EURL-PROFICIENCY TEST-FV-SC05, 2021-2022

Pesticide Residues in dried white beans

Final Report

Organiser:

Dr. Amadeo R. Fernández-Alba
Co-Head of EURL-FV
University of Almería, Edificio Químicas CITE I
Ctra. Sacramento s/n
04120 Almería, SPAIN
Phone: +34 950015034
E-mail: amadeo@ual.es
http://www.eurl-pesticides.eu

Organising team at the University of Almería:

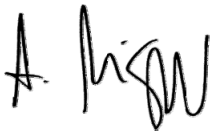
Carmen Ferrer, Chemist.	University of Almería
Octavio Malato, Chemist.	University of Almería
Mª del Mar Gómez, Agronomist.	University of Almería
Víctor Cutillas, Chemist.	University of Almería
Francisco José Diaz, Chemist.	University of Almería
María Murcia, Chemist.	University of Almería
Lorena Manzano, Chemist.	University of Almería
Patricia Blanco, Chemist.	University of Almería

Scientific Committee:

Antonio Valverde, Senior Chemist (QCG).	University of Almería, Spain.
Paula Medina, Senior Chemist (QCG).	European Food Safety Authority, Italy.
Michelangelo Anastassiades, Senior Chemist (AG).	EURL-SRM, CVUA Stuttgart, Fellbach, Germany.
Björn Hardebusch, Senior Chemist (AG).	EURL-AO, CVUA Freiburg, Freiburg, Germany.
Magnus Jezussek, Senior Chemist (AG).	LGL, Erlangen, Germany.
André de Kok, Senior Chemist (AG).	Formerly Wageningen Food Safety Research, Wageningen, The Netherlands.
Ralf Lippold, Senior Chemist (AG).	CVUA Freiburg, Germany.
Hans Mol, Senior Chemist (AG).	Wageningen Food Safety Research, Wageningen, The Netherlands.
Finbarr O'Regan, Senior Chemist (AG).	Pesticide Registration Division, DAFM, Kildare, Ireland.
Patrizia Pelosi, Senior Chemist (AG).	Istituto Superiore di Sanità, Rome, Italy.
Tuija Pihlström, Senior Chemist (AG).	Swedish Food Agency, SFA, Uppsala, Sweden.
Mette Erecius Poulsen, Senior Chemist (AG).	EURL-CF, DTU National Food Institute, Lyngby, Denmark.
Radim Štěpán, Senior Chemist (AG).	Czech Agriculture and Food Inspection Authority, Prague, Czech Republic.
Hermann Unterluggauer, Senior Chemist (AG).	AGES GmbH, Institute for Food Safety Innsbruck, Austria.

QCG: Quality Control Group
AG: Advisory Group

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



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EURL-EUROPEAN UNION PROFICIENCY TEST SC05
FOR THE DETERMINATION OF PESTICIDES IN SPECIAL COMMODITIES USING MULTIRESIDUE METHODS
2021-2022

According to Article 28 of Regulation 396/2005/EC (23rd February 2005) of the European Parliament and of the Council, concerning maximum residue levels for pesticides in or on food and feed of plant and animal origin[1], all laboratories analysing samples for the official control of pesticide residues shall participate in the European Union Proficiency Tests (EUPTs) for pesticide residues organised by the European Union. These proficiency tests are carried out on an annual basis in order to continuously improve the quality, accuracy and comparability of the residue data reported by EU Member States to the European Union, as well as by other Member States, within the framework of the EU multi-annual coordinated control programme and national monitoring programmes.

Regulation (EU) 2017/625[2] lays down the general tasks, duties and requirements for European Union Reference Laboratories (EURLs)[3] for Food, Feed and Animal Health. Among these tasks is the provision for independently organised comparative tests. European Proficiency Test FV-SC04 has been organised by the EURL in Fruits and Vegetables at the University of Almería, Spain[4].

Participation in European Proficiency Test FV-SC05 was on a voluntary basis. The invitation was sent to 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 non-EU/non-EFTA countries were invited to take part.

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

[1] Regulation (EC) No 396/2005, published in the OJ of the EU L70 on 16.03.2005, last amended by Regulation 839/2008 published in the OJ of the EU L234 on 30.08.2008.
[2] Regulation (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.
[3] The Community Reference Laboratory (CRL) changed its name to the European Union Reference Laboratory (EURL) on 1st December 2009 as a result of the Treaty of Lisbon. OJ of the EU C306 on 17.12.2007.
[4] Commission Regulation (EC) No 776/2006 of 23rd May 2006 - amending Annex VII to Regulation (EC) No 882/2004 of the European Parliament and of the Council as regards European Union Reference Laboratories.

1. INTRODUCTION

Sixty laboratories agreed to participate in EUPT-FV-SC05.

The proficiency test was performed at the end of 2021 and beginning of 2022 using dried white beans. Blank beans were purchased in the local market in Almería (Spain) and they were spiked with analytical standards. Participating laboratories were not provided with a 'blank' of dried beans sample.

The test item, 125 g of white dried beans flour containing spiked pesticide residues, was shipped to participants on 29th November 2021. The deadline for results submission to the Organiser was 10th January 2022. The participants were asked to determine the residue levels of all the pesticides that they detected in the white beans and to report the concentrations in mg/kg. The participants were provided with a target pesticide list, which contained 215 target pesticides. The pesticide target list is detailed in Annex A. The lists of target pesticides also contained the MRRL for each pesticide fixed at 0.01 mg/kg, except for the following pesticides which have lower MRRLs based on Regulation (EU) No. 396/2005 and EU Directive 2006/125/EC, 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).

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 (σ) 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-SC05.

2. TEST ITEM

2.1 Preparation of the treated test item

Dried white beans were purchased in the local market in Almería. They were analysed in the EURL-FV laboratory in Almería for all the pesticides included in the target list. None of the compounds were detected at concentrations above the MRRL, so they had to be spiked with a mixture of analytical standards.

1 kg portions of white beans were weighed into disposable aluminium trays and covered with 1 L of a standard solution mixture in acetonitrile under the fume hood. Once the solvent had evaporated completely and the beans were completely dry, all the beans were mixed in a large container and ground into a fine flour in the blender. Again, all the flour was well homogenised by hand, and 125 g portions of the test item were weighed out into polyethylene plastic bottles 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. 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. The quantification by GC-MS/MS and LC-MS/MS was performed using matrix matched calibration curves prepared with blank white dried beans.

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**. All the pesticides evaluated in the dried beans test item passed the homogeneity test. The acceptance criteria for the test item to be sufficiently homogenous for the proficiency test were that: $Ss^2 < c$, where Ss is the between-bottle sampling standard deviation and $c = F_1\sigma^2_{all} + F_2S^2_{an}$; F_1 and F_2 being constant values of 1.88 and 1.01, respectively, from the ten samples taken, and σ^2_{all} = (0.3 x FFP-RSD(25 %) x mean concentration)^2. This was used to demonstrate that the between-bottle variance was not higher than the within-bottle variance.

2.3 Stability tests

The stability tests were also carried out by the EURL-FV laboratory at the University of Almería. 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 $|x_1 - y_i| \leq 0.3\times\sigma$, where x_1 is the mean value of the Day 1 stability test, yi the mean value of the Day 2 stability test and σ the standard deviation used for proficiency assessment (typically 25 % of the assigned value).

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

Moreover, regarding the stability of the sample during shipment, a duplicate analysis of three bottles reproducing the delivery conditions that the samples experienced for 48 hours was performed (Day 3). All the pesticides passed this second stability test.

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 29th November 2021.

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. This ensured that confidentiality was maintained throughout the duration of Proficiency Test SC05. The Target Pesticide List was the same as for EUPT-FV23, and it was 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. 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 EUPTs. The same

target RSD has been applied to all the pesticides, independent of concentration. For informative purposes the robust relative standard deviation (CVs*) is calculated according to ISO 13528:2015 Chapter 7.7 (Consensus value from participant results) following Algorithm A in Annex C, and it can be compared to the FFP-RSD in **Table 7**.

3.4 z scores

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

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

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.
- σ_{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'.

$\lvert AZ^2 \rvert \leq 2.0$	Good
$2.0 < \lvert AZ^2 \rvert < 3.0$	Satisfactory
$\lvert AZ^2 \rvert \geq 3.0$	Unsatisfactory

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 2**.

Sixty laboratories agreed to participate in this proficiency test. The results reported by all the laboratories are presented in this report. However, only results reported by laboratories from EU/EFTA-countries have been included in the statistical treatment. Four laboratories from non-EU/EFTA countries (UK, Uruguay and Peru) participated in EUPT-SC05. Their results have not been included for the calculation of the assigned value.

Seventeen pesticides from the pesticide target list were used to spike the sample and were present in the test item at concentrations above the MRRL. Dichlorvos showed long-term stability and analytical problems in the test item, and for this reason, the Scientific Committee decided not to use it for the evaluation of the results. Dichlorvos results will be shown for information purposes only.

A summary of the reported results for both the pesticides evaluated and those informative can be seen below in **Table 1**.

Table 1. Summary of Reported Results for pesticides evaluated

Pesticides	No. of Reported Results	No. of False Negative Results	No. of Not Analysed Results	Percentage of Reported Results[a] (out of 56)
Acephate	50	2	4	89
Aclonifen	38	0	18	68
Azoxystrobin	53	2	1	95
Boscalid	54	0	2	96
Carbaryl	53	0	3	95
Carbendazim	52	0	4	93
Chlorpyrifos	55	1	0	98
Cyprodinil	53	2	1	95
Dichlorvos ⓘ	39	10	7	70
Dimethoate	54	1	1	96
Fenitrothion	53	0	3	95
Fludioxonil	52	2	2	93
Imidacloprid	53	0	3	95
Pendimethalin	54	1	1	96
Pyrimethanil	55	0	1	98
Tebuconazole	54	1	1	96
Trichlorfon	44	3	9	79

[a] The percentage of Reported Results comes from 56 laboratories. It does not take into account results reported by laboratories in the UK, Uruguay and Peru.
ⓘ For information purposes only.

4.1.1 False positives

Two laboratories reported one result each for additional pesticides that were not present in the test item. These pesticides and the residue levels reported are presented in **Table 2**, 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.

Table 2. Laboratories that reported as quantitative results for pesticides that were not present in the treated test item

Lab Code	Pesticide	Reporting level (mg/kg)	Concentration (mg/kg)	Determination technique
Lab026	Flonicamid	0.01	0.091	LC-MS/MS (QQQ)
Lab002	Phosmet	0.01	0.04	-

4.1.2 False negatives

Table 3 summarises the results from laboratories (including non-EU/EFTA laboratories) that reported false negatives, presented as 'Not Detected' (ND). Those pesticides for which their assigned value is below three times the MRRL are not included in this table, as in that case, false negatives cannot be assigned. Dichlorvos is not considered in this table, but the total number of false negative results for that compound is shown in **Table 1**.

Table 3. Laboratories that failed to report pesticides that were present in the treated test item.

Laboratory	Acephate	Aclonifen	Azoxystrobin	Carbaryl	Chlorpyrifos	Cyprodinil	Dimethoate	Fludioxonil	Pendimethalin	Tebuconazole	Trichlorfon
Lab002	—	—	—	ND	—	—	ND	ND	—	ND	—
Lab013						ND					
Lab018			ND			ND			ND	ND	
Lab022								ND			ND
Lab025	ND		ND				ND	ND			ND
Lab027					ND						
Lab032											ND
Lab050											ND
Lab055	ND										
Lab057		ND									ND

ND: Not detected

4.2 Assigned values and target standard deviations

The assigned values are based on the robust mean values calculated using all the results reported by laboratories from EU and EFTA countries. The assigned values for the 17 pesticides and their uncertainties are presented in **Table 4**.

The target standard deviation was calculated using a fixed FFP-RSD value of 25 %. For comparison, a robust standard deviation (CV*) was also calculated for informative purposes, employing also this value for the calculation of the uncertainty. These RSDs can be seen in **Table 4**.

Table 4. Robust mean values, uncertainty and % RSDs for all pesticides evaluated.

Pesticides	n	Robust mean (mg/kg)	CV(%)	MRRL (mg/kg)	Uncertainty (mg/kg)
Acephate	50	0.072	22.6	0.01	0.003
Aclonifen	38	0.039	22.8	0.01	0.002
Azoxystrobin	53	0.036	18.9	0.01	0.001
Boscalid	54	0.058	19.2	0.01	0.002
Carbaryl	53	0.049	17.9	0.005	0.001
Carbendazim	52	0.064	29.4	0.01	0.003
Chlorpyrifos	55	0.076	15.2	0.005	0.002
Cyprodinil	53	0.051	13.8	0.01	0.001
Dichlorvosⓘ	39	0.027	43.2	0.005	0.002
Dimethoate	54	0.033	15.5	0.003	0.001
Fenitrothion	53	0.060	22.3	0.01	0.002
Fludioxonil	52	0.078	17.2	0.01	0.002
Imidacloprid	53	0.081	17.7	0.01	0.002
Pendimethalin	54	0.034	14.3	0.01	0.001
Pyrimethanil	55	0.035	12.8	0.01	0.001
Tebuconazole	54	0.044	11.9	0.01	0.001
Trichlorfon	44	0.068	17.4	0.01	0.002

ⓘFor information purposes only.

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 2** the individual z scores are presented for each laboratory, together with the concentrations reported for each pesticide. The z scores of the non-EU/EFTA laboratories have

been included in **Appendix 2** but have not been considered in **Table 5.**, where the classification of z scores reported by EU/EFTA laboratories is shown.

Table 5.
Classification of z scores for the pesticides evaluated (only EU/EFTA participants)

	Robust Mean (mg/kg)	Acceptable %	Questionable %	Unacceptable %
Acephate	0.072	88	6	6
Aclonifen	0.039	97	0	3
Azoxystrobin	0.036	95	0	5
Boscalid	0.058	96	2	2
Carbaryl	0.049	96	4	0
Carbendazim	0.064	88	6	6
Chlorpyrifos	0.076	93	0	7
Cyprodinil	0.051	95	0	5
Dichlorvosⓘ	0.027	61	8	31
Dimethoate	0.033	96	2	2
Fenitrothion	0.060	92	6	2
Fludioxonil	0.078	96	2	2
Imidacloprid	0.081	98	0	2
Pendimethalin	0.034	94	0	6
Pyrimethanil	0.035	96	4	0
Tebuconazole	0.044	96	2	2
Trichlorfon	0.068	95	5	0

ⓘFor information purposes only.

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 3**, 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 4** shows the values of individual z scores for each evaluated pesticide and the combined 'Average of the Squared z scores' (AZ^2) for all EU/EFTA laboratories in Category A (including non-EU/EFTA countries), which were those laboratories that were able to analyse at least 90 % of the pesticides in the target pesticides list (193), to detect and quantify at least 90 % of the pesticides present in the Test Item (14), 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 5**.

Forty of the 56 EU and EFTA laboratories were classified into Category A (71 %).

From the AZ^2, 92.5 % were classed as 'good', 5 % as 'satisfactory' and 2.5 % as 'unsatisfactory' (Only considering EU and EFTA laboratories).

Of the 16 EU and EFTA laboratories in Category B, one had reported a false positive result, and it would have been classified into Category A if not for that false positive result.

Table 6 shows all the laboratories in Category A (including non-EU laboratories), the number of evaluated pesticides reported, the percentage of pesticides analysed from the target list, the AZ^2 values and their subclassifications. Laboratories that reported false negative results in Category A are marked with the symbol Θ.

Table 6. Performance and Classification of laboratories in Category A using the AZ^2 formula

Lab Code	No. of pesticides detected (max.16)	% of pesticides analysed from target list	AZ^2	Classification
Lab001	16	99.5	0.2	Good
Lab003	16	99.5	0.2	Good
Lab004	16	97.2	1.6	Good
Lab005	16	100	0.4	Good
Lab007	16	100	0.3	Good
Lab008	16	99.1	1.5	Good
Lab009	16	100	0.8	Good
Lab014	16	99.5	0.1	Good
Lab015	16	93.5	1.2	Good
Lab016	16	100	0.5	Good
Lab017	16	100	0.3	Good
Lab019	16	98.6	0.2	Good
Lab020	16	98.1	0.2	Good
Lab021	16	100	2.4	Satisfactory
Lab022 Θ	14	100	6.4	Unsatisfactory
Lab023	16	99.5	0.5	Good
Lab024	16	91.2	0.2	Good
Lab028	16	100	0.3	Good

Lab Code	No. of pesticides detected (max.16)	% of pesticides analysed from target list	AZ^2	Classification
Lab029	16	100	0.6	Good
Lab030	16	100	0.5	Good
Lab031	16	100	0.5	Good
Lab032 Θ	15	97.7	1.6	Good
Lab033	16	100	0.3	Good
Lab034	15	97.7	0.4	Good
Lab035	16	100	0.6	Good
Lab036	16	98.6	0.3	Good
Lab038	15	95.3	0.4	Good
Lab039	16	99.1	0.5	Good
Lab041	16	96.7	1.0	Good
Lab042	16	99.5	0.5	Good
Lab043	16	97.2	0.3	Good
Lab044	16	99.1	0.3	Good
Lab045	16	100	0.8	Good
Lab046	16	99.1	0.6	Good
Lab049	16	100	1.0	Good
Lab050 Θ	15	98.6	1.7	Good
Lab051	16	100	0.2	Good
Lab052	16	100	0.6	Good
Lab053	16	98.1	1.6	Good
Lab054	16	97.7	1.1	Good
Lab058	16	99.1	2.1	Satisfactory
Lab060	15	94.9	0.5	Good

Θ Laboratories reporting a false negative result.

Table 7 shows all the laboratories in Category B, the number of results reported, the percentage of pesticides analysed from the target list and the number of acceptable z scores. Laboratories reporting a false negative are marked with the symbol Θ and laboratories reporting a false positive are marked with a '+'.

The AZ^2 graphical representation for EU/EFTA laboratories classified into Category A can be seen in Appendix 5.

Table 7. 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)
Lab002 ⊖+	9	56	48	13	8
Lab006	14	88	80	14	14
Lab010	12	75	60	12	12
Lab011	11	69	62	11	11
Lab012	13	81	50	13	13
Lab013 ⊖	15	94	89	16	15
Lab018 ⊖	11	69	81	15	11
Lab025 ⊖	5	31	34	10	4
Lab026 +	15	94	98	15	15
Lab027 ⊖	12	75	81	13	12
Lab037	15	94	89	15	13
Lab040	3	19	16	3	2
Lab047	11	69	54	11	11
Lab048	13	81	67	13	12
Lab055 ⊖	13	81	72	14	1
Lab056	14	88	67	14	14
Lab057 ⊖	13	81	87	15	13
Lab059	15	94	77	15	14

⊖ Laboratories reporting a false negative result.
+ Laboratories reporting a false positive result.

5. CONCLUSIONS

Sixty laboratories agreed to participate in EUPT-FV-SC05. Four of them did not belong to EU nor EFTA countries, so their results were not considered for the estimation of the assigned value.

Seventeen pesticides were present in EUPT-FV-SC05 test item at concentrations above the MRRL.

Of a total number of 896 possible determinations from EU/EFTA laboratories (56 laboratories by 16 evaluated pesticides), 92.3 % were reported, 6.0 % were not analysed and 1.7 % were not detected (false negative results).

71 % of the EU and EFTA laboratories that submitted results were classified into Category A. Of them, 92.5 % were classed as 'good', 5 % as 'satisfactory' and 2.5 % as 'unsatisfactory'.

