.8	18	0.6
73	0.0	-0.5	-0.7	-0.7	-0.9	-0.4	-1.7	-0.8	-0.2	-1.2	0.1	-1.1	-0.8	0.7	-0.6	-0.8	-0.9	-0.9	18	0.7
77	-0.1	-0.5	0.0	-0.5	0.0	0.0	0.0	-0.4	0.0	0.3	-0.1	-0.2	-0.5	-0.5	-0.1	-0.7	0.4	0.1	18	0.1
83	0.0	-0.6	-1.0	-0.8	-0.5	0.1	-0.6	-0.9	-1.1	0.8	0.1	-0.8	-0.8	-1.6	-0.6	0.1	-0.2	-0.7	18	0.6
85	0.0	0.5	-0.1	0.4	-0.3	0.3	0.5	-0.3	0.2	-0.1	1.0	0.8	0.7	0.9	-0.2	0.7	0.1	0.6	18	0.3
87	-0.1	-0.2	-0.2	0.9	-0.2	-0.1	0.4	-0.1	-0.3	-0.3	-0.1	-0.2	0.0	-0.4	-0.2	-0.2	0.4	-0.3	18	0.1
89	-0.3	-0.2	0.0	-0.5	3.0	-0.1	-0.1	2.1	0.2	0.2	-0.2	1.3	-0.2	0.1	0.2	-0.5	-2.0	-0.1	18	1.1
93	0.8	0.7	0.1	-0.3	0.3	-0.1	-0.8	-0.2	0.5	0.5	-0.5	0.5	-0.2	-0.6	-0.1	-0.4	-0.4	0.6	18	0.2
95	-0.1	0.7	1.4	0.6	-0.5	0.4	-0.2	-0.6	-0.1	0.1	-1.5	2.8	-0.1	3.1	-0.7	-0.3	1.1	0.1	18	1.4
97	2.6	-0.3	0.4	-1.0	0.5	0.2	-0.3	-0.1	0.5	-1.4	1.9	0.8	0.4	0.4	0.5	0.1	1.2	1.2	18	1.0
99	-0.5	0.9	-0.3	-0.5	-0.4	0.2	0.5	-0.5	-0.4	-0.8	-0.5	-0.2	-0.2	1.5	-0.1	-0.4	-0.5	-0.1	18	0.3
109	0.0	1.5	1.1	0.3	-0.2	-0.2	1.1	0.0	-0.9	-0.3	-0.8	-0.1	-1.3	2.3	-0.2	-0.1	0.3	0.1	18	0.8
111	-0.5	0.4	-1.8	-1.2	0.8	-0.2	0.0	0.4	1.3	-0.7	-0.5	-1.0	0.7	0.7	-1.2	0.1	-0.3	-1.0	18	0.7
113	0.0	-0.4	-0.7	0.3	-1.1	-0.1	-0.3	-0.8	-0.5	-1.0	-0.2	-0.4	-0.1	-0.2	-0.2	-0.2	-0.5	-0.4	18	0.2
117	0.0	-0.9	-0.9	-0.8	-1.1	-0.5	-0.6	-1.6	-0.5	-0.7	-0.2	-1.1	-1.1	-1.0	-0.8	-0.7	-1.1	-1.3	18	0.8
119	-0.4	-0.6	-0.1	-0.5	-1.6	-0.9	-0.6	-1.2	-0.6	-3.1	-0.8	-1.0	2.4	-0.8	-1.0	-1.8	-0.7		17	1.7
121	-0.7	-0.6	-0.3	3.5	-0.1	0.0	-0.5	-0.5	0.7	0.3	0.2	-0.4	0.6	-0.6	0.4	0.5	-0.7	-0.1	18	0.9
125	-0.1	0.1	-0.2	-0.4	0.9	-0.2	0.7	0.0	-0.5	0.9	-0.1	-0.1	0.5	0.9	0.1	0.3	1.1	0.1	18	0.3
127	0.4	-0.1	0.4	0.2	0.6	0.5	-3.9	0.9	0.2	0.8	0.1	0.2	1.6	0.7	0.4	0.3	0.5	0.2	18	1.2
129	0.8	0.0	-0.2	-2.5	-0.4	-0.2	0.1	-0.1	-0.3	-0.7	-0.6	-0.3	-0.5	0.1	-1.2	-0.7	-0.6	-0.6	18	0.6
131	0.0	-0.6	-1.3	-0.2	-0.4	-0.3	-0.4	-0.4	0.0	-1.2	0.0	-0.6	0.0	0.3	-0.3	-0.5	-0.7	0.0	18	0.3
133	0.0	-2.2	1.2	-0.4	-0.1	-0.6	1.3	1.1		0.1	0.2	-0.7	0.3	1.2	-0.6	-0.5	-0.4	-1.0	17	0.8
147	0.2	0.5	-0.2	0.7	0.6	0.4	0.3	0.1	-0.2	0.7	-1.5	0.5	0.5	0.2	0.2	-0.1	1.3	0.4	18	0.4
149	0.2	-0.6	0.5	0.4	0.5	0.5	0.2	0.1	0.5	0.2	0.6	0.6	0.5	0.3	-0.1	0.7	0.2	0.6	18	0.2

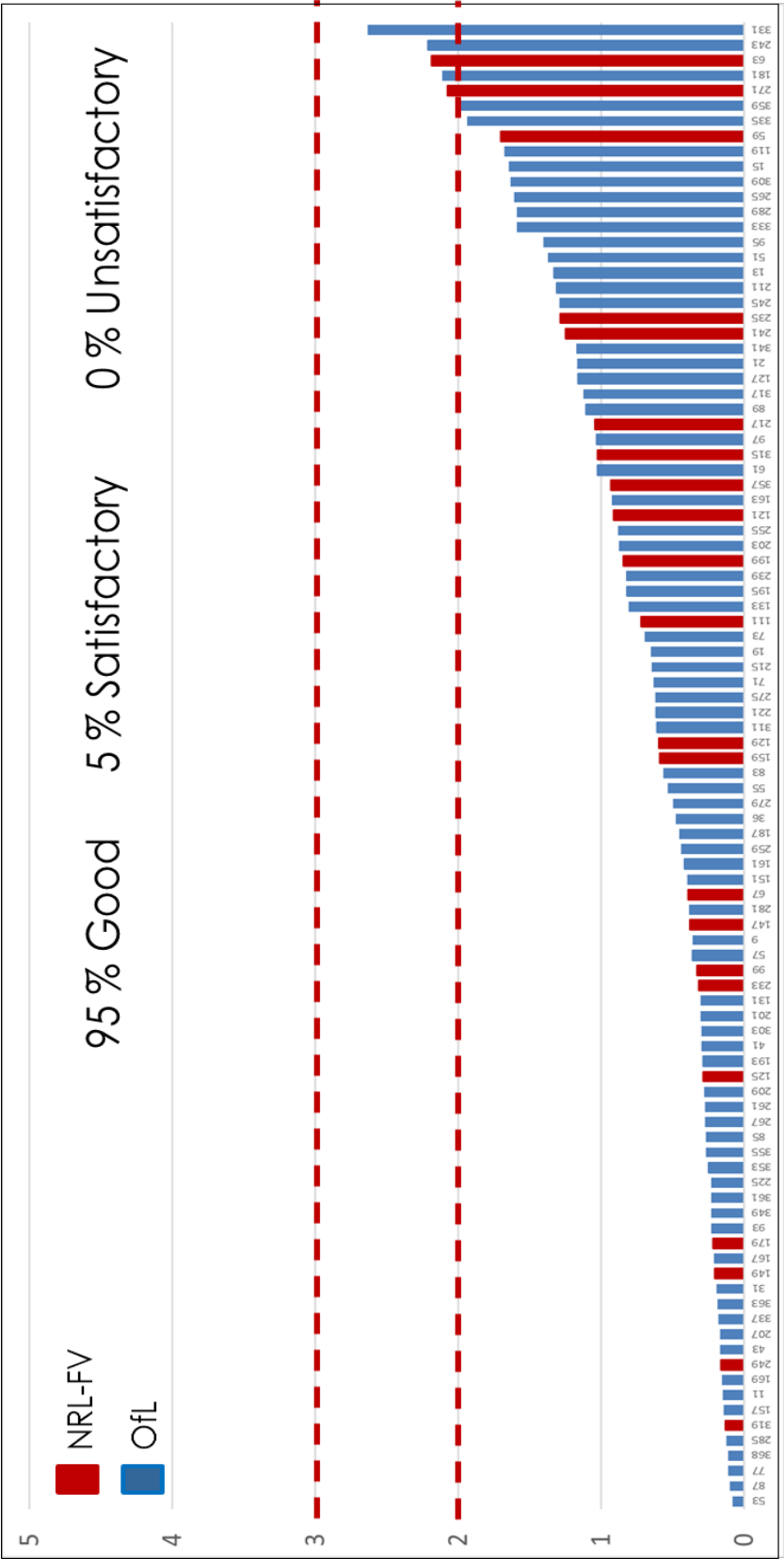


APPENDIX 5. Average of the Squared z scores (AZ2) for laboratories in Category A.

