EURL-PROFICIENCY TEST-FV-SC07, 2023-2024

Pesticide Residues in ground green coffee beans

Final Report

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EURL-EUROPEAN UNION PROFICIENCY TEST SC07
FOR THE DETERMINATION OF PESTICIDES IN SPECIAL COMMODITIES USING MULTIRESIDUE METHODS
2023-2024

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-SC07 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

Forty-two laboratories agreed to participate in EUPT-FV-SC07.

The proficiency test was performed at the end of 2023 and beginning of 2024 using ground green coffee beans. Coffee 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 coffee beans sample.

The test item, 100 g of ground green coffee beans containing spiked pesticide residues, was shipped to participants on 20th November 2023. The deadline for results submission to the Organiser was 20th December 2023. The participants were asked to determine the residue levels of all the pesticides that they detected in the coffee beans and to report the concentrations in mg/kg. The participants were provided with a target pesticide list, which contained 212 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), chlorpyrifos methyl (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.

2. TEST ITEM

2.1 Preparation of the treated test item

Green coffee 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 coffee 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 coffee 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 100 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 green coffee.

The statistical evaluation was performed according to the International Harmonized Protocol published by IUPAC, ISO and AOAC [1]. The individual residues data from the homogeneity tests are given in **Appendix 1**. The results of the statistical analyses (for the evaluated compounds) are given in **Table 1**. All the pesticides evaluated in the coffee 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.

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

Pesticide	Mean Conc.	Ss^2	c	$Ss^2 < c$
Acetamiprid	0.070	2.66E-06	6.00E-05	Pass
Bifenthrin	0.067	4.14E-06	6.00E-05	Pass
Carbaryl	0.128	1.80E-05	2.20E-04	Pass
Carbofuran	0.412	0.00E+00	2.22E-03	Pass
Chlorpyrifos	0.045	2.52E-06	3.00E-05	Pass
Clothianidin	0,176	0.00E+00	5,70E-04	Pass
Cyproconazole	0.124	2.72E-05	1.90E-04	Pass
Dichlorvos	0.042	0.00E+00	4.00E-05	Pass
Dimethoate	0.103	1.48E-06	1.60E-04	Pass

Pesticide	Mean Conc.	Ss^2	c	$Ss^2 < c$
Flutriafol	0.171	1.45E-05	4.40E-04	Pass
Fluxapyroxad	0.056	2.08E-06	5.00E-05	Pass
Imidacloprid	0.277	0.00E+00	1.59E-03	Pass
Lufenuron	0.104	4.58E-05	2.00E-04	Pass
Methidathion	0.188	8.59E-05	6.60E-04	Pass
Profenofos	0.066	2.12E-06	6.00E-05	Pass
Thiamethoxam	0.094	0.00E+00	2.00E-04	Pass

S_s: Between-Sampling Standard Deviation

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).

The individual results for the evaluated compounds are given in **Table 2**. 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.

Table 2. Statistical test for analytical precision and to demonstrate results stability after the interval of time-elapse between the shipment of the test item and the deadline for reporting of results.

(mg/kg)	*Day 1*							*Day 2*							*(M2 – M1)*	*M2-M1 ≤ 0.3*σ*
	Sample 58_A	Sample 58_B	Sample 6_A	Sample 6_B	Sample 20_A	Sample 20_B	Mean 1	Sample 23_A	Sample 23_B	Sample 3_A	Sample 3_B	Sample 44_A	Sample 44_B	Mean 2		
Acetamiprid	0.070	0.071	0.071	0.069	0.074	0.067	0.070	0.074	0.067	0.073	0.073	0.074	0.071	0.072	0.002	Pass
Bifenthrin	0.071	0.070	0.071	0.066	0.066	0.070	0.069	0.063	0.067	0.070	0.067	0.069	0.067	0.067	-0.002	Pass
Carbaryl	0.136	0.136	0.135	0.138	0.124	0.131	0.133	0.135	0.128	0.126	0.128	0.138	0.126	0.130	-0.003	Pass
Carbofuran	0.419	0.392	0.423	0.390	0.409	0.397	0.405	0.408	0.397	0.405	0.416	0.397	0.404	0.405	-0.001	Pass
Chlorpyrifos	0.048	0.046	0.050	0.041	0.048	0.051	0.047	0.050	0.043	0.050	0.051	0.050	0.047	0.049	0.001	Pass
Clothianidin	0.172	0.162	0.184	0.182	0.183	0.173	0.176	0.161	0.167	0.171	0.173	0.194	0.172	0.173	-0.003	Pass
Cyproconazole	0.126	0.129	0.129	0.122	0.110	0.127	0.124	0.130	0.114	0.120	0.132	0.115	0.111	0.120	-0.004	Pass
Dichlorvos	0.043	0.046	0.040	0.039	0.035	0.045	0.041	0.044	0.043	0.036	0.046	0.045	0.036	0.042	0.000	Pass

(mg/kg)	Day 1							Day 2							(M2 – M1)	M2-M1 ≤ 0.3*σ
	Sample 58_A	Sample 58_B	Sample 6_A	Sample 6_B	Sample 20_A	Sample 20_B	Mean 1	Sample 23_A	Sample 23_B	Sample 3_A	Sample 3_B	Sample 44_A	Sample 44_B	Mean 2		
Dimethoate	0.110	0.101	0.103	0.109	0.099	0.098	0.103	0.107	0.102	0.101	0.097	0.114	0.098	0.103	0.000	Pass
Flutriafol	0.169	0.157	0.183	0.177	0.158	0.159	0.167	0.181	0.170	0.162	0.176	0.185	0.175	0.175	0.008	Pass
Fluxapyroxad	0.061	0.062	0.056	0.056	0.049	0.062	0.058	0.058	0.057	0.063	0.061	0.056	0.050	0.058	0.000	Pass
Imidacloprid	0.268	0.292	0.277	0.286	0.292	0.286	0.284	0.284	0.290	0.275	0.265	0.292	0.268	0.279	-0.005	Pass
Lufenuron	0.107	0.101	0.112	0.098	0.098	0.108	0.104	0.108	0.109	0.101	0.102	0.103	0.105	0.105	0.001	Pass
Methidathion	0.183	0.203	0.204	0.194	0.175	0.189	0.191	0.178	0.187	0.195	0.183	0.203	0.189	0.189	-0.002	Pass
Profenofos	0.068	0.064	0.068	0.063	0.061	0.062	0.064	0.062	0.063	0.061	0.071	0.070	0.068	0.066	0.002	Pass
Thiamethoxam	0.105	0.090	0.086	0.090	0.081	0.087	0.090	0.092	0.082	0.093	0.093	0.100	0.103	0.094	0.004	Pass

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 (**Table 3**).

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

(mg/kg)	Day 1							Day 3							(M2 – M1)	M3-M1 ≤ 0.3*σ
	Sample 58_A	Sample 58_B	Sample 6_A	Sample 6_B	Sample 20_A	Sample 20_B	Mean 1	Sample 59_A	Sample 59_B	Sample 15_A	Sample 15_B	Sample 41_A	Sample 41_B	Mean 2		
Acetamiprid	0.070	0.071	0.071	0.069	0.074	0.067	0.070	0.075	0.073	0.075	0.067	0.069	0.070	0.072	0.001	Pass
Bifenthrin	0.071	0.070	0.071	0.066	0.066	0.070	0.069	0.068	0.067	0.064	0.068	0.069	0.070	0.068	-0.001	Pass
Carbaryl	0.136	0.136	0.135	0.138	0.124	0.131	0.133	0.132	0.138	0.137	0.124	0.135	0.131	0.133	-0.001	Pass
Carbofuran	0.419	0.392	0.423	0.390	0.409	0.397	0.405	0.396	0.409	0.400	0.402	0.419	0.403	0.405	0.000	Pass
Chlorpyrifos	0.048	0.046	0.050	0.041	0.048	0.051	0.047	0.044	0.047	0.045	0.046	0.040	0.045	0.045	-0.003	Pass
Clothianidin	0.172	0.162	0.184	0.182	0.183	0.173	0.176	0.167	0.161	0.188	0.162	0.183	0.185	0.174	-0.002	Pass
Cyproconazole	0.126	0.129	0.129	0.122	0.110	0.127	0.124	0.117	0.110	0.118	0.128	0.124	0.111	0.118	-0.006	Pass
Dichlorvos	0.043	0.046	0.040	0.039	0.035	0.045	0.041	0.047	0.043	0.038	0.039	0.044	0.040	0.042	0.001	Pass
Dimethoate	0.110	0.101	0.103	0.109	0.099	0.098	0.103	0.102	0.103	0.109	0.106	0.091	0.087	0.100	-0.004	Pass
Flutriafol	0.169	0.157	0.183	0.177	0.158	0.159	0.167	0.169	0.182	0.156	0.157	0.160	0.172	0.166	-0.001	Pass
Fluxapyroxad	0.061	0.062	0.056	0.056	0.049	0.062	0.058	0.059	0.050	0.057	0.059	0.051	0.058	0.056	-0.002	Pass
Imidacloprid	0.268	0.292	0.277	0.286	0.292	0.286	0.284	0.276	0.266	0.270	0.271	0.283	0.283	0.275	-0.009	Pass
Lufenuron	0.107	0.101	0.112	0.098	0.098	0.108	0.104	0.113	0.102	0.098	0.107	0.111	0.107	0.106	0.002	Pass
Methidathion	0.183	0.203	0.204	0.194	0.175	0.189	0.191	0.182	0.185	0.207	0.204	0.175	0.202	0.193	0.001	Pass
Profenofos	0.068	0.064	0.068	0.063	0.061	0.062	0.064	0.066	0.066	0.065	0.063	0.069	0.062	0.065	0.001	Pass
Thiamethoxam	0.105	0.090	0.086	0.090	0.081	0.087	0.090	0.082	0.105	0.086	0.105	0.091	0.081	0.092	0.002	Pass

2.4 Distribution of test items and protocol to participants

One bottle of frozen treated test item was shipped to each participant. The test items were sent out on 20th November 2023.

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 SC07. The Target Pesticide List was the same as for EUPT-FV25, 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.

All false negatives have been assigned a z score of -4.0. However, these z scores have not been 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'.

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

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

All results reported by the participants are given in **Appendix 2**.

Forty-two 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. Three laboratories from non-EU/EFTA countries (Serbia, Tanzania and United Kingdom) participated in EUPT-SC07. Their results have not been included for the calculation of the assigned value.

Sixteen pesticides from the pesticide target list were used to spike the sample and were present in the test item at concentrations above the MRRL.
A summary of the reported results for both the pesticides evaluated and those informative can be seen below in **Table 4**.

Table 4. 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 39)
Acetamiprid	38	0	1	97
Bifenthrin	35	3	1	90
Carbaryl	38	1	0	97
Carbofuran	37	1	1	95
Chlorpyrifos	37	2	0	95
Clothianidin	37	0	2	95
Cyproconazole	39	0	0	100
Dichlorvos	29	9	1	74
Dimethoate	38	1	0	97
Flutriafol	39	0	0	100
Fluxapyroxad	31	4	4	79
Imidacloprid	38	0	1	97
Lufenuron	31	1	7	79
Methidathion	37	2	0	95
Profenofos	36	2	1	92
Thiamethoxam	36	2	1	92

[a] The percentage of Reported Results comes from 39 laboratories. It does not take into account results reported by laboratories in Serbia, Tanzania and United Kingdom.

4.1.1 False positives

Three laboratories reported fourteen results for additional pesticides that were not present in the test item. These pesticides and the residue levels reported are presented in **Table 5**, 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 5. 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
Lab002	Fenazaquin	0.01	0.029	GC-MS (Q)
Lab029	Pirimicarb	0.01	0.022	LC-MS (QQQ)
Lab039	Chlorothalonil	0.0563	0.0734	GC-MS (Q)
Lab039	Cypermethrin	0.1599	0.1735	GC-MS (Q)
Lab039	Fipronil	0.287	0.321	GC-MS (Q)
Lab039	Hexaconazole	0.2339	0.2512	GC-MS (Q)
Lab039	Lambda-Cyhalothrin	0.0967	0.103	GC-MS (Q)

Lab Code	Pesticide	Reporting level (mg/kg)	Concentration (mg/kg)	Determination technique
Lab039	Malathion	0.0781	0.0904	GC-MS (Q)
Lab039	Metalaxyl	0.0831	0.123	GC-MS (Q)
Lab039	Methoxyfenozide	0.0754	0.0812	GC-MS (Q)
Lab039	Pendimethalin	0.1205	0.1297	GC-MS (Q)
Lab039	Tebuconazole	0.1033	0.1231	GC-MS (Q)
Lab039	Triadimefon	0.0344	0.0876	GC-MS (Q)
Lab039	Triadimenol	0.0602	0.0829	GC-MS (Q)

4.1.2 False negatives

Table 6 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.

Table 6. Laboratories that failed to report pesticides that were present in the treated test item.

Laboratory	Acetamiprid	Bifenthrin	Carbaryl	Carbofuran	Chlorpyrifos	Clothianidin	Cyproconazole	Dichlorvos	Dimethoate	Flutriafol	Fluxapyroxad	Imidacloprid	Lufenuron	Methidathion	Profenofos	Thiamethoxam
Lab003											ND					
Lab007													ND			
Lab010				ND									ND			
Lab014													ND			
Lab015		ND													ND	
Lab021											ND					
Lab025											ND					
Lab029													ND			
Lab030								ND			ND		ND			
Lab035						ND							ND			
Lab036	ND					ND						ND	ND			ND
Lab039	ND	ND	ND	ND		ND	ND	ND		ND	ND	ND	ND	ND		ND
Lab043													ND			

ND: Not detected

4.2 Assigned values and target standard deviations

In a first evaluation of the assigned values, they were calculated as the robust mean values of all the results reported by EU and EFTA laboratories. However, a high number of evaluated compounds (nine) presented a robust standard deviation (CV*) above 25 %. The organisers conducted internal studies and concluded that if water was not added to the coffee sample prior to extraction, the recoveries of the more polar compounds were low. in view of the results, the

Scientific Committee decided that for the calculation of the assigned value, results obtained without the addition of water prior to extraction should be removed. Therefore, the assigned values are based on the robust mean values calculated using only the results of EU and EFTA laboratories that added water to the sample prior to extraction. The assigned values for the 16 pesticides and their uncertainties are presented in **Table 7**.

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 7**.

Table 7. Robust mean values, uncertainty and % RSDs for all pesticides evaluated.

Pesticides	n	Robust mean (mg/kg)	CV(%)	MRRL (mg/kg)	Uncertainty (mg/kg)
Acetamiprid	33	0.071	22.5	0.01	0.003
Bifenthrin	30	0.066	23.6	0.01	0.004
Carbaryl	33	0.131	25.4	0.005	0.007
Carbofuran	32	0.416	32.7	0.005	0.030
Chlorpyrifos	32	0.045	17.0	0.005	0.002
Clothianidin	32	0.177	31.3	0.01	0.012
Cyproconazole	34	0.121	16.1	0.01	0.004
Dichlorvos	26	0.041	27.2	0.005	0.003
Dimethoate	34	0.103	34.5	0.003	0.008
Flutriafol	34	0.171	23.4	0.01	0.009
Fluxapyroxad	27	0.056	22.8	0.01	0.003
Imidacloprid	33	0.277	26.8	0.01	0.016
Lufenuron	26	0.106	22.5	0.01	0.006
Methidathion	32	0.191	22.8	0.01	0.010
Profenofos	31	0.066	21.0	0.01	0.003
Thiamethoxam	31	0.091	23.7	0.01	0.005

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 8**, where the classification of z scores reported by EU/EFTA laboratories is shown.

Table 8. Classification of z scores for the pesticides evaluated (only EU/EFTA participants)

Pesticide	Robust Mean (mg/kg)	Acceptable %	Questionable %	Unacceptable %
Acetamiprid	0.071	84	11	5
Bifenthrin	0.066	92	0	8
Carbaryl	0.131	82	10	8
Carbofuran	0.416	84	3	13
Chlorpyrifos	0.045	85	5	10
Clothianidin	0.177	78	11	11
Cyproconazole	0.121	85	10	5
Dichlorvos	0.041	61	11	29
Dimethoate	0.103	77	15	8
Flutriafol	0.171	92	3	5
Fluxapyroxad	0.056	83	3	14
Imidacloprid	0.277	76	8	16
Lufenuron	0.106	81	13	6
Methidathion	0.191	92	3	5
Profenofos	0.066	87	5	8
Thiamethoxam	0.091	71	11	18

z scores for false negative results have been assigned the fixed value of -4.0.
The high percentage of questionable and unacceptable z scores is related, in most of the cases, to the non-addition of water prior to extraction.