The robust standard deviation (CV*) was below 25 % for most of the pesticides, except for carbendazim (CV*= 29.4 %) and dichlorvos (CV*= 43.2%). The average value of CV* of the 16 pesticides evaluated was 18.1 %.

Participation in this EUPT-FV-SC05 involved laboratories from 21 EU Member States and 1 EFTA country (Norwey). 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. Four non-European laboratories (from UK, Uruguay and Peru) participated in EUPT-FV-SC05.

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APPENDIX 1. Homogeneity data.

Acephate (mg/kg)		Aclonifen (mg/kg)		Azoxystrobin (mg/kg)		Boscalid (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.076	0.084	0.046	0.046	0.039	0.039	0.064	0.063
0.085	0.087	0.048	0.044	0.040	0.038	0.067	0.062
0.084	0.084	0.045	0.044	0.038	0.037	0.060	0.060
0.078	0.085	0.038	0.044	0.035	0.038	0.057	0.062
0.073	0.084	0.039	0.039	0.034	0.038	0.055	0.062
0.082	0.062	0.044	0.033	0.042	0.029	0.065	0.046
0.073	0.075	0.034	0.039	0.031	0.034	0.050	0.057
0.080	0.083	0.041	0.045	0.037	0.037	0.060	0.060
0.081	0.084	0.045	0.045	0.040	0.038	0.065	0.059
0.081	0.087	0.042	0.044	0.038	0.040	0.062	0.061

Carbaryl (mg/kg)		Carbendazim (mg/kg)		Chlorpyrifos (mg/kg)		Cyprodinil (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.053	0.055	0.071	0.061	0.080	0.089	0.056	0.060
0.056	0.058	0.074	0.074	0.085	0.081	0.061	0.060
0.056	0.061	0.074	0.072	0.078	0.083	0.055	0.056
0.056	0.059	0.070	0.073	0.079	0.079	0.051	0.060
0.051	0.057	0.065	0.063	0.067	0.080	0.051	0.057
0.057	0.042	0.074	0.050	0.079	0.081	0.060	0.045
0.050	0.052	0.065	0.060	0.075	0.066	0.046	0.055
0.054	0.058	0.072	0.063	0.074	0.092	0.056	0.056
0.056	0.059	0.072	0.070	0.081	0.070	0.057	0.057
0.057	0.057	0.073	0.074	0.073	0.083	0.057	0.058

Dimethoate (mg/kg)		Fenitrothion (mg/kg)		Fludioxonil (mg/kg)		Imidacloprid (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.038	0.037	0.072	0.074	0.085	0.088	0.092	0.094
0.038	0.038	0.076	0.073	0.093	0.085	0.095	0.091
0.037	0.038	0.068	0.070	0.082	0.085	0.089	0.095
0.037	0.038	0.064	0.072	0.083	0.090	0.093	0.093
0.036	0.041	0.062	0.072	0.079	0.092	0.079	0.094
0.037	0.027	0.076	0.054	0.090	0.083	0.092	0.091
0.034	0.035	0.058	0.062	0.066	0.079	0.081	0.080
0.037	0.038	0.067	0.072	0.086	0.084	0.090	0.089
0.040	0.039	0.073	0.072	0.091	0.082	0.093	0.091
0.037	0.043	0.071	0.071	0.088	0.086	0.091	0.093

Pendimethalin (mg/kg)		Pyrimethanil (mg/kg)		Tebuconazole (mg/kg)		Trichlorfon (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.040	0.043	0.037	0.038	0.048	0.050	0.078	0.079
0.043	0.043	0.039	0.039	0.051	0.049	0.077	0.080
0.037	0.038	0.036	0.038	0.046	0.048	0.076	0.082
0.039	0.041	0.034	0.040	0.043	0.048	0.072	0.080
0.037	0.042	0.033	0.038	0.043	0.050	0.069	0.076
0.043	0.032	0.038	0.028	0.051	0.036	0.077	0.060
0.034	0.039	0.028	0.035	0.038	0.044	0.069	0.071
0.039	0.041	0.037	0.037	0.047	0.047	0.078	0.076
0.041	0.045	0.037	0.037	0.051	0.049	0.078	0.079
0.040	0.041	0.039	0.036	0.048	0.049	0.077	0.077

APPENDIX 2. Results (mg/kg) and z scores for FFP-RSD (25 %).

Results reported by the laboratories for the evaluated pesticides (mg/kg) and their calculated z score value using FFP-RSD 25 %

Lab Code	Acephate	z score (FFP-RSD 25 %)	Aclonifen	z score (FFP-RSD 25 %)	Azoxystrobin	z score (FFP-RSD 25 %)	Boscalid	z score (FFP-RSD 25 %)	Carbaryl	z score (FFP-RSD 25 %)	Carbendazim	z score (FFP-RSD 25 %)	Chlorpyrifos	z score (FFP-RSD 25 %)
MRRL (mg/kg)	**0.01**		**0.01**		**0.01**		**0.01**		**0.005**		**0.01**		**0.005**	
Robust mean (mg/kg)	**0.072**		**0.229**		**1.073**		**0.035**		**0.105**		**0.05**		**0.047**	
Lab001	0.086	0.8	0.036	-0.3	0.036	0.0	0.054	-0.3	0.052	0.3	0.065	0.1	0.075	-0.1
Lab002	0.04	-1.8	NA		0.03	-0.6	0.06	0.1	ND	-3.6	0.05	-0.9	0.08	0.2
Lab003	0.078	0.3	0.037	-0.2	0.036	0.0	0.051	-0.5	0.051	0.2	0.056	-0.5	0.074	-0.1
Lab004	0.0725	0.0	0.087	4.9	0.038	0.3	0.057	-0.1	0.044	-0.4	0.05	-0.9	0.075	-0.1
Lab005	0.064	-0.4	0.04	0.1	0.047	1.3	0.063	0.3	0.046	-0.2	0.063	-0.1	0.094	0.9
Lab006	0.073	0.1	NA		0.032	-0.4	0.05	-0.6	0.063	1.2	0.071	0.4	0.068	-0.4
Lab007	0.089	0.9	0.029	-1.0	0.032	-0.4	0.05	-0.6	0.042	-0.6	0.06	-0.3	0.07	-0.3
Lab008	0.027	-2.5	0.031	-0.8	0.027	-1.0	0.048	-0.7	0.033	-1.3	0.037	-1.7	0.062	-0.7
Lab009	0.072	0.0	0.05	1.1	0.034	-0.2	0.056	-0.1	0.053	0.4	0.062	-0.1	0.076	0.0
Lab010	0.069	-0.2	NA		0.039	0.4	0.059	0.1	0.051	0.2	0.061	-0.2	0.075	-0.1
Lab011	NA		NA		0.043	0.8	0.065	0.5	0.062	1.1	NA		0.076	0.0
Lab012	NA		NA		0.026	-1.1	0.048	-0.7	0.04	-0.7	0.09	1.6	0.074	-0.1
Lab013	0.072	0.0	0.051	1.2	0.034	-0.2	0.061	0.2	0.057	0.7	0.069	0.3	0.092	0.8
Lab014	0.084	0.7	0.046	0.7	0.04	0.5	0.058	0.0	0.047	-0.1	0.067	0.2	0.083	0.4
Lab015	0.03	-2.3	0.036	-0.3	0.03	-0.6	0.043	-1.0	0.029	-1.6	0.046	-1.1	0.081	0.3
Lab016	0.064	-0.4	0.035	-0.4	0.025	-1.2	0.047	-0.8	0.047	-0.1	0.059	-0.3	0.06	-0.8
Lab017	0.09	1.0	0.034	-0.5	0.038	0.3	0.059	0.1	0.052	0.3	0.072	0.5	0.084	0.4
Lab018	0.07	-0.1	NA		ND	-3.5	0.084	1.8	0.047	-0.1	0.052	-0.8	0.086	0.5
Lab019	0.098	1.4	0.038	-0.1	0.038	0.3	0.06	0.1	0.052	0.3	0.07	0.4	0.075	-0.1
Lab020	0.086	0.8	0.047	0.8	0.037	0.2	0.055	-0.2	0.055	0.5	0.072	0.5	0.072	-0.2
Lab021	0.073	0.1	0.042	0.3	0.026	-1.1	0.06	0.1	0.059	0.8	0.084	1.2	0.139	3.3
Lab022	0.145	4.1	0.032	-0.7	0.035	-0.1	0.08	1.5	0.075	2.2	0.054	-0.6	0.172	5.0
Lab023	0.064	-0.4	0.028	-1.1	0.038	0.3	0.052	-0.4	0.048	-0.1	0.055	-0.6	0.068	-0.4
Lab024	0.062	-0.6	0.032	-0.7	0.033	-0.3	0.061	0.2	0.044	-0.4	0.052	-0.8	0.065	-0.6
Lab025	ND	-3.4	NA		ND	-3.5	NA		NA		NA		0.067	-0.5
Lab026	0.074	0.1	NA		0.033	-0.3	0.075	1.2	0.042	-0.6	0.087	1.4	0.074	-0.1
Lab027	0.056	-0.9	NA		0.022	-1.5	0.042	-1.1	NA		0.072	0.5	ND	-3.7
Lab028	0.068	-0.2	0.028	-1.1	0.034	-0.2	0.045	-0.9	0.041	-0.6	0.062	-0.1	0.064	-0.6
Lab029	0.09	1.0	0.05	1.1	0.04	0.5	0.06	0.1	0.05	0.1	0.07	0.4	0.08	0.2
Lab030	0.077	0.3	0.049	1.0	0.041	0.6	0.057	-0.1	0.05	0.1	0.082	1.1	0.073	-0.2
Lab031	0.058	-0.8	0.043	0.4	0.039	0.4	0.066	0.5	0.049	0.0	0.042	-1.4	0.085	0.5
Lab032	0.046	-1.4	0.032	-0.7	0.029	-0.7	0.043	-1.0	0.045	-0.3	0.1	2.2	0.091	0.8
Lab033	0.064	-0.4	0.043	0.4	0.039	0.4	0.056	-0.1	0.049	0.0	0.064	0.0	0.085	0.5
Lab034	0.0937	1.2	NA		0.0364	0.1	0.0585	0.0	0.0487	0.0	0.0804	1.0	0.0869	0.6

APPENDIX 2. Results (mg/kg) and z scores for FFP-RSD (25 %).

Lab Code	Acephate	z score (FFP-RSD 25 %)	Aclonifen	z score (FFP-RSD 25 %)	Azoxystrobin	z score (FFP-RSD 25 %)	Boscalid	z score (FFP-RSD 25 %)	Carbaryl	z score (FFP-RSD 25 %)	Carbendazim	z score (FFP-RSD 25 %)	Chlorpyrifos	z score (FFP-RSD 25 %)
MRRL (mg/kg)	**0.01**		**0.01**		**0.01**		**0.01**		**0.005**		**0.01**		**0.005**	
Robust mean (mg/kg)	**0.072**		**0.229**		**1.073**		**0.035**		**0.105**		**0.05**		**0.047**	
Lab035	0.094	1.2	0.047	0.8	0.042	0.7	0.06	0.1	0.049	0.0	0.083	1.2	0.089	0.7
Lab036	0.091	1.1	0.049	1.0	0.041	0.6	0.054	-0.3	0.049	0.0	0.065	0.1	0.087	0.6
Lab037	0.044	-1.6	NA		0.047	1.3	0.066	0.5	0.051	0.2	0.12	3.5	0.071	-0.3
Lab038	0.066	-0.3	NA		0.037	0.2	0.068	0.7	0.049	0.0	0.039	-1.6	0.058	-0.9
Lab039	0.073	0.1	0.036	-0.3	0.028	-0.9	0.049	-0.6	0.036	-1.0	0.05	-0.9	0.082	0.3
Lab040	NA		NA		NA		NA		NA		NA		0.045	-1.6
Lab041	0.050	-1.2	0.035	-0.4	0.028	-0.9	0.051	-0.5	0.039	-0.8	0.047	-1.1	0.069	-0.4
Lab042	0.089	0.9	0.03	-0.9	0.033	-0.3	0.071	0.9	0.052	0.3	0.052	-0.8	0.071	-0.3
Lab043	0.0794	0.4	0.0432	0.4	0.0307	-0.6	0.0524	-0.4	0.0469	-0.2	0.0907	1.7	0.0711	-0.3
Lab044	0.09	1.0	0.047	0.8	0.038	0.3	0.062	0.3	0.048	-0.1	0.082	1.1	0.084	0.4
Lab045	0.063	-0.5	0.032	-0.7	0.044	0.9	0.066	0.5	0.072	1.9	0.037	-1.7	0.071	-0.3
Lab046	0.07	-0.1	0.034	-0.5	0.043	0.8	0.076	1.2	0.037	-1.0	0.071	0.4	0.076	0.0
Lab047	NA		NA		0.041	0.6	0.056	-0.1	0.028	-1.7	NA		0.072	-0.2
Lab048	0.066	-0.3	NA		0.038	0.3	0.074	1.1	0.05	0.1	0.016	-3.0	0.075	-0.1
Lab049	0.075	0.2	0.051	1.2	0.033	-0.3	0.091	2.3	0.06	0.9	0.075	0.7	0.089	0.7
Lab050	0.037	-1.9	0.035	-0.4	0.038	0.3	0.048	-0.7	0.048	-0.1	0.08	1.0	0.08	0.2
Lab051	0.081	0.5	0.031	-0.8	0.035	-0.1	0.044	-1.0	0.046	-0.2	0.071	0.4	0.072	-0.2
Lab052	0.071	-0.1	0.044	0.5	0.04	0.5	0.056	-0.1	0.052	0.3	0.108	2.7	0.072	-0.2
Lab053	0.046	-1.4	0.045	0.6	0.041	0.6	0.071	0.9	0.078	2.4	0.098	2.1	0.085	0.5
Lab054	0.0543	-1.0	0.0256	-1.4	0.0334	-0.2	0.0667	0.6	0.0439	-0.4	0.0543	-0.6	0.0503	-1.4
Lab055	ND	-3.4	NA		0.0085	-3.0	0.013	-3.1	0.0257	-1.9	0.0082	-3.5	0.012	-3.4
Lab056	0.068	-0.2	NA		0.028	-0.9	0.056	-0.1	0.052	0.3	0.064	0.0	0.052	-1.3
Lab057	NA		ND	-3.0	0.035	-0.1	0.054	-0.3	0.043	-0.5	0.055	-0.6	0.077	0.1
Lab058	0.02	-2.9	0.026	-1.4	0.026	-1.1	0.036	-1.5	0.029	-1.6	0.043	-1.3	0.052	-1.3
Lab059	0.084	0.7	NA		0.047	1.3	0.07	0.8	0.055	0.5	0.053	-0.7	0.098	1.2
Lab060	0.063	-0.5	NA		0.044	0.9	0.071	0.9	0.063	1.2	0.047	-1.1	0.082	0.3

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

APPENDIX 2. Results (mg/kg) and z scores for FFP-RSD (25 %).

Lab Code	Cyprodinil	z score (FFP-RSD 25 %)	Dimethoate	z score (FFP-RSD 25 %)	Fenitrothion	z score (FFP-RSD 25 %)	Fludioxonil	z score (FFP-RSD 25 %)	Imidacloprid	z score (FFP-RSD 25 %)	Pendimethalin	z score (FFP-RSD 25 %)	Pyrimethanil	z score (FFP-RSD 25 %)
MRRL (mg/kg)	**0.01**		**0.003**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Robust mean (mg/kg)	**0.051**		**0.033**		**0.060**		**0.078**		**0.081**		**0.034**		**0.035**	
Lab001	0.054	0.2	0.03	-0.3	0.065	0.3	0.064	-0.7	0.1	0.9	0.032	-0.3	0.035	0.0
Lab002	0.02	-2.4	ND	-3.6	0.07	0.7	ND	-3.5	0.05	-1.5	0.04		0.03	-0.6
Lab003	0.059	0.6	0.028	-0.6	0.059	-0.1	0.076	-0.1	0.077	-0.2	0.031	-0.4	0.038	0.3
Lab004	0.0396	-0.9	0.0334	0.1	0.0656	0.4	0.0776	0.0	0.0783	-0.2	0.0349	0.0	0.0318	-0.4
Lab005	0.058	0.5	0.035	0.3	0.076	1.1	0.086	0.4	0.102	1.0	0.035	0.1	0.036	0.1
Lab006	0.048	-0.3	0.031	-0.2	0.036	-1.6	0.07	-0.4	0.072	-0.5	0.033	-0.2	0.033	-0.2
Lab007	0.048	-0.3	0.03	-0.3	0.068	0.5	0.075	-0.2	0.074	-0.4	0.035	0.1	0.026	-1.0
Lab008	0.049	-0.2	0.021	-1.4	0.041	-1.3	0.077	-0.1	0.042	-1.9	0.031	-0.4	0.031	-0.5
Lab009	0.063	0.9	0.038	0.6	0.101	2.8	0.079	0.0	0.08	-0.1	0.044	1.1	0.043	0.9
Lab010	0.051	0.0	0.034	0.1	NA		NA		0.077	-0.2	0.036	0.2	0.041	0.7
Lab011	0.056	0.4	0.034	0.1	0.054	-0.4	NA		0.097	0.8	0.039	0.5	0.039	0.5
Lab012	0.041	-0.8	0.03	-0.3	0.056	-0.3	0.066	-0.6	0.073	-0.4	0.03	-0.5	0.035	0.0
Lab013	ND	-3.2	0.035	0.3	0.085	1.7	0.081	0.1	0.08	-0.1	0.039	0.5	0.037	0.2
Lab014	0.058	0.5	0.035	0.3	0.065	0.3	0.077	-0.1	0.072	-0.5	0.038	0.4	0.035	0.0
Lab015	0.055	0.3	0.029	-0.5	0.053	-0.5	0.077	-0.1	0.065	-0.8	0.036	0.2	0.034	-0.1
Lab016	0.046	-0.4	0.024	-1.1	0.056	-0.3	0.064	-0.7	0.063	-0.9	0.033	-0.2	0.025	-1.1
Lab017	0.058	0.5	0.037	0.5	0.046	-0.9	0.076	-0.1	0.087	0.3	0.03	-0.5	0.043	0.9
Lab018	ND	-3.2	0.037	0.5	0.073	0.9	0.076	-0.1	0.067	-0.7	ND	-3.5	0.043	0.9
Lab019	0.056	0.4	0.035	0.3	0.07	0.7	0.088	0.5	0.087	0.3	0.034	-0.1	0.039	0.5
Lab020	0.052	0.0	0.036	0.4	0.058	-0.1	0.09	0.6	0.072	-0.5	0.042	0.9	0.037	0.2
Lab021	0.05	-0.1	0.034	0.1	0.05	-0.7	0.096	0.9	0.085	0.2	0.075	4.7	0.032	-0.3
Lab022	0.057	0.4	0.048	1.9	0.053	-0.5	ND	-3.7	0.108	1.3	0.067	3.8	0.054	2.2
Lab023	0.047	-0.3	0.03	-0.3	0.027	-2.2	0.086	0.4	0.08	-0.1	0.029	-0.6	0.033	-0.2
Lab024	0.049	-0.2	0.028	-0.6	0.055	-0.3	0.067	-0.6	0.08	-0.1	0.029	-0.6	0.037	0.2
Lab025	0.041	-0.8	ND	-3.6	0.101	2.8	ND	-3.5	NA		NA		0.045	1.1
Lab026	0.06	0.7	0.036	0.4	0.034	-1.7	0.076	-0.1	0.087	0.3	0.035	0.1	0.035	0.0
Lab027	0.043	-0.7	0.03	-0.3	0.047	-0.9	0.049	-1.5	0.072	-0.5	0.035	0.1	0.032	-0.3
Lab028	0.041	-0.8	0.027	-0.7	0.06	0.0	0.09	0.6	0.081	0.0	0.029	-0.6	0.03	-0.6
Lab029	0.06	0.7	0.04	0.9	0.05	-0.7	0.06	-0.9	0.09	0.4	0.02	-1.7	0.03	-0.6
Lab030	0.055	0.3	0.04	0.9	0.068	0.5	0.094	0.8	0.115	1.7	0.037	0.3	0.039	0.5
Lab031	0.053	0.1	0.032	-0.1	0.09	2.0	0.083	0.3	0.074	-0.4	0.039	0.5	0.039	0.5
Lab032	0.051	0.0	0.03	-0.3	0.046	-0.9	0.083	0.3	0.091	0.5	0.031	-0.4	0.033	-0.2
Lab033	0.054	0.2	0.036	0.4	0.082	1.5	0.091	0.7	0.081	0.0	0.042	0.9	0.035	0.0
Lab034	0.0549	0.3	0.0351	0.3	0.0782	1.2	0.0683	-0.5	0.0768	-0.2	0.0378	0.4	0.0398	0.6
Lab035	0.071	1.5	0.039	0.8	0.062	0.1	0.094	0.8	0.098	0.8	0.035	0.1	0.034	-0.1
Lab036	0.052	0.0	0.032	-0.1	0.057	-0.2	0.098	1.0	0.078	-0.2	0.033	-0.2	0.033	-0.2

APPENDIX 2. Results (mg/kg) and z scores for FFP-RSD (25 %).