Lab Code	Acetamiprid	Chlorfenapyr	Chlorpyrifos	Clofentezine	Diazinon	Dimethoate	Endosulfan sulfate	Fenarimol	Flonicamid	Imazalil	Methomyl	Quinoxifen	Spinosad	Tau-Fluvalinate	Tetraconazole	Thiabendazole	Triazophos	Zoxamide	No. of z scores	Az <sup>2</sup>
	z score																			
151	-0.1	0.0	-0.6	-0.7	-0.6	0.1	-1.2	-0.9	-0.2	0.7	-0.3	-0.1	-0.3	-1.2	-0.9	0.1	-0.6	-0.4	18	0.4
157	-0.3	-0.4	-0.2	0.0	0.2	-0.3	0.4	-0.3	-0.3	-0.4	0.2	-0.2	0.0	0.1	-0.9	-0.3	-0.7	-0.4	18	0.1
159	-0.3	0.0	-0.2	0.4	-0.1	-0.1	0.3	-0.1	3.0	-0.2	-0.2	-0.1	-0.1	0.0	-0.4	-0.9	-0.2	-0.5	18	0.6
161	-0.7	-0.4	-0.7	-1.0	-0.3	-0.4	-0.1	-1.5	-0.3	-0.4	0.2	-0.1	-0.6	-0.9	-0.9	0.3	-0.7	0.1	18	0.4
163	0.4	0.1	0.0	-3.0	-0.5	0.2	0.6	-0.3	0.0	0.5	-0.7	-0.2	-0.8	1.8	0.6	0.5	-0.9	1.0	18	0.9
167	0.6	-0.3	-0.3	-0.5	-0.5	0.2	0.0	-0.7	0.6	0.0	-0.8	-0.1	0.4	-0.2	-0.8	-0.4	-0.4	-0.1	18	0.2
169	0.2	0.2	0.0	0.1	-0.1	0.4	1.2	-0.3	0.5	-0.6	0.0	-0.4	-0.3	-0.1	0.1	0.1	-0.2	-0.2	18	0.2
179	-0.5	-0.5	-0.4	-0.6	-0.4	-0.1	-0.5	-0.5	-0.2	0.2	-0.6	-0.5	0.0	-0.5	-0.5	-0.5	-0.1	-0.9	18	0.2
181	0.0	0.7	0.9	0.8	0.3	0.4	0.0	2.5	5.0	1.6	0.6	0.5	-0.1	0.2	0.6	0.4	0.7	0.4	18	2.1
187	-0.3	-0.2	-0.7	-0.1	-1.0	0.1	-1.4	-0.8	-0.5	-0.8	0.2	-0.5	-0.3	-1.4	-0.4	-0.1	-0.4	-0.6	18	0.5
193	0.1	-0.2	0.6	0.8	-0.2	-0.4	0.7	-0.5	-0.2	-0.7	0.0	0.6	-0.4	0.3	1.3	-0.1	-0.4	0.3	18	0.3
195	-0.1	-1.3	-0.7	-0.1	-0.7	0.0	0.1	-1.0		0.5	0.1	0.1		-1.0	0.1	0.1	-2.8	0.0	16	0.8
199	-0.1	0.5	-0.3	1.4	0.8	0.7	0.5	-0.2	0.3	0.9	0.1	0.5	-0.3	-1.0	-0.1	2.7	1.1	0.6	18	0.8
201	-1.1	-0.5	-0.5	-0.6	-0.7	-0.2	-0.4	-1.2	0.2	0.2	-0.1	-0.4	-0.5	0.2	-0.6	-0.4	-0.2	-0.5	18	0.3
203	-0.8	-0.9	-0.5	-0.2	-1.3	-1.3	-0.8	-1.5	-0.8	-1.2	-1.5	-0.7	-0.9	-0.6	-0.9	-0.3	-0.6	-0.7	18	0.9
205	0.6	0.9	-0.2	0.0	-0.4	-0.1	-0.3	1.8	-0.2	-0.2	0.1	-0.1	0.5	-0.3	0.6	0.3	-0.6	0.1	18	0.3
207	-0.6	0.1	-0.1	-0.5	-0.3	-0.5	0.2	-0.5	-0.4	-0.1	-0.8	-0.3	-0.2	-0.4	-0.4	-0.3	-0.3	-0.6	18	0.2
209	-0.7	0.0	-0.5	-0.8	-0.5	-0.1	0.6	0.0	-0.6	-1.0	-0.3	-0.5	-0.2	0.0	-0.8	-0.5	-0.4	-0.7	18	0.3
211	-0.5	-0.2	-0.2	-0.8	-0.3	0.7	0.2	-0.4	-3.6	0.1	-2.8	-0.3	-0.1	-0.1	-0.6	-0.3	-0.4	-0.7	18	1.3
215	-0.8	1.5	1.2	-0.9	0.1	-0.8	1.6	0.5	-0.7	0.4	-0.6	0.5	0.3	1.0	0.3	-0.3	-0.1	0.5	18	0.6
217	0.1	0.1	2.0	1.3	-0.2	1.0	1.3	0.6	0.6	2.1	0.6	0.5	0.4	-0.1	0.4	0.7	1.8	0.8	18	1.0
221	-0.4	-0.1	-0.2	0.1	-0.3	1.3	0.2	-1.2	0.2	-0.7	-0.1	0.1	0.4	2.2	-0.1	-0.4	1.4	-0.2	18	0.6
225	-0.1	0.6	0.2	0.3	0.0	-0.2	1.0	-0.7	-0.3	-1.1	-0.2	0.0	0.4	0.1	-0.5	-0.2	-0.4	0.5	18	0.2
233	-0.3	-0.4	-0.2	0.4	-0.6	-0.5	0.3	1.2	-0.9	-0.2	-0.6	-0.1	0.7	0.8	-0.3	-0.1	-0.7	0.2	18	0.3
235	-0.4	-1.2	-0.7	0.4	-1.5	0.3	-1.3	-1.7	0.0	-0.9	-0.3	-0.7	-0.8	-3.0	-1.3	-0.6	-0.7	-0.8	18	1.3
239	-0.2	0.8	0.8	1.4	0.5	-0.6	1.6	0.9	-0.5	0.7	-0.7	0.1	-0.4	1.8	1.3	0.3	0.8	1.0	18	0.8
241	0.9	0.3	0.4	0.2	0.1	2.1	1.3	1.1	1.6	2.7	1.6	-0.3	-0.3	0.3	0.2	0.9	-0.8	-0.3	18	1.2
243	0.1	0.2	0.5	-0.9	0.1	-0.1	-2.7	-0.9	-3.6	0.5	-0.7	-0.2	4.0	0.3	0.2	-0.5	0.2	0.3	18	2.2
245	0.2	0.5	0.6	-1.7	0.0	0.0	-0.2	-0.3	0.0	0.1	0.6	0.2	0.8	2.7	-0.1	1.8	-0.4	2.8	18	1.3
249	-0.2	0.2	0.1	-0.1	0.5	-0.9	-0.1	-0.1	-0.2	-0.6	0.0	0.0	-0.2	0.5	0.8	-0.4	0.4	0.0	18	0.2
255	-0.1	0.2	-0.7	-3.0	-0.8	-0.1	0.1	-1.5	-0.3	-0.5	0.9	-0.8	0.0	-0.1	-1.0	-0.2	-0.6	-0.6	18	0.9
259	-0.5	-0.6	-0.4	-1.2	0.4	-1.4	-0.6	0.4	-0.3	0.3	0.3	-0.4	1.3	0.1	-0.2	-0.5	-0.5	-0.5	18	0.4
261	0.2	1.1	0.1	-0.4	-0.3	-0.4	0.0	-0.8	-0.4	0.5	0.1	0.5	0.5	0.3	-0.9	-0.3	0.8	-0.3	18	0.3
265	1.3	-0.5	-0.9	0.8	-0.3	0.4	-2.4	-0.5	1.3	0.8	1.0	0.8	0.9	-2.6	1.3	0.7	-2.2	1.0	18	1.6
267	-0.4	0.2	0.0	-0.3	-0.5	0.0	-0.6	0.2	-0.5	-0.7	-0.7	-0.1	-0.6	-1.0	0.0	-0.5	0.0	-1.0	18	0.3
271	0.3	0.2	0.2	0.8	-0.5	0.2	-3.9	-0.5	0.3	0.5	0.6	-0.2	4.4	0.6	0.0	0.8	0.5	-0.4	18	2.1
275	-0.3	0.3	0.0	0.1	1.3	-0.5	0.8	-0.7	-0.9	-1.4	-0.5	-0.9	-1.1	1.2	0.1	-1.0	0.4	0.1	18	0.6
279	-0.1	0.7	1.5	-0.5	-0.6	-0.5	0.2	0.5	0.3	-0.6	1.1	-0.3	-0.9	-0.6	0.1	0.1	-1.3	-0.4	18	0.5
281	0.5	0.4	0.5	0.8	0.5	0.8	0.5	0.7	0.7	0.5	0.1	-0.1	1.6	0.3	0.6	0.3	0.1	0.5	18	0.4
285	0.0	-0.6	-0.3	-0.4	-0.1	-0.2	-0.4	-0.3	-0.2	0.2	0.0	-0.7	0.0	0.0	-0.4	-0.1	-0.4	-0.6	18	0.1
289	-1.2	-0.8	-1.1	-0.2	-1.1	-1.9	-1.5	0.1	-1.4	-1.0	-1.6	-0.9	-1.7	-1.0	-1.6	-0.8	-1.9	-1.2	18	1.6
303	-0.1	-0.2	0.6	-0.2	0.8	0.4	0.0	0.8	0.2	-0.5	0.4	0.4	0.5	0.9	0.7	0.1	0.9	0.5	18	0.3
309	-0.3	2.5	3.3	0.5	-0.1	-0.6	2.4	-0.2	-0.4	-0.5	-0.7	-0.4	-0.6	-1.0	-1.0	-0.9	-1.3	0.1	18	1.6