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.

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 (191), 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**.

Twenty five of the 39 EU and EFTA laboratories were classified into Category A (64 %).

From the AZ^2, 68 % were classed as 'good', 12 % as 'satisfactory' and 20 % as 'unsatisfactory' (Only considering EU and EFTA laboratories). Again, this unusual high number of unsatisfactory combined z scores is related to those results obtained without adding water before extraction.

Of the 14 EU and EFTA laboratories in Category B, one would have been classified into Category A if not for a false positive result.

Table 9 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 9. 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
Lab004 Θ	14	100.0	3.7	Unsatisfactory
Lab005	16	100.0	0.2	Good
Lab008	16	100.0	0.7	Good
Lab009	16	100.0	0.4	Good
Lab012	16	99.5	1.1	Good
Lab013 Θ	14	93.9	3.8	Unsatisfactory
Lab014	15	90.1	0.3	Good
Lab015	14	96.2	0.4	Good
Lab016	16	98.1	0.1	Good
Lab017	16	100.0	2.3	Satisfactory
Lab018	16	100.0	0.7	Good
Lab019	16	97.2	2.7	Satisfactory
Lab020	16	97.6	0.4	Good
Lab021 Θ	14	99.5	3.3	Unsatisfactory
Lab022	16	98.1	5.3	Unsatisfactory
Lab023	16	99.5	0.2	Good
Lab024	16	99.5	0.2	Good
Lab025	15	91.5	1.0	Good
Lab026	16	91.5	1.4	Good
Lab031	16	99.5	3.1	Unsatisfactory
Lab034 Θ	15	100.0	1.5	Good
Lab037	16	100.0	0.1	Good
Lab040 Θ	14	96.2	2.2	Satisfactory
Lab041	16	100.0	4.0	Unsatisfactory
Lab042	16	99.5	1.4	Good
Lab043	15	95.8	6.1	Unsatisfactory
Lab044	16	99.5	0.3	Good

Θ Laboratories reporting a false negative result.

Table 10 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 ⊖ 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 10. 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)
Lab001 ⊖	13	81	100	16	4
Lab002 ⊖ +	14	88	96	16	1
Lab003 ⊖	11	69	75	15	6
Lab006	16	100	79	16	16
Lab007 ⊖	14	88	82	15	14
Lab010 ⊖	10	63	74	14	7
Lab011	16	100	8	16	16
Lab028	16	100	81	16	16
Lab029 ⊖ +	14	88	77	15	9
Lab030	13	81	76	13	13
Lab032 ⊖	15	94	85	16	12
Lab033 ⊖	13	81	100	16	6
Lab035 ⊖	11	69	70	14	10
Lab036	11	69	52	11	11
Lab039 +	3	19	7	3	1

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

5. CONCLUSIONS

Forty-two laboratories agreed to participate in EUPT-FV-SC07. Three of them did not belong to EU nor EFTA countries, so their results were not considered for the estimation of the assigned value.

Sixteen pesticides were present in EUPT-FV-SC07 test item at concentrations above the MRRL.

Of a total number of 624 possible determinations from EU/EFTA laboratories (39 laboratories by 16 evaluated pesticides), 92.3 % were reported, 3.2 % were not analysed and 4.5 % were not detected (false negative results).

64 % of the EU and EFTA laboratories that submitted results were classified into Category A. Of them, 68 % were classed as 'good', 12 % as 'satisfactory' and 20 % as 'unsatisfactory'.

The addition of water prior to extraction proved to be essential for the recovery of polar compounds.

Participation in this EUPT-FV-SC07 involved laboratories from 18 EU Member States and 2 EFTA countries (Iceland and Norway). 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. Three non-European laboratories (from Serbia, Tanzania and United Kingdom) participated in EUPT-FV-SC07.

6. REFERENCES

1. M. Thompson, S. L. R. Ellison, and R. Wood. The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories. Pure Appl. Chem., 2006, 78 (1), 145–196.

2. ISO 13528:2015. Statistical methods for use in proficiency testing by interlaboratory comparison. International Organization for Standardization.

3. P. Medina-Pastor, C. Rodríguez-Torreblanca, A. Andersson, A. R. Fernández-Alba. European Commission proficiency tests for pesticide residues in fruits and vegetables. Trends in Analytical Chemistry, 2010, 29 (1), 70-83.

4. P. Medina Pastor, A. Valverde, T. Pihlström, S. Masselter, M. Gamón, M. Mezcua, C. Rodríguez Torreblanca, A. R. Fernández-Alba. Comparative Study of the Main Top-down Approaches for the Estimation of Measurement Uncertainty in Multiresidue Analysis of Pesticides in Fruits and Vegetables. J. Agric. Food Chem., 2011, 59 (14), 7609-7619.

5. P. Medina-Pastor, M. Mezcua, C. Rodríguez-Torreblanca, A. R. Fernández-Alba. Laboratory assessment by combined z-score values in proficiency tests: experience gained through the European Union proficiency tests for pesticide residues in fruits and vegetables. Anal. Bioanal. Chem., 2010, 397, 3061–3070.

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Acetamiprid (mg/kg)		**Bifenthrin (mg/kg)**		**Carbaryl (mg/kg)**		**Carbofuran (mg/kg)**	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.074	0.075	0.062	0.071	0.122	0.128	0.404	0.387
0.069	0.068	0.066	0.070	0.119	0.125	0.398	0.412
0.068	0.070	0.070	0.068	0.128	0.126	0.432	0.428
0.069	0.073	0.063	0.060	0.138	0.123	0.407	0.424
0.069	0.071	0.070	0.061	0.136	0.124	0.393	0.446
0.066	0.069	0.072	0.072	0.140	0.137	0.415	0.442
0.066	0.068	0.072	0.068	0.131	0.118	0.442	0.390
0.071	0.072	0.068	0.063	0.133	0.126	0.404	0.406
0.072	0.066	0.063	0.065	0.140	0.134	0.385	0.408
0.072	0.069	0.064	0.064	0.134	0.124	0.426	0.400

Chlorpyrifos (mg/kg)		**Clothianidin (mg/kg)**		**Cyproconazole (mg/kg)**		**Dichlorvos (mg/kg)**	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.045	0.046	0.182	0.165	0.124	0.133	0.039	0.045
0.042	0.045	0.202	0.152	0.134	0.123	0.046	0.042
0.048	0.040	0.162	0.160	0.117	0.132	0.042	0.042
0.050	0.049	0.172	0.195	0.117	0.120	0.045	0.037
0.050	0.043	0.164	0.174	0.112	0.119	0.043	0.038
0.050	0.047	0.153	0.168	0.116	0.111	0.036	0.044
0.042	0.042	0.176	0.180	0.134	0.131	0.047	0.041
0.046	0.047	0.199	0.177	0.127	0.130	0.040	0.045
0.043	0.042	0.186	0.192	0.119	0.120	0.035	0.045
0.043	0.046	0.163	0.189	0.124	0.127	0.047	0.035

Dimethoate (mg/kg)		**Flutriafol (mg/kg)**		**Fluxapyroxad (mg/kg)**		**Imidacloprid (mg/kg)**	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.093	0.098	0.156	0.169	0.054	0.057	0.258	0.295
0.110	0.094	0.158	0.165	0.054	0.053	0.279	0.246
0.100	0.109	0.153	0.186	0.057	0.054	0.279	0.289
0.107	0.101	0.172	0.187	0.056	0.050	0.274	0.262
0.109	0.098	0.185	0.190	0.062	0.057	0.305	0.258
0.112	0.100	0.184	0.163	0.053	0.052	0.290	0.279
0.108	0.112	0.160	0.177	0.062	0.054	0.307	0.250
0.113	0.099	0.176	0.166	0.049	0.062	0.282	0.272
0.113	0.102	0.157	0.160	0.050	0.053	0.305	0.243
0.093	0.094	0.184	0.170	0.060	0.063	0.252	0.309

APPENDIX 1. Homogeneity data.

Lufenuron (mg/kg)		Methidathion (mg/kg)		Profenofos (mg/kg)		Thiamethoxam (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 1	Replicate 2	Replicate 2	Replicate 1	Replicate 2
0.097	0.116	0.211	0.226	0.071	0.064	0.094	0.099
0.098	0.088	0.200	0.199	0.066	0.059	0.102	0.101
0.120	0.107	0.177	0.197	0.069	0.069	0.086	0.094
0.095	0.097	0.198	0.165	0.060	0.064	0.081	0.109
0.098	0.105	0.166	0.213	0.070	0.073	0.098	0.105
0.104	0.116	0.158	0.192	0.065	0.060	0.100	0.086
0.086	0.089	0.196	0.200	0.065	0.070	0.087	0.077
0.087	0.117	0.173	0.189	0.066	0.066	0.086	0.084
0.110	0.114	0.169	0.158	0.067	0.065	0.111	0.090
0.117	0.115	0.184	0.194	0.073	0.062	0.102	0.079

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	Acetamiprid	z score (FFP-RSD 25 %)	Bifenthrin	z score (FFP-RSD 25 %)	Carbaryl	z score (FFP-RSD 25 %)	Carbofuran	z score (FFP-RSD 25 %)	Chlorpyrifos	z score (FFP-RSD 25 %)	Clothianidin	z score (FFP-RSD 25 %)	Cyproconazole	z score (FFP-RSD 25 %)
MRRL (mg/kg)	**0.01**		**0.01**		**0.005**		**0.005**		**0.005**		**0.01**		**0.01**	
Robust mean (mg/kg)	**0.071**		**0.066**		**0.131**		**0.416**		**0.045**		**0.177**		**0.121**	
Lab001	0.169	5.0	0.098	1.9	ND	-4.0	0.167	-2.3	0.099	4.9	0.455	5.0	0.198	2.7
Lab002	0.011	-3.3	0.067	0.0	0.031	-3.0	0.074	-3.2	0.017	-2.5	0.022	-3.5	0.043	-2.6
Lab003	0.053	-0.9	0.07	0.2	0.06	-2.1	0.342	-0.5	ND	-4.0	0.088	-1.8	0.207	3.0
Lab004	0.057	-0.6	ND	-4.0	0.104	-0.7	0.293	-1.0	0.04	-0.4	0.026	-3.4	0.1	-0.6
Lab005	0.072	0.3	0.076	0.6	0.139	0.5	0.515	1.2	0.047	0.2	0.195	0.8	0.133	0.5
Lab006	0.057	-0.6	0.058	-0.5	0.161	1.2	0.208	-1.9	0.044	0.0	0.177	0.4	0.102	-0.6
Lab007	0.061	-0.4	0.047	-1.2	0.094	-1.0	0.23	-1.7	0.024	-1.8	0.15	-0.3	0.1	-0.6
Lab008	0.07	0.1	0.077	0.6	0.157	1.0	0.593	2.0	0.056	1.0	0.173	0.3	0.13	0.4
Lab009	0.075	0.4	0.071	0.3	0.136	0.4	0.513	1.2	0.046	0.1	0.205	1.0	0.137	0.6
Lab010	0.061	-0.4	ND	-4.0	0.127	0.1	NA		0.035	-0.9	0.108	-1.3	0.043	-2.6
Lab011	0.07	0.1	0.065	-0.1	0.12	-0.1	0.45	0.6	0.045	0.0	0.17	0.2	0.1	-0.6
Lab012	0.086	1.1	0.07	0.2	0.152	0.9	0.59	2.0	0.05	0.5	0.201	0.9	0.137	0.6
Lab013	0.078	0.6	0.032	-2.1	0.207	2.7	0.344	-0.5	0.018	-2.4	0.172	0.2	0.09	-1.0
Lab014	0.0645	-0.2	0.0765	0.6	0.133	0.3	0.357	-0.4	0.0415	-0.3	0.154	-0.2	0.11	-0.3
Lab015	0.056	-0.7	NA		0.101	-0.8	0.381	-0.1	0.05	0.5	0.114	-1.2	0.126	0.2
Lab016	0.067	0.0	0.054	-0.7	0.138	0.4	0.421	0.3	0.04	-0.4	0.187	0.6	0.129	0.3
Lab017	0.054	-0.8	0.056	-0.6	0.098	-0.9	0.409	0.1	0.044	0.0	0.106	-1.4	0.11	-0.3
Lab018	0.059	-0.5	0.056	-0.6	0.093	-1.0	0.339	-0.6	0.038	-0.6	0.157	-0.1	0.113	-0.2
Lab019	0.0242	-2.6	0.0667	0.0	0.0907	-1.1	0.314	-0.8	0.0488	0.4	0.0594	-2.5	0.106	-0.4
Lab020	0.054	-0.8	0.0437	-1.4	0.111	-0.4	0.413	0.2	0.0406	-0.3	0.155	-0.2	0.112	-0.2
Lab021	0.027	-2.4	0.0713	0.3	0.0805	-1.4	0.295	-1.0	0.0452	0.1	0.0843	-1.9	0.106	-0.4
Lab022	0.0941	1.6	0.0827	1.0	0.247	3.9	0.824	4.4	0.0419	-0.2	0.212	1.2	0.115	-0.1
Lab023	0.063	-0.3	0.049	-1.0	0.11	-0.5	0.361	-0.3	0.043	-0.1	0.159	-0.1	0.141	0.7
Lab024	0.0638	-0.2	0.0779	0.7	0.123	0.0	0.391	0.0	0.0552	1.0	0.173	0.3	0.112	-0.2
Lab025	0.106	2.3	0.0513	-0.9	0.117	-0.2	0.359	-0.4	0.0412	-0.3	0.226	1.6	0.0995	-0.7
Lab026	0.087	1.1	0.08	0.8	0.152	0.9	0.528	1.4	0.052	0.7	0.221	1.4	0.138	0.6
Lab028	0.073	0.3	0.087	1.2	0.129	0.1	0.46	0.7	0.048	0.3	0.183	0.5	0.127	0.3
Lab029	0.092	1.4	0.065	-0.1	0.227	3.3	0.812	4.2	0.037	-0.7	0.233	1.7	0.121	0.1
Lab030	0.085	1.0	0.084	1.1	0.159	1.1	0.337	-0.6	0.05	0.5	0.205	1.0	0.117	-0.1
Lab031	0.073	0.3	0.054	-0.7	0.126	0.0	0.452	0.6	0.047	0.2	0.26	2.4	0.288	5.0
Lab032	0.061	-0.4	0.04	-1.6	0.098	-0.9	0.048	-3.5	0.02	-2.2	0.12	-1.1	0.11	-0.3
Lab033	0.05	-1.0	ND	-4.0	0.07	-1.8	ND	-4.0	0.16	5.0	0.05	-2.8	0.03	-3.0
Lab034	0.078	0.6	0.075	0.5	0.143	0.6	0.422	0.3	0.057	1.1	0.205	1.0	0.135	0.5
Lab035	0.055	-0.7	0.091	1.5	0.205	2.6	0.27	-1.3	ND	-4.0	NA		0.097	-0.7

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

Lab Code	Acetamiprid	z score (FFP-RSD 25 %)	Bifenthrin	z score (FFP-RSD 25 %)	Carbaryl	z score (FFP-RSD 25 %)	Carbofuran	z score (FFP-RSD 25 %)	Chlorpyrifos	z score (FFP-RSD 25 %)	Clothianidin	z score (FFP-RSD 25 %)	Cyproconazole	z score (FFP-RSD 25 %)
MRRL (mg/kg)	**0.01**		**0.01**		**0.005**		**0.005**		**0.005**		**0.01**		**0.01**	
Robust mean (mg/kg)	**0.071**		**0.066**		**0.131**		**0.416**		**0.045**		**0.177**		**0.121**	
Lab036	NA		0.059	-0.4	0.125	0.0	0.466	0.7	0.05	0.5	NA		0.124	0.2
Lab037	0.071	0.2	0.0647	-0.1	0.134	0.3	0.475	0.8	0.0457	0.1	0.174	0.3	0.131	0.4
Lab039	NA		NA		NA		NA		0.123	5.0	NA		NA	
Lab040	0.069	0.1	0.061	-0.3	0.123	0.0	0.363	-0.3	0.042	-0.2	0.186	0.6	0.125	0.2
Lab041	0.022	-2.7	0.098	1.9	0.073	-1.7	0.225	-1.7	0.029	-1.4	0.05	-2.8	0.094	-0.8
Lab042	0.074	0.4	0.058	-0.5	0.168	1.4	0.5	1.1	0.043	-0.1	0.212	1.2	0.148	1.0
Lab043	0.113	2.7	0.065	-0.1	0.207	2.7	0.572	1.8	0.04	-0.4	0.457	5.0	0.128	0.3
Lab044	0.083	0.9	0.048	-1.1	0.119	-0.2	0.343	-0.5	0.041	-0.3	0.154	-0.2	0.12	0.0