Lab Code	Cyprodinil	z score (FFP-RSD 25 %)	Dimethoate	z score (FFP-RSD 25 %)	Fenitrothion	z score (FFP-RSD 25 %)	Fludioxonil	z score (FFP-RSD 25 %)	Imidacloprid	z score (FFP-RSD 25 %)	Pendimethalin	z score (FFP-RSD 25 %)	Pyrimethanil	z score (FFP-RSD 25 %)
MRRL (mg/kg)	**0.01**		**0.003**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Robust mean (mg/kg)	**0.051**		**0.033**		**0.060**		**0.078**		**0.081**		**0.034**		**0.035**	
Lab037	0.051	0.0	0.03	-0.3	0.065	0.3	0.12	2.1	0.057	-1.2	0.029	-0.6	0.034	-0.1
Lab038	0.06	0.7	0.033	0.0	0.055	-0.3	0.078	0.0	0.094	0.6	0.031	-0.4	0.035	0.0
Lab039	0.052	0.0	0.028	-0.6	0.072	0.8	0.055	-1.2	0.057	-1.2	0.039	0.5	0.03	-0.6
Lab040	NA		NA		NA		0.058	-1.0	NA		0.017	-2.0	NA	
Lab041	0.044	-0.6	0.017	-1.9	0.047	-0.9	0.070	-0.4	0.085	0.2	0.028	-0.8	0.032	-0.3
Lab042	0.053	0.1	0.035	0.3	0.057	-0.2	0.07	-0.4	0.079	-0.1	0.034	-0.1	0.033	-0.2
Lab043	0.0475	-0.3	0.0315	-0.2	0.0708	0.7	0.0731	-0.3	0.0765	-0.2	0.0369	0.3	0.033	-0.2
Lab044	0.054	0.2	0.034	0.1	0.066	0.4	0.095	0.9	0.086	0.2	0.035	0.1	0.036	0.1
Lab045	0.052	0.0	0.037	0.5	0.06	0.0	0.088	0.5	0.089	0.4	0.03	-0.5	0.033	-0.2
Lab046	0.05	-0.1	0.038	0.6	0.057	-0.2	0.074	-0.2	0.12	1.9	0.031	-0.4	0.035	0.0
Lab047	0.045	-0.5	0.029	-0.5	0.067	0.5	0.077	-0.1	NA		0.033	-0.2	0.033	-0.2
Lab048	0.052	0.0	0.033	0.0	NA		0.112	1.7	0.076	-0.3	0.035	0.1	0.038	0.3
Lab049	0.068	1.3	0.048	1.9	0.074	0.9	0.081	0.1	0.077	-0.2	0.041	0.8	0.036	0.1
Lab050	0.048	-0.3	0.023	-1.2	0.036	-1.6	0.1	1.1	0.123	2.0	0.035	0.1	0.038	0.3
Lab051	0.054	0.2	0.03	-0.3	0.06	0.0	0.084	0.3	0.084	0.1	0.038	0.4	0.036	0.1
Lab052	0.047	-0.3	0.026	-0.8	0.058	-0.1	0.09	0.6	0.083	0.1	0.034	-0.1	0.038	0.3
Lab053	0.059	0.6	0.051	2.2	0.043	-1.1	0.078	0.0	0.1	0.9	0.045	1.2	0.043	0.9
Lab054	0.0486	-0.2	0.0274	-0.7	0.0394	-1.4	0.0985	1.0	0.0947	0.7	0.0373	0.3	0.0456	1.2
Lab055	0.013	-3.0	0.0071	-3.1	0.013	-3.1	0.017	-3.1	0.0121	-3.4	0.0056	-3.4	0.01	-2.9
Lab056	0.048	-0.3	0.03	-0.3	0.059	-0.1	0.088	0.5	0.073	-0.4	0.032	-0.3	0.031	-0.5
Lab057	0.051	0.0	0.019	-1.7	0.043	-1.1	0.072	-0.3	0.076	-0.3	0.038	0.4	0.032	-0.3
Lab058	0.029	-1.7	0.021	-1.4	0.052	-0.5	0.06	-0.9	0.051	-1.5	0.032	-0.3	0.023	-1.4
Lab059	0.033	-1.4	0.035	0.3	0.065	0.3	0.042	-1.8	0.107	1.3	0.035	0.1	0.019	-1.8
Lab060	0.053	0.1	0.038	0.6	0.053	-0.5	0.082	0.2	0.11	1.4	0.039	0.5	0.035	0.0

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

APPENDIX 2. Results (mg/kg) and z scores for FFP-RSD (25 %).

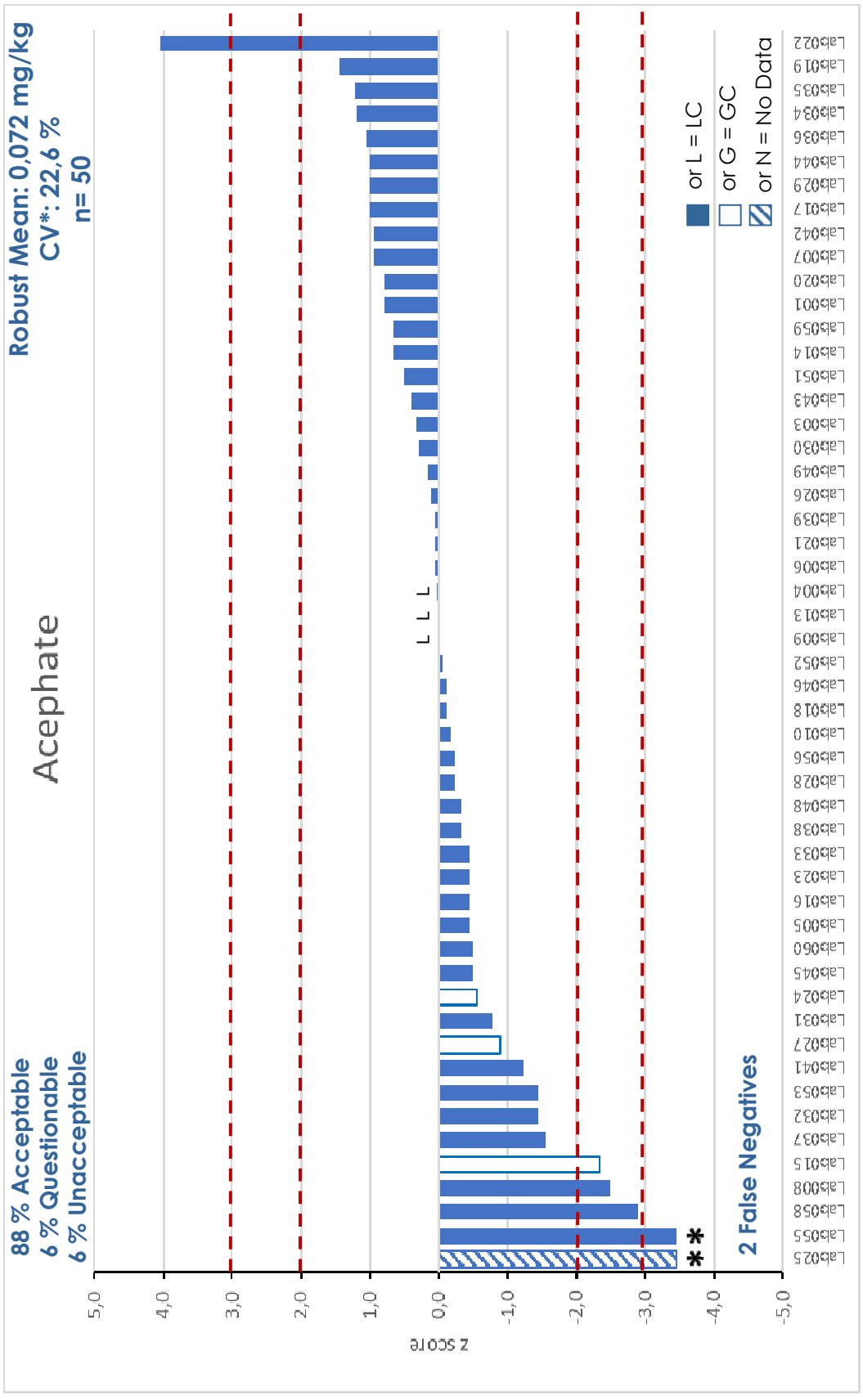
Lab Code	Tebuconazole	z score (FFP-RSD 25 %)	Trichlorfon	z score (FFP-RSD 25 %)
MRRL (mg/kg)	**0.01**		**0.01**	
Robust mean (mg/kg)	**0.044**		**0.068**	
Lab001	0.045	0.1	0.071	0.2
Lab002	ND	-3.1	NA	
Lab003	0.051	0.6	0.055	-0.8
Lab004	0.0441	0.0	0.0638	-0.3
Lab005	0.045	0.1	0.075	0.4
Lab006	0.043	-0.1	NA	
Lab007	0.042	-0.2	0.066	-0.1
Lab008	0.039	-0.5	0.04	-1.7
Lab009	0.046	0.2	0.072	0.2
Lab010	0.041	-0.3	NA	
Lab011	0.04	-0.4	NA	
Lab012	0.039	-0.5	NA	
Lab013	0.046	0.2	0.057	-0.7
Lab014	0.044	0.0	0.074	0.3
Lab015	0.047	0.2	0.021	-2.8
Lab016	0.038	-0.6	0.057	-0.7
Lab017	0.049	0.4	0.074	0.3
Lab018	ND	-3.1	0.076	0.5
Lab019	0.045	0.1	0.077	0.5
Lab020	0.042	-0.2	0.075	0.4
Lab021	0.044	0.0	0.081	0.7
Lab022	0.042	-0.2	ND	-3.7
Lab023	0.048	0.3	0.063	-0.3
Lab024	0.043	-0.1	0.08	0.7
Lab025	0.048	0.3	ND	-3.4
Lab026	0.056	1.1	0.067	-0.1
Lab027	0.03	-1.3	NA	
Lab028	0.042	-0.2	0.07	0.1
Lab029	0.05	0.5	0.08	0.7
Lab030	0.046	0.2	0.067	-0.1
Lab031	0.048	0.3	0.057	-0.7
Lab032	0.049	0.4	ND	-3.7
Lab033	0.051	0.6	0.071	0.2
Lab034	0.0397	-0.4	0.0621	-0.4
Lab035	0.051	0.6	0.076	0.5
Lab036	0.041	-0.3	0.055	-0.8

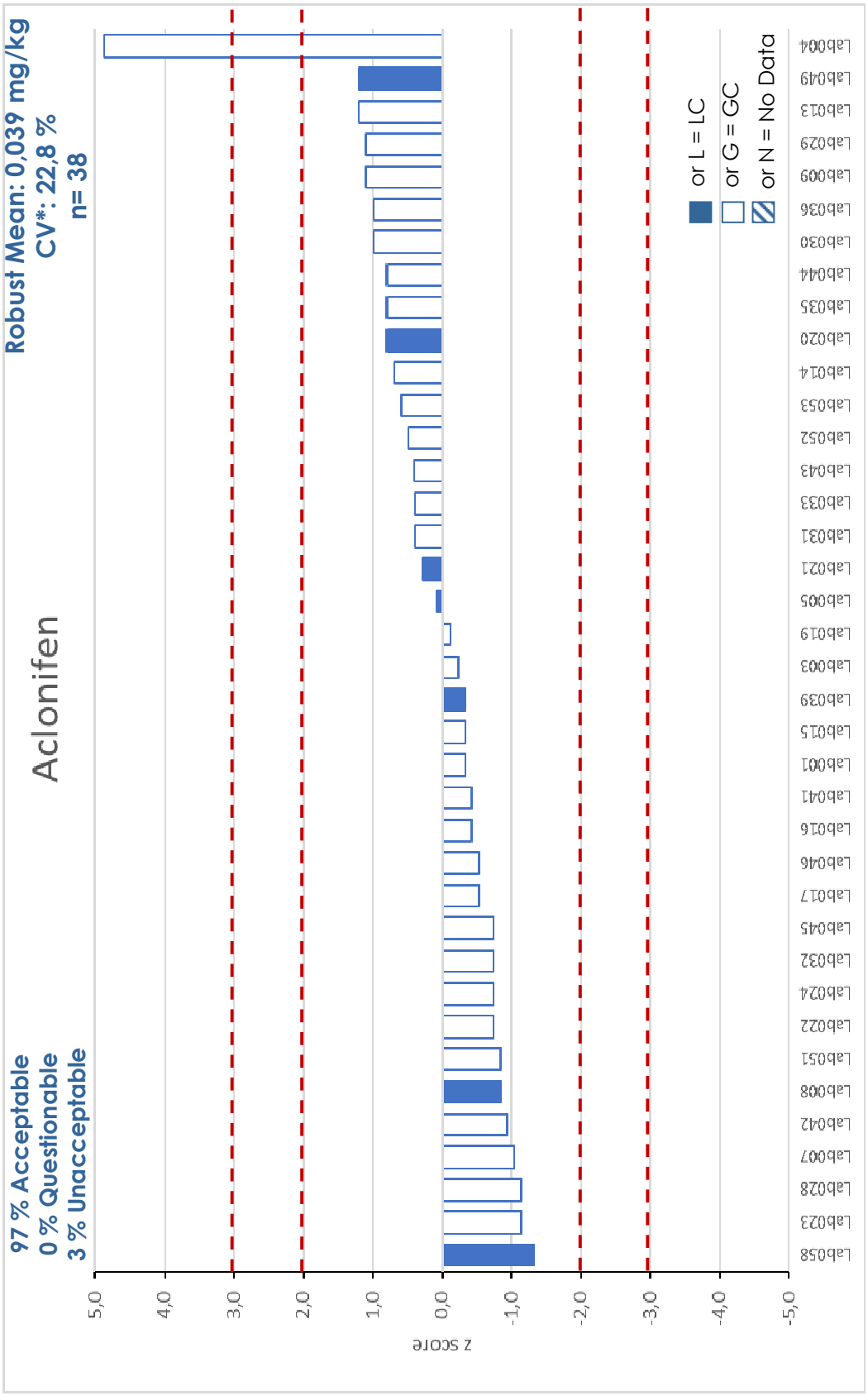
APPENDIX 2. Results (mg/kg) and z scores for FFP-RSD (25 %).

Lab Code	Tebuconazole	z score (FFP-RSD 25 %)	Trichlorfon	z score (FFP-RSD 25 %)
MRRL (mg/kg)	**0.01**		**0.01**	
Robust mean (mg/kg)	**0.044**		**0.068**	
Lab037	0.042	-0.2	0.037	-1.8
Lab038	0.046	0.2	0.08	0.7
Lab039	0.039	-0.5	0.064	-0.3
Lab040	NA		NA	
Lab041	0.030	-1.3	0.031	-2.2
Lab042	0.066	2.0	0.072	0.2
Lab043	0.0415	-0.3	0.0635	-0.3
Lab044	0.045	0.1	0.068	0.0
Lab045	0.051	0.6	0.101	1.9
Lab046	0.046	0.2	0.095	1.6
Lab047	0.041	-0.3	NA	
Lab048	0.042	-0.2	0.062	
Lab049	0.058	1.2	0.067	-0.1
Lab050	0.045	0.1	ND	-3.4
Lab051	0.044	0.0	0.075	0.4
Lab052	0.048	0.3	0.065	-0.2
Lab053	0.053	0.8	0.091	1.3
Lab054	0.0695	2.3	0.0559	-0.7
Lab055	0.0084	-3.2	NA	
Lab056	0.041	-0.3	NA	
Lab057	0.043	-0.1	ND	-3.4
Lab058	0.028	-1.5	0.045	-1.4
Lab059	0.02	-2.2	0.073	0.3
Lab060	0.045	0.1	0.083	0.9

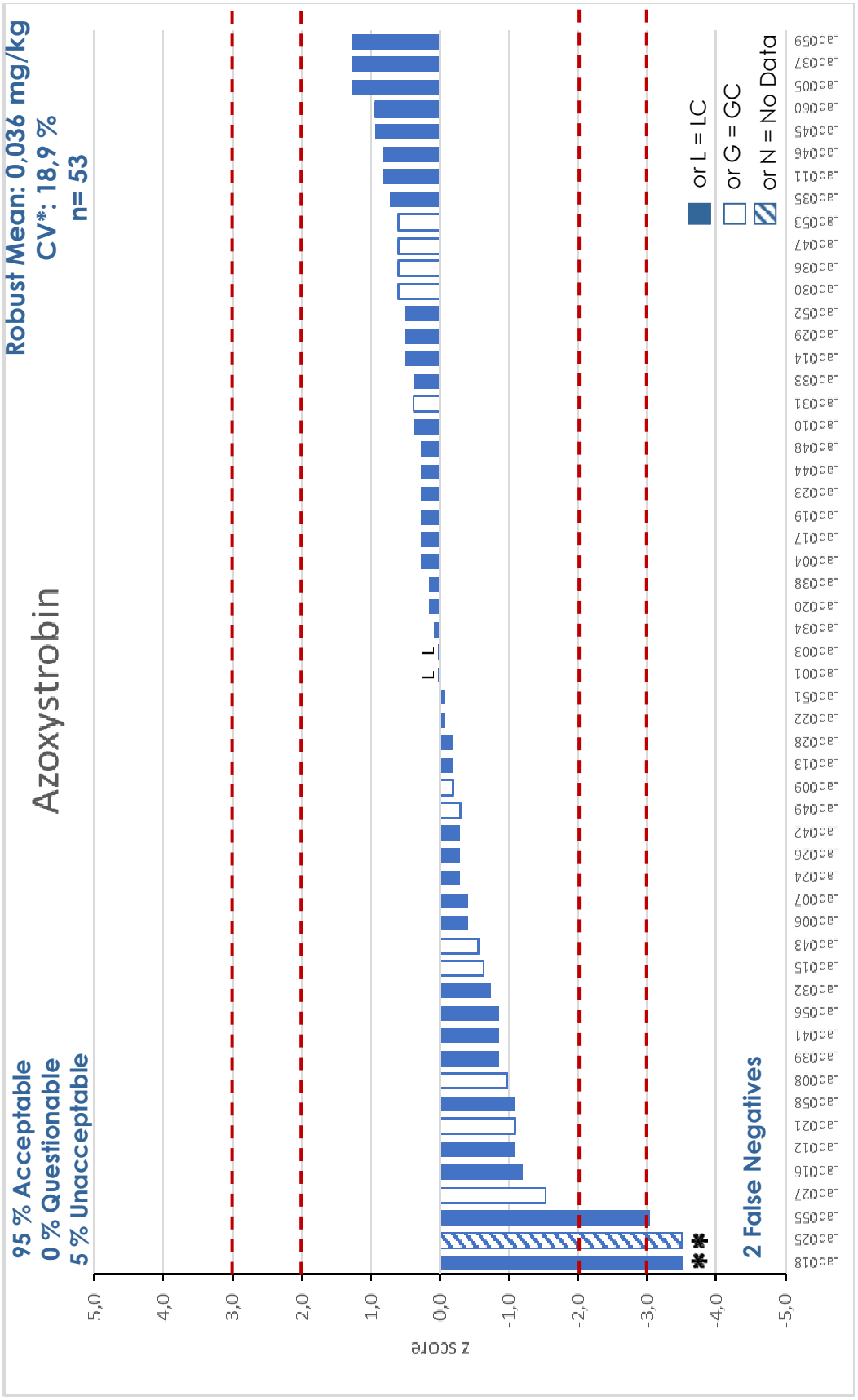
NA: Not analysed ND: Not detected (False negative)

APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).

