

EURL-PROFICIENCY TEST-FV-SC08, 2024-2025

Pesticide Residues in green tea

Final Report

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EURL-EUROPEAN UNION PROFICIENCY TEST SC08
FOR THE DETERMINATION OF PESTICIDES IN SPECIAL COMMODITIES USING
MULTIRESIDUE METHODS
2024-2025

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 origin¹, 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) 2017/625² 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, Spain⁴.

Participation in European Proficiency Test FV-SC08 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

Fifty-three laboratories agreed to participate in EUPT-FV-SC08.

The proficiency test was performed at the end of 2024 using ground green tea. Tea was purchased in the local market in Almería (Spain) and it was spiked with analytical standards. Participating laboratories were not provided with a 'blank' of tea sample.

The test item, 30 g of ground green tea containing both spiked and incurred pesticide residues, was shipped to participants on 18th November 2024. The deadline for results submission to the Organiser was 18th December 2024. The participants were asked to determine the residue levels of all the pesticides that they detected in the tea and to report the concentrations in mg/kg. The participants were provided with a target pesticide list, which contained 213 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.

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 tea was purchased in the local market in Almería. It was analysed in the EURL-FV laboratory in Almería for all the pesticides included in the target list, and incurred residues of bifenthrin, chlorfenapyr, deltamethrin, flufenoxuron, indoxacarb, thiacloprid and thiamethoxam were detected above their corresponding MRRLs.

1 kg portions of tea were weighed into disposable aluminium trays and covered with 1 L of a standard solution mixture in acetonitrile containing analytical standards of eight pesticides different than those already present in the tea. The trays were disposed under the fume hood to allow evaporation of the solvent. Once the solvent had evaporated completely and the tea was completely dry, all the tea was mixed in a large container and ground into a fine powder in the blender. Again, all the powder was thoroughly mixed by hand, and 30 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 tea.

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 green tea test item passed the homogeneity test. The acceptance criteria for the test item to be sufficiently homogenous for the proficiency test were that: $S_s^2 < c$, where S_s is the between-bottle sampling standard deviation and $c = F_1\sigma_{all}^2 + F_2s_{an}^2$; F_1 and F_2 being constant values of 1.88 and 1.01, respectively, from the ten samples taken, and $\sigma_{all}^2 = (0.3 \times \text{FFP-RSD}(25\%) \times \text{mean concentration})^2$. This was used to demonstrate that the between-bottle variance was not higher than the within-bottle variance.

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

Pesticide	Mean Conc.	S_s^2	c	$S_s^2 < c$
Acetamiprid	0.036	3.45E-07	2.00E-05	Pass
Bifenthrin	0.257	1.75E-04	9.80E-04	Pass
Biphenyl	0.084	1.21E-05	1.00E-04	Pass
Chlorfenapyr	0.193	6.13E-05	5.80E-04	Pass
Chlorpyrifos	0.070	6.34E-06	7.00E-05	Pass
Clothianidin	0.094	1.22E-05	1.10E-04	Pass
Cypermethrin	0.091	3.47E-05	1.20E-04	Pass

Pesticide	Mean Conc.	Ss ²	c	Ss ² < c
Deltamethrin	0.057	1.18E-05	5.00E-05	Pass
Difenoconazole	0.065	2.05E-06	9.00E-05	Pass
Flufenoxuron	0.280	0.00E+00	8.90E-03	Pass
Hexythiazox	0.066	0.00E+00	1.10E-04	Pass
Indoxacarb	0.077	1.15E-05	1.10E-04	Pass
Lambda-cyhalothrin	0.077	2.12E-05	8.00E-05	Pass
Thiacloprid	0.036	1.39E-07	2.00E-05	Pass
Thiamethoxam	0.069	0.00E+00	7.00E-05	Pass

Ss: 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, y_i 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 51_A	Sample 51_B	Sample 33_A	Sample 33_B	Sample 82_A	Sample 82_B	Mean 1	Sample 04_A	Sample 04_B	Sample 43_A	Sample 43_B	Sample 63_A	Sample 63_B	Mean 2		
Acetamidrid	0,033	0,040	0,037	0,036	0,041	0,038	0,038	0,040	0,036	0,037	0,041	0,035	0,037	0,038	0,000	Pass
Bifenthrin	0,261	0,318	0,281	0,301	0,299	0,271	0,289	0,298	0,276	0,267	0,304	0,289	0,296	0,289	0,000	Pass
Biphenyl	0,062	0,075	0,073	0,071	0,074	0,073	0,072	0,084	0,062	0,056	0,086	0,079	0,082	0,075	0,003	Pass
Chlorfenapyr	0,182	0,212	0,198	0,207	0,200	0,194	0,199	0,196	0,187	0,190	0,215	0,194	0,215	0,200	0,001	Pass
chlorpyrifos	0,070	0,081	0,072	0,078	0,079	0,073	0,076	0,075	0,074	0,066	0,080	0,072	0,078	0,074	-0,002	Pass
Clothianidin	0,072	0,091	0,093	0,080	0,100	0,096	0,089	0,100	0,080	0,084	0,101	0,082	0,080	0,088	-0,001	Pass
Cypermethrin	0,096	0,117	0,104	0,118	0,110	0,101	0,108	0,108	0,104	0,098	0,111	0,106	0,111	0,106	-0,001	Pass