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

Lab Code	Dichlorvos	z score (FFP-RSD 25 %)	Dimethoate	z score (FFP-RSD 25 %)	Flutriafol	z score (FFP-RSD 25 %)	Fluxapyroxad	z score (FFP-RSD 25 %)	Imidacloprid	z score (FFP-RSD 25 %)	Lufenuron	z score (FFP-RSD 25 %)	Methidathion	z score (FFP-RSD 25 %)
MRRL (mg/kg)	**0.005**		**0.003**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Robust mean (mg/kg)	**0.041**		**0.103**		**0.171**		**0.056**		**0.277**		**0.106**		**0.191**	
Lab001	0.084	4.7	0.288	5.0	0.231	1.7	ND	-4.0	0.625	5.0	ND	-4.0	0.282	2.1
Lab002	ND	-4.0	ND	-4.0	0.038	-3.1	0.02	-2.5	0.035	-3.5	0.041	-2.4	0.065	-2.6
Lab003	ND	-4.0	0.037	-2.5	0.334	4.2	NA		0.381	1.9	0.212	4.3	0.21	0.5
Lab004	0.018	-2.1	0.038	-2.4	0.148	-0.4	0.047	-0.5	0.179	-1.2	0.093	-0.3	ND	-4.0
Lab005	0.041	0.3	0.111	0.6	0.18	0.4	0.053	-0.1	0.262	0.0	0.11	0.3	0.19	0.1
Lab006	0.05	1.2	0.091	-0.3	0.134	-0.7	0.053	-0.1	0.252	-0.1	0.13	1.1	0.182	-0.1
Lab007	ND	-4.0	0.075	-0.9	0.16	-0.1	0.046	-0.6	0.29	0.5	NA		0.14	-1.0
Lab008	0.043	0.5	0.11	0.5	0.19	0.7	0.06	0.5	0.25	-0.2	0.117	0.6	0.224	0.8
Lab009	0.041	0.3	0.114	0.7	0.188	0.6	0.054	0.0	0.285	0.4	0.107	0.2	0.208	0.5
Lab010	ND	-4.0	0.213	4.8	0.136	-0.7	0.042	-0.9	0.239	-0.3	NA		ND	-4.0
Lab011	0.035	-0.4	0.11	0.5	0.145	-0.4	0.045	-0.6	0.24	-0.3	0.065	-1.4	0.155	-0.6
Lab012	0.028	-1.1	0.125	1.1	0.21	1.2	0.079	1.9	0.303	0.7	0.13	1.1	0.189	0.1
Lab013	ND	-4.0	0.098	0.0	0.217	1.3	0.047	-0.5	0.299	0.6	0.081	-0.8	ND	-4.0
Lab014	0.0381	0.0	0.1	0.1	0.17	0.2	0.0693	1.2	0.232	-0.4	NA		0.174	-0.2
Lab015	0.03	-0.9	0.091	-0.3	0.151	-0.3	0.049	-0.3	0.192	-1.0	0.083	-0.7	0.161	-0.5
Lab016	0.038	-0.1	0.11	0.5	0.178	0.4	0.052	-0.1	0.257	0.0	0.107	0.2	0.192	0.2
Lab017	0.042	0.4	0.098	0.0	0.13	-0.8	0.071	1.3	0.556	4.5	0.166	2.5	0.126	-1.3

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

Lab Code	Dichlorvos	z score (FFP-RSD 25 %)	Dimethoate	z score (FFP-RSD 25 %)	Flutriafol	z score (FFP-RSD 25 %)	Fluxapyroxad	z score (FFP-RSD 25 %)	Imidacloprid	z score (FFP-RSD 25 %)	Lufenuron	z score (FFP-RSD 25 %)	Methidathion	z score (FFP-RSD 25 %)
MRRL (mg/kg)	**0.005**		**0.003**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Robust mean (mg/kg)	**0.041**		**0.103**		**0.171**		**0.056**		**0.277**		**0.106**		**0.191**	
Lab018	0.018	-2.1	0.081	-0.7	0.159	-0.1	0.04	-1.0	0.2	-0.9	0.09	-0.5	0.149	-0.8
Lab019	0.0189	-2.0	0.0486	-2.0	0.106	-1.4	0.0476	-0.5	0.0788	-2.8	0.105	0.1	0.152	-0.7
Lab020	0.0337	-0.5	0.08	-0.7	0.156	-0.2	0.051	-0.2	0.222	-0.6	0.0786	-0.9	0.167	-0.4
Lab021	ND	-4.0	0.0401	-2.4	0.111	-1.3	NA		0.119	-2.2	0.085	-0.7	0.153	-0.7
Lab022	0.106	5.0	0.145	2.0	0.23	1.7	0.0825	2.1	0.356	1.5	0.134	1.3	0.253	1.5
Lab023	0.03	-0.9	0.102	0.2	0.138	-0.6	0.056	0.2	0.259	0.0	0.108	0.2	0.156	-0.6
Lab024	0.04	0.2	0.0913	-0.2	0.152	-0.3	0.0511	-0.2	0.227	-0.5	0.0993	-0.1	0.191	0.1
Lab025	0.04	0.2	0.149	2.1	0.119	-1.1	NA		0.258	0.0	0.0912	-0.4	0.183	0.0
Lab026	0.06	2.2	0.123	1.1	0.212	1.2	0.063	0.7	0.301	0.6	0.114	0.5	0.261	1.6
Lab028	0.034	-0.5	0.107	0.4	0.175	0.3	0.055	0.1	0.245	-0.2	0.112	0.4	0.214	0.6
Lab029	0.043	0.5	0.16	2.6	0.191	0.7	ND	-4.0	0.531	4.2	NA		0.177	-0.2
Lab030	NA		0.092	-0.2	0.145	-0.4	NA		0.311	0.8	NA		0.234	1.1
Lab031	0.034	-0.5	0.063	-1.4	0.256	2.3	0.047	-0.5	0.353	1.4	0.164	2.4	0.154	-0.7
Lab032	ND	-4.0	0.062	-1.5	0.121	-1.0	0.043	-0.8	0.299	0.6	0.055	-1.8	0.14	-1.0
Lab033	ND	-4.0	0.06	-1.5	0.075	-2.2	0.12	4.9	0.06	-3.1	0.1	-0.1	0.15	-0.8
Lab034	0.057	1.9	0.117	0.8	0.178	0.4	0.054	0.0	0.281	0.3	0.1	-0.1	0.199	0.3
Lab035	ND	-4.0	0.054	-1.8	0.125	-0.9	ND	-4.0	0.16	-1.5	NA		0.133	-1.1
Lab036	0.031	-0.8	0.106	0.4	0.18	0.4	0.047	-0.5	NA		NA		0.228	0.9
Lab037	0.0447	0.6	0.104	0.3	0.174	0.3	0.0513	-0.2	0.26	0.0	0.0923	-0.4	0.186	0.0
Lab039	NA		0.0678	-1.2	NA		NA		NA		NA		NA	
Lab040	ND	-4.0	0.102	0.2	0.169	0.2	ND	-4.0	0.298	0.6	0.103	0.0	0.234	1.1
Lab041	0.018	-2.1	0.038	-2.4	0.087	-1.9	0.038	-1.2	0.075	-2.8	0.051	-2.0	0.158	-0.6
Lab042	0.048	1.0	0.116	0.8	0.222	1.5	0.074	1.5	0.285	0.4	0.091	-0.4	0.262	1.7
Lab043	0.045	0.7	0.152	2.2	0.156	-0.2	0.075	1.6	0.672	5.0	NA		0.215	0.7
Lab044	0.039	0.0	0.085	-0.5	0.204	1.0	0.053	-0.1	0.239	-0.3	0.102	0.0	0.171	-0.3

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

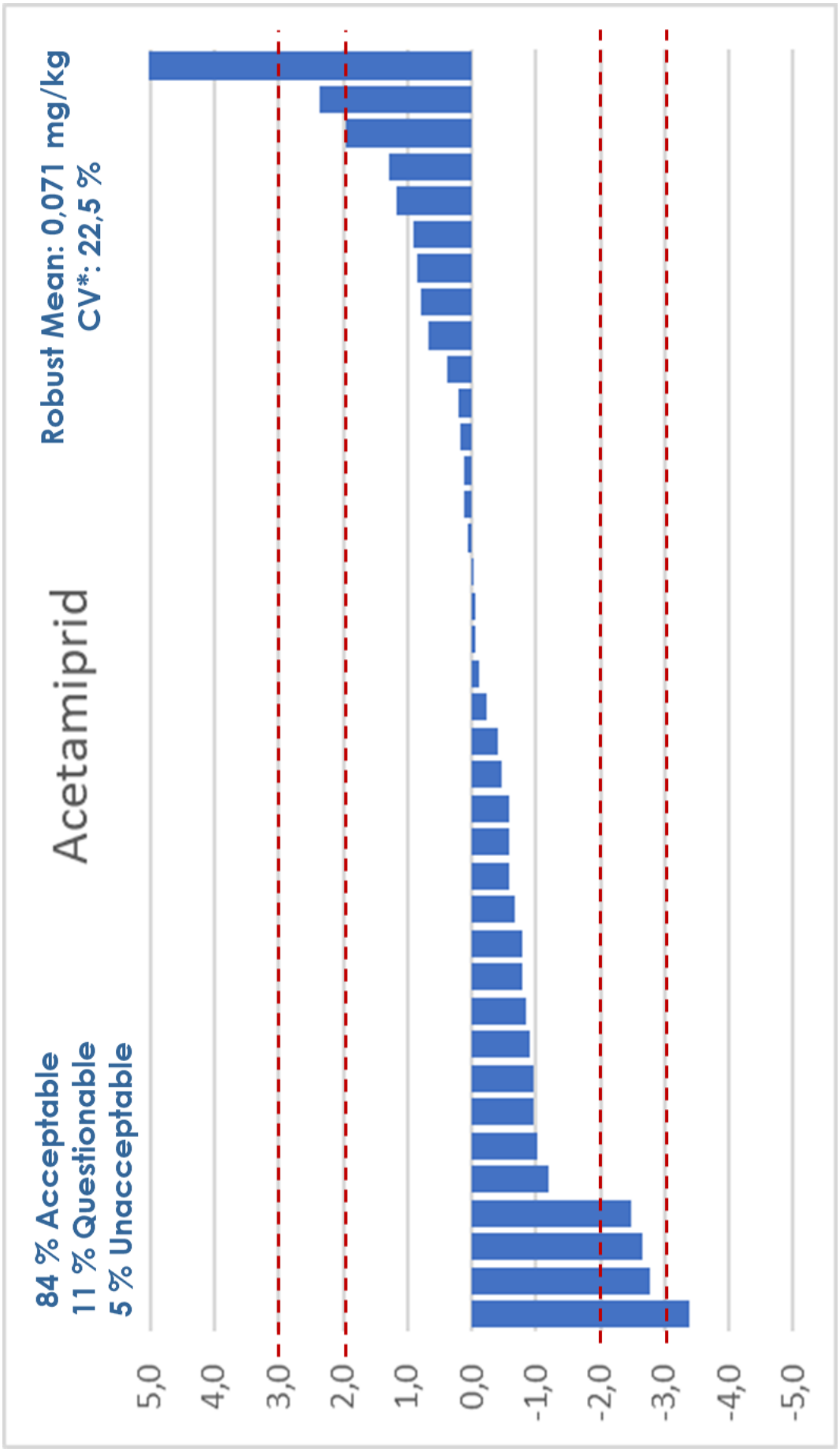
APPENDIX 2. Results (mg/kg) and z scores for FFP-RSD (25 %).

Lab Code	Profenofos	z score (FFP-RSD 25 %)	Thiamethoxam	z score (FFP-RSD 25 %)
MRRL (mg/kg)	0.01		0.01	
Robust mean (mg/kg)	0.066		0.091	
Lab001	0.091	1.5	0.101	0.9
Lab002	0.021	-2.7	0.011	-3.5
Lab003	ND	-4.0	ND	-4.0
Lab004	0.062	-0.2	0.07	-0.6
Lab005	0.073	0.4	0.089	0.3
Lab006	0.07	0.3	0.092	0.5
Lab007	0.047	-1.1	0.037	-2.2
Lab008	0.082	1.0	0.099	0.8
Lab009	0.07	0.3	0.104	1.0
Lab010	ND	-4.0	0.029	-2.6
Lab011	0.06	-0.4	0.085	0.1
Lab012	0.068	0.1	0.1	0.8
Lab013	0.028	-2.3	0.056	-1.3
Lab014	0.0743	0.5	0.111	1.4
Lab015	NA		0.081	-0.1
Lab016	0.071	0.3	0.089	0.3
Lab017	0.055	-0.7	0.106	1.1
Lab018	0.054	-0.7	0.071	-0.6
Lab019	0.0746	0.5	0.023	-2.9
Lab020	0.0533	-0.8	0.0866	0.2
Lab021	0.0689	0.2	0.0243	-2.8

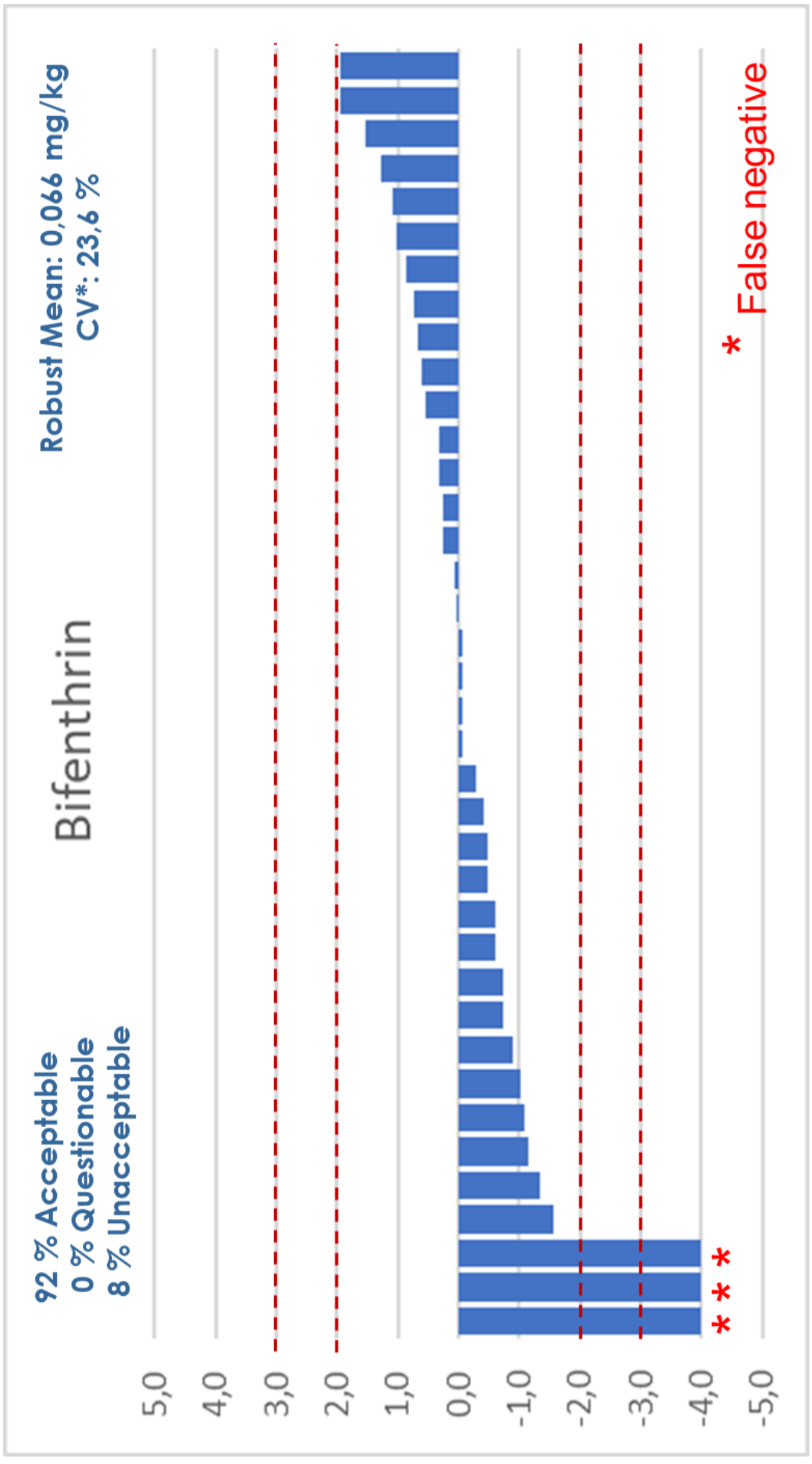
Lab Code	Profenofos	z score (FFP-RSD 25 %)	Thiamethoxam	z score (FFP-RSD 25 %)
MRRL (mg/kg)	0.01		0.01	
Robust mean (mg/kg)	0.066		0.091	
Lab022	0.0647	-0.1	0.116	1.6
Lab023	0.066	0.0	0.075	-0.4
Lab024	0.0785	0.8	0.0908	0.4
Lab025	0.0637	-0.1	0.099	0.8
Lab026	0.084	1.1	0.111	1.4
Lab028	0.072	0.4	0.086	0.2
Lab029	0.084	1.1	0.153	3.4
Lab030	0.057	-0.5	0.115	1.6
Lab031	0.068	0.1	0.114	1.5
Lab032	0.036	-1.8	0.016	-3.2
Lab033	0.19	7.6	0.08	-0.1
Lab034	0.073	0.4	ND	-4.0
Lab035	0.043	-1.4	0.059	-1.1
Lab036	0.078	0.7	NA	
Lab037	0.0673	0.1	0.086	0.2
Lab039	0.105	2.4	NA	
Lab040	0.06	-0.4	0.093	0.5
Lab041	0.057	-0.5	0.022	-2.9
Lab042	0.023	-2.6	0.105	1.1
Lab043	0.066	0.0	0.162	3.8
Lab044	0.066	0.0	0.071	-0.6