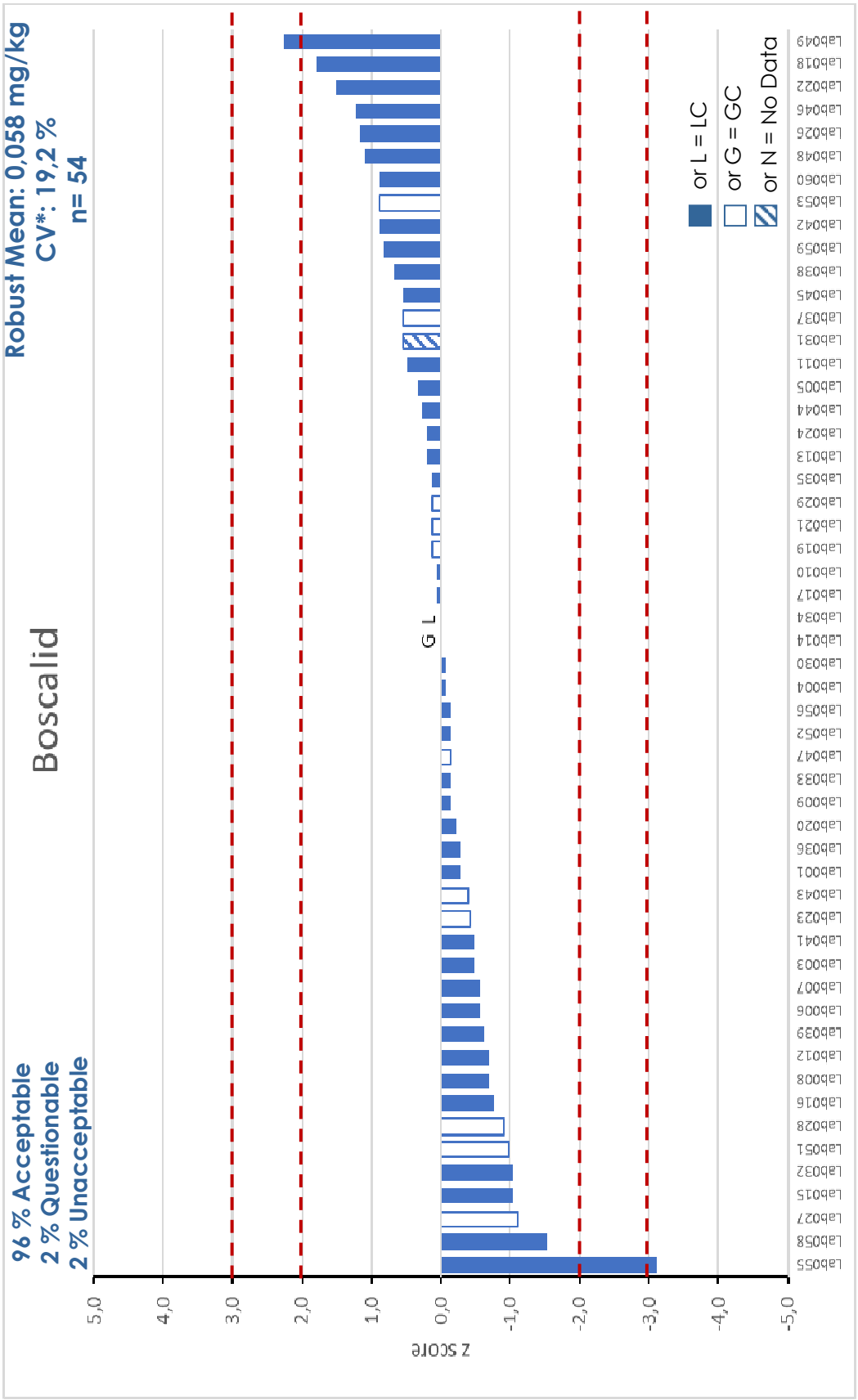



Robust Mean: 0,039 mg/kg
CV*: 22,8 %
n= 38
Aclonifen
97 % Acceptable
0 % Questionable
3 % Unacceptable
or L = LC
or G = GC
or N = No Data
z score
5,0
4,0
3,0
2,0
1,0
0,0
-1,0
-2,0
-3,0
-4,0
-5,0
lab004
lab049
lab013
lab029
lab009
lab036
lab030
lab044
lab035
lab020
lab014
lab053
lab052
lab043
lab033
lab031
lab021
lab005
lab019
lab003
lab039
lab015
lab001
lab041
lab016
lab046
lab017
lab045
lab032
lab024
lab022
lab051
lab008
lab042
lab007
lab028
lab023
lab058

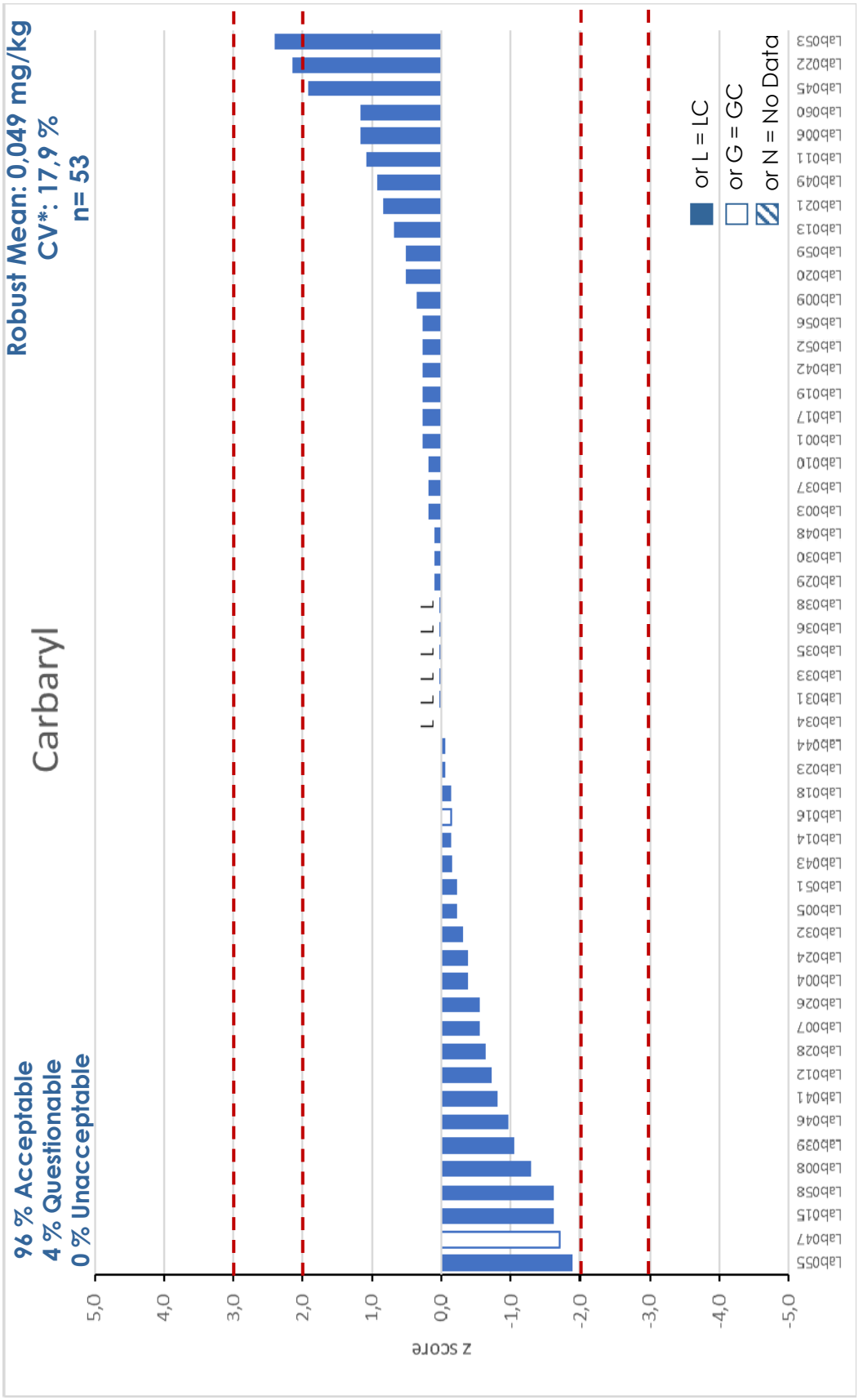
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



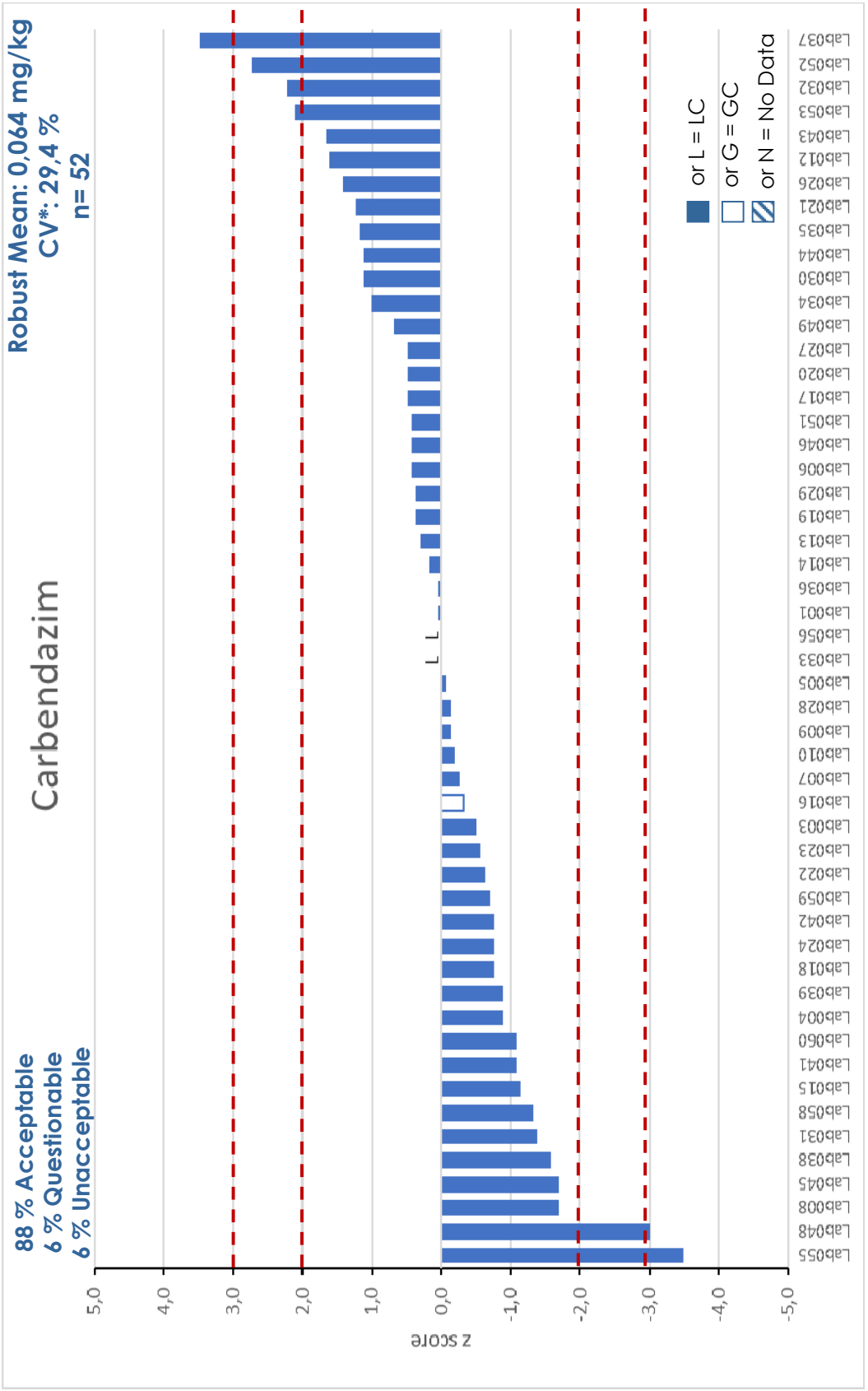
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



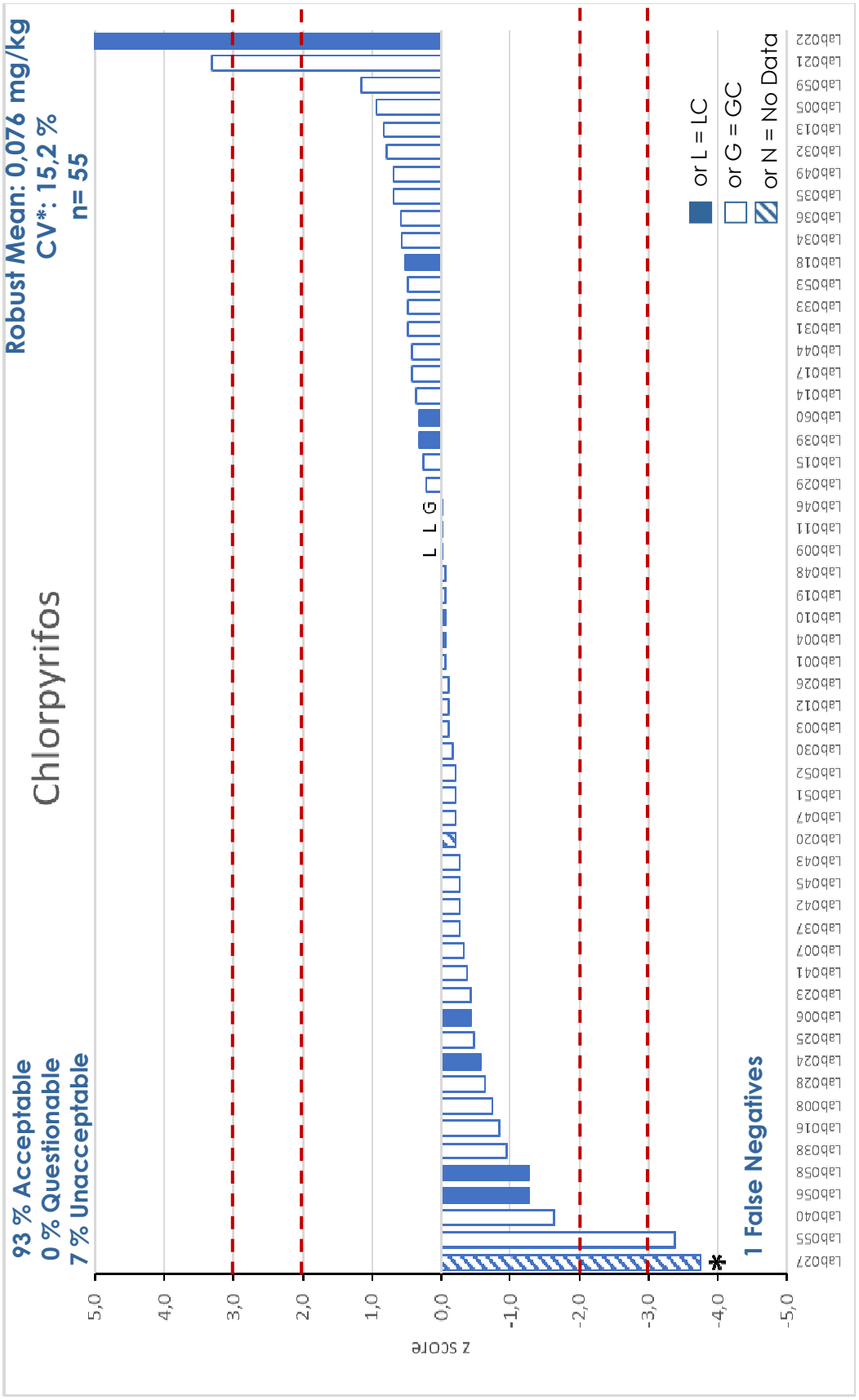
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