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

Lab Code	Acetamiprid	Chlorfenapyr	Chlorpyrifos	Clofentazine	Diazinon	Dimethoate	Endosulfan sulfate	Fenarimol	Flonicamid	Imazalil	Methomyl	Quinoxifen	Spinosad	Tau-Fluvalinate	Tetraconazole	Thiabendazole	Triazophos	Zoxamide	No. of z scores	AZ <sup>2</sup>
z score																				
311	-0.1	-0.3	-0.5	-0.9	-0.4	-0.3	-1.6	-0.6	-0.9	0.1	-0.5	-0.1	0.1	-1.7	0.1	-0.5	-1.0	-1.2	18	0.6
315	1.2	1.1	0.9	1.1	0.7	0.8	-0.5	2.0	1.0	0.4	0.2	1.2	-1.5	-0.3	1.0	0.4	1.5	-0.3	18	1.0
317	-0.1	-2.1	-1.9	-1.5	0.6	0.0	-1.7	-1.2	0.1	-0.5	-0.2	-0.6	0.0	0.0	0.8	0.0	2.1	-0.1	18	1.1
319	-0.4	-0.2	0.4	0.3	0.0	-0.1	0.5	0.4	-0.2	1.0	-0.2	0.3	0.0	-0.3	0.2	0.2	-0.1	0.4	18	0.1
321	0.5	0.1	-1.8	-0.7	-1.4	1.3	-1.0	-0.7	-3.6	-0.5	-0.1	-0.1	0.0	-0.2	-0.7	0.0	-0.8	-0.3	18	1.3
323	0.7	-0.9	-1.1	-2.0	-0.7	0.3	-0.8	-1.5	0.5	-0.5	-0.6	-1.2	1.5	-1.5	-0.8	0.0	-1.0	-0.1	18	1.0
331	-0.8	0.4	1.7	2.6	1.8	-0.1	-0.2	5.0	-0.5	-1.2	-0.3	-0.1	-2.0	-0.6	0.4	-0.3	0.9	1.1	18	2.6
333	1.6	-1.0	-0.9	1.6	-0.9	-0.5	0.3	0.6	2.7	1.6	0.4	-1.4	0.1	-1.3	-1.4	1.6	1.2	-0.7	18	1.6
335	0.4	-0.4	0.0	-0.8	0.5	-0.6	1.3	1.1	0.1	1.0	0.4	-0.3	5.0	0.4	-0.2	-0.5	0.4	1.9	18	1.9
337	0.5	0.9	0.5	-0.4	-0.1	-0.1	0.6	0.3	0.2	-0.1	0.5	0.4	-0.1	0.6	0.2	0.0	0.3	0.4	18	0.2
341	-0.1	-2.0	-1.6	0.3	-2.0	0.0	-1.3	-0.7	-0.9	0.2	0.7	-1.3	0.5	-1.5	0.0	0.3	-1.5	-0.7	18	1.2
349	0.1	-0.2	-0.7	-0.6	-0.3	0.2	0.1	-0.4	-0.3	-1.0	0.5	-0.1	0.2	0.0	0.0	-0.2	-0.7	-0.9	18	0.2
353	0.7	-0.6	-0.6	0.6	-0.1	0.0	-0.2	-0.6	1.1	0.2	0.3	0.0	0.2	-0.4	-0.3	-0.3	-0.4	-0.8	18	0.2
355	0.2	-0.5	-0.4	0.2	-0.3	-0.4	-0.1	0.5	1.3	-0.9	-0.5	-0.1	0.2	-0.8	0.0	-0.4	0.5	-0.3	18	0.3
357	0.7	-0.7	-0.3	0.6	0.7	0.7	-1.7	0.3	1.1	0.8	0.8	-0.7	1.6	-2.1	0.6	-0.5	0.0	0.9	18	0.9
359	1.1	-0.7	-1.8	-3.6	0.2	-0.1	1.2	0.8	1.1	2.5	0.5	-1.3	1.0	-0.4	-0.6	1.0	2.1	0.6	18	2.0
361	0.2	1.1	0.8	-0.1	-0.1	-0.5	0.5	0.1	0.2	-0.5	0.3	0.5	0.0	0.8	0.8	0.2	0.0	0.1	18	0.2
363	0.1	-0.1	0.4	0.6	0.7	0.4	0.2	0.0	0.3	-0.2	-0.1	-0.3	0.7	-0.9	0.4	-0.5	0.2	-0.4	18	0.2
368	-0.2	0.0	-0.3	0.7	-0.3	-0.4	-0.1	0.0	-0.2	-0.8	0.0	-0.3	-0.1	0.1	-0.2	0.4	0.1	-0.4	18	0.1



## GENERAL PROTOCOL

### for EU Proficiency Tests on Pesticide Residues in Food and Feed

#### Introduction

This protocol contains general procedures valid for all European Union Proficiency Tests (EUTs) organised on behalf of the European Commission, DG-SANTE<sup>6</sup> by the four European Union Reference Laboratories (EURLs) responsible for pesticide residues in food and feed. These EUTs are directed at laboratories belonging to the Network<sup>7</sup> of National Reference Laboratories (NRLs) and Official Laboratories (OLs) of the EU Member States. OLs from EFTA countries and EU-Candidate countries are also welcome to participate in the EUTs. OLs from Third countries may be permitted to participate on a case-by-case basis.

The following four EURLs for pesticide residues were appointed by DG-SANTE based on regulation 882/2004/EC that was repealed by regulation 625/2017/EC<sup>8</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 EUTs 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>9</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.

#### EUT-Organisers and Scientific Committee

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

An **Organising Team** (in the following named Organisers) 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, the production of the PT-material (Test Item), the undertaking of homogeneity and stability tests, the packing and shipment of the PT-materials, the handling and evaluation of the results and method information submitted by the participants, the drafting of the preliminary and final reports as well as generation and distribution of EUT-participation certificates.