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

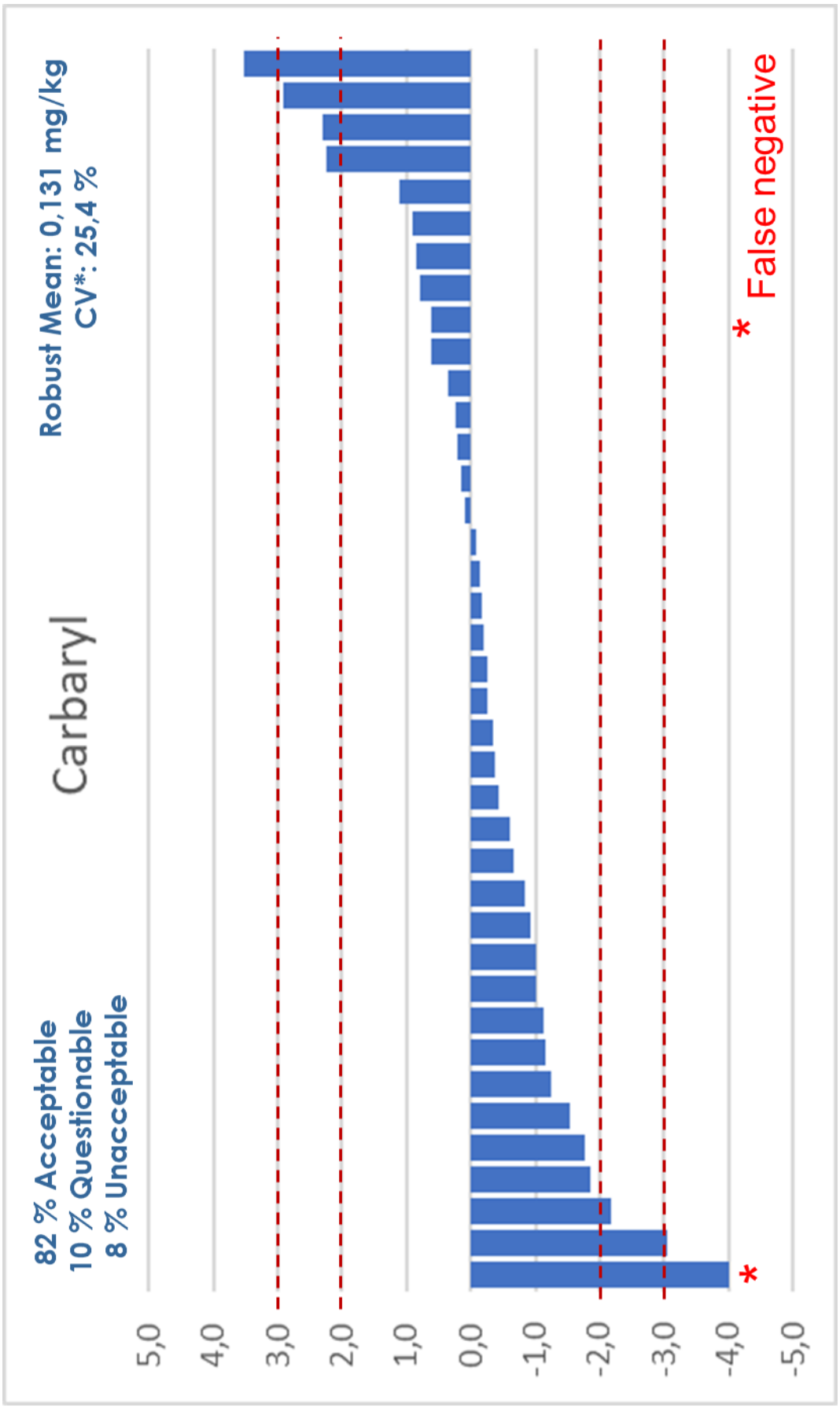
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



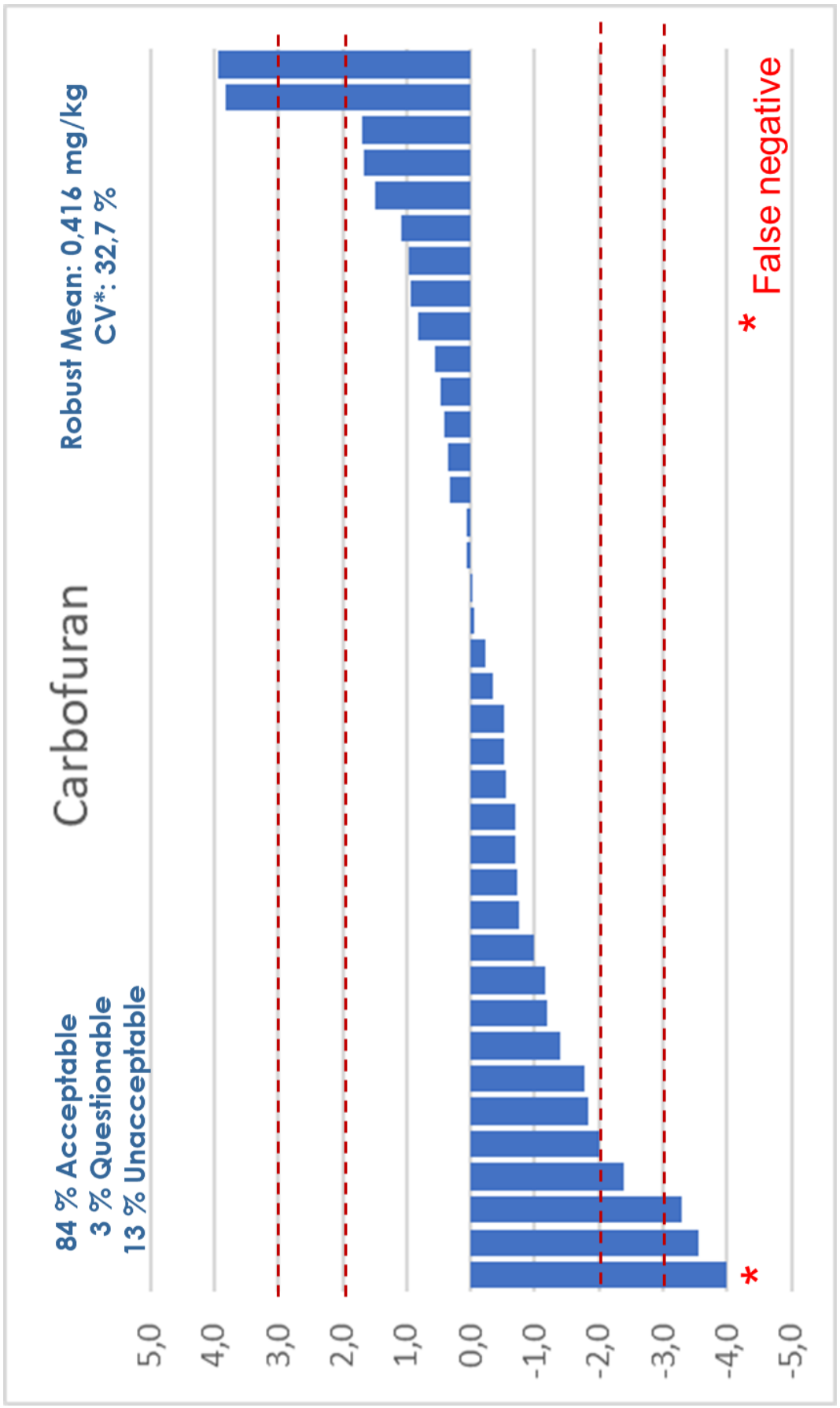
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



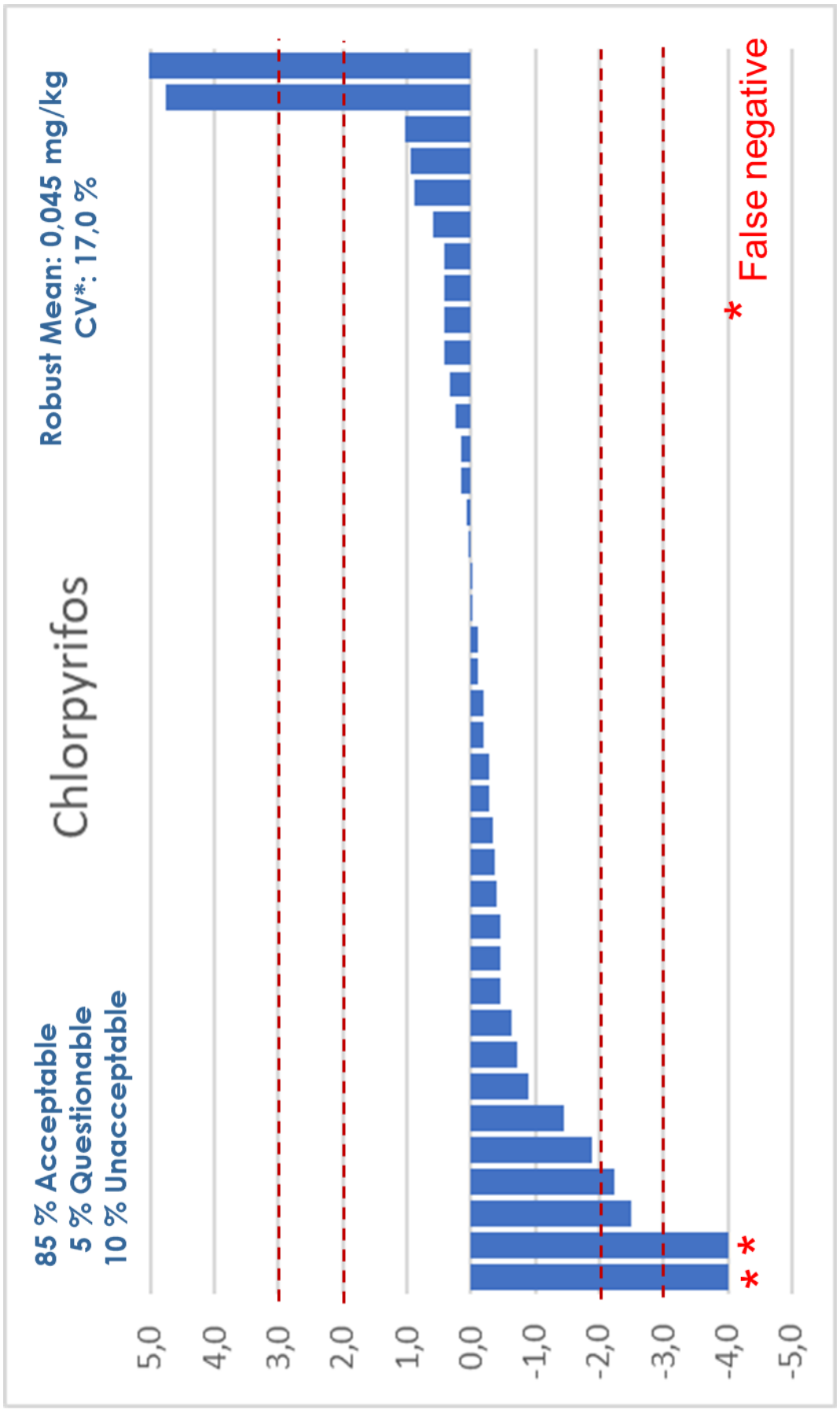
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



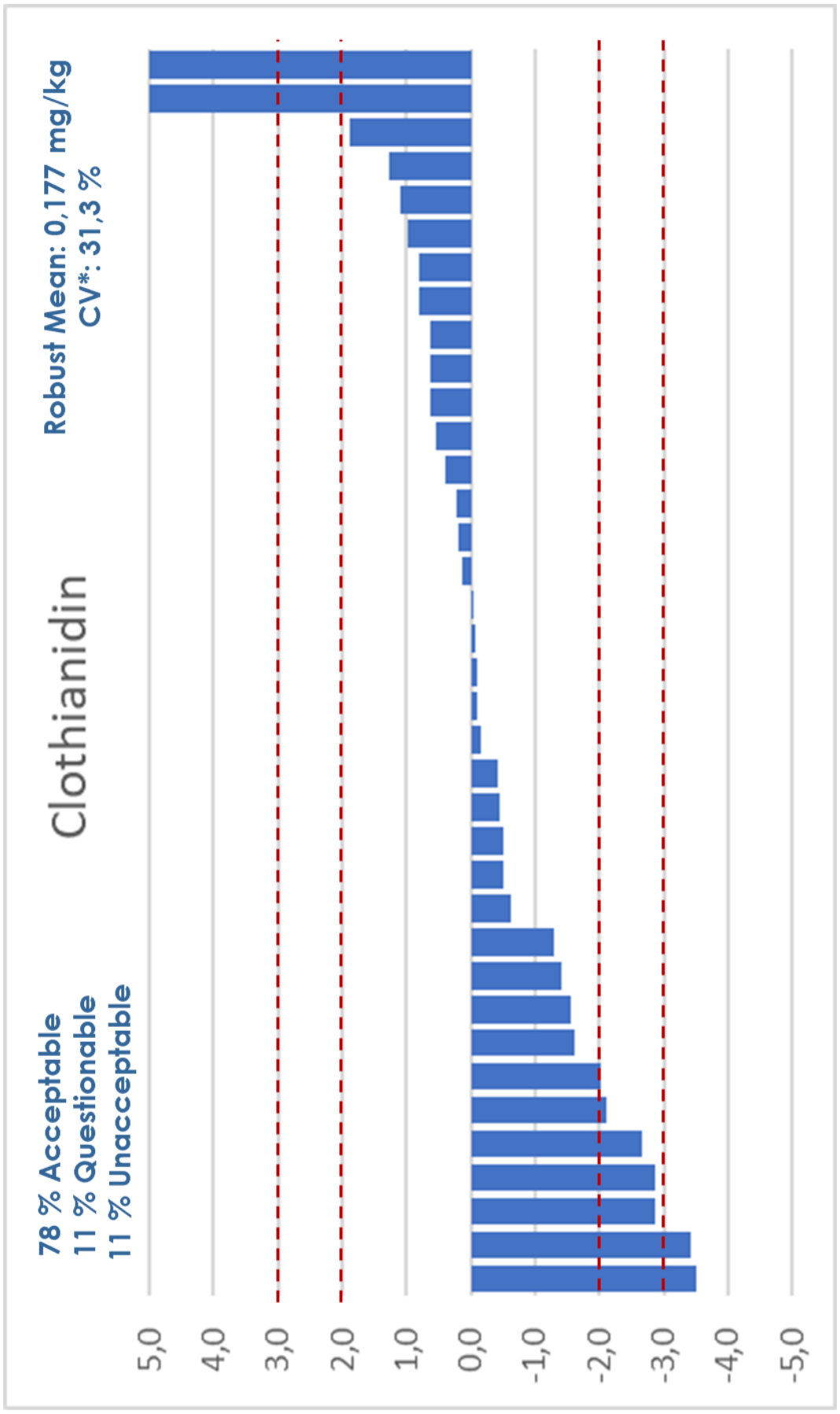
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