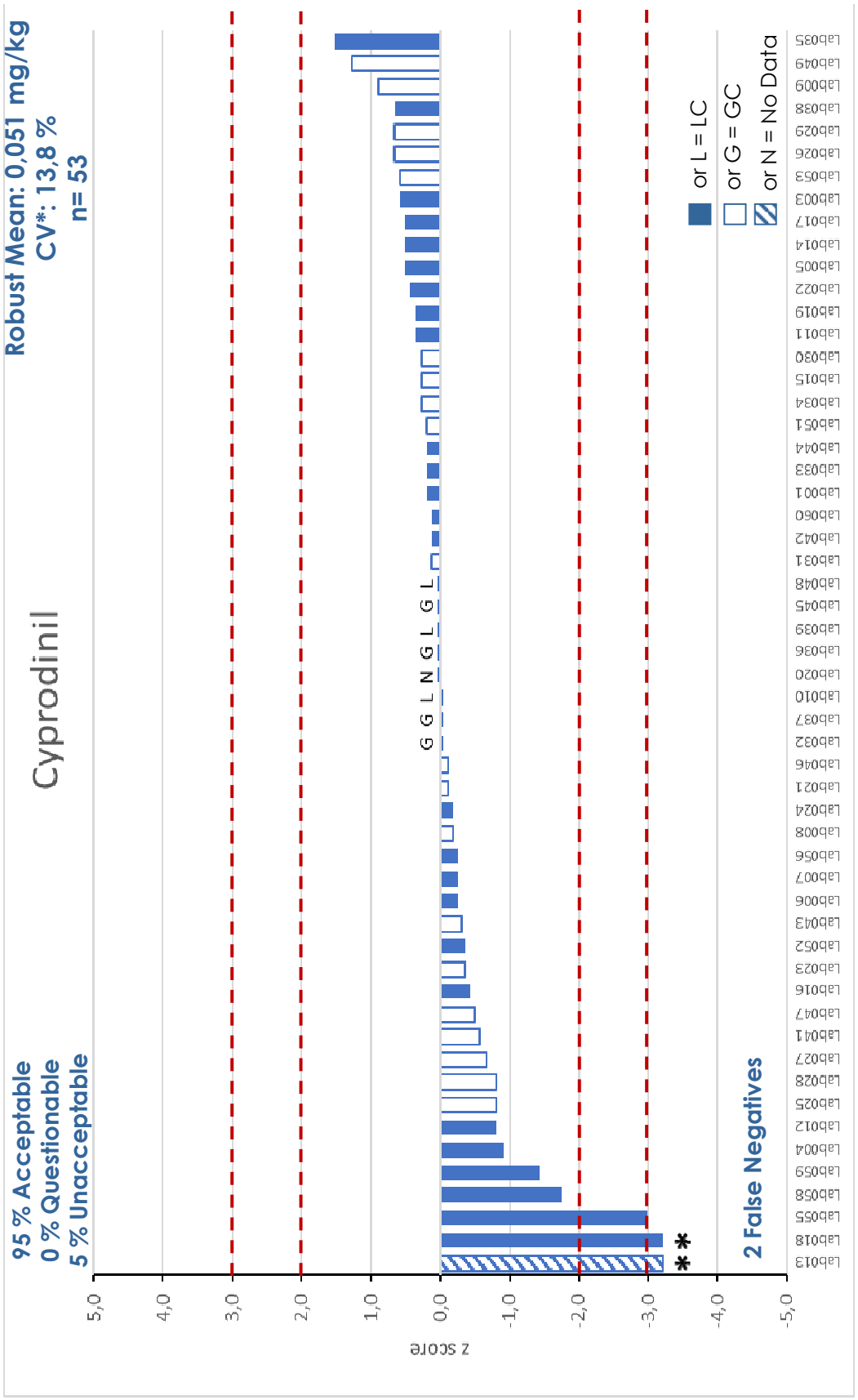
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



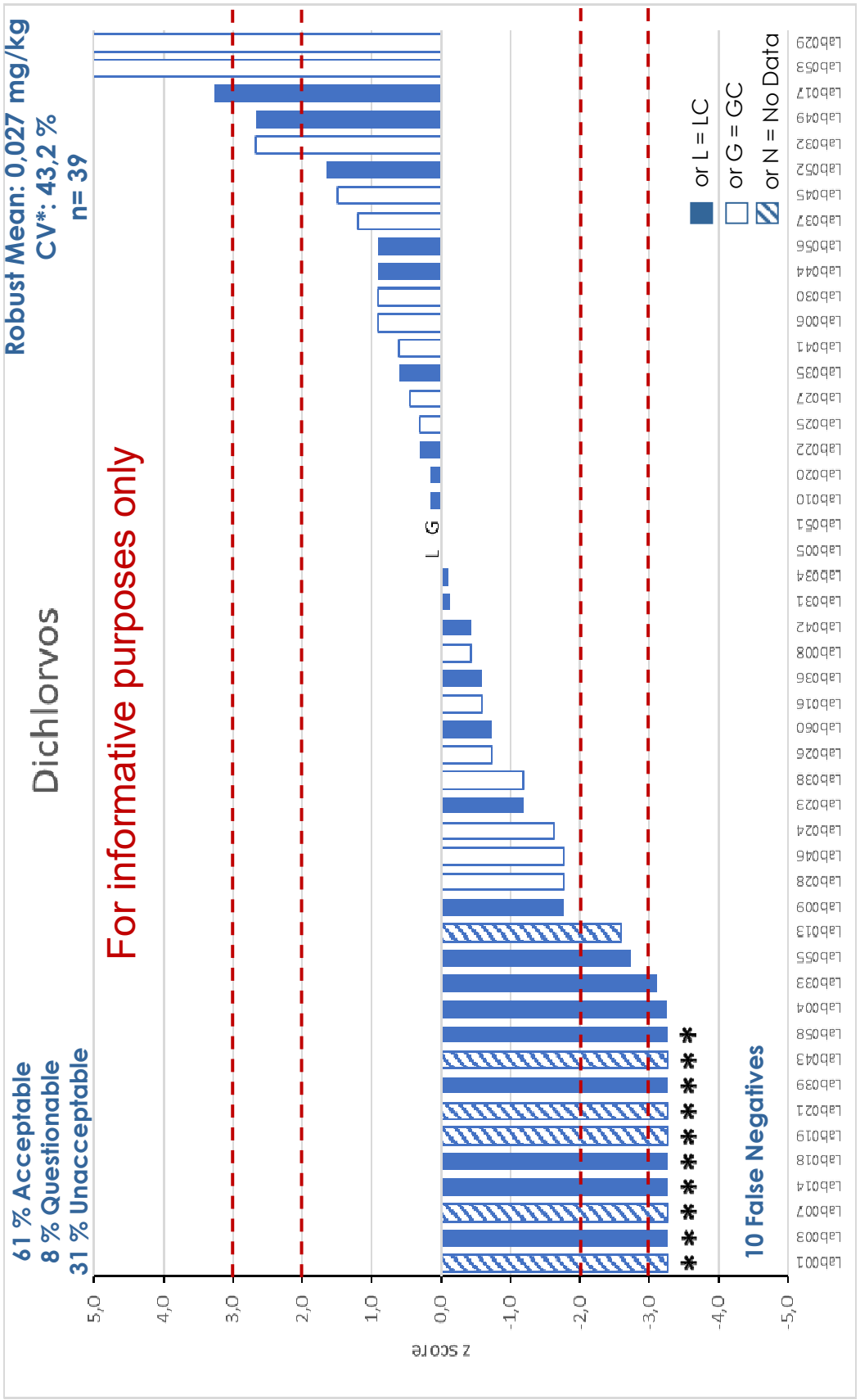
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).

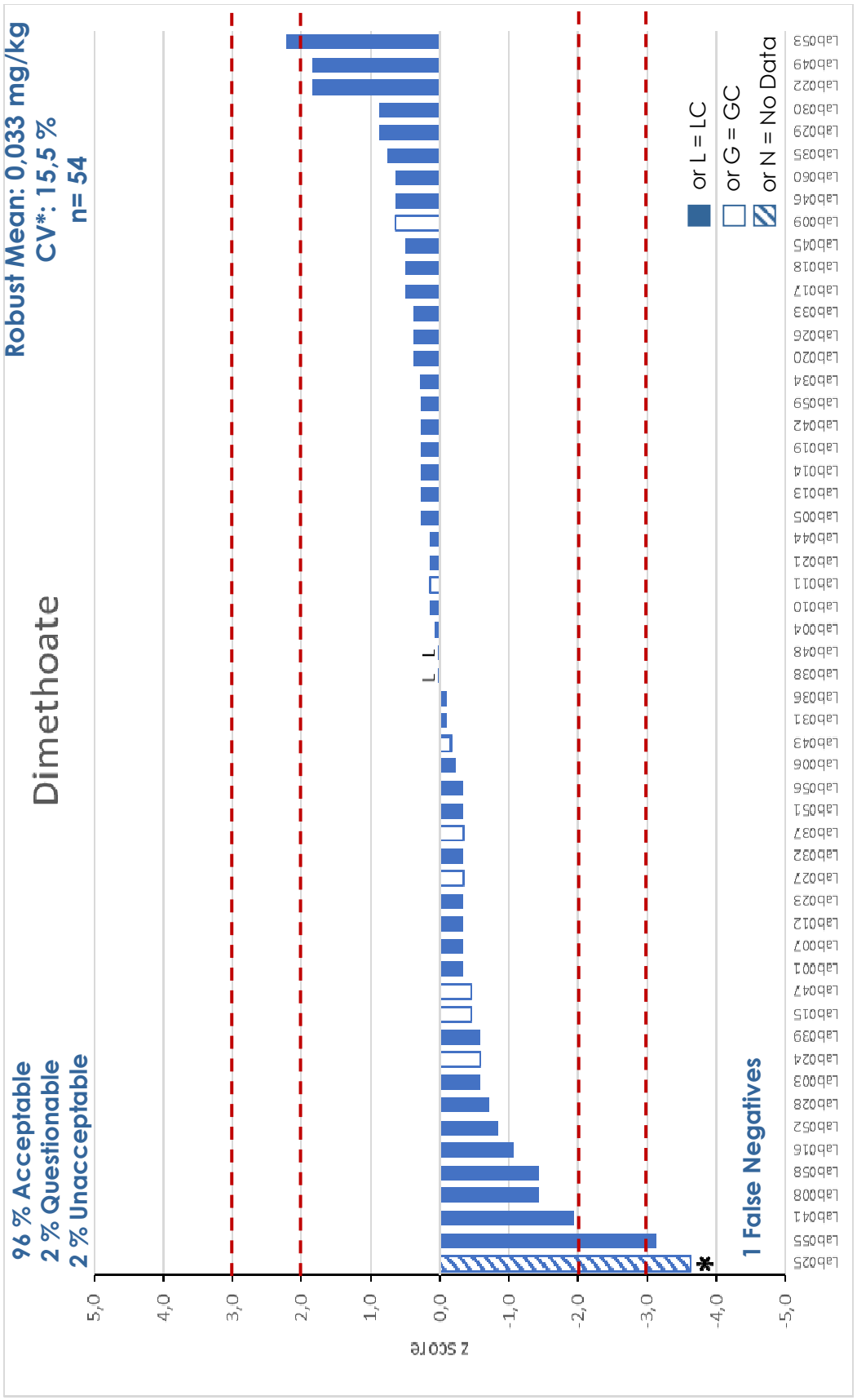


APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).

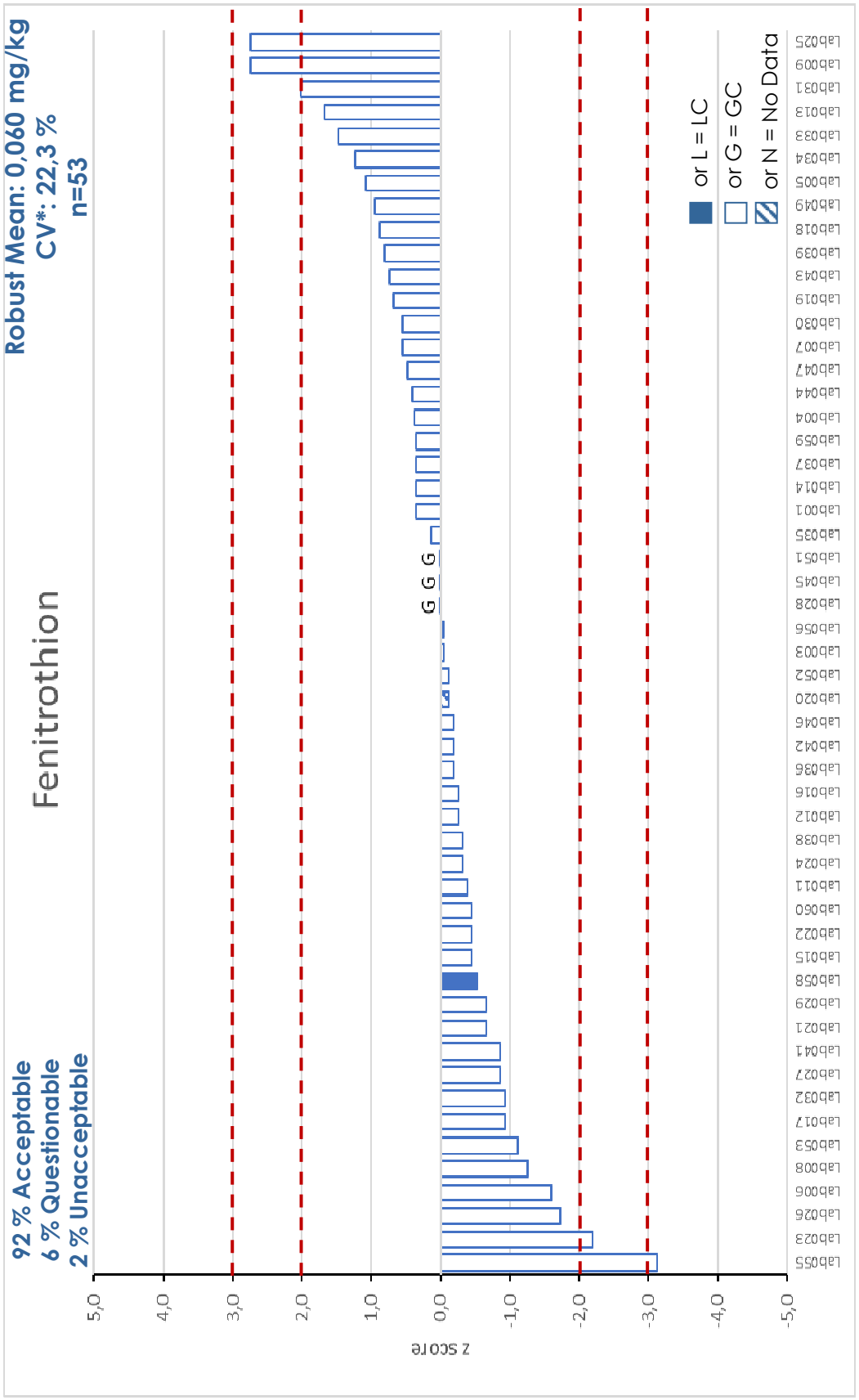


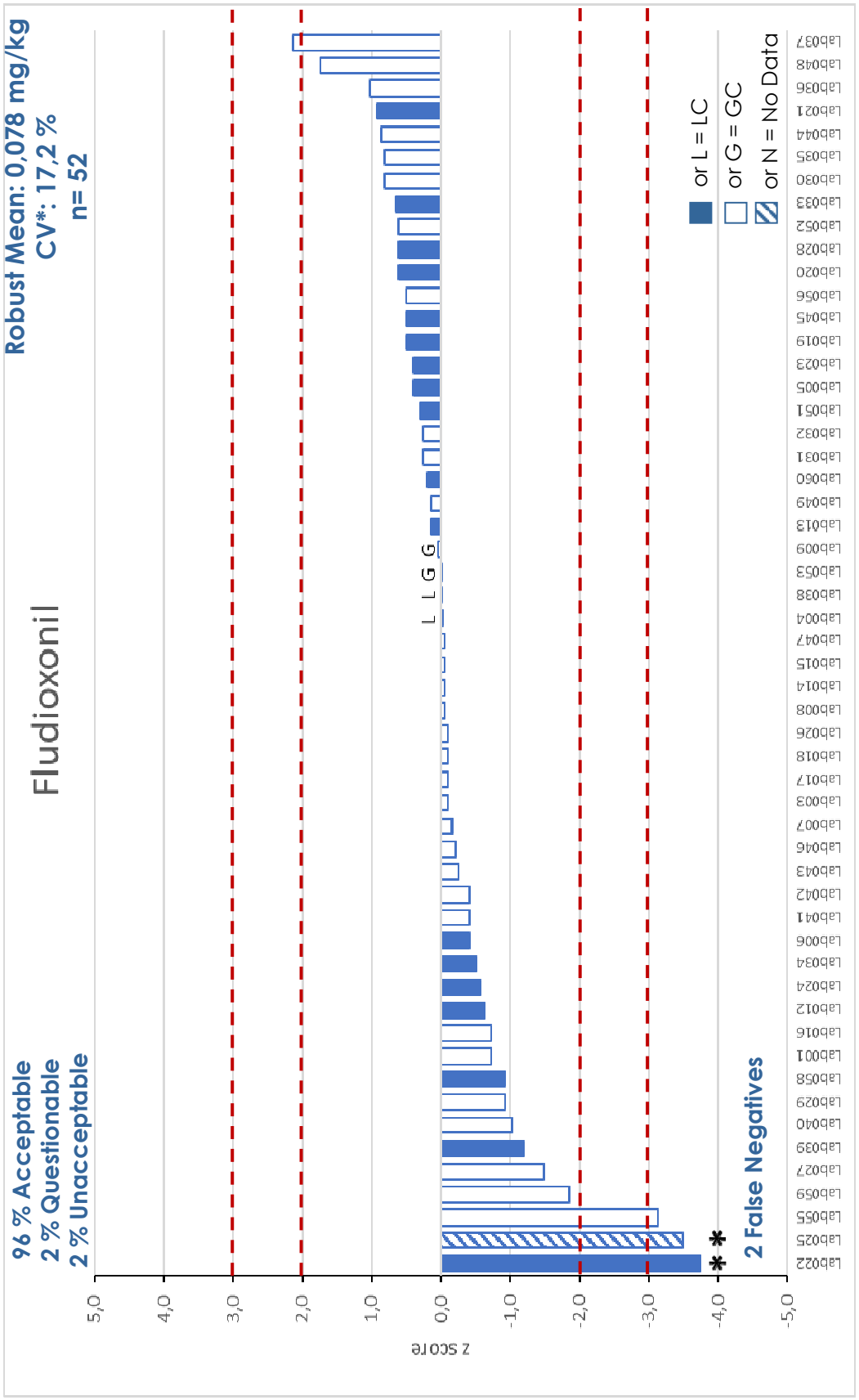
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



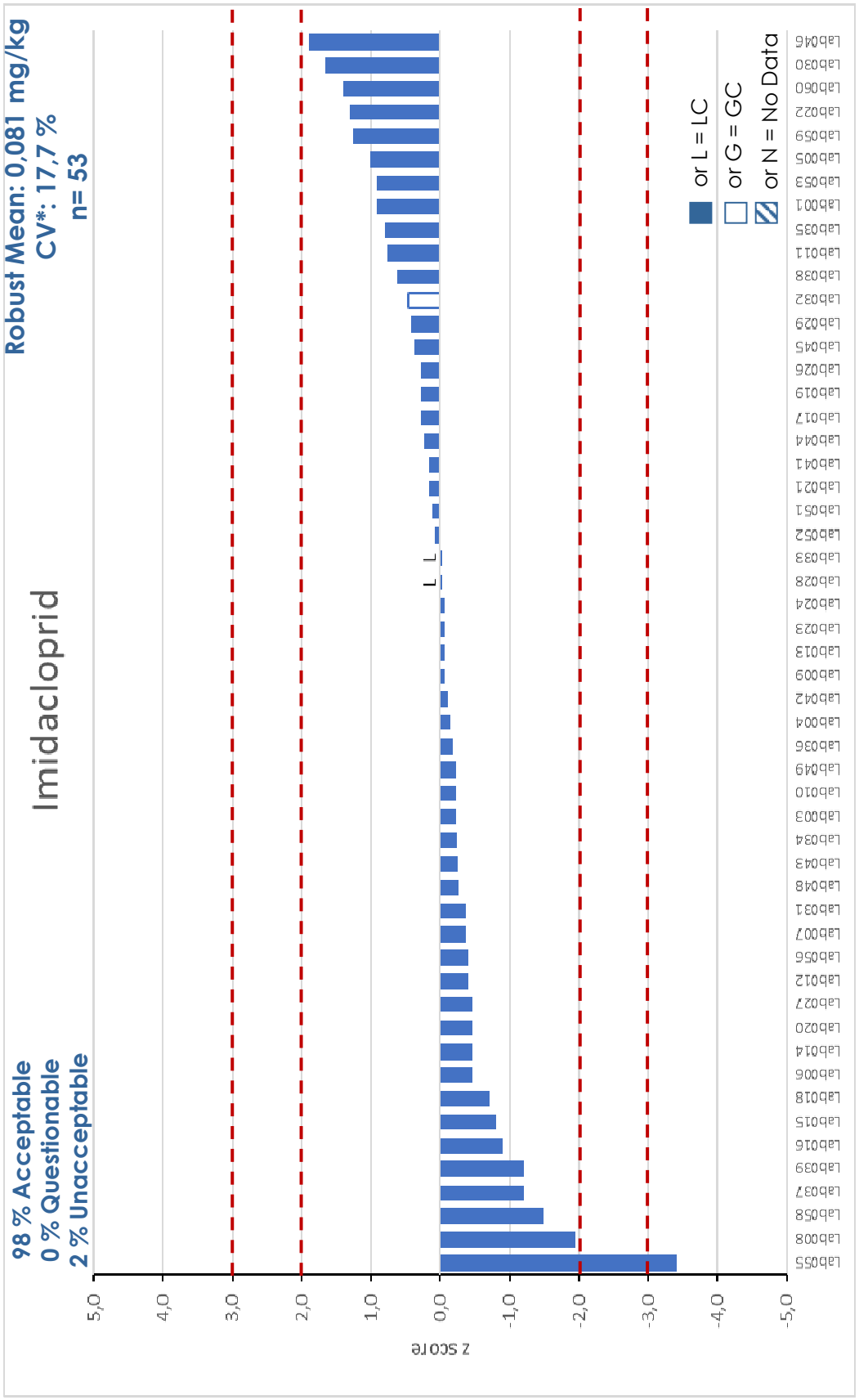

Robust Mean: 0,033 mg/kg
CV*: 15,5 %
n= 54
Dimethoate
96 % Acceptable
2 % Questionable
2 % Unacceptable
1 False Negatives
or L = LC
or G = GC
or N = No Data
Z score
5,0
4,0
3,0
2,0
1,0
0,0
-1,0
-2,0
-3,0
-4,0
-5,0

APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).