To complement the internal expertise of the EURLs, a group of external consultants forming the **EUT-Scientific Committee** (EUT-SC)<sup>10</sup> has been established and approved by DG-SANTE. The EUT-SC consists of expert scientists with many years of experience in PTs and/or pesticide residue analysis. The actual composition of the EUT-SC and the affiliation of each of its members is shown on the EURL-Website. The members of the EUT-SC are also listed in the Specific Protocol and the Final Report of each EUT.

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

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

The EUT-SC's role is to help the Organisers make decisions regarding the EUT 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 the participants' results (in anonymous form), and the drafting and updating of documents, such as the General and Specific PT Protocols and the Final EUT-Reports.

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

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

<sup>6</sup> DG-SANTE = European Commission, Health and Food Safety Directorate-General

<sup>7</sup> For more information about the EURL/NRL/OL-Network please refer to the EURL-Web-portal under: "<http://www.eurl-pesticides.eu>"

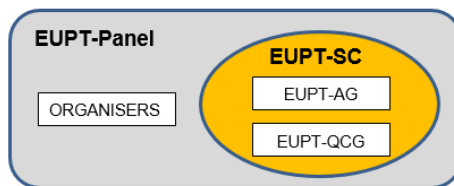
<sup>8</sup> Regulation (EU) 2017/625 of the European Parliament and of the Council on official controls and other official activities performed to ensure the application of food and feed law, rules on animal health and welfare, plant health and plant protection products. Published at OJ of the EU L95 of 07.04.2017

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

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

## ANNEX A. Protocols and Target lists of pesticides to be sought.

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.

### 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 38 (b) of Reg. 625/2017/EC and Art. 28 of Reg. 396/2005/EC<sup>11</sup> (for all OfLs analysing for pesticide residues within the framework of official controls<sup>12</sup> of food or feed)
- Art. 101 (1)(a) of Reg. 625/2017/EC (for all NRLs)

The four EURLs will annually issue and distribute, via the EURL-website, a joint list of all OfLs that must participate in each of the EUPTs to be conducted within a given year. The list of obliged labs will be updated every year to take account of any changes in the lab profiles. Interim updates will be issued to eliminate any possible errors. NRLs are responsible for checking whether all relevant OfLs within their network are included in the list of obligated laboratories with their actual commodity-scopes and contact information.

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. This also applies to any participating laboratories that fail to report results.

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 corresponding NRL 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.

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

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

For each EUPT the organising EURL prepares a specific EUPT-Website where all PT-relevant documents in their latest version are linked. In case of important modifications on any of these documents, the participating laboratories will be informed via e-mail. In any case, as soon as the PT-period starts the participants are encouraged to visit the particular EUPT-Website, to make sure that they are using the latest versions of all PT-relevant documents.

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 for, 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).

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<sup>11</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>12</sup> Official controls in the sense of Reg. 625/2017/EC. This includes labs involved in controls within the framework of national and/or EU-controlled programmes as well as labs involved in import controls according to Regulation 669/2009/EC.

## ANNEX A. Protocols and Target lists of pesticides to be sought.

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 usually 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-Panel, considering all relevant aspects (e.g. the homogeneity results of other pesticides spiked at the same time, the overall distribution of the participants' results (CV\*), 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. For certain analytes with comparable properties, an equivalent distribution within the sample can be expected if they were spiked/used at simultaneously. The homogeneity test, of one or more of these analytes, may thus be skipped or simplified. If, however, the distribution of participants' results for an analyte that was not or not fully tested for homogeneity, is found to be atypically broad, compared to the tested analytes, the EUPT-SC may decide that a homogeneity test should be performed *a posteriori* by the EURL.

### 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-Panel will finally decide whether analytes for which the stability test was not undertaken will be included in the Final EUPT-Report, considering all relevant aspects such as the distribution of the participant's results (CV\*).

A pesticide is considered to be adequately stable if  $|y_i - \bar{y}| \leq 0.3 \times \sigma_{pt}$ , with  $y_i$  being the mean value of the results of the last phase of the stability test,  $\bar{y}$  being the mean value of the results of the first phase of the stability test and  $\sigma_{pt}$  being 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.

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.

**Stability during shipment:** 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 keep track of the shipment duration and then decide whether it is reasonable to 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 by the EUPT-Panel considering all relevant aspects including the duration and conditions of the shipment to the laboratory as well as the feedback by the 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. Laboratories should not report results below their reporting limits.

## ANNEX A. Protocols and Target lists of pesticides to be sought.

### Correction of results for recovery

Correction of results for recovery is recommended if the average recovery rate significantly deviates from 100 % (typically if outside the 80–120% range). Approaches for recovery correction explicitly stated in the DG-SANTE document are

- a) the use of recovery correction factors,
- b) the use of stable isotope labelled analogues of the target analytes as Internal Standards (ILISs),
- c) the 'procedural calibration' approach as well as
- d) 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. If one or more of the approaches b), c) and d) were employed, in which correction for recovery is inherent to the procedures, the apparent recovery figures obtained during validation experiments are not mandatory, and the approach followed are to be reported in the appropriate fields within the data submission tool.

### 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 EUPT-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 Organisers reserve 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 Positive results

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

#### – False Negative results

These are results for pesticides reported by the laboratories as 'analysed' but without reporting numerical values although they were: a) used by the Organiser to treat the Test Item and b) detected by the Organiser as well as the majority of the participants that had targeted these specific pesticides at or above the respective 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 the robust estimate of the participant's mean ( $x^*$ ) as described in ISO 13528:2015<sup>13</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 or similar compounds 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), inappropriate storage or transport conditions (in case of susceptible compounds), and the use of inappropriate analytical steps or procedures that demonstrably lead to significantly biased results (e.g. employing inappropriate internal standards or analytical steps or conditions leading to considerable losses, due to degradations, adsorptions, incomplete extractions, partitioning etc.). 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

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<sup>13</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).

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").

Results reported by laboratories from non EU member states are typically excluded from the population that is used to derive the assigned value (see also "Estimation of the assigned value").

#### Uncertainty of the assigned value

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

$$u(x_{pt}) = 1,25 \times \frac{s^*}{\sqrt{p}}$$

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).

Based on experience from previous EUPTs<sup>14</sup>, a percentage FFP-RSD of 25 % is currently used for all analyte-matrix combination, with the target standard deviation being calculated as follows:

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

The EUPT-Panel reserves the right to also employ other FFP-RSDs or other approaches for setting the assigned value on a case-by-case basis, considering analytical difficulties and experience gained from previous proficiency tests.

For informative purposes the robust relative standard deviation ( $CV^*$ ) of the participants results is calculated according to ISO 13528:2015; Chapter 7.7 following Algorithm A in Annex C (so called "consensus approach").

#### – 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 the 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 the combined z-scores will be 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>15</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  $RL < MRRL$ . Where, using this approach, the calculated z scores for false negatives are > -3 (still questionable), they will be fixed at -3.5 to underline that these are unacceptable results. These z-scores will typically appear in the z-score histograms and used in the calculation of combined z-scores.

#### – Collection of measurement uncertainty (MU) figures

The participating labs will be asked to report the MU figure they would routinely report with each EUPT result. The EUPT-Panel will decide whether and how to evaluate these figures and whether indications will be made to the laboratories in this respect.

#### – Category classification

The EUPT-Panel will decide if and how to classify the laboratories into categories based on their scope and/or performance. Currently a scope-based classification into Category A and Category B is employed.

<sup>14</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>15</sup> ISO/IEC 17043:2010. Conformity assessment – General requirements for proficiency testing



## ANNEX A. Protocols and Target lists of pesticides to be sought.

Laboratories that a) are able to analyse at least 90% of the compulsory pesticides in the target pesticides list, b) have correctly detected and quantified a sufficiently high percentage of the pesticides present in the Test Item (at least 90 %) and c) reported no false positives, will have demonstrated 'sufficient scope' and will be therefore classified into Category A. For the 90% criterion the number of pesticides needed to be correctly analysed to have sufficient scope will be calculated by multiplying the number of compulsory pesticides from the Target Pesticides List by 0.9 and rounding to the nearest full number with 0.5 decimals being rounded downwards (see some examples in Table 1).

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

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

The EUPT-Panel reserves the right to develop and apply alternative classification rules.