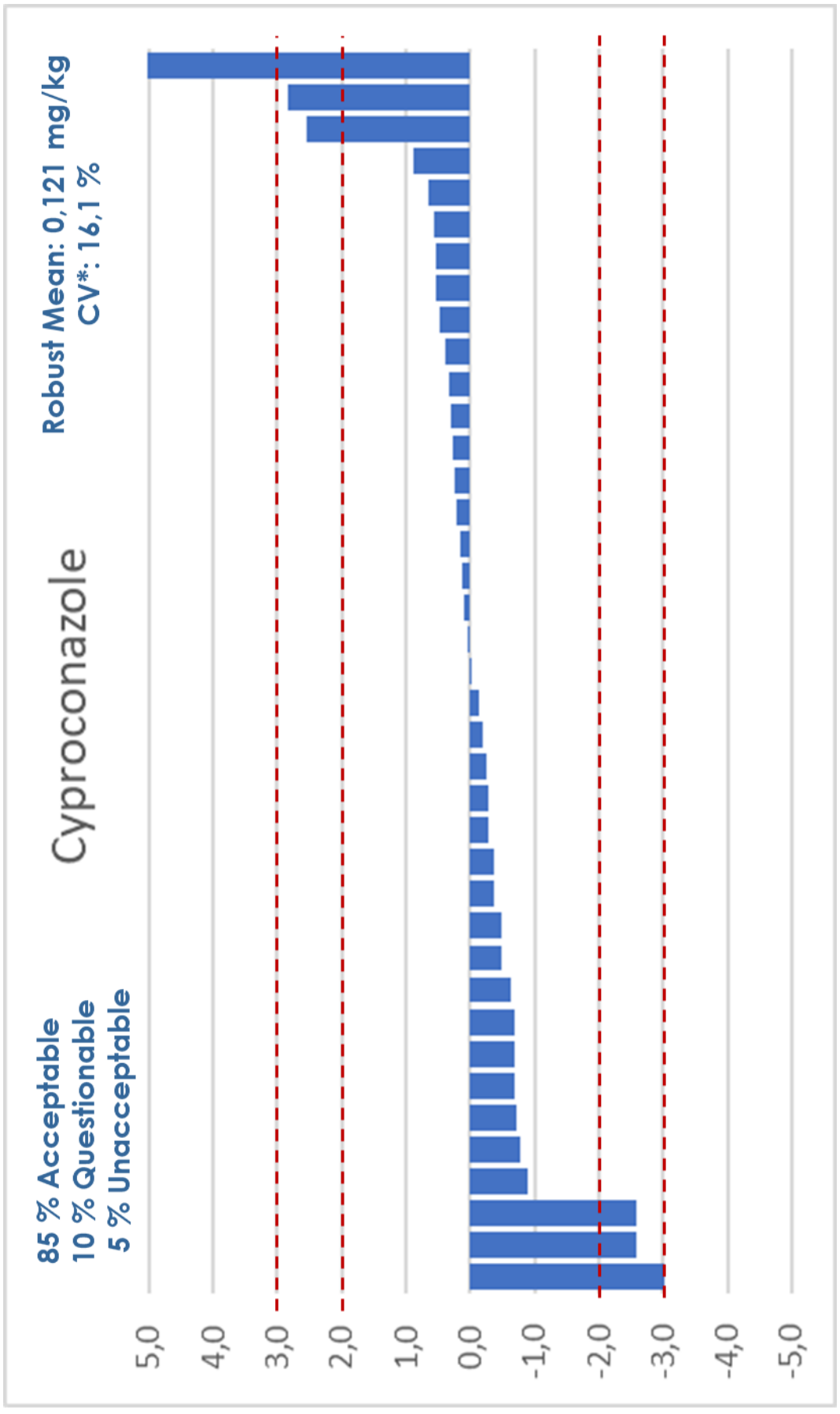
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



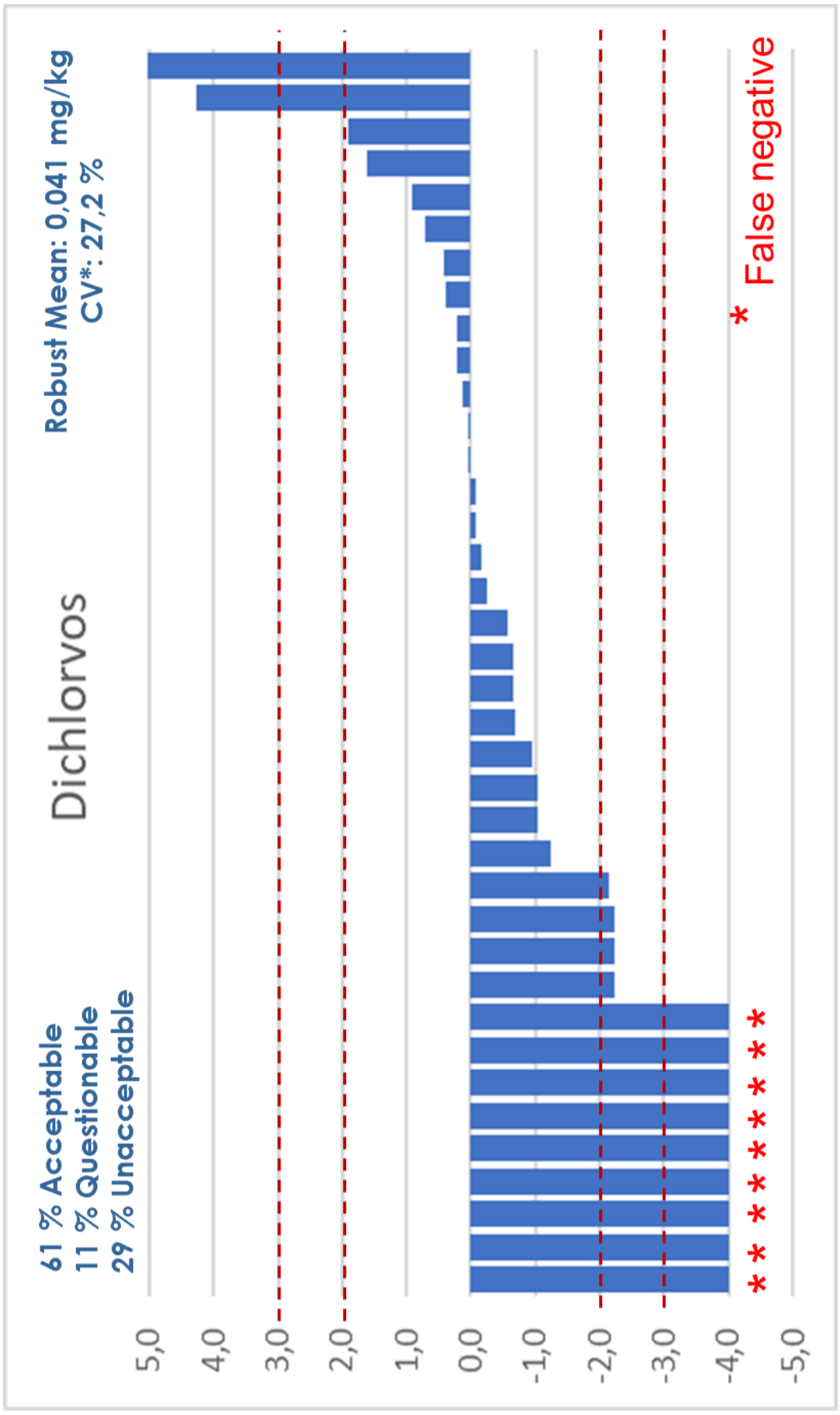
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



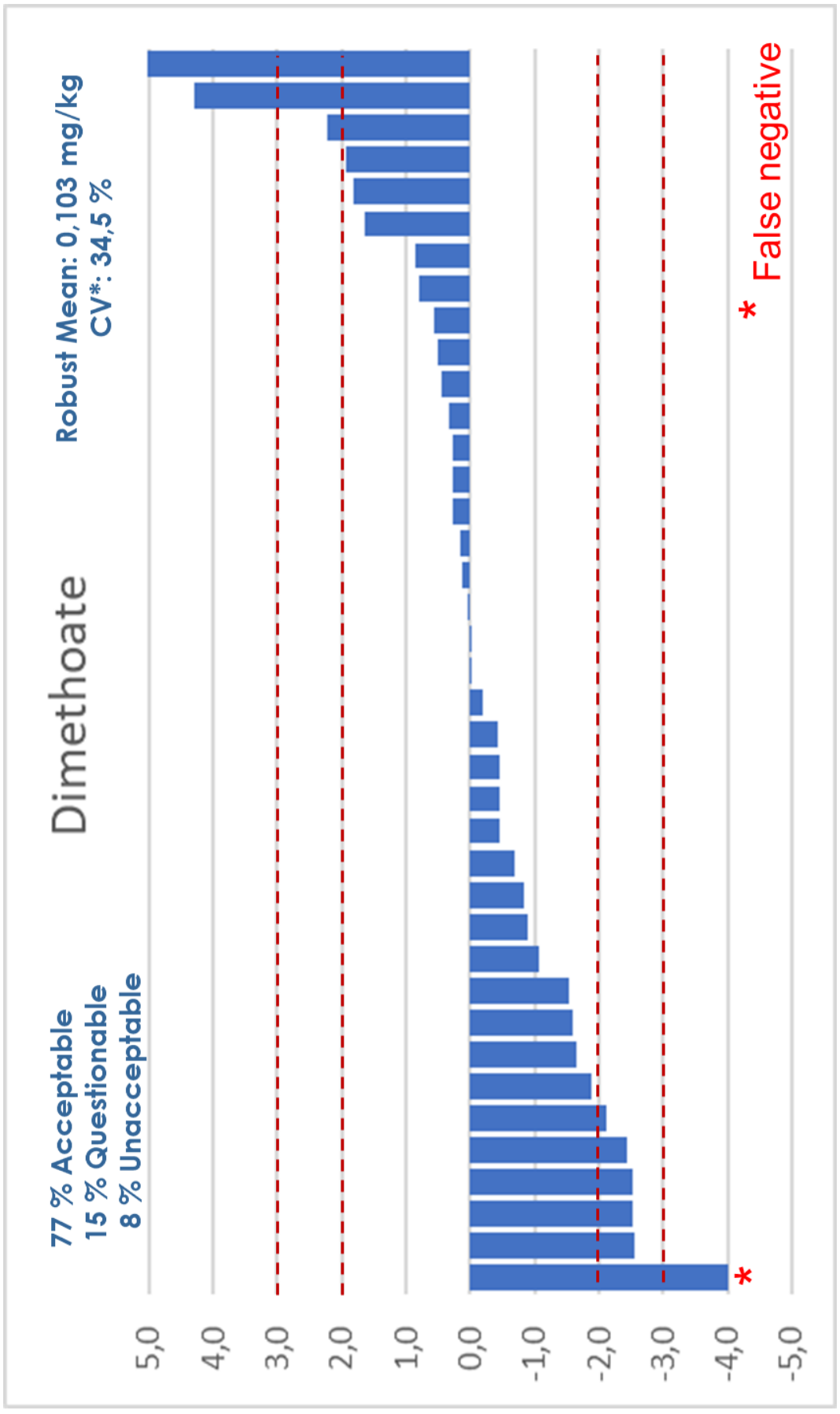
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



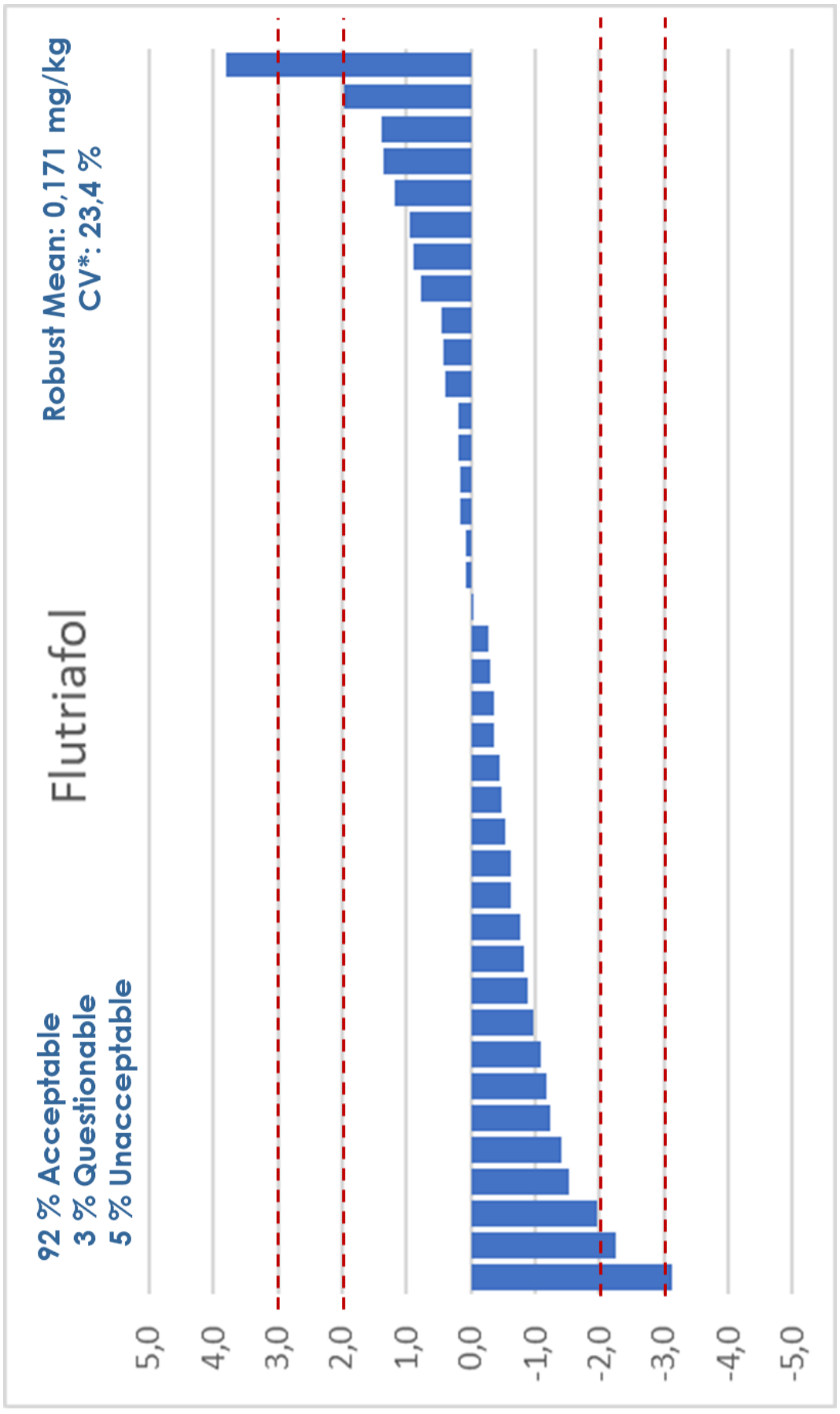
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