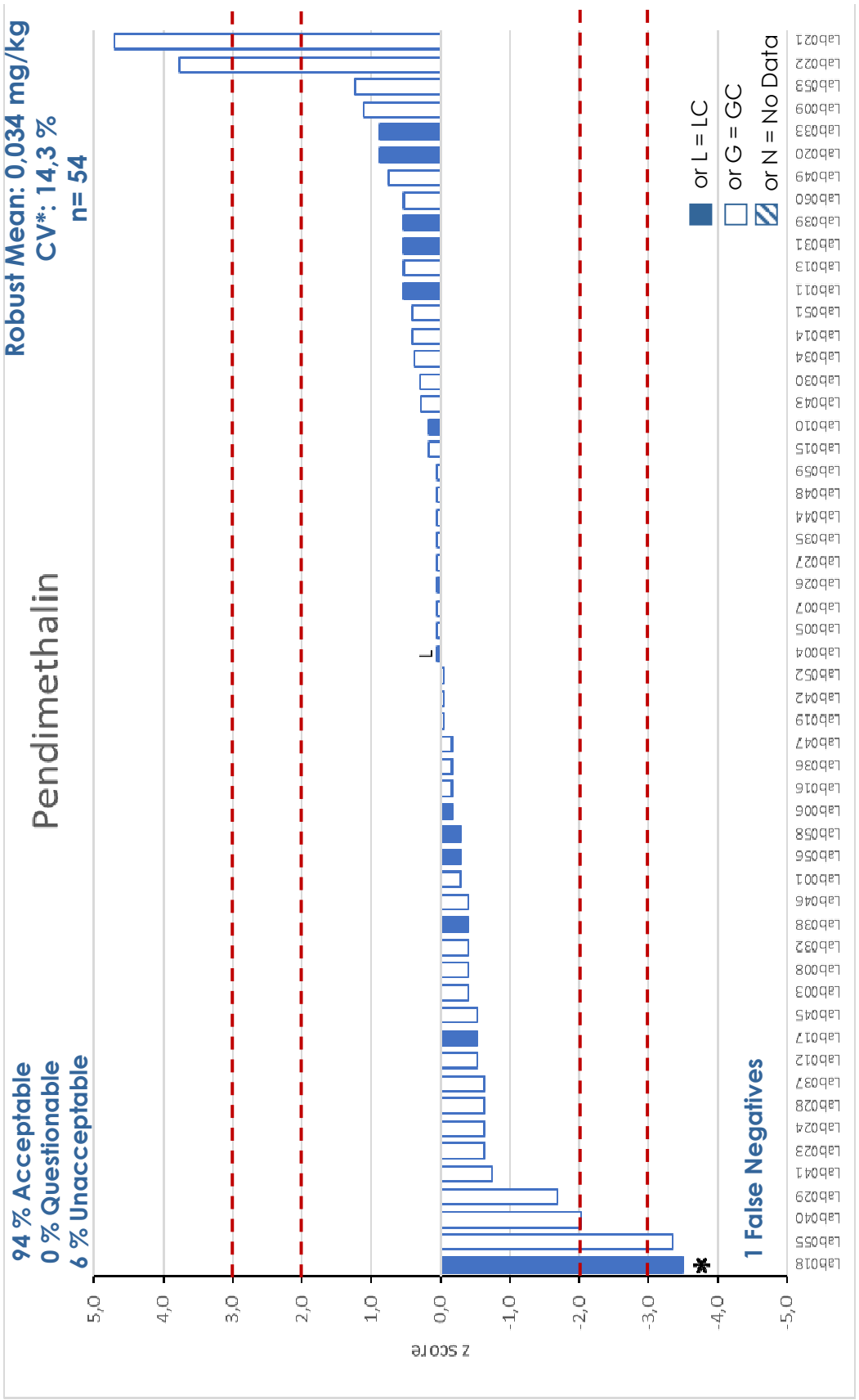



Robust Mean: 0,078 mg/kg
CV*: 17,2 %
n= 52
Fludioxonil
96 % Acceptable
2 % Questionable
2 % Unacceptable
2 False Negatives
or L = LC
or G = GC
or N = No Data
Z score
5,0
4,0
3,0
2,0
1,0
0,0
-1,0
-2,0
-3,0
-4,0
-5,0

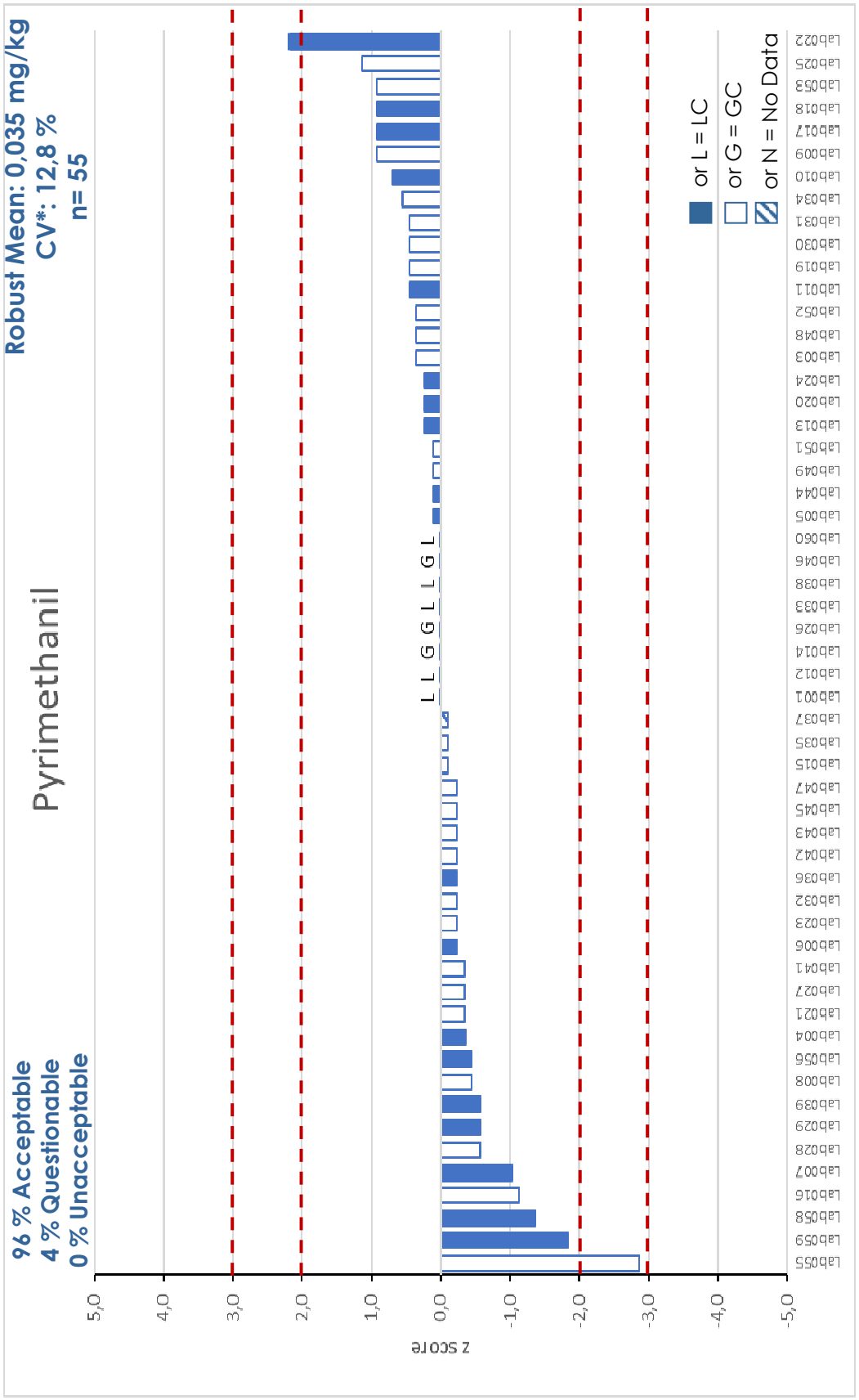
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



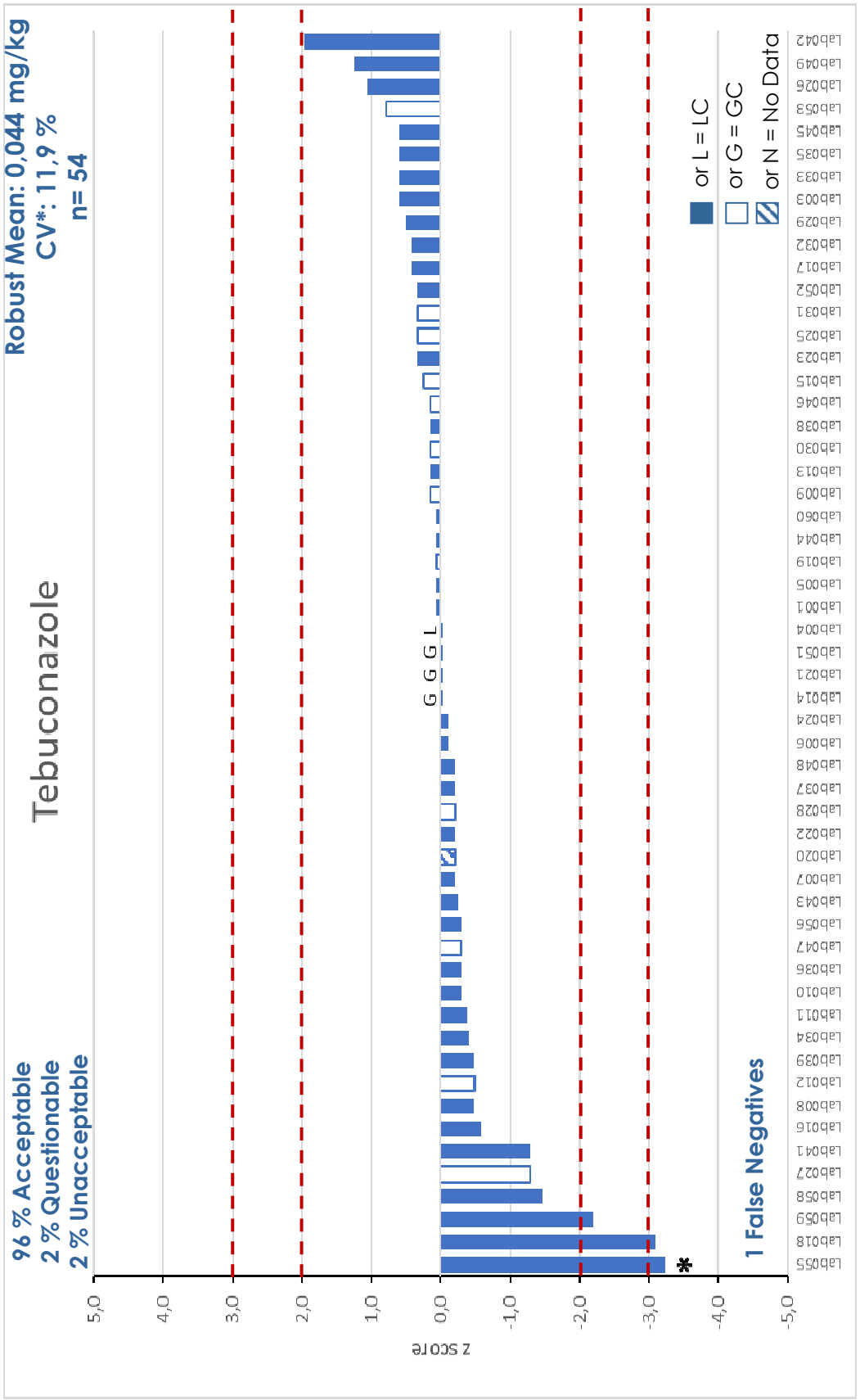
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



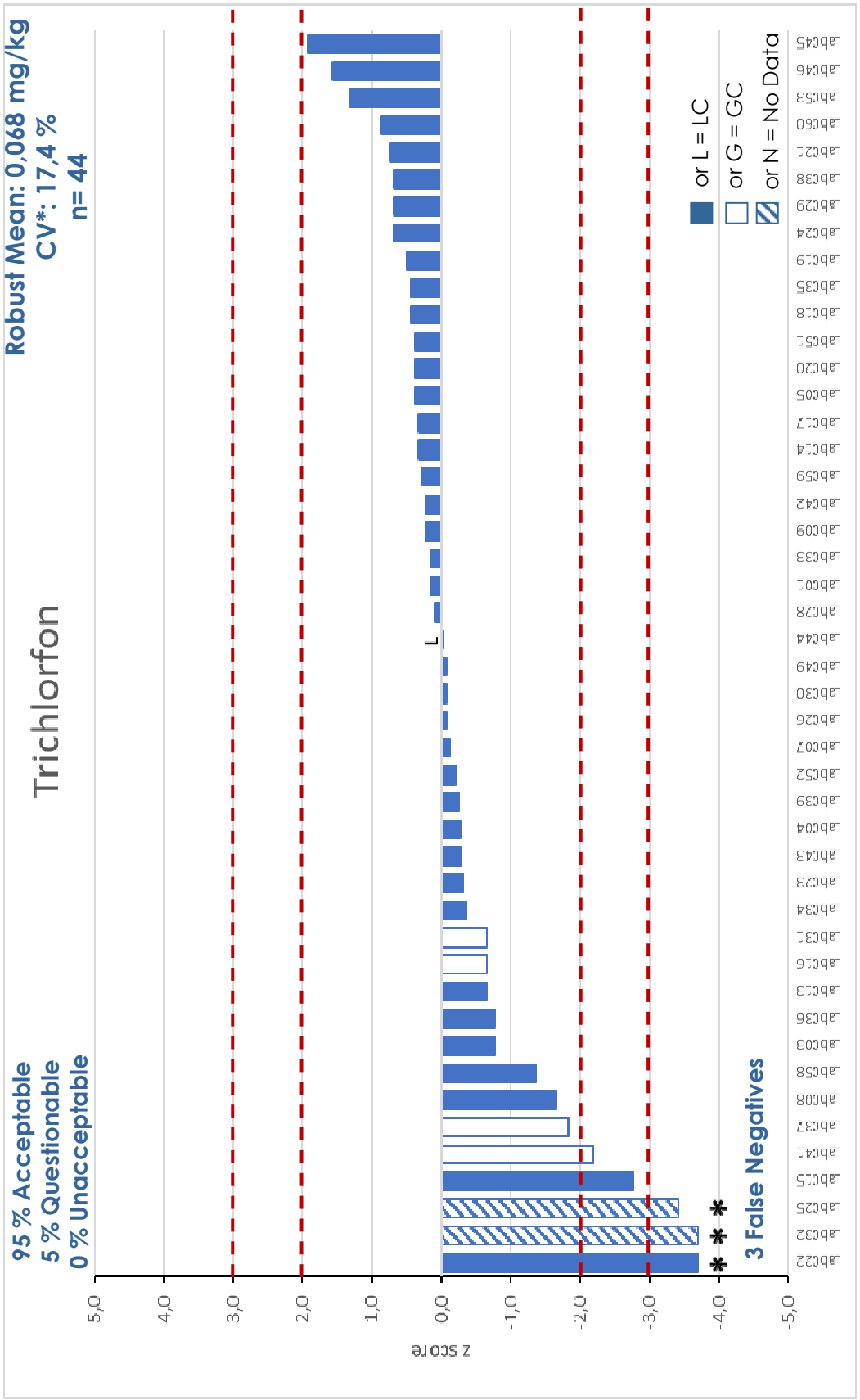
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



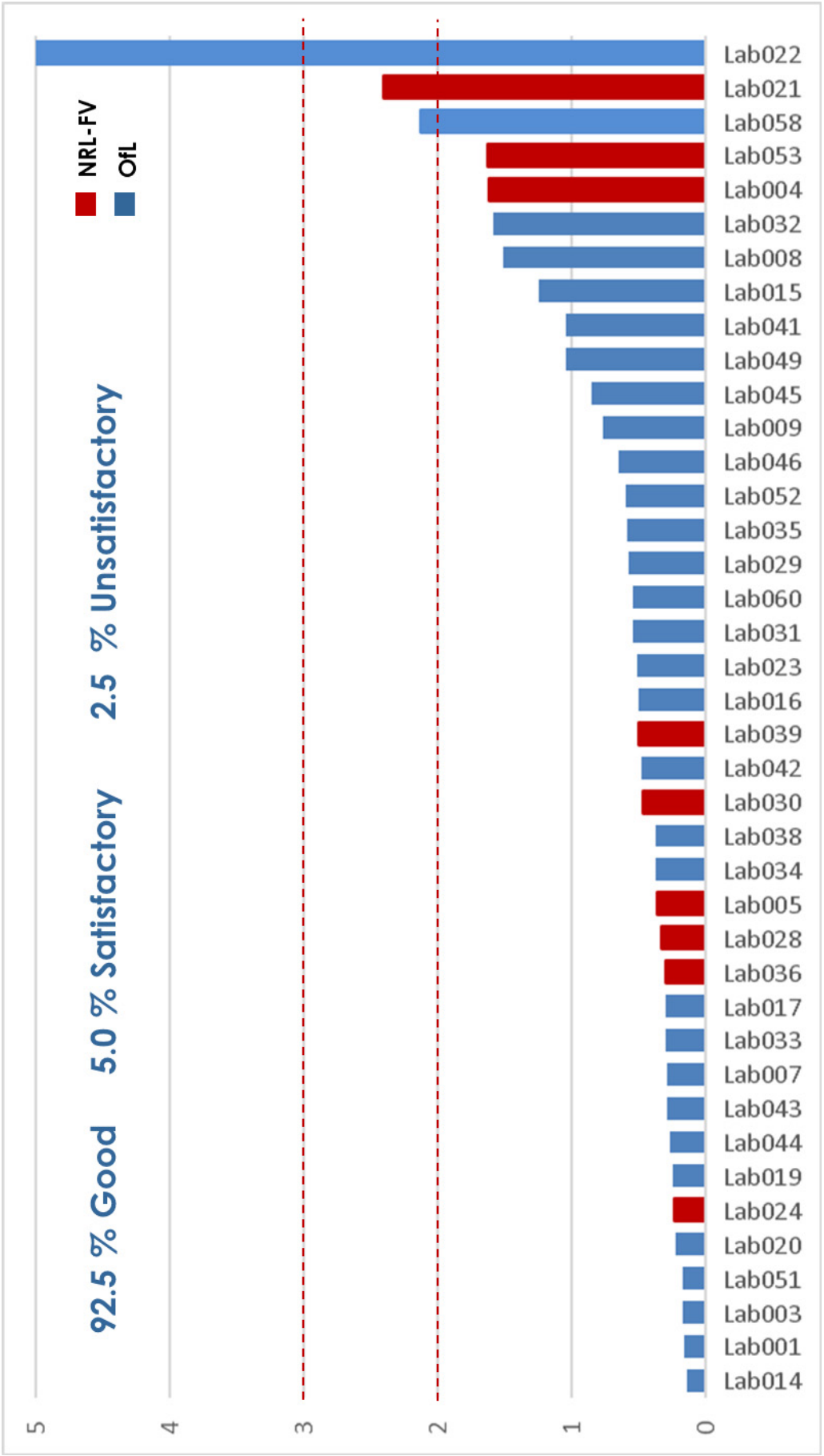
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



APPENDIX 4. Average of the Squared z scores (AZ^2) for laboratories in Category A.