### – 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>16,17</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 set 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 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

<sup>16</sup> Formerly named "Sum of squared z scores (SZ<sup>2</sup>)"

<sup>17</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.

to obtain 5 or more z scores. For the calculation of the AAZ, z scores higher than 5 will also be set as 5. The z-scores appointed to false negatives will be also included in the calculation of the combined z-scores. Laboratories within Category B will be typically ranked according to the total number of pesticides 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 EUPT-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 EUPT-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 Categories.

### 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 like. The online version of the Final EUPT 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. In exceptional cases, follow-up activities may even be indicated for results within  $|z| \leq 2.0$  (e.g. where two errors with opposed tendency cancel each other leading to acceptable results).

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 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 AAZ<sup>2</sup> higher than 3 (AAZ higher than 1.3 for SRM-compounds) 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>18</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.

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<sup>18</sup> Article 101 of Regulation (EC) 625/2017

## **ANNEX A. Protocols and Target lists of pesticides to be sought.**

- 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.

## **EUPT-FV23 SPECIFIC PROTOCOL**

### **European Union Proficiency Test for Pesticide Residues in Fruits and Vegetables (2021)**

#### **Introduction**

This protocol is complementary to the General Protocol of EU Proficiency Tests (EUP) for Pesticide Residues in Food and Feed (9<sup>th</sup> 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 (EUPs) for pesticide residues organised by the European Union.

These proficiency tests are carried out in order to improve the quality, accuracy and comparability of the residue data and to evaluate the laboratory capacity to report results that covers the entire range of maximum residue limits (0.005 - 15 mg/kg) in all groups of fruit and vegetable matrices (high water, acid and fat content). Bearing that 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 **aubergine**. The aubergines were grown in a greenhouse in Almeria. 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 was stored frozen (-20°C) prior to shipment to participants.

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

Blank material will not be distributed to the participants.

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

Participants will receive:

- Approximately 200 g of aubergine test item treated with pesticides.

#### **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 1<sup>st</sup> February 2021. 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).

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.

#### **Target List**

Participants will be provided with two target pesticide lists, one with pesticides that have to be analysed on a compulsory basis, and a second one with pesticides to be analysed voluntarily. 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.

## ANNEX A. Protocols and Target lists of pesticides to be sought.

### Laboratory assessment

For the assessment of the overall laboratory performance, the Average of the Squared z-Score ( $AZ^2$ ) will be used as in the last Proficiency Test, but only for those laboratories in Category A, which will be those laboratories that are able to analyse at least 90% of the pesticides in the target list, that are able to detect at least 90% of the pesticides evaluated 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 Lists from this web site. Laboratories should carefully read the Target Pesticide Lists, where the Minimum Required Reporting Limits (MRRLs) are given. The MRRLs do not always correspond with the EU MRLs set for aubergine.

2. The participation fee 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 the invoice number identifying them will not be considered as paid.**

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

4. **Scope Form** will be placed in the restricted area and will be open to participants from the 18<sup>th</sup> January – 29<sup>th</sup> January 2021, prior to test item shipment. The aim is that laboratories provide information regarding their scope of analysis before receipt of the test item. As default, all compounds of the target lists are selected and the MRRL is listed in the scope. Laboratories will be asked to deselect the compounds they will not include in their PT scope and insert their Reporting Limits for each pesticide. If a laboratory does not select their scope, the default values will be considered for its evaluation.

5. When the scope is completed, laboratories will receive an e-mail with their user name and password for the restricted area of submission of results.

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. If no test item has been received by 5<sup>th</sup> February 2021, 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 – 2<sup>nd</sup> March 2021- using the tabs **Detected**, **Edit results** and **Edit Methods** 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 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.

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/12682/2019. 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.

When all fields are filled out, laboratories must accept and submit their final results by clicking the check box and then click on Final submission, before 2<sup>nd</sup> March 2021.

**IMPORTANT: After the final submission it will NOT be possible to edit the results.**

Participants will receive an email confirming the submission of their results, and with an attached excel file with their submitted data.

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

8. One final tab, **Additional Info**, will be accessible after the deadline for submission of results has passed. In this Form it will be possible to submit the method information of false negative results. The deadline for this form will

## ANNEX A. Protocols and Target lists of pesticides to be sought.

be 11<sup>th</sup> March 2021. 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. When necessary, the Organiser will ask the participants by e-mail specific details about the methods of analysis used. 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 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	7 <sup>th</sup> December 2020 - 8 <sup>th</sup> January 2021
Specific Protocol published on the Web site	18 <sup>th</sup> January 2021 at the latest
Selection of the scope	18 <sup>th</sup> - 29 <sup>th</sup> January 2021
Sample distribution	1 <sup>st</sup> February 2021
Deadline for receiving sample acceptance	5 <sup>th</sup> February 2021
Deadline for receiving results	2 <sup>nd</sup> March 2021
Deadline Filling in additional information, if necessary (false negatives)	11 <sup>th</sup> March 2021
Preliminary Report: (containing preliminary assigned values and z scores)	March 2021
Final Report distributed to the Laboratories	August 2021

### 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 you receive the invoice.**

**Remember to include your invoice number 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

### 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.):

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### Quality Control Group

Dr. Antonio Valverde, University of Almería, Spain

Dr. Paula Medina, European Food Safety Authority, Italy.

### Advisory Group

Dr. Michelangelo Anastassiades, CVUA, Stuttgart, Germany.

Dr. Magnus Jezussek, Bavarian Health and Food Safety Authority, Erlangen, Germany.

Dr. André de Kok, Wageningen Food Safety Research, Wageningen, The Netherlands.

Mr. Ralf Lippold, CVUA, Freiburg, Germany.

Dr. Sonja Masselter, AGES GmbH Institute for Food Safety, Innsbruck, Austria.

Dr. Hans Mol, Wageningen Food Safety Research, Wageningen, The Netherlands.

Mr. Finbarr O'Regan, Pesticide Control Laboratory, Celbridge, Ireland.

Ms. Patrizia Pelosi, Istituto Superiore di Sanità, Roma, Italy.

Dr. Tuija Pihlström, National Food Agency, Uppsala, Sweden.

Dr. Mette Erecius Poulsen, DTU, Copenhagen, Denmark.

**TARGET PESTICIDE LIST FOR EUPT-FV-23****Compulsory Compounds (will be considered in Category A/B classification)**

Pesticide	MRRL (mg/Kg)
Acephate	0.01
Acetamiprid	0.01
<b>Aclonifen</b>	<b>0.01</b>
Acrinathrin	0.01
Aldicarb	0.01
Aldicarb Sulfone	0.01
Aldicarb Sulfoxide	0.01
Aldrin	0.005
Ametoctradin	0.01
Azinphos-methyl	0.005
Azoxystrobin	0.01
Bifenthrin (sum of isomers)	0.01
Biphenyl	0.01
Bitertanol (sum of isomers)	0.01
Boscalid	0.01
Bromopropylate	0.01
Bromuconazole (sum of diastereoisomers)	0.01
Bupirimate	0.01
Buprofezin	0.01
Cadusafos	0.005
Carbaryl	0.005
Carbendazim	0.01
Carbofuran	0.005
Carbofuran-3-hydroxy	0.005
Chlorantraniliprole	0.01
Chlorfenapyr	0.01
Chlorfenvinphos	0.01
Chlorobenzilate	0.01
Chlorothalonil	0.01
Chlorpropham	0.01
Chlorpyrifos	0.005
Chlorpyrifos-methyl	0.01
Clofentezine	0.01
Clothianidin	0.01
<b>Cyantraniliprole</b>	<b>0.01</b>
Cyazofamid	0.01
Cyflufenamid: sum of cyflufenamid (Z-isomer) and its E-isomer	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	0.005
Diazinon	0.005
Dichlofluanid	0.01
Dichlorvos	0.005
Dicloran	0.01
Dicofol (sum of p, p' and o,p' isomers)	0.01
Dieldrin	0.005
Diethofencarb	0.01
Difenoconazole	0.01
Diflubenzuron	0.01
Dimethoate	0.003
Dimethomorph (sum of isomers)	0.01

ANNEX A. Protocols and Target lists of pesticides to be sought.