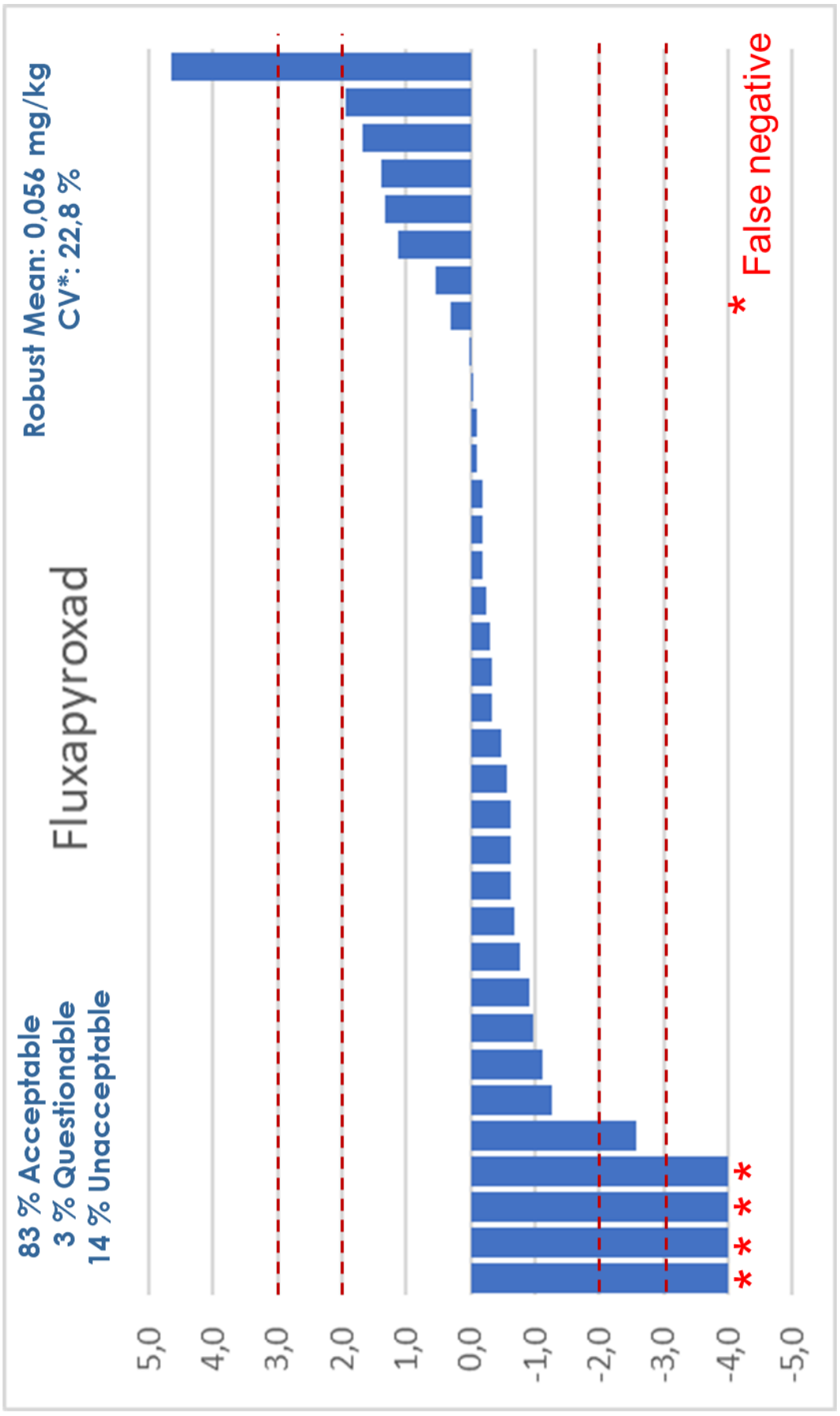
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



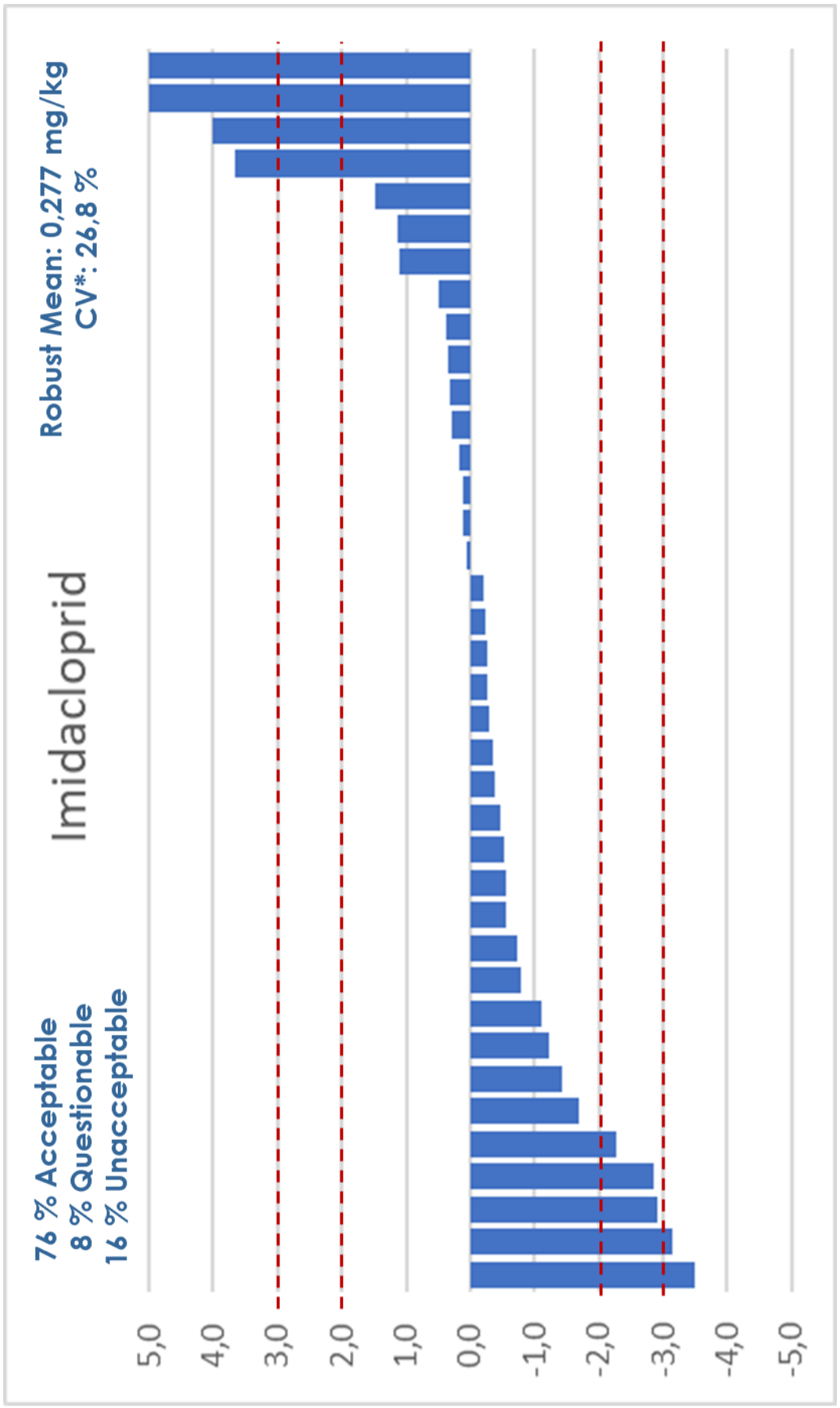
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



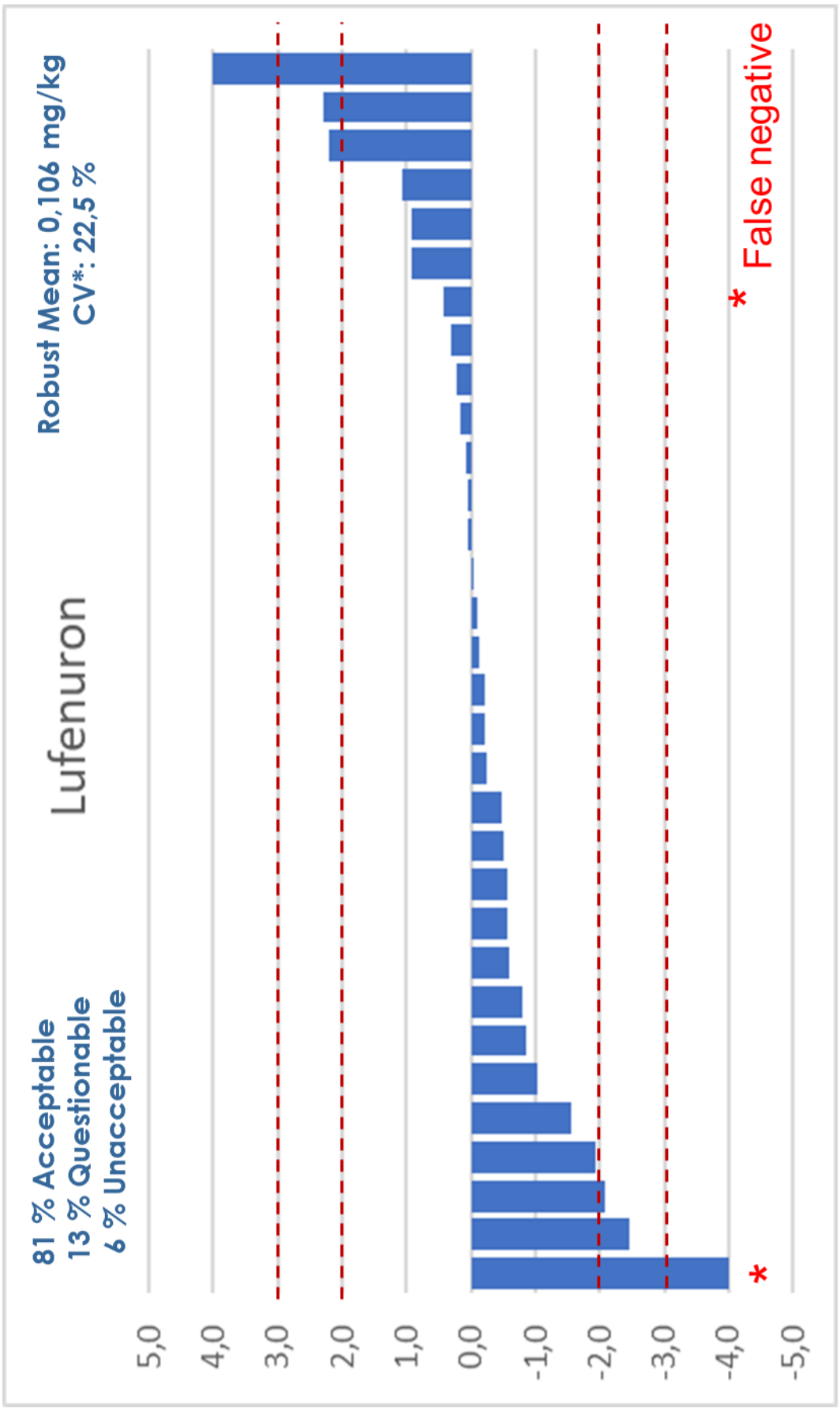
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



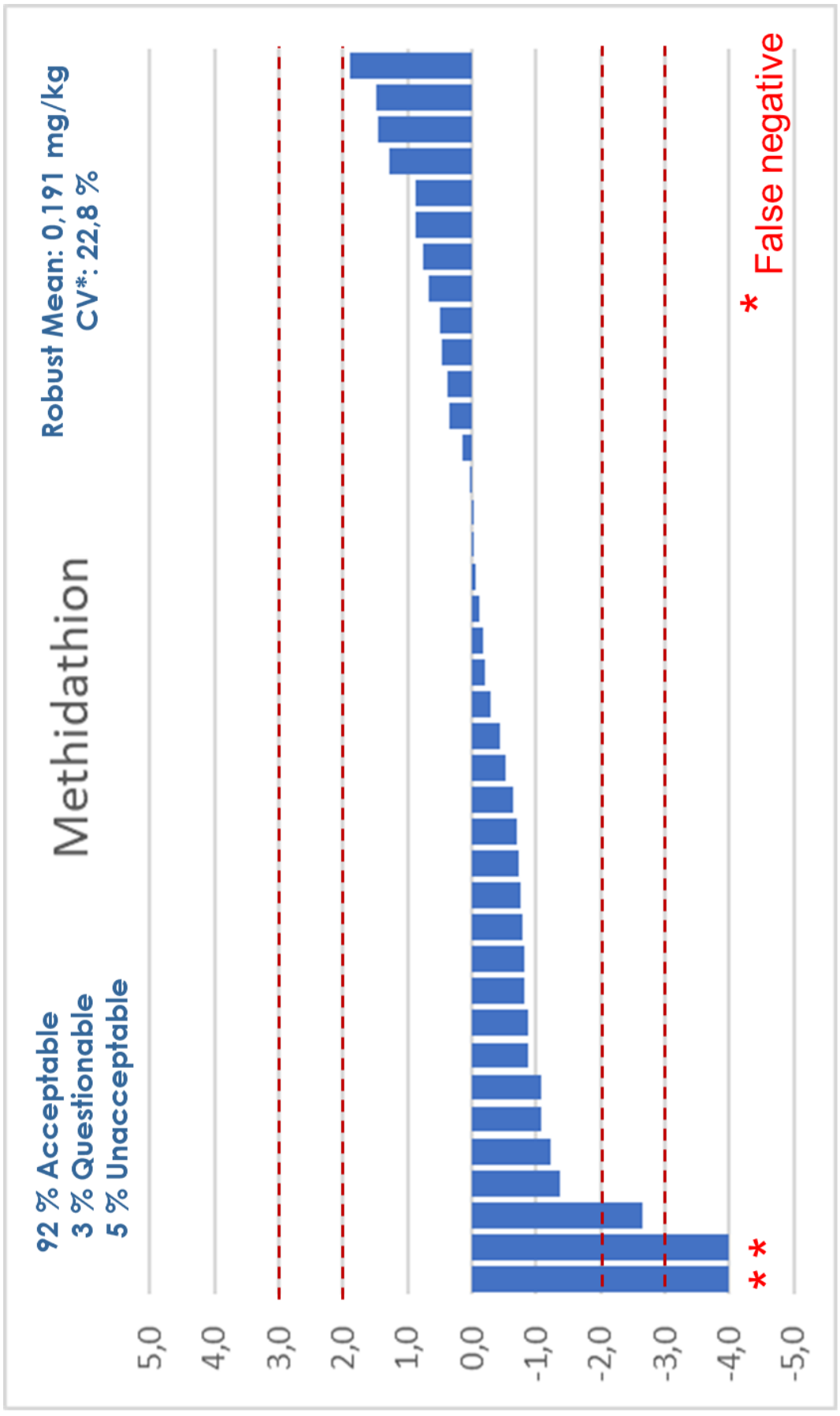
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