Lab Code	Acephate	Aclonifen	Azoxystrobin	Boscalid	Carbaryl	Carbendazim	Chlorpyrifos	Cyprodinil	Dimethoate	Fenitrothion	Fludioxonil	Imidacloprid	Pendimethalin	Pyrimethanil	Tebuconazole	Trichlorfon	No. of z scores	AZ^2
Lab001	0.8	-0.3	0.0	-0.3	0.3	0.1	-0.1	0.2	-0.3	0.3	-0.7	0.9	-0.3	0.0	0.1	0.2	16	0.2
Lab003	0.3	-0.2	0.0	-0.5	0.2	-0.5	-0.1	0.6	-0.6	-0.1	-0.1	-0.2	-0.4	0.3	0.6	-0.8	16	0.2
Lab004	0.0	4.9	0.3	-0.1	-0.4	-0.9	-0.1	-0.9	0.1	0.4	0.0	-0.2	0.0	-0.4	0.0	-0.3	16	1.6
Lab005	-0.4	0.1	1.3	0.3	-0.2	-0.1	0.9	0.5	0.3	1.1	0.4	1.0	0.1	0.1	0.1	0.4	16	0.4
Lab007	0.9	-1.0	-0.4	-0.6	-0.6	-0.3	-0.3	-0.3	-0.3	0.5	-0.2	-0.4	0.1	-1.0	-0.2	-0.1	16	0.3
Lab008	-2.5	-0.8	-1.0	-0.7	-1.3	-1.7	-0.7	-0.2	-1.4	-1.3	-0.1	-1.9	-0.4	-0.5	-0.5	-1.7	16	1.5
Lab009	0.0	1.1	-0.2	-0.1	0.4	-0.1	0.0	0.9	0.6	2.8	0.0	-0.1	1.1	0.9	0.2	0.2	16	0.8
Lab014	0.7	0.7	0.5	0.0	-0.1	0.2	0.4	0.5	0.3	0.3	-0.1	-0.5	0.4	0.0	0.0	0.3	16	0.1
Lab015	-2.3	-0.3	-0.6	-1.0	-1.6	-1.1	0.3	0.3	-0.5	-0.5	-0.1	-0.8	0.2	-0.1	0.2	-2.8	16	1.2
Lab016	-0.4	-0.4	-1.2	-0.8	-0.1	-0.3	-0.8	-0.4	-1.1	-0.3	-0.7	-0.9	-0.2	-1.1	-0.6	-0.7	16	0.5
Lab017	1.0	-0.5	0.3	0.1	0.3	0.5	0.4	0.5	0.5	-0.9	-0.1	0.3	-0.5	0.9	0.4	0.3	16	0.3
Lab019	1.4	-0.1	0.3	0.1	0.3	0.4	-0.1	0.4	0.3	0.7	0.5	0.3	-0.1	0.5	0.1	0.5	16	0.2
Lab020	0.8	0.8	0.2	-0.2	0.5	0.5	-0.2	0.0	0.4	-0.1	0.6	-0.5	0.9	0.2	-0.2	0.4	16	0.2
Lab021	0.1	0.3	-1.1	0.1	0.8	1.2	3.3	-0.1	0.1	-0.7	0.9	0.2	4.7	-0.3	0.0	0.7	16	2.4
Lab022	4.1	-0.7	-0.1	1.5	2.2	-0.6	5.0	0.4	1.9	-0.5	-3.7	1.3	3.8	2.2	-0.2	-3.7	16	6.4
Lab023	-0.4	-1.1	0.3	-0.4	-0.1	-0.6	-0.4	-0.3	-0.3	-2.2	0.4	-0.1	-0.6	-0.2	0.3	-0.3	16	0.5
Lab024	-0.6	-0.7	-0.3	0.2	-0.4	-0.8	-0.6	-0.2	-0.6	-0.3	-0.6	-0.1	-0.6	0.2	-0.1	0.7	16	0.2
Lab028	-0.2	-1.1	-0.2	-0.9	-0.6	-0.1	-0.6	-0.8	-0.7	0.0	0.6	0.0	-0.6	-0.6	-0.2	0.1	16	0.3
Lab029	1.0	1.1	0.5	0.1	0.1	0.4	0.2	0.7	0.9	-0.7	-0.9	0.4	-1.7	-0.6	0.5	0.7	16	0.6
Lab030	0.3	1.0	0.6	-0.1	0.1	1.1	-0.2	0.3	0.9	0.5	0.8	1.7	0.3	0.5	0.2	-0.1	16	0.5
Lab031	-0.8	0.4	0.4	0.5	0.0	-1.4	0.5	0.1	-0.1	2.0	0.3	-0.4	0.5	0.5	0.3	-0.7	16	0.5
Lab032	-1.4	-0.7	-0.7	-1.0	-0.3	2.2	0.8	0.0	-0.3	-0.9	0.3	0.5	-0.4	-0.2	0.4	-3.7	16	1.6
Lab033	-0.4	0.4	0.4	-0.1	0.0	0.0	0.5	0.2	0.4	1.5	0.7	0.0	0.9	0.0	0.6	0.2	16	0.3
Lab034	1.2		0.1	0.0	0.0	1.0	0.6	0.3	0.3	1.2	-0.5	-0.2	0.4	0.6	-0.4	-0.4	15	0.4
Lab035	1.2	0.8	0.7	0.1	0.0	1.2	0.7	1.5	0.8	0.1	0.8	0.8	0.1	-0.1	0.6	0.5	16	0.6
Lab036	1.1	1.0	0.6	-0.3	0.0	0.1	0.6	0.0	-0.1	-0.2	1.0	-0.2	-0.2	-0.2	-0.3	-0.8	16	0.3
Lab038	-0.3		0.2	0.7	0.0	-1.6	-0.9	0.7	0.0	-0.3	0.0	0.6	-0.4	0.0	0.2	0.7	15	0.4
Lab039	0.1	-0.3	-0.9	-0.6	-1.0	-0.9	0.3	0.0	-0.6	0.8	-1.2	-1.2	0.5	-0.6	-0.5	-0.3	16	0.5
Lab041	-1.2	-0.4	-0.9	-0.5	-0.8	-1.1	-0.4	-0.6	-1.9	-0.9	-0.4	0.2	-0.8	-0.3	-1.3	-2.2	16	1.0
Lab042	0.9	-0.9	-0.3	0.9	0.3	-0.8	-0.3	0.1	0.3	-0.2	-0.4	-0.1	-0.1	-0.2	2.0	0.2	16	0.5
Lab043	0.4	0.4	-0.6	-0.4	-0.2	1.7	-0.3	-0.3	-0.2	0.7	-0.3	-0.2	0.3	-0.2	-0.3	-0.3	16	0.3
Lab044	1.0	0.8	0.3	0.3	-0.1	1.1	0.4	0.2	0.1	0.4	0.9	0.2	0.1	0.1	0.1	0.0	16	0.3
Lab045	-0.5	-0.7	0.9	0.5	1.9	-1.7	-0.3	0.0	0.5	0.0	0.5	0.4	-0.5	-0.2	0.6	1.9	16	0.8
Lab046	-0.1	-0.5	0.8	1.2	-1.0	0.4	0.0	-0.1	0.6	-0.2	-0.2	1.9	-0.4	0.0	0.2	1.6	16	0.6
Lab049	0.2	1.2	-0.3	2.3	0.9	0.7	0.7	1.3	1.9	0.9	0.1	-0.2	0.8	0.1	1.2	-0.1	16	1.0
Lab050	-1.9	-0.4	0.3	-0.7	-0.1	1.0	0.2	-0.3	-1.2	-1.6	1.1	2.0	0.1	0.3	0.1	-3.4	16	1.7
Lab051	0.5	-0.8	-0.1	-1.0	-0.2	0.4	-0.2	0.2	-0.3	0.0	0.3	0.1	0.4	0.1	0.0	0.4	16	0.2
Lab052	-0.1	0.5	0.5	-0.1	0.3	2.7	-0.2	-0.3	-0.8	-0.1	0.6	0.1	-0.1	0.3	0.3	-0.2	16	0.6
Lab053	-1.4	0.6	0.6	0.9	2.4	2.1	0.5	0.6	2.2	-1.1	0.0	0.9	1.2	0.9	0.8	1.3	16	1.6
Lab054	-1.0	-1.4	-0.2	0.6	-0.4	-0.6	-1.4	-0.2	-0.7	-1.4	1.0	0.7	0.3	1.2	2.3	-0.7	16	1.1
Lab058	-2.9	-1.4	-1.1	-1.5	-1.6	-1.3	-1.3	-1.7	-1.4	-0.5	-0.9	-1.5	-0.3	-1.4	-1.5	-1.4	16	2.1
Lab060	-0.5		0.9	0.9	1.2	-1.1	0.3	0.1	0.6	-0.5	0.2	1.4	0.5	0.0	0.1	0.9	15	0.5

APPENDIX 5. EUPT-FV-SC05-AZ²-Graphical representation for EU/EFTA laboratories in Category A.



9th Edition
Revised:15th November 2019

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 (EUPTs) organised on behalf of the European Commission, DG-SANTE[5] by the four European Union Reference Laboratories (EURLs) responsible for pesticide residues in food and feed. These EUPTs are directed at laboratories belonging to the Network[6] of National Reference Laboratories (NRLs) and Official Laboratories (OfLs) of the EU Member States. OfLs from EFTA countries and EU-Candidate countries are also welcome to participate in the EUPTs. OfLs from Third countries may be permitted to participate on a case-by-case basis.
The following four EURLs for pesticide residues were appointed by DG-SANTE based on regulation 882/2004/EC that was repealed by regulation 625/2017/EC[7]:

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

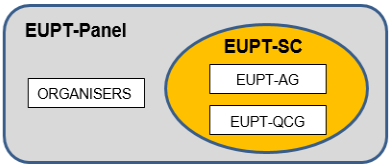
The aim of these EUPTs is to obtain information regarding the quality, accuracy and comparability of pesticide residue data in food and feed reported to the European Union within the framework of the national control programmes and the EU multiannual co-ordinated control programme[8]. Participating laboratories will be provided with an assessment of their analytical performance that they can use to demonstrate their analytical performance and compare themselves with other participating laboratories.

EUPT-Organisers and Scientific Committee
EUPTs 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 EUPT-participation certificates.
To complement the internal expertise of the EURLs, a group of external consultants forming the **EUPT-Scientific Committee** (EUPT-SC)[9] has been established and approved by DG-SANTE. The EUPT-SC consists of expert scientists with many years of experience in PTs and/or pesticide residue analysis. The actual composition of the EUPT-SC and the affiliation of each of its members is shown on the EURL-Website. The members of the EUPT-SC are also listed in the Specific Protocol and the Final Report of each EUPT.
The EUPT-SC is made up of the following two subgroups:

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

The EUPT-SC's role is to help the Organisers make decisions regarding the EUPT design: the selection of the commodity, the selection of pesticides to be included in the Target Pesticide List (see below), the establishment of the Minimum Required Reporting Levels (MRRLs), the statistical treatment and evaluation of the participants' results (in anonymous form), and the drafting and updating of documents, such as the General and Specific PT Protocols and the Final EUPT-Reports.
The EUPT-QCG has the additional function of supervising the quality of EUPTs and of assisting the EURLs in confidential aspects such as the choice of the pesticides to be present in the Test Item and the approximate concentrations at which they should be present.
The EUPT-SC typically meets once a year, after the EUPTs of all four pesticide EURLs have been conducted, to discuss the evaluation of the EUPT-results and to assist the EURLs in their decision making. Upcoming EUPTs are also planned during these meetings.

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



The decisions of the EUPT-Panel will be documented.
This present EUPT General Protocol was jointly drafted by the EUPT-SC and the EURLs.

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[5] DG-SANTE = European Commission, Health and Food Safety Directorate-General
[6] For more information about the EURL/NRL/OfL-Network please refer to the EURL-Web-portal under: "http://www.eurl-pesticides.eu"
[7] Regulation (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
[8] European Commission Proficiency Tests for Pesticide Residues in Fruits and Vegetables, Trends in Analytical Chemistry, 2010, 29 (1), 70 – 83.
[9] Link to the List of current members of the EUPT Scientific Committee:
http://www.eurl-pesticides.eu/library/docs/allcrl/EUPT-SC.pdf

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

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[10] (for all OfLs analysing for pesticide residues within the framework of official controls[11] 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).
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

[10] Regulation (EC) No 396/2005, published at OJ of the EU L70 of 16.03.2005, as last amended by Regulation 839/2008 published at OJ of the EU L234 of 30.08.2008.
[11] Official controls in the sense of Reg. 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.

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 $|y_i - y| \leq 0.3 \times \sigma_{pt}$, with y_i being the mean value of the results of the last phase of the stability test, y being the mean value of the results of the first phase of the stability test and σ_{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.

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 approached 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[12], 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 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:

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

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-σ_{pt}) will be calculated using a Fit-For-Purpose approach with a fixed Relative Standard Deviation (FFP-RSD).
Based on experience from previous EUPTs13, a percentage FFP-RSD of 25 % is currently used for all analyte-matrix combination, with the target standard deviation being calculated as follows:

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

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:

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

where x_i is the value reported by the laboratory, x_{pt} is the assigned value, and FFP-σ_{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[14]:

$\lvert z \rvert \leq 2.0$	Acceptable
$2.0 < \lvert z \rvert < 3.0$	Questionable
$\lvert z \rvert \geq 3.0$	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.

[12] 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).
[13] 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.
[14] ISO/IEC 17043:2010. Conformity assessment – General requirements for proficiency testing

– **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. 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	N - 1
6	5.4	5	
7	6.3	6	
8	7.2	7	
9	8.1	8	
10	9.0	9	
11	9.9	10	
12	10.8	11	
13	11.7	12	
14	12.6	13	
15	13.5	13	N - 2
16	14.4	14	
17	15.3	15	
18	16.2	16	
19	17.1	17	
20	18	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^2)[15,16] (see below) will be used. The AZ^2 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^2, z scores higher than 5 will be set as 5. Based on the AZ^2 achieved, the laboratories are classified as follows:

$AZ^2 \leq 2.0$	Good
$2.0 < AZ^2 < 3.0$	Satisfactory
$AZ^2 \geq 3.0$	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^2 if it is considered as not being useful or if the number of results reported by any participant is considered to be too low.

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

[15] Formerly named "Sum of squared z scores (SZ^2)"

[16] Laboratory assessment by combined z score values in proficiency tests: experience gained through the EUPT for pesticide residues in fruits and vegetables. Anal. Bioanal. Chem., 2010, 397, 3061–3070.

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

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 AZ^2 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[17]:

Phase 1:
- Identifying the origin of the bad results (failure in EUPTs).
- Actions: On the spot visits and training if necessary and repetition of the comparative test if feasible and close the assessment of results by the EURL.

Phase 2:
- If the results still reveal underperformance the Commission shall be informed officially by the EURL including a report of the main findings and corrective actions.
- The Commission shall inform the Competent Authority and require that appropriate actions are taken.

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

Disclaimer
The EUPT-Panel retains the right to change any parts of this EUPT – General Protocol based on new scientific or technical information. Any changes will be communicated in due course.

[17] Article 101 of Regulation (EC) 625/2017

EUPT-FV-SC05 SPECIFIC PROTOCOL

European Union Proficiency Test for Pesticide Residues in dried white beans (2021)

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

Test item
This proficiency test is based on the analysis of **dried white beans** containing pesticide residues. The test item will consist of **dried white beans powder.**
The test item will be homogenised and sub-sampled into coded bottles. Ten of those bottles containing the test item will be chosen randomly and analysed to check for homogeneity.
The test item will be stored frozen (–20ºC) prior to shipment to participants.
Three bottles, again chosen randomly, will be analysed by the Organiser over a period of time to confirm the stability of the pesticides in the test item (firstly, when the test items are shipped, then a few days after the receipt deadline for participants´ results).
No blank material will be provided.

Steps to follow
This Proficiency Test will be made up of the following steps:

Participation in this proficiency test remains on a voluntary basis. To participate, each laboratory must complete the Application Form, uploaded in the EURL-FV webpage, before the deadline stipulated on the Calendar. The participants will also receive the Target Pesticide List, containing the Minimum Required Reporting Limits (MRRLs). Given the limited material available, the registration forms will be accepted on a first come first served basis.

2.Laboratories will then receive an e-mail confirming their participation in this exercise and assigning them each a Laboratory Code.

3.The sample delivery will be 250 euros for EU national reference laboratories and EU official laboratories for pesticide residues and 350 euros for the rest of laboratories.

4.The sample will be delivered to the participant laboratories on November 29th 2021. The Excel file to report the results will be uploaded to the EURL-FV webpage.

5.The deadline for submitting the results of this proficiency test is 10th January 2022.

6.The Organiser will evaluate the results at the end of the proficiency test, once the deadline for the receipt of results has passed. The Organiser will upload an electronic version onto the EURL-FV website and will send the electronic copy of the Final Report to each participant laboratory. This report will include information regarding the design of the test, the homogeneity and stability results, a statistical evaluation of the participant's results as well as graphical displays of the results and any conclusions. Further relevant information considered to be of value may also be included.