Pesticide	MRRL (mg/Kg)
Dimethylaminosulfotoluidide (DMST)	0.01
Diniconazole (sum of isomers)	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	0.005
Etofenprox	0.01
Etoxazole	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	0.005
Fenhexamid	0.01
Fenitrothion	0.01
Fenoxycarb	0.01
Fenpropathrin	0.01
Fenpropidin	0.01
Fenpropimorph (sum of isomers)	0.01
Fenpyrazamine	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	0.004
Fipronil sulfone	0.004
Flonicamid	0.01
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
Fluxapyroxad	0.01
Formetanate (expressed as formetanate (hydrochloride))	0.01
Fosthiazate	0.01
Hexaconazole	0.01
Hexythiazox	0.01
Imazalil	0.005
Imidacloprid	0.01
Indoxacarb (sum of indoxacarb and its R enantiomer)	0.01
Iprodione	0.01
Iprovalicarb	0.01



**ANNEX A. Protocols and Target lists of pesticides to be sought.**

Pesticide	MRRL (mg/Kg)
Isocarbophos	0.01
Isofenphos-methyl	0.01
Isoprothiolane	0.01
Kresoxim-methyl	0.01
Lambda-Cyhalothrin	0.01
Linuron	0.01
Lufenuron (any proportion of constituent isomers)	0.01
Malaoxon	0.01
Malathion	0.01
Mandipropamid	0.01
Mepanipyrin	0.01
Metaflumizone (sum of E- and Z- isomers)	0.01
Metalaxyl and metalaxyl-M	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
Metrafenone	0.01
Monocrotophos	0.005
Myclobutanyl	0.01
Omethoate	0.003
Orthophenylphenol (Free compound only)	0.01
Oxadixyl	0.01
Oxamyl	0.01
Oxydemeton-methyl	0.005
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 (only parent compound)	0.01
Propargite	0.01
Propiconazole (sum of isomers)	0.01
Propyzamide	0.01
Proquinazid	0.01
Prosulfocarb	0.01
Prothioconazole (Prothioconazole-desthio) (sum of isomers)	0.01
Prothiofos	0.01
Pymetrozine	0.01
Pyraclostrobin	0.01
Pyridaben	0.01

ANNEX A. Protocols and Target lists of pesticides to be sought.

Pesticide	MRRL (mg/Kg)
<b>Pyridalyl</b>	<b>0.01</b>
Pyrimethanil	0.01
Pyriproxyfen	0.01
Quinoxifen	0.01
<b>Spinetoram (XDE-175)</b>	<b>0.01</b>
Spinosad (sum of spinosyn A and spinosyn D, expr. as spinosad)	0.01
Spirodiclofen	0.01
Spiromesifen	0.01
Spirotetramat	0.01
Spirotetramat metabolite BYI08330 enol-glucoside	0.01
Spirotetramat metabolite BYI08330-enol	0.01
Spirotetramat metabolite BYI08330-ketohydroxy	0.01
Spirotetramat metabolite BYI08330-monohydroxy	0.01
Spiroxamine (sum of isomers)	0.01
<b>Sulfoxaflor (sum of isomers)</b>	<b>0.01</b>
Tau-Fluvalinate	0.01
Tebuconazole	0.01
Tebufozide	0.01
Tebufozpyrad	0.01
Teflubenzuron	0.01
Tefluthrin	0.01
Terbutylazine	0.01
Tetraconazole	0.01
Tetradifon	0.01
Thiabendazole	0.01
Thiacloprid	0.01
Thiamethoxam	0.01
Thiodicarb	0.01
Thiophanate-methyl	0.01
Tolclofos-methyl	0.01
Tolyfluanid	0.01
Triadimefon	0.01
Triadimenol (any proportion of constituent isomers)	0.01
Triazophos	0.005
Trichlorfon	0.01
Tricyclazole	0.01
Trifloxystrobin	0.01
<b>Triflumizole</b>	<b>0.01</b>
<b>Triflumizole metabolite (FM-6-1)</b>	<b>0.01</b>
Triflumuron	0.01
Trifluralin	0.01
Triticonazole	0.01
Vinclozolin (only parent compound)	0.01
Zoxamide	0.01

In red: new pesticides this year

This list is based on Commission Implementing Regulation (EU) 2020/585 of 27 April 2020.  
The MRRLs are based on Regulation (EC) No. 396/2005 and Commission Directive 2006/125/EC.

**VOLUNTARY PESTICIDE LIST FOR EUPT-FV-23**Voluntary Compounds (will **NOT** be considered in Category A/B classification)

Pesticide	MRRL (mg/Kg)
Benalaxyl and benalaxyl-M	0.01
Benzovindiflupyr	0.01
Chlorfluazuron	0.01
Clomazone	0.01
<b>Cyhalofop-butyl</b>	<b>0.01</b>
Dinotefuran	0.01
Fenobucarb	0.01
Fenpicoxamid	0.01
<b>Florpyrauxifen-benzyl</b>	<b>0.01</b>
Fluensulfone	0.01
Flufenacet (only parent compound)	0.01
<b>Flutianil</b>	<b>0.01</b>
Heptachlor	0.01
Heptachlor epoxide	0.01
<b>Isofetamid</b>	<b>0.01</b>
Isopyrazam	0.01
Isoxaflutole	0.01
Isoxaflutole diketonitrile degradate	0.01
<b>Mefentrifluconazole</b>	<b>0.01</b>
Metconazole (sum of isomers)	0.01
Molinate	0.01
Novaluron	0.01
<b>Oxadiargyl</b>	<b>0.01</b>
Oxathiapiprolin	0.01
<b>Oxyfluorfen</b>	<b>0.01</b>
Penflufen	0.01
Pentachloro-aniline	0.01
Penthiopyrad	0.01
Picolinafen	0.01
Propaquizafop	0.01
Pyrethrins	0.01
<b>Pyridate (only parent compound)</b>	<b>0.01</b>
Pyriofenone	0.01
Quinalphos	0.01
Quinoclamine	0.01
Quintozene	0.01
Rotenone	0.01
Tetramethrin	0.01
Tolfenpyrad	0.01
Tri-allate	0.01
Tritosulfuron	0.01

In red: new pesticides this year

This list is based on the working document SANCO/12745/2013 of 23 – 24 November 2020 rev. 12(2)

**ANNEX B. List of laboratories that agreed to participate in EUPT-FV-23.**

ON BEHALF OF	LABORATORY NAME	CITY	COUNTRY	REPORTED RESULTS
Austria	Department for Pesticide and Food Analytics (PLMA)	Innsbruck	Austria	Yes
Belgium	LOVAP NV - Belgium, Geel	Geel	Belgium	Yes
Belgium	Sciensano - Pesticide Lab	Brussels	Belgium	Yes
Belgium	Phytocontrol (Nimes) - Pesticide Lab	NIMES	France	Yes
Belgium	Groen Agro Control - Netherlands	Delfgauw	The Netherlands	Yes
Belgium	AGROLAB LUFA GmbH	Kiel	Germany	Yes
Belgium, Bulgaria, France, Luxembourg	PRIMORIS (Phytolab) - Belgium, Gent	Gent - Zwijnaarde	Belgium	Yes
Belgium, The Netherlands	Eurofins Lab Zeeuws-Vlaanderen B.V. - Pesticiden	Graauw	The Netherlands	Yes
Bulgaria	Euro Lab- Interpreted Eurologistic Ltd	Svilengrad	Bulgaria	Yes
Bulgaria	Central Laboratory for Chemical Testing and Control	Sofia	Bulgaria	Yes
Bulgaria	Primoris - Bulgaria, Plovdiv	Plovdiv	Bulgaria	Yes
China	Beijing Uni-Star Inspection - Pesticide Lab	Beijing	China	Yes
China	Agro-product Safety Research Center - Guofang Pang	Beijing	China	Yes
China	Shanghai Municipal Center For Disease Control and Prevention	Shanghai	China	Yes
China	Agro-Environmental Quality Supervision & Testing Center, Ministry of Agriculture and Rural Affairs, Tianjin	Tianjin	China	Yes
China	Lanzhou Institute, Food & Drug Control - China	Lanzhou	China	Yes
Costa Rica	Servicio Fitosanitario del Estado - San Jose	San Jose	Costa Rica	Yes
Croatia	Teaching Institute of Public Health of Primorsko-goranska County	Rijeka	Croatia	Yes
Croatia	Center for Food Control - PBF, Zagreb	Zagreb	Croatia	Yes
Croatia	Eurofins Croatiakontrola - Croatia, Zagreb	Zagreb	Croatia	Yes