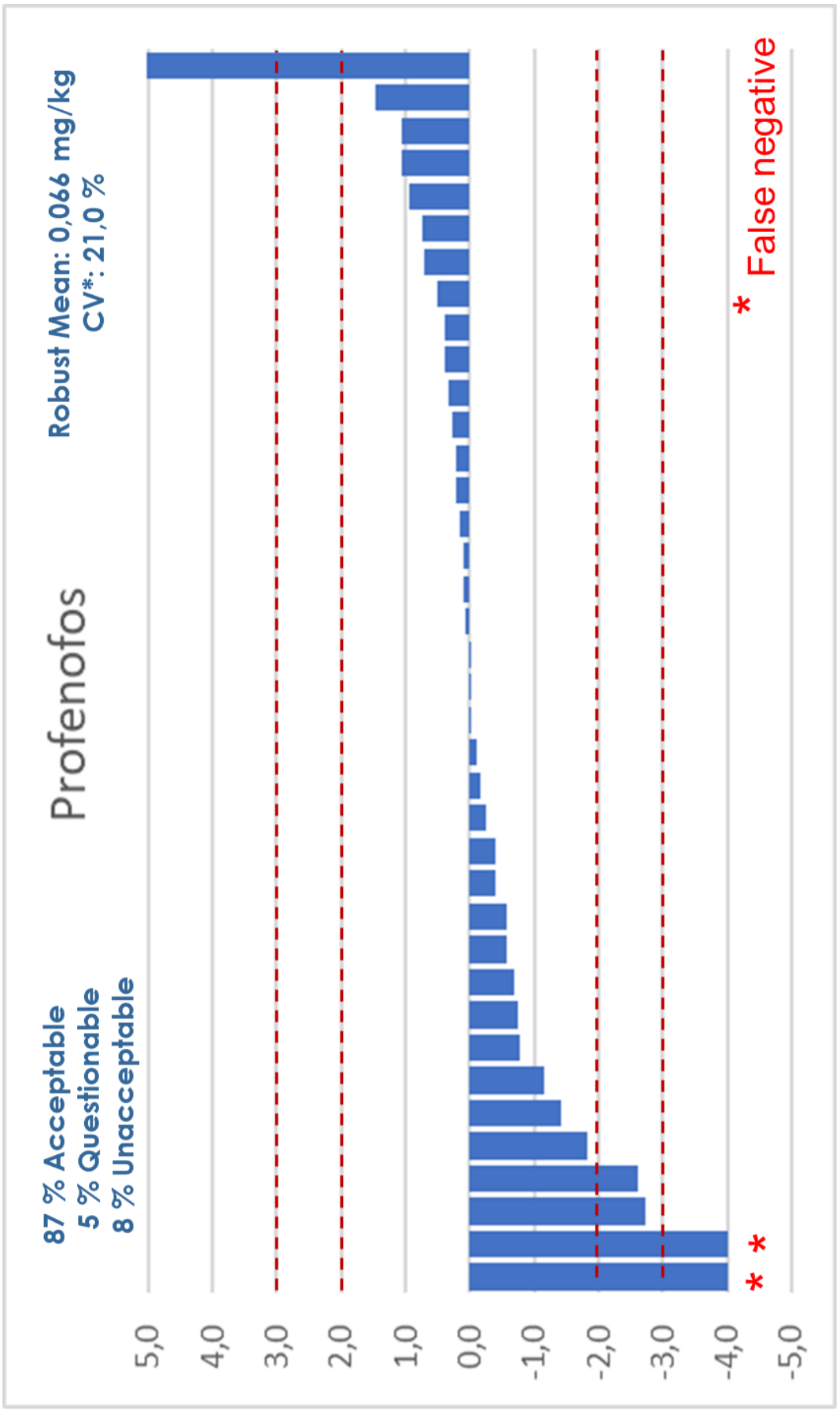
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



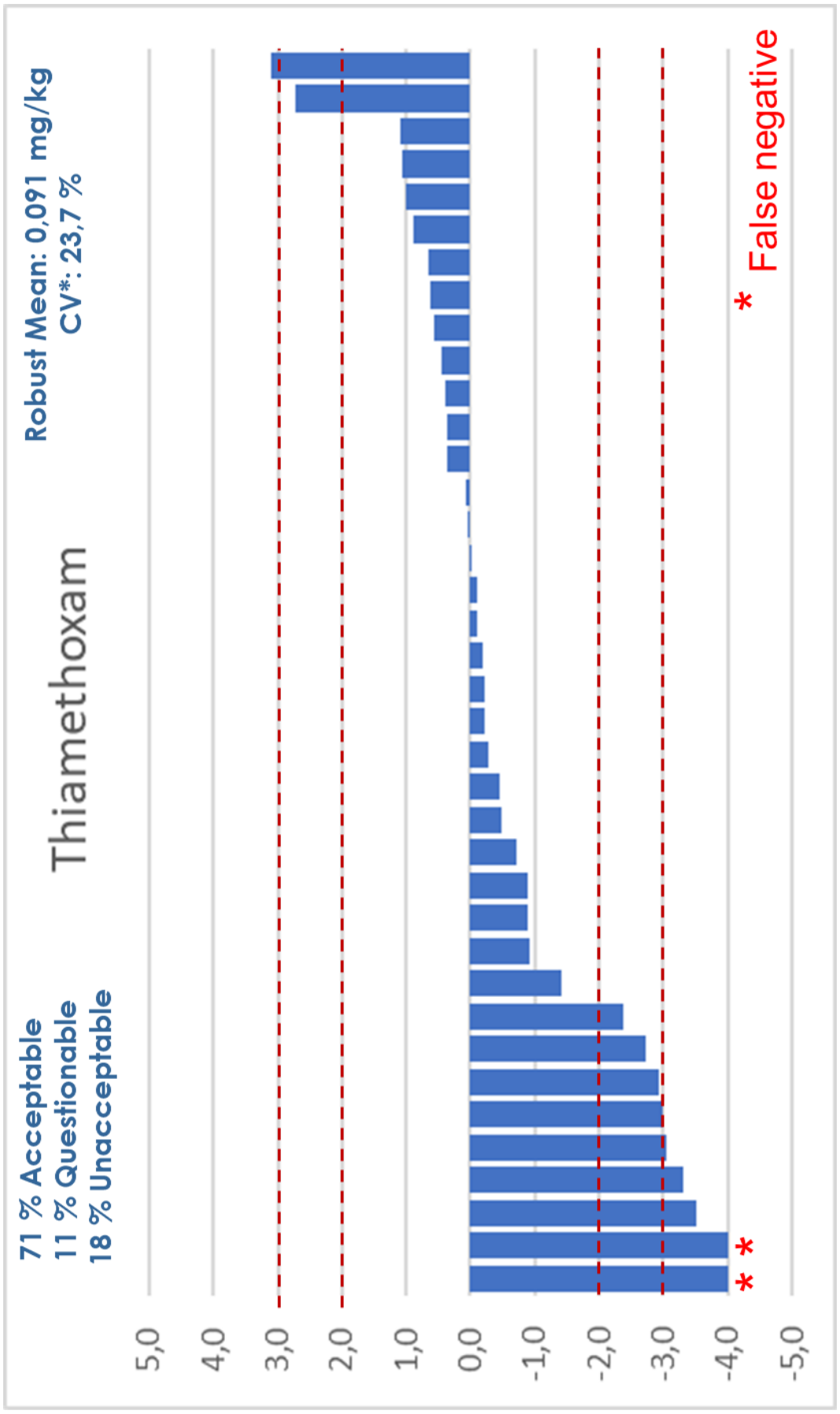
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



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



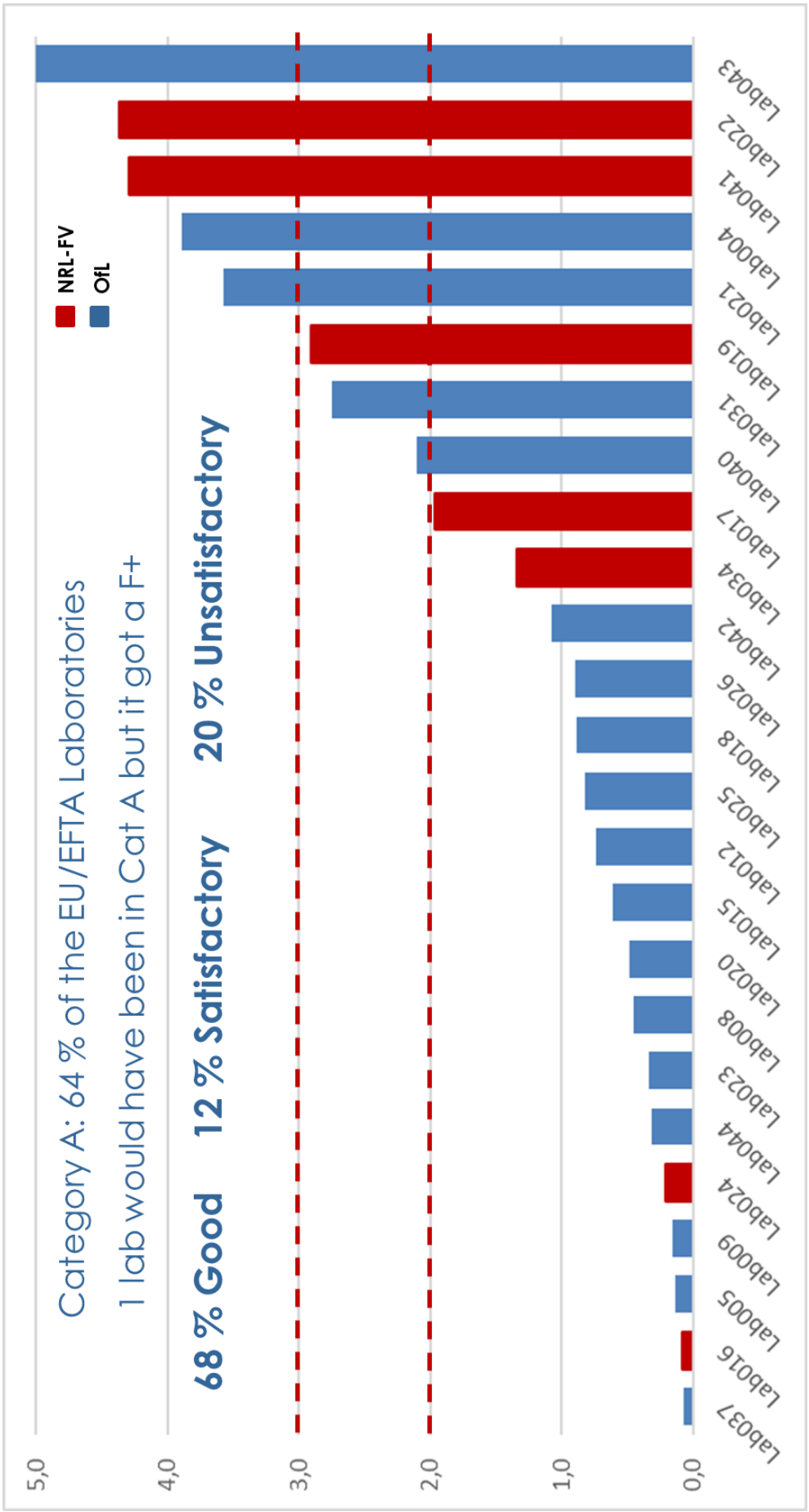
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



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

Lab Code	Acetamiprid	Bifenthrin	Carbaryl	Carbofuran	Chlorpyrifos	Clothianidin	Cyproconazole	Dichlorvos	Dimethoate	Flutriafol	Fluxapyroxad	Imidacloprid	Lufenuron	Methidathion	Profenofos	Thiamethoxam	No. of z scores	AZ^2
Lab004	-0.6	-4.0	-0.7	-1.0	-0.4	-3.4	-0.6	-2.1	-2.4	-0.4	-0.5	-1.2	-0.3	-4.0	-0.2	-0.6	16	3.7
Lab005	0.3	0.6	0.5	1.2	0.2	0.8	0.5	0.3	0.6	0.4	-0.1	0.0	0.3	0.1	0.4	0.3	16	0.2
Lab008	0.1	0.6	1.0	2.0	1.0	0.3	0.4	0.5	0.5	0.7	0.5	-0.2	0.6	0.8	1.0	0.8	16	0.7
Lab009	0.4	0.3	0.4	1.2	0.1	1.0	0.6	0.3	0.7	0.6	0.0	0.4	0.2	0.5	0.3	1.0	16	0.4
Lab012	1.1	0.2	0.9	2.0	0.5	0.9	0.6	-1.1	1.1	1.2	1.9	0.7	1.1	0.1	0.1	0.8	16	1.1
Lab013	0.6	-2.1	2.7	-0.5	-2.4	0.2	-1.0	-4.0	0.0	1.3	-0.5	0.6	-0.8	-4.0	-2.3	-1.3	16	3.8
Lab014	-0.2	0.6	0.3	-0.4	-0.3	-0.2	-0.3	0.0	0.1	0.2	1.2	-0.4		-0.2	0.5	1.4	15	0.3
Lab015	-0.7		-0.8	-0.1	0.5	-1.2	0.2	-0.9	-0.3	-0.3	-0.3	-1.0	-0.7	-0.5		-0.1	14	0.4
Lab016	0.0	-0.7	0.4	0.3	-0.4	0.6	0.3	-0.1	0.5	0.4	-0.1	0.0	0.2	0.2	0.3	0.3	16	0.1
Lab017	-0.8	-0.6	-0.9	0.1	0.0	-1.4	-0.3	0.4	0.0	-0.8	1.3	4.5	2.5	-1.3	-0.7	1.1	16	2.3
Lab018	-0.5	-0.6	-1.0	-0.6	-0.6	-0.1	-0.2	-2.1	-0.7	-0.1	-1.0	-0.9	-0.5	-0.8	-0.7	-0.6	16	0.7
Lab019	-2.6	0.0	-1.1	-0.8	0.4	-2.5	-0.4	-2.0	-2.0	-1.4	-0.5	-2.8	0.1	-0.7	0.5	-2.9	16	2.7
Lab020	-0.8	-1.4	-0.4	0.2	-0.3	-0.2	-0.2	-0.5	-0.7	-0.2	-0.2	-0.6	-0.9	-0.4	-0.8	0.2	16	0.4
Lab021	-2.4	0.3	-1.4	-1.0	0.1	-1.9	-0.4	-4.0	-2.4	-1.3		-2.2	-0.7	-0.7	0.2	-2.8	15	3.3
Lab022	1.6	1.0	3.9	4.4	-0.2	1.2	-0.1	5.0	2.0	1.7	2.1	1.5	1.3	1.5	-0.1	1.6	16	5.3
Lab023	-0.3	-1.0	-0.5	-0.3	-0.1	-0.1	0.7	-0.9	0.2	-0.6	0.2	0.0	0.2	-0.6	0.0	-0.4	16	0.2
Lab024	-0.2	0.7	0.0	0.0	1.0	0.3	-0.2	0.2	-0.2	-0.3	-0.2	-0.5	-0.1	0.1	0.8	0.4	16	0.2
Lab025	2.3	-0.9	-0.2	-0.4	-0.3	1.6	-0.7	0.2	2.1	-1.1		0.0	-0.4	0.0	-0.1	0.8	15	1.0
Lab026	1.1	0.8	0.9	1.4	0.7	1.4	0.6	2.2	1.1	1.2	0.7	0.6	0.5	1.6	1.1	1.4	16	1.4
Lab031	0.3	-0.7	0.0	0.6	0.2	2.4	5.0	-0.5	-1.4	2.3	-0.5	1.4	2.4	-0.7	0.1	1.5	16	3.1
Lab034	0.6	0.5	0.6	0.3	1.1	1.0	0.5	1.9	0.8	0.4	0.0	0.3	-0.1	0.3	0.4	-4.0	16	1.5
Lab037	0.2	-0.1	0.3	0.8	0.1	0.3	0.4	0.6	0.3	0.3	-0.2	0.0	-0.4	0.0	0.1	0.2	16	0.1
Lab040	0.1	-0.3	0.0	-0.3	-0.2	0.6	0.2	-4.0	0.2	0.2	-4.0	0.6	0.0	1.1	-0.4	0.5	16	2.2
Lab041	-2.7	1.9	-1.7	-1.7	-1.4	-2.8	-0.8	-2.1	-2.4	-1.9	-1.2	-2.8	-2.0	-0.6	-0.5	-2.9	16	4.0
Lab042	0.4	-0.5	1.4	1.1	-0.1	1.2	1.0	1.0	0.8	1.5	1.5	0.4	-0.4	1.7	-2.6	1.1	16	1.4
Lab043	2.7	-0.1	2.7	1.8	-0.4	5.0	0.3	0.7	2.2	-0.2	1.6	5.0		0.7	0.0	3.8	15	6.1
Lab044	0.9	-1.1	-0.2	-0.5	-0.3	-0.2	0.0	0.0	-0.5	1.0	-0.1	-0.3	0.0	-0.3	0.0	-0.6	16	0.3

APPENDIX 5. EUPT-FV-SC07-AZ²-Graphical representation for EU/EFTA laboratories in Category A.