Amount of Test Item
Participants will receive:
• Approximately 125 g of dried white beans powder.

Shipment of Test item
The shipment of the test item will be on 29th November 2021. The Organiser will try to ensure that all the packages arrive on the same day at each laboratory. An information message will be sent out by e-mail before shipment. Laboratories must make their own arrangements for the receipt of the package. They must inform the Organiser of any public holidays in their country/city during the delivery period given in the calendar, as well as making the necessary arrangements for receiving the shipment, even if the laboratory is closed.

Advice on Test item Handling
Once received, the test item should be stored deeply frozen (-18ºC 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.

Test item Receipt
Once the laboratory has received the test item, its arrival must be reported to the Organiser by e-mail. The deadline for acceptance (or non-acceptance) is 3rd December 2021. If the laboratory does not respond by this date, the Organiser will assume that the test item has been received and accepted.
If any laboratory has not received the test item by 3rd December 2021, they must inform the Organiser by e-mail (cferrer@ual.es)

Submission of results:
Once the laboratory has analysed the test item and is ready to submit their data, they must enter their results in the Excel file provided by the Organisers and send it to the following e-mail address: cferrer@ual.es.
All analyte concentrations must be expressed in mg/kg together with the associated recovery expressed as a percentage.
The number of significant figures should be based on the guidelines provided in SANTE/12682/2019. Additional significant figures may be recorded for the purpose of statistical analysis. Please bear this in mind when reporting data:
- Residue levels above the reporting level and < 10 mg/kg should be rounded to two significant figures.
- Residue levels ≥ 10 mg/kg may be rounded to three significant figures or to a whole number.

Results should not be reported where a pesticide was not detected or was detected below the laboratory's LOQ. In both cases, this will be considered as 'ND' (Not Detected). If a pesticide was not sought, it will be considered as 'NA' (Not Analysed). The actual results/residue levels measured must be reported as numbers.

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

Further instructions on how to fill in the Excel file will be provided in the same file.

False Negatives
After the receipt of results, participant laboratories that have reported that they sought a pesticide present in the test item but did not find it (false negative) will be asked via e-mail about the analytical method used to determine that specific pesticide.

Calendar

ACTIVITY	DATE
Opening Registration period	26th October 2021
Deadline for receiving Application Form from laboratories.	15th November 2021
Sample distribution	29th November 2021
Deadline for receiving results	10th January 2022
Preliminary Report with statistical treatment	February 2022
Final Report	August 2022

Cost of test item shipment.
The sample delivery will be 250 € for EU National Reference Laboratories and EU Official Laboratories and 350 € for the rest of laboratories. Regarding payment procedures, each laboratory can specify their details and invoice requests when applying for the test.

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

Payment details are as follows:
BANK NAME: CAJAMAR - Caja Rural Sociedad Corporativa de Crédito
BANK ACCOUNT HOLDER: Universidad de Almeria
BANK ADDRESS: Office Number 990. Universidad de Almeria. Spain
IBAN: ES0730580130172731005000
SWIFT: CCRIES2A
REFERENCE: Invoice No. or Lab Code

Contact information
The official organising group details are as follows:
Universidad de Almería. Edificio Químicas CITE I
Ctra. Sacramento s/n
04120 La Cañada de San Urbano Almería - Spain
Phone No.: +34 950214102

Organising team (e-mails and phone no.s):
Amadeo R. Fernández-Alba EURL-FV amadeo@ual.es +34 950015034
Carmen Ferrer AmateEURL-FV cferrer@ual.es +34 950214102
Octavio Malato RodríguezEURL-FV omalato@ual.es +34 950214423
María Murcia Morales EURL-FV mariamurcia@ual.es +34 950214102

Scientific Committee
Michelangelo Anastassiades, EURL-SRM, CVUA Stuttgart, Fellbach, Germany.
Magnus Jezussek, Bavarian Health and Food Safety Authority, Erlangen, Germany.
André de Kok, Wageningen Food Safety Research, Wageningen, The Netherlands.
Ralf Lippold, EURL-AO, CVUA Freiburg, Germany.
Sonja Masselter, AGES, Innsbruck, Austria.
Paula Medina Pastor, EFSA, Parma, Italy
Hans Mol, Wageningen Food Safety Research, Wageningen, The Netherlands.
Finbarr O'Regan, The Pesticide Control Laboratory, Celbridge, Ireland.
Patrizia Pelosi, Istituto Superiore di Sanità, Rome, Italy.
Tuija Pihlström, National Food Agency, Uppsala, Sweden.
Mette Erecius Poulsen, EURL-CF, National Food Institute (DTU), Søborg, Denmark.
Antonio Valverde, University of Almería, Spain

TARGET PESTICIDE LIST FOR THE EUPT-FV-SC05

Pestide no.	Pesticides	MRRL (mg/kg)
1	Acephate	0.01
2	Acetamiprid	0.01
3	**Aclonifen**	**0.01**
4	Acrinathrin	0.01
5	Aldicarb	0.01
6	Aldicarb Sulfone	0.01
7	Aldicarb Sulfoxide	0.01
8	Aldrin	0.005
9	Ametoctradin	0.01
10	Azinphos-methyl	0.005
11	Azoxystrobin	0.01
12	Bifenthrin (sum of isomers)	0.01
13	Biphenyl	0.01
14	Bitertanol (sum of isomers)	0.01
15	Boscalid	0.01
16	Bromopropylate	0.01
17	Bromuconazole (sum of diastereoisomers)	0.01
18	Bupirimate	0.01
19	Buprofezin	0.01
20	Cadusafos	0.005
21	Carbaryl	0.005

Pestide no.	Pesticides	MRRL (mg/kg)
22	Carbendazim	0.01
23	Carbofuran	0.005
24	Carbofuran-3-hydroxy	0.005
25	Chlorantraniliprole	0.01
26	Chlorfenapyr	0.01
27	Chlorfenvinphos	0.01
28	Chlorobenzilate	0.01
29	Chlorothalonil	0.01
30	Chlorpropham	0.01
31	Chlorpyrifos	0.005
32	Chlorpyrifos-methyl	0.01
33	Clofentezine	0.01
34	Clothianidin	0.01
35	**Cyantraniliprole**	**0.01**
36	Cyazofamid	0.01
37	Cyflufenamid: sum of cyflufenamid (Z-isomer) and its E-isomer	0.01
38	Cyfluthrin (cyfluthrin incl. other mixtures of constituent isomers (sum of isomers))	0.01
39	Cymoxanil	0.01
40	Cypermethrin (cypermethrin incl. other mixtures of constituent isomers (sum of isomers))	0.01
41	Cyproconazole	0.01
42	Cyprodinil	0.01
43	Deltamethrin (cis-deltamethrin)	0.01
44	Demeton-S-methylsulfone	0.005
45	Diazinon	0.005
46	Dichlofluanid	0.01
47	Dichlorvos	0.005
48	Dicloran	0.01
49	Dicofol (sum of p, p´ and o,p´ isomers)	0.01
50	Dieldrin	0.005
51	Diethofencarb	0.01
52	Difenoconazole	0.01
53	Diflubenzuron	0.01
54	Dimethoate	0.003
55	Dimethomorph (sum of isomers)	0.01
56	Dimethylaminosulfotoluidide (DMST)	0.01
57	Diniconazole (sum of isomers)	0.01
58	Diphenylamine	0.01
59	Endosulfan alpha	0.01
60	Endosulfan beta	0.01
61	Endosulfan sulfate	0.01
62	EPN	0.01
63	Epoxiconazole	0.01
64	Ethion	0.01
65	Ethirimol	0.01
66	Ethoprophos	0.005
67	Etofenprox	0.01
68	Etoxazole	0.01
69	Famoxadone	0.01
70	Fenamidone	0.01
71	Fenamiphos	0.01
72	Fenamiphos sulfone	0.01
73	Fenamiphos sulfoxide	0.01
74	Fenarimol	0.01
75	Fenazaquin	0.01
76	Fenbuconazole	0.005
77	Fenhexamid	0.01
78	Fenitrothion	0.01
79	Fenoxycarb	0.01
80	Fenpropathrin	0.01
81	Fenpropidin	0.01
82	Fenpropimorph (sum of isomers)	0.01
83	Fenpyrazamine	0.01
84	Fenpyroximate	0.01
85	Fenthion	0.01
86	Fenthion oxon	0.01
87	Fenthion oxon sulfone	0.01
88	Fenthion oxon sulfoxide	0.01
89	Fenthion sulfone	0.01
90	Fenthion sulfoxide	0.01
91	Fenvalerate (any ratio of constituent isomers (RR, SS, RS & SR) including esfenvalerate)	0.01
92	Fipronil	0.004

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

Pestide no.	Pesticides	MRRL (mg/kg)
93	Fipronil sulfone	0.004
94	Flonicamid	0.01
95	Flubendiamide	0.01
96	Fludioxonil	0.01
97	Flufenoxuron	0.01
98	Fluopicolide	0.01
99	Fluopyram	0.01
100	Fluquinconazole	0.01
101	Flusilazole	0.01
102	Flutolanil	0.01
103	Flutriafol	0.01
104	Fluxapyroxad	0.01
105	Formetanate (expressed as formetanate (hydrochloride))	0.01
106	Fosthiazate	0.01
107	Hexaconazole	0.01
108	Hexythiazox	0.01
109	Imazalil	0.005
110	Imidacloprid	0.01
111	Indoxacarb (sum of indoxacarb and its R enantiomer)	0.01
112	Iprodione	0.01
113	Iprovalicarb	0.01
114	Isocarbophos	0.01
115	Isofenphos-methyl	0.01
116	Isoprothiolane	0.01
117	Kresoxim-methyl	0.01
118	Lambda-Cyhalothrin	0.01
119	Linuron	0.01
120	Lufenuron (any proportion of constituent isomers)	0.01
121	Malaoxon	0.01
122	Malathion	0.01
123	Mandipropamid	0.01
124	Mepanipyrim	0.01
125	Metaflumizone (sum of E- and Z- isomers)	0.01
126	Metalaxyl and metalaxyl-M	0.01
127	Methamidophos	0.01
128	Methidathion	0.01
129	Methiocarb	0.01
130	Methiocarb sulfone	0.01
131	Methiocarb sulfoxide	0.01
132	Methomyl	0.01
133	Methoxyfenozide	0.01
134	Metrafenone	0.01
135	Monocrotophos	0.005
136	Myclobutanyl	0.01
137	Omethoate	0.003
138	Orthophenylphenol (Free compound only)	0.01
139	Oxadixyl	0.01
140	Oxamyl	0.01
141	Oxydemeton-methyl	0.005
142	Paclobutrazole	0.01
143	Paraoxon-methyl	0.01
144	Parathion-ethyl	0.01
145	Parathion-methyl	0.01
146	Penconazole	0.01
147	Pencycuron	0.01
148	Pendimethalin	0.01
149	Permethrin (sum of isomers)	0.01
150	Phenthoate	0.01
151	Phosalone	0.01
152	Phosmet	0.01
153	Phosmet oxon	0.01
154	Phoxim	0.01
155	Pirimicarb	0.01
156	Pirimicarb-desmethyl	0.01
157	Pirimiphos-methyl	0.01
158	Prochloraz (only parent compound)	0.01
159	Procymidone	0.01
160	Profenofos	0.01
161	Propamocarb (only parent compound)	0.01
162	Propargite	0.01
163	Propiconazole (sum of isomers)	0.01
164	Propyzamide	0.01
165	Proquinazid	0.01
166	Prosulfocarb	0.01

Pestide no.	Pesticides	MRRL (mg/kg)
167	Prothioconazole (Prothioconazole-desthio) (sum of isomers)	0.01
168	Prothiofos	0.01
169	Pymetrozine	0.01
170	Pyraclostrobin	0.01
171	Pyridaben	0.01
172	**Pyridalyl**	**0.01**
173	Pyrimethanil	0.01
174	Pyriproxyfen	0.01
175	Quinoxyfen	0.01
176	**Spinetoram (XDE-175)**	**0.01**
177	Spinosad (sum of spinosyn A and spinosyn D, expr. as spinosad)	0.01
178	Spirodiclofen	0.01
179	Spiromesifen	0.01
180	Spirotetramat	0.01
181	Spirotetramat metabolite BYI08330 enol-glucoside	0.01
182	Spirotetramat metabolite BYI08330-enol	0.01
183	Spirotetramat metabolite BYI08330-ketohydroxy	0.01
184	Spirotetramat metabolite BYI08330-monohydroxy	0.01
185	Spiroxamine (sum of isomers)	0.01
186	**Sulfoxaflor (sum of isomers)**	**0.01**
187	Tau-Fluvalinate	0.01
188	Tebuconazole	0.01
189	Tebufenozide	0.01
190	Tebufenpyrad	0.01
191	Teflubenzuron	0.01
192	Tefluthrin	0.01
193	Terbuthylazine	0.01
194	Tetraconazole	0.01
195	Tetradifon	0.01
196	Thiabendazole	0.01
197	Thiacloprid	0.01
198	Thiamethoxam	0.01
199	Thiodicarb	0.01
200	Thiophanate-methyl	0.01
201	Tolclofos-methyl	0.01
202	Tolylfluanid	0.01
203	Triadimefon	0.01
204	Triadimenol (any proportion of constituent isomers)	0.01
205	Triazophos	0.005
206	Trichlorfon	0.01
207	Tricyclazole	0.01
208	Trifloxystrobin	0.01
209	**Triflumizole**	**0.01**
210	**Triflumizole metabolite (FM-6-1)**	**0.01**
211	Triflumuron	0.01
212	Trifluralin	0.01
213	Triticonazole	0.01
214	Vinclozolin (only parent compound)	0.01
215	Zoxamide	0.01

New pesticides this year

MRRL: Minimum Required Reporting Level
This list is based on Commission Implementing Regulation (EU) EU) 2020/585 of 27 April 2020
MRRLs are based on Regulation (EC) No. 396/2005, Regulation (EU) 2016/127 and on toxicity data of each compound.
Low MRRLs allow evaluation of pesticides at low concentration levels."

ANNEX B. List of laboratories that agreed to participate in EUPT-FV-SC05

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
Austria	AGES - Innsbruck (Austrian Agency for Health and Food Safety), Institute for Food Safety	Innsbruck	Yes
Belgium	Groen Agro Control	Delfgauw	Yes
Belgium	Eurofins Lab Zeeuws-Vlaanderen	Graauw	Yes
Belgium, Luxemburg, France	Primoris Belgium	Zwijnaarde	Yes
Bulgaria	Primoris Bulgaria AD	Plovdiv	Yes
Croatia	Sample Control d.o.o.	Zagreb-Lučko	Yes
Croatia	Bioinstitut Ltd.	Cakovec	Yes
Cyprus	Pesticide residues Lab of State General Laboratory of Cyprus	Nicosia	Yes
Estonia	Agricultural Research Centre, Laboratory for Feeds and Residues	Tallinn	Yes
Finland	Finnish Customs Laboratory	Espoo	Yes
France	SCL paris	Massy	Yes
Germany	Landeslabor Berlin Brandenburg	Berlin	Yes
Germany	LUFA Nord-West	Hameln	Yes
Germany	Niedersächsisches Landesamt für Verbraucherschutz und Lebensmittelsicherheit, Lebensmittelinstitut Oldenburg	Oldenburg	Yes
Germany	Landesamt für Verbraucherschutz Sachsen-Anhalt	Halle/S.	Yes
Germany	State Office for Agriculture, Food Safety and Fisheries	Rostock	Yes
Germany	CVUA Stuttgart	Fellbach	Yes
Germany	Intertek Food Services GmbH	Bremen	Yes
Germany	Eurofins - Dr.Specht Express GmbH	Hamburg	Yes
Germany	LGL Erlangen	Erlangen	Yes
Greece	GENERAL CHEMICAL STATE LABORATORY	ATHENS	Yes
Ireland	Pesticide Residues Laboratory, Food Chemistry Division, Dept of Agriculture, Food & Marine	Celbridge	Yes
Italy	ARPA FVG	Udine	Yes
Italy	ARPAL - U.O. Analisi Chimche e fisiche - Sett. Chimica Levante	La Spezia	Yes
Italy	Laboratorio di Prevenzione	Milan	Yes
Italy	Istituto zooprofilattico della Sicilia "A.Mirri"	Palermo	Yes
Italy	Arpae Emilia Romagna,	Ferrara	Yes
Italy	Laboratorio Sanita Pubblica	Firenze	Yes
Lithuania	National Food and Veterinary Risk Assessment Institute (NFVRAI)	Vilnius	Yes
Luxembourg	Laboratoire National de Santé du Luxembourg	Dudelange	Yes
Netherlands	Wageningen Food Safety Research	Wageningen	Yes
Norway	NIBIO, Pesticides and Natural Products Chemistry	Aas	Yes
Peru	Servicio Nacional de Sanidad Agraria (SENASA) -Unidad del Centro de Control de Insumos y Residuos Tóxicos (UCCIRT)	Lima	Yes
Peru	Bureau Veritas - Lab Lima	Lima	Yes

ANNEX B. List of laboratories that agreed to participate in EUPT-FV-SC05

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
Poland	Food Safety Laboratory, The National Institute of Horticultural Research	Skierniewice	Yes
Poland	Jars S.A.	Łajski	Yes
Poland	SGS Polska	Pszczyna	Yes
Poland	Hamilton- UO Technologia	Grójec	Yes
Poland	Intertek Poland Sp. z o.o.	Gostynin	Yes
Romania	Sanitary Veterinary and Food Safety Directorate, Pesticides Residues Laboratory	Bucharest	Yes
Romania	Fotometric Research Laboratory	Voluntari	Yes
Slovakia	Veterinary and Food Institute in Bratislava	Bratislava	Yes
Spain	EUROFINS ECOSUR, S.A.	Murcia	Yes
Spain	AINIA	Valencia	Yes
Spain	INSTITUTO TECNOLÓGICO DE CANARIAS, S. A. LABORATORIO DE RESIDUOS. DEPARTAMENTO DE ANÁLISIS AMBIENTAL	Agüimes	Yes
Spain	EUROFINS SICA AGRIQ, SLU	Almería	Yes
Spain	Labcolor-Coexphal - Spain, Almeria	La Mojonera, Almería	Yes
Spain	Laboratorio Químico Microbiologico, S.L	Murcia	Yes
Spain	Laboratori Agroalimentari	Cabrils	Yes
Spain	Laboratorio Analitico Bioclinico, SLU	Almeria	Yes
Spain	Laboratorio Agrario y Fitopatologico de Galicia	Abegondo. (A Coruña)	Yes
Spain	Laboratorio Regional de la CCAA de La Rioja	Logroño	Yes
Spain	Laboratorio SOIVRE Almería Dirección Provincial de Comercio de Almería	Almería	Yes
Spain	Laboratorio Agroambiental de Zaragoza (Gobierno de Aragón)	Zaragoza	Yes
Spain	Analytica Alimentaria GMBH sucursal España	Almería	Yes
Spain	Dolmar Innova (Laboratorio Dolmar)	Gimileo (La Rioja)	Yes
Spain	LABORATORIO QUÍMICO MICROBIOLÓGICO, S.L. (SEVILLE)	Alcalá de Guadaira (Seville)	Yes
Sweden	Eurofins Food and Feed Testning Sweden AB	Lidköping	Yes
United Kingdom	Fera Science Ltd	York	Yes
Uruguay	GACT/FARMACOGNOSIA	Montevideo	Yes