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Croatia	INSPECTO d.o.o. Laboratorij (Osijek)	Osijek	Croatia	Yes
Croatia	Croatian National Institute of Public Health-HZJZ	Zagreb	Croatia	Yes
Croatia	Bioinstitut d.o.o., Cakovec	Cakovec	Croatia	Yes
Croatia	Sample Control d.o.o.	Lučko	Croatia	Yes
Croatia	Dr. Andrija Štampar - Pesticide Lab	Zagreb	Croatia	Yes
Cyprus	Pesticide Residues Laboratory of the State General Laboratory	Nicosia	Cyprus	Yes
Czech Republic	Czech Agriculture and Food Inspection Authority	Praha	Czech Republic	Yes
Czech Republic	Central Institute for Supervising and Testing in Agriculture	Brno	Czech Republic	Yes
Czech Republic	University of Chemistry and Technology Prague, Dept. of Food Analysis.	Praha 6	Czech Republic	Yes
Denmark	Laboratoriet Ringsted - Pesticide Lab	ringsted	Denmark	Yes
Denmark	DTU National Food Institute	Lyngby	Denmark	Yes
Estonia	Agricultural Research Center - Estonia, Saku	Tallinn	Estonia	Yes
Estonia	Tartu laboratory of Health Board	Tartu	Estonia	Yes
Finland	Finnish Food Authority	Helsinki	Finland	Yes
Finland	Finnish Customs Laboratory	Espoo	Finland	Yes
Finland	Metropolilab	Helsinki	Finland	Yes
France	CAMP Méditerranée (Perpignan)	Perpignan	France	Yes
France	INOVALYS - Le Mans	LE MANS	France	Yes
France	Capinov	Landerneau Cedex	France	Yes
France	Intertek Food Services - Bremen	Bremen	Germany	Yes
France	GIRPA	BEAUCOUZE	France	Yes
France	CERECO (GARONS)	GARONS	France	Yes
France	SCL Montpellier	MONTPELLIER	France	Yes
France	SCL Paris	MASSY Cedex	France	Yes
Germany	Landeslabor Berlin-Brandenburg	Frankfurt (Oder)	Germany	Yes

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Germany	CVUA Stuttgart - Pesticide Lab	Fellbach	Germany	Yes
Germany	Chemisches Labor Dr. Mang	Frankfurt am Main	Germany	Yes
Germany	LUA Bremen	Bremen	Germany	Yes
Germany	Landesamt für Verbraucherschutz FB 3 Lebensmittelsicherheit	Halle/Saale	Germany	Yes
Germany	BVL Unit 504 NRL for Pesticide Residues	Berlin	Germany	Yes
Germany	Thüringer Landesamt für Verbraucherschutz	Bad Langensalza	Germany	Yes
Germany	LUA Saarbrücken	Saarbrücken	Germany	Yes
Germany	Chemisches und Veterinäruntersuchungsamt Rhein-Ruhr-Wupper	Krefeld	Germany	Yes
Germany	Landesuntersuchungsanstalt Sachsen	Dresden	Germany	Yes
Germany	Zentrales Institut des Sanitätsdienstes der Bundeswehr Kiel	Kronshagen	Germany	Yes
Germany	LUFA Nord-West Standort Hameln	Hameln	Germany	Yes
Germany	Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit	Erlangen	Germany	Yes
Germany	Landeslabor Schleswig-Holstein	Neumünster	Germany	Yes
Germany	CVUA-MEL Muenster	Muenster	Germany	Yes
Germany	Niedersächsisches Landesamt für Verbraucherschutz und Lebensmittelsicherheit, LVI Oldenburg	Oldenburg	Germany	Yes
Germany	Landesuntersuchungsamt Institut für Lebensmittelchemie Speyer	Speyer	Germany	Yes
Germany	Institut für Hygiene und Umwelt	Hamburg	Germany	Yes
Germany	Bundeswehr - Pesticide Lab (Garching-Hochbrück)	Garching-Hochbrück	Germany	Yes
Germany	Eurofins - Dr. Specht Express GmbH	Hamburg	Germany	Yes
Germany	Landesamt für Landwirtschaft, Lebensmittelsicherheit und Fischerei MV	Rostock	Germany	Yes

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ON BEHALF OF	LABORATORY NAME	CITY	COUNTRY	REPORTED RESULTS
Germany	Kwalis Qualitätsforschung Fulda GmbH	Dipperz	Germany	Yes
Germany	LUFA Nord-West Rückstandsanalytik	Oldenburg	Germany	Yes
Germany	Analytica Alimentaria GmbH Germany	Kleinmachnow	Germany	Yes
Germany	Hessisches Landeslabor Kassel	Kassel	Germany	Yes
Germany	Landwirtschaftliches Technologiezentrum Augustenberg (LTZ)	Karlsruhe	Germany	Yes
Germany	Labor Friedle - Germany, Tegernheim	Tegernheim	Germany	Yes
Germany	GALAB Laboratories GmbH - Hamburg	Hamburg	Germany	Yes
Greece	GENERAL CHEMICAL STATE LABORATORY	Athens	Greece	Yes
Greece	Pesticide Residue Laboratory, NRL/Benaki Phytopathological Institute	KIFISSIA	Greece	Yes
Greece	LABORATORY OF PESTICIDE RESIDUES ANALYSIS/REGIONAL CENTRE OF PLANT PROTECTION, QUALITY AND PHYSOSANITARY CONTROL	THESSALONIKI	Greece	Yes
Hungary	Food Chain Safety Centre Non-profit Ltd., Pesticide Residue Analytical Laboratory, Szolnok	Szolnok	Hungary	Yes
Hungary	National Food Chain Safety Office Food Chain Safety Laboratory Directorate Pesticide Analytical National Reference Laboratory, Velence	Velence	Hungary	Yes
Hungary	Food Chain Safety Centre Non-profit Ltd. Pesticide Residue Analytical Laboratory, Hódmezővásárhely	Hódmezővásárhely	Hungary	Yes
Hungary	FCSCN Ltd. Pesticide Residue Analytical Laboratory, Miskolc	Miskolc	Hungary	Yes
Iceland	Matis - Iceland, Reykjavík	Reykjavík	Iceland	Yes
Ireland	Dept. of Agriculture, Food & Marine Food Chemistry Division	Celbridge	Ireland	Yes
Italy	DAP Bari Polo Alimenti	Bari	Italy	Yes

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ON BEHALF OF	LABORATORY NAME	CITY	COUNTRY	REPORTED RESULTS
Italy	APPA Bolzano	Bolzano	Italy	Yes
Italy	LSP Usl Toscana Centro	FIRENZE	Italy	Yes
Italy	APPA-SL Trento - Pesticide Lab	Trento	Italy	Yes
Italy	ARPA Lazio (sez. Latina) - Pesticide Lab	Latina	Italy	Yes
Italy	ATS Milano - Laboratorio di Prevenzione	Milano	Italy	Yes
Italy	ARPA-ER - Pesticide Lab	Ferrara	Italy	No
Italy	ARPA VDA - Pesticide Lab	Saint-Christophe	Italy	Yes
Italy	ARPAL Sez. La Spezia - Sett. Chimico Levante	La Spezia	Italy	Yes
Italy	IZSUM - Italy, Perugia	Perugia	Italy	Yes
Italy	Istituto Zooprofilattico Sperimentale del Piemonte e Liguria e Valle D'Aosta	Cuneo	Italy	Yes
Italy	LABORATORIO DI PREVENZIONE ATS BERGAMO	Bergamo	Italy	Yes
Italy	ARPA FVG - Pesticide Lab (Udine)	Udine	Italy	No
Italy	Istituto Superiore di Sanità - Roma	ROMA	Italy	Yes
Italy	ISTITUTO ZOOPROFILATTICO SPERIMENTALE LOMBARDIA EMILIA ROMAGNA (IZSLER) - LABORATORIO PESTICIDI	Brescia	Italy	Yes
Italy	IZS Mezzogiorno	PORTICI	Italy	Yes
Italy	ARPAM - Pesticide Lab	MACERATA	Italy	Yes
Italy	IZS PB - Pesticide Lab	FOGGIA	Italy	Yes
Italy	IZSAM - Pesticide Lab	Teramo	Italy	Yes
Italy	IZS LT (sezione Firenze) - Pesticide Lab	Firenze	Italy	Yes
Italy	IZSLT - Roma	Roma	Italy	Yes
Italy	ARPAC - Pesticide Lab	Naples	Italy	No
Italy	IZS Sicilia - Pesticide Lab	Palermo	Italy	Yes
Italy	IZS Sardegna - Pesticide Lab	Sassari	Italy	Yes
Italy	ARPA Veneto (Laboratorio di Verona)	Verona	Italy	Yes
Kenya	KEPHIS - Kenya, Nairobi	Nairobi	Kenya	Yes
Kenya	SGS (Mombasa) - Pesticide Lab	Mombasa	Kenya	No