10th Edition
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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[1] by the four European Union Reference Laboratories (EURLs) responsible for pesticide residues in food and feed. These EUPTs are organised for laboratories belonging to the Network[2] 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 the official controls Regulation (EU) No. 2017/625[3]:

- EURL for Fruits and Vegetables (EURL-FV),
- EURL for Cereals and Feeding stuff (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[4]. 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 Proficiency Test (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)[5] 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 the analytes 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 analytes 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 all EUPTs of the season have been conducted and preliminarily evaluated by the four pesticide EURLs. The aim of these meetings is to discuss the EUPT-results, especially where case-by-case decisions are needed. PT plans for the next EUPT season and, if needed, possible changes in the EUPT-General Protocol are also discussed during these meetings. The main topics and decisions on these meetings are documented.

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1 DG-SANTE = European Commission, Health and Food Safety Directorate-General

2 For more information about the EURL/NRL/OfL-Network please refer to the EURL-Web-portal under: "*http://www.eurl-pesticides.eu*"

3 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

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

5 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.

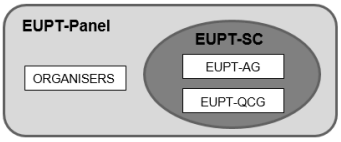

Figure 1: *Composition of EUPT-Scientific Committee*

The present EUPT General Protocol (EUPT-GP) was drafted by the EURLs and reviewed by the EUPT-SC. Follow the link to access a website giving an *overview of the GP-versions.*

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 Regulation (EU) No. 2017/6253 Art. 28 (3) of Reg. (EC) No. 2005/396 (for all OfLs analysing for pesticide residues within the framework of official controls of food or feed[6])
- Art. 101 (1)(a) of Regulation (EU) No. 2017/6253 (for all NRLs)

Every year, shortly before launching the registration period of the first of the four EUPTs in a given EUPT-Season, all OfLs and NRLs are asked to update their routine scope of commodities as well their contact information within the EURL-DataPool. Based on this information the OfLs are classified into those that are obliged and those that are eligible participate in each of the EUPTs to be conducted within a given year..
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 (EU) No. 2017/6253, 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 preliminary report distribution.
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 (EC) No. 2005/396 and Regulation (EU) No. 2016/ 128 (Baby Food Directive). Labs must express their results as stated in the Target Pesticides List.

[6] Official controls in the sense of Regulation (EU) 2017/625. This includes labs involved in controls within the framework of national and/or EU programs, as well as labs involved in import controls according to Regulation (EU) 2019/1793 (which repealed Regulation (EC) No. 2009/669).

Specific Protocol
For each EUPT the organising 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 analysis of two replicate analytical portions, taken from at least ten randomly chosen units of treated Test Item. Measurements should be conducted in random order. The homogeneity test data are statistically evaluated according to ISO 13528:2022, Annex B[7] or to the International Harmonized Protocols jointly published by ISO, AOAC and IUPAC[8]. The results of all homogeneity tests are presented to the EUPT-SC. In special cases, where the above homogeneity test criteria are not met, the EUPT-SC, considering all relevant aspects (e.g. the homogeneity results of other analytes 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 compound in 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.

Stability of the analytes contained in the Test Item
The Test Items will also be tested for stability - according to ISO 13528:2022, Annex B[7] . The time delay between the first and the last stability test must exceed the period of the EUPT-exercise. Typically the first analysis is carried out shortly before the shipment of the Test Items and the last one shortly after the deadline for submission of results. To better recognise trends and gain additional certainty one or more additional tests may be conducted by the organisers. At least 6 subsamples (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 analytes 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 EUPTQCG, may decide to omit a specific stability test. The EUPT-SC will finally decide whether analytes for which the stability test was not undertaken will be included in the Final EUPT-Report, considering all relevant aspects such as the distribution of the participant's results (CV*).
An analyte 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 compound in 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 analytes in the Test Item to possible losses, the organisers will choose the shipment conditions to be such that analyte losses are minimised (e.g. shipment of frozen samples, addition of dry ice). As shipmentduration 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 analytes, the EUPTSC will be informed (or the EUPT-QCG before or during the test). Case-by-case decisions may be taken by the EUPT-SC 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.

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[7] ISO 13528:2022: 'Statistical methods for use in proficiency testing by interlaboratory comparisons", International Organization for Standardization.

[8] Thompson M., Ellison S.L.R., Wood R., "The International Harmonized Protocol for the proficiency testing of analytical chemistry laboratories" (IUPAC Technical Report). Pure Appl. Chem. 2006, 78, 145 □ 196

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

General procedures for reporting results
Participating laboratories are responsible for reporting their own quantitative results to the organiser within the stipulated deadline. Any analyte 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 analytes 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 bias
According to the DG-SANTE Guidelines, the result of an analyte needs to be adjusted for method bias if the bias exceeds 20%. Unless a method is used that inherently accounts for method bias (see cases a-c below), laboratories are required to report the recovery (in percent), and whether their results were corrected mathematically using a recovery factor reflecting the reported recovery.
The EUPT-Panel will examine whether results, for which no correction for recovery was undertaken, should be omitted from the population used for calculating the assigned value.
When the laboratory uses any of the following approaches inherently accounting for method bias, this needs to be indicated in the appropriate fields within the Web-Tool. In such cases, reporting of the recovery rate is not mandatory.

a. use of stable isotope labelled analogues of the target analytes as Internal Standard (ILISs),added to the analytical portion at an early stage of the procedure
b. 'procedural calibration' approach
c. 'standard addition' approach with additions of analyte(s) to the analytical portions before extraction.

Methodology information
All laboratories are requested to provide information on the analytical method(s) they have used. The Web-Tool, which also serves for submitting analytical results, is typically used for collecting method information.
The collection of method information is considered very important by the EUPT-SC, as it facilitates the interpretation of results and the identification of analytical patterns associated with systematically biased results. A compilation of the methodology information submitted by all participants may be presented in an Annex of the Final EUPT-Report or in a separate report. Where the initial method information provided by the participating laboratories is not sufficient for evaluating methodology related errors, or where additional information critical for results evaluation is needed, the EURLs and/or the EUPT-Panel may decide to conduct specific follow-up surveys among the concerned laboratories. 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.
Where necessary the methods are evaluated and discussed within the EUPT-SC, especially in those cases where the result distribution is not unimodal or very broad (e.g. CV* > 35 %)

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

False Positive (FP) results
These are results of analytes 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 analytes. In certain instances, case-by-case decisions by the EUPT-SC 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 (FN) results
These are results for analytes 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 analytes 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, caseby-case decisions by the EUPT-SC 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-SC may decide to take case-by-case decisions in this respect after considering all relevant factors such as the result distribution and the RLs of the affected labs.

Estimation of the assigned value (xpt)
To minimise the influence of out-lying results on the statistical evaluation, the assigned value xpt (= consensus concentration) will typically be estimated using the robust estimate of the participant's mean (x*) as described in ISO 13528:2022[9], taking into account the results reported by EU and EFTA countries laboratories only. In special justifiable cases, the EUPT-Panel may decide including results submitted by laboratories not belonging to the EU-/EFTA-OfLs network or to even to only use the results of a subgroup of ('expert') laboratories that have previously repeatedly demonstrated good performance for the specific or similar compounds.

[9] ISO 13528:2022 '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).

Furthermore, the EUPT-Panel may decide to eliminate certain results traceably associated with bias or gross errors for establishing the assigned value (see 'Omission or Exclusion of results' below).
Since the assigned values of the EUPT analytes are typically generated using robust mean concentrations of participant results (xpt), which are generated by a variety of analytical standards and methods, the assigned values of EUPTs are typically metrologically not traceable.

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(xpt) is calculated according to ISO 13528:2022 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-SC 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 13528:2022 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-σpt) will be calculated using a Fit-ForPurpose approach with a fixed Relative Standard Deviation (FFP-RSD).
Based on experience from previous EUPTs[10], 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-SC 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 13528:2022; 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 xi is the value reported by the laboratory, xpt is the assigned value, and FFP-σpt 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).

[10] 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. DOI:10.1021/jf104060h

[11] ISO/IEC 17043:2010. Conformity assessment – General requirements for proficiency testing

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

Following ISO 17043:2010[11], z scores will be classified as follows::

$|z| \leq 2.0$ Acceptable

$2.0 < |z| < 3.0$ Questionable

$|z| \geq 3.0$ Unacceptable

All false negatives will be assigned a z score of -4. These z scores will typically appear in the z score histograms and will be used in the calculation of combined z scores.

Collection of measurement uncertainty (MU) figures

The participating labs will be asked to report the MU figure they would routinely report with each EUPT result. The EUPT-SC 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-SC 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 analytes in the target pesticides list, b) have correctly detected and quantified a sufficiently high percentage of the analytes 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 analytes needed to be correctly analysed to have sufficient scope will be calculated by multiplying the number of compulsory analytes 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 analytes from the Target Pesticides List needed to be targeted or analytes present in the Test Item that need to be correctly detected and quantified to have sufficient scope.

No. of compulsory analytes present in the Test Item / Target Pesticides List (N)	90 %	No. of analytes 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-SC 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 (AZ2)[12,13] (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 AZ^2 , z scores > 5 will be set as 5. Based on the AZ2 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 EUPTSC 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.

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

[13] 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.

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

Publication of results
The EURLs will publish a preliminary report, containing tentative assigned values and z score values for all analytes present in the Test Item, within 2 months of the deadline for result submission.
The Final EUPT-Report will be published after the EUPT-SC has discussed the results. Taking into account that the EUPT-SC 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 12 months after the deadline for results submission. Results submitted by non-EU/EFTA laboratories might not always be used in the tables or figures in the Final Report.

Certificates of participation
Together with the Final EUPT-Report, the EUPT organiser will deliver a Certificate of Participation to each participating laboratory showing the z scores achieved for each individual analyte, the classification into Categories, and if deemed necessary also combined z scores. The certificates of participation will be uploaded onto the EURL-DataPool where they can be accessed by the concerned laboratories only.

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-SC 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. if 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. In accordance with the 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 analytes in the target lists (80% for SRM-compounds), or c) detected less than 90% of the compulsory compounds present in the test items (80% for SRM-compounds).
Additionally, NRLs that obtained AZ2 higher than 3 (AAZ higher than 1.3 for SRM-compounds) in two consecutive EUPTs of the last four EUPTs, will be considered as underperforming in accuracy. As soon as underperformance of an NRL is detected, a two-step protocol established by DG-SANTE will be applied[14]:
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 iffeasible 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-SC 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.

[14] Article 101 of Regulation (EU) 2017/625

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

EUPT-FV-SC07 SPECIFIC PROTOCOL

European Union Proficiency Test for Pesticide Residues in ground green coffee beans (2023)

Introduction

This protocol is complementary to the General Protocol of EU Proficiency Tests (EUPTs) for Pesticide Residues in Food and Feed (10th Edition). 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 **green coffee beans** containing pesticide residues. The test item will consist of **ground green coffee beans**.

The Organiser, will check the Test Items for sufficient homogeneity and for stability at conditions reproducing sample shipment and storage during the duration of the test, according to ISO 13528, Annex B. All these tests will be conducted by the organiser, the EURL-FV. No blank material will be provided.

Steps to follow

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

1.**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 400 euros for the rest of laboratories.

4.The sample will be delivered to the participant laboratories on November 20th 2023. 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 20th December 2023.

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 100 g of ground green coffee beans containing pesticide residues.

No blank material will be provided.

Shipment of Test item

The shipment of the test item will be on 20th November 2023, and will be made at room temperature. 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 as regards shipment. Laboratories must make their own arrangements for the receipt of the package. They must inform the Organiser of any public holidays in their country/city during the delivery period given in the calendar, as well as making the necessary arrangements for receiving the shipment, even if the laboratory is closed.

Advice on Test item Handling

Once received, the test item should be stored deeply frozen (-18°C or less) prior to analysis thus avoiding any possible deterioration/spoilage. The test item should be mixed thoroughly before taking the analytical portion(s).

All participants should use their own routine standard operating procedures for extraction, clean-up and analytical measurement and their own reference standards for identification and quantification.

Test item Receipt

If any laboratory has not received the test item by 24th November 2023, 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:
- Two, for residue levels <0.010 mg/kg (e.g. 0.0086 mg/kg).
- Three, for residue levels ≥ 0.010 mg/kg (e.g. 0.0673, 0.245, 1.32, 10.1 mg/kg).

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. 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
Registration period	13th October 2023 - 31st October 2023
Specific Protocol published on the Web site.	6th November 2023 at the latest
Sample distribution.	20th November 2023
Deadline for receiving results	20th December 2023
Preliminary Report: (containing preliminary assigned valuesand z scores)	January 2024
Final Report distributed to the Laboratories.	August 2024

Cost of test item shipment.
The sample delivery will be **250 € for EU National Reference Laboratories and EU Official Laboratories and 400 € 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 Química CITE I
Carretera de Sacramento s/n
04120 La Cañada de San Urbano Almería - Spain
Phone No.: +34 950214102

Organising team (e-mails and phone numbers):

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Scientific Committee

Antonio Valverde, Senior Chemist (QCG).	University of Almería, Spain.
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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.
Marine Lambert, Senior Chemist (AG)	ANSES, French Agency for Food, Environmental and Occupational Health & Safety.
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).	SLV, Swedish Food Agency, 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

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

TARGET PESTICIDE LIST FOR THE EUPT-FV-SC07

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	0.01
13	Biphenyl	0.01
14	Bitertanol	0.01
15	Boscalid	0.01
16	Bromopropylate	0.01
17	Bromuconazole	0.01
18	Bupirimate	0.01
19	Buprofezin	0.01
20	Cadusafos	0.005
21	Carbaryl	0.005
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	0.005
33	Clofentezine	0.01
34	Clothianidin	0.01
35	Cyantraniliprole	0.01
36	Cyazofamid	0.01
37	Cyflufenamid	0.01
38	Cyfluthrin	0.01
39	Cymoxanil	0.01
40	Cypermethrin	0.01
41	Cyproconazole	0.01
42	Cyprodinil	0.01
43	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	0.01
50	Dieldrin	0.005
51	Diethofencarb	0.01
52	Difenoconazole	0.01
53	Diflubenzuron	0.01
54	Dimethoate	0.003
55	Dimethomorph	0.01
56	Dimethylaminosulfotoluidide (DMST)	0.01
57	Diniconazole	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	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	0.01
92	Fipronil	0.004
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	0.01
106	Fosthiazate	0.01
107	Hexaconazole	0.01
108	Hexythiazox	0.01
109	Imazalil	0.005
110	Imidacloprid	0.01
111	Indoxacarb	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	0.01
126	Metalaxyl	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	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	0.01

Pestide no.	Pesticides	MRRL (mg/kg)
145	Parathion-methyl	0.01
146	Penconazole	0.01
147	Pencycuron	0.01
148	Pendimethalin	0.01
149	Permethrin	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	0.01
159	Procymidone	0.01
160	Profenofos	0.01
161	Propamocarb	0.01
162	Propargite	0.01
163	Propiconazole	0.01
164	Propyzamide	0.01
165	Proquinazid	0.01
166	Prosulfocarb	0.01
167	Prothioconazole	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	0.01
177	Spinosad	0.01
178	Spirodiclofen	0.01
179	Spiromesifen	0.01
180	Spirotetramat	0.01
181	Spirotetramat metabolite BYI08330-enol	0.01
182	Spiroxamine	0.01
183	Sulfoxaflor	0.01
184	Tau-Fluvalinate	0.01
185	Tebuconazole	0.01
186	Tebufenozide	0.01
187	Tebufenpyrad	0.01
188	Teflubenzuron	0.01
189	Tefluthrin	0.01
190	Terbuthylazine	0.01
191	Tetraconazole	0.01
192	Tetradifon	0.01
193	Thiabendazole	0.01
194	Thiacloprid	0.01
195	Thiamethoxam	0.01
196	Thiodicarb	0.01
197	Thiophanate-methyl	0.01
198	Tolclofos-methyl	0.01
199	Tolylfluanid	0.01
200	Triadimefon	0.01
201	Triadimenol	0.01
202	Triazophos	0.005
203	Trichlorfon	0.01
204	Tricyclazole	0.01
205	Trifloxystrobin	0.01
206	Triflumizole	0.01
207	Triflumizole metabolite (FM-6-1)	0.01
208	Triflumuron	0.01
209	Trifluralin	0.01
210	Triticonazole	0.01
211	Vinclozolin	0.01
212	Zoxamide	0.01

MRRL: Minimum Required Reporting Level
This list is based on Commission Implementing Regulation (EU) EU) 2022/741 of 13 May 2022
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.