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Latvia	BIOR (Riga) - Pesticide Lab	Riga	Latvia	Yes
Lithuania	NMVRVI - Pesticide Lab (Vilnius)	VILNIUS	Lithuania	Yes
Luxembourg	LNS Food lab	Dudelange	Luxembourg	Yes
Malta	Eurofins - Germany, Hamburg	Hamburg	Germany	Yes
Norway	NIBIO, Pesticides and Natural Products Chemistry	Aas	Norway	Yes
Peru	Inspectorate Services Perú SAC	Chorrillos Lima	Peru	Yes
Poland	Laboratory of Food & Feed Safety in Bialystok	Bialystok	Poland	Yes
Poland	Wojewódzka Stacja Sanitarno-Epidemiologiczna w Łodzi, Dział Laboratoryjny, Oddział Laboratoryjny Badania Żywności i Produktów Kosmetycznych	Łódź (Lodz)	Poland	Yes
Poland	Jars S.A.	Łojski	Poland	Yes
Poland	Wojewódzka Stacja Sanitarno-Epidemiologiczna w Opolu	Opole	Poland	Yes
Poland	Central Laboratory Main Inspectorate of Plant Health and Seed Inspection	Torun	Poland	Yes
Poland	Wojewodzka Stacja Sanitarno-Epidemiologiczna we Wrocławiu - Dział Laboratoryjny	Wroclaw	Poland	Yes
Poland	IPP-NRI - Pesticide Lab (Poznan)	60-318	Poland	Yes
Poland	IPP-NRI - Pesticide Lab (Sosnicowice)	Sosnicowice	Poland	Yes
Poland	Food Safety Laboratory Research Institute of Horticulture	Skierniewice	Poland	Yes
Poland	VSES Warszawa - Pesticide Lab	Warsaw	Poland	Yes
Poland	Hamilton UO-Technologia	Grójec	Poland	Yes
Poland	Agrolab Polska Sp. z o.o.	Dęblin	Poland	No
Poland	SGS Sp. z o.o. Laboratorium Środowiskowe	Pszczyna	Poland	Yes
Portugal	Pesticide Lab (Funchal - Madeira Island)	Funchal	Portugal	Yes
Portugal	INIAV-Pesticide Laboratory (vegetable and animal products)	Vairao- Vila do Conde	Portugal	Yes

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ON BEHALF OF	LABORATORY NAME	CITY	COUNTRY	REPORTED RESULTS
Portugal	Labiagro – Laboratório Químico	Oeiras	Portugal	Yes
Portugal, Spain	Labs & Technological Services AGQ - Burguillos	Burguillos	Spain	Yes
Romania	National Phytosanitary Authority-Laboratory for Pesticides Residues Control in Plants and Vegetable Products	Bucuresti, Sect.1	Romania	Yes
Romania	LRCRPPV Mures	Targu Mures	Romania	Yes
Romania	DSVSA OLT - Lab	Slatina	Romania	No
Romania	Sanitary Veterinary and Food Safety Directorate	Bucharest	Romania	Yes
Serbia	SP Laboratorija - Pesticide Lab	BECEJ	Serbia	Yes
Singapore	National Centre for Food Science	Singapore	Singapore	Yes
Slovakia	State Veterinary and Food Institute (Bratislava)	Bratislava	Slovakia	Yes
Slovakia	Pesticide Lab of PHA SR - Bratislava	Bratislava	Slovakia	Yes
Slovenia	Pesticide Lab - Maribor	Maribor	Slovenia	Yes
Spain	Laboratori Agroalimentari	CABRILS	Spain	Yes
Spain	SES-Gerencia del Area de Salud de Badajoz	BADAJOS	Spain	No
Spain	LABORATORIO KUDAM, S.L.	PILAR DE LA HORADADA (ALICANTE)	Spain	Yes
Spain	LABORATORIO AGRARIO REGIONAL de CASTILLA Y LEÓN	Burgos	Spain	Yes
Spain	SALUD PUBLICA (Almería) - Pesticide Lab	ALMERIA	Spain	Yes
Spain	Laboratorio Químico Microbiológico, S.L	Murcia	Spain	Yes
Spain	Laboratorio Analítico Bioclínico - Spain, Almería	Almeria	Spain	Yes
Spain	LSP Ayto Madrid. MadridSalud	Madrid	Spain	Yes
Spain	Laboratorio Agroalimentario y de Sanidad Animal	EL PALMAR (MURCIA)	Spain	Yes
Spain	National Centre for Food (Majadahonda)	Majadahonda-Madrid	Spain	Yes
Spain	Lab Arbitral Agroalimentario	MADRID	Spain	Yes

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ON BEHALF OF	LABORATORY NAME	CITY	COUNTRY	REPORTED RESULTS
Spain	Laboratorio Agroalimentario - Spain, Valencia	Burjassot (Valencia)	Spain	Yes
Spain	Laboratorio Agroalimentario de Granada	ATARFE (GRANADA)	Spain	Yes
Spain	Laboratorio de Producción y Sanidad Vegetal de Almería	La Mojonera (Almería)	Spain	Yes
Spain	Laboratorio Agroambiental de Zaragoza (Gobierno de Aragón)	Zaragoza	Spain	Yes
Spain	National Center for Technology and Food Safety	SAN ADRIAN (NAVARRA)	Spain	Yes
Spain	Agricultural and Phytopathological Lab. of Galicia	Abegondo. A Coruña	Spain	No
Spain	AINIA	Paterna	Spain	Yes
Spain	Laboratorio SOIVRE Almería	ALMERIA	Spain	No
Spain	Laboratorio Regional de la CCAA de La Rioja	Logroño	Spain	Yes
Spain	Laboratorio de Salud Pública de Galicia, Lugo	LUGO	Spain	Yes
Spain	Laboratorio Agroalimentario de Extremadura	CÁCERES	Spain	Yes
Spain	LABORATORIO DE SALUD PUBLICA DE CUENCA	CUENCA	Spain	Yes
Spain	Nasertic - Spain, Villava	Villava	Spain	Yes
Spain	Analytica Alimentaria GmbH	Almería	Spain	Yes
Spain	Laboratori Agència Salut Pública Barcelona	Barcelona	Spain	Yes
Spain	EUROFINS ECOSUR, S.A.	LORQUI - MURCIA	Spain	Yes
Spain	Laboratorio de Producción y Sanidad Vegetal (Jaén)	Mengibar (Jaen)	Spain	Yes
Spain	Laboratorio de Residuos, Inst. Tecnol. de Canarias	AGÜIMES, GRAN CANARIA	Spain	Yes
Spain	Labcolor-Coexphal - Spain, Almería	LA MOJONERA - ALMERIA	Spain	Yes
Spain	EUROFINS SICAAGRIQ	Vicar	Spain	Yes
Sweden	Eurofins Food and Feed Testing Sweden AB	Lidköping	Sweden	Yes
Sweden	National Food Agency	Uppsala	Sweden	Yes
Switzerland	Amt fuer Verbraucherschutz Aargau (Cantonal Office of Consumer Protection Aargau)	Aarau	Switzerland	Yes
Switzerland	SCAV Genève	Genève	Switzerland	Yes

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Switzerland	Kantonaies Laboratorium Bern	Bern	Switzerland	Yes
Switzerland	Kantonaies Labor Zürich	Zurich	Switzerland	Yes
The Netherlands	Wageningen Food Safety Research	Wageningen	The Netherlands	Yes
United Kingdom	Fera Science Ltd	York	United Kingdom	Yes
United Kingdom	Concept Life Sciences	Bar Hill	United Kingdom	Yes
United Kingdom	SASA	Edinburgh	United Kingdom	Yes
United Kingdom	Eurofins Food Testing UK Limited i54 Business Park, Valiant Way Wolverhampton WV9 5GB	Wolverhampton	United Kingdom	Yes