(mg/kg)	Day 1							Day 2							(M2 - M1)	M2-M1 ≤ 0.3*σ
	Sample 51_A	Sample 51_B	Sample 33_A	Sample 33_B	Sample 82_A	Sample 82_B	Mean 1	Sample 04_A	Sample 04_B	Sample 43_A	Sample 43_B	Sample 63_A	Sample 63_B	Mean 2		
Deltamethrin	0,054	0,064	0,064	0,061	0,069	0,062	0,062	0,070	0,060	0,066	0,070	0,060	0,061	0,065	0,002	Pass
Difenoconazole	0,066	0,083	0,082	0,077	0,086	0,072	0,078	0,084	0,081	0,076	0,076	0,080	0,076	0,079	0,001	Pass
Flufenoxuron	0,260	0,230	0,294	0,288	0,325	0,294	0,282	0,298	0,250	0,268	0,294	0,315	0,330	0,293	0,011	Pass
Hexythiazox	0,066	0,075	0,078	0,076	0,082	0,074	0,075	0,081	0,082	0,077	0,083	0,078	0,077	0,080	0,005	Pass
Indoxacarb	0,069	0,077	0,074	0,078	0,076	0,074	0,075	0,085	0,085	0,075	0,076	0,075	0,080	0,079	0,005	Pass
Lambda-cyhalothrin	0,081	0,100	0,088	0,096	0,092	0,085	0,090	0,094	0,087	0,081	0,096	0,090	0,091	0,090	0,000	Pass
Thiacloprid	0,035	0,041	0,039	0,038	0,043	0,038	0,039	0,039	0,037	0,039	0,042	0,037	0,039	0,039	0,000	Pass
Thiamethoxam	0,066	0,090	0,081	0,076	0,081	0,084	0,080	0,093	0,070	0,075	0,081	0,071	0,075	0,077	-0,002	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 51_A	Sample 51_B	Sample 33_A	Sample 33_B	Sample 82_A	Sample 82_B	Mean 1	Sample 65_A	Sample 65_B	Sample 64_A	Sample 64_B	Sample 59_A	Sample 59_B	Mean 2		
Acetamiprid	0,033	0,040	0,037	0,036	0,041	0,038	0,038	0,036	0,035	0,032	0,037	0,037	0,039	0,036	-0,002	Pass
Bifenthrin	0,261	0,318	0,281	0,301	0,299	0,271	0,289	0,287	0,286	0,208	0,288	0,300	0,313	0,281	-0,008	Pass
Biphenyl	0,062	0,075	0,073	0,071	0,074	0,073	0,072	0,074	0,066	0,038	0,076	0,076	0,100	0,072	0,000	Pass
Chlorfenapyr	0,182	0,212	0,198	0,207	0,200	0,194	0,199	0,198	0,191	0,127	0,198	0,230	0,241	0,197	-0,001	Pass
chlorpyrifos	0,070	0,081	0,072	0,078	0,079	0,073	0,076	0,073	0,071	0,053	0,078	0,075	0,085	0,073	-0,003	Pass
Clothianidin	0,072	0,091	0,093	0,080	0,100	0,096	0,089	0,081	0,078	0,079	0,088	0,089	0,096	0,085	-0,004	Pass
Cypermethrin	0,096	0,117	0,104	0,118	0,110	0,101	0,108	0,108	0,105	0,079	0,107	0,112	0,114	0,104	-0,003	Pass
Deltamethrin	0,054	0,064	0,064	0,061	0,069	0,062	0,062	0,056	0,059	0,056	0,063	0,067	0,069	0,062	-0,001	Pass
Difenoconazole	0,066	0,083	0,082	0,077	0,086	0,072	0,078	0,072	0,067	0,071	0,071	0,077	0,080	0,073	-0,005	Pass
Flufenoxuron	0,260	0,230	0,294	0,288	0,325	0,294	0,282	0,311	0,294	0,294	0,316	0,294	0,271	0,297	0,015	Pass
Hexythiazox	0,066	0,075	0,078	0,076	0,082	0,074	0,075	0,077	0,073	0,072	0,080	0,077	0,082	0,077	0,002	Pass
Indoxacarb	0,069	0,077	0,074	0,078	0,076	0,074	0,075	0,078	0,080	0,074	0,072	0,080	0,078	0,077	0,002	Pass
Lambda-cyhalothrin	0,081	0,100	0,088	0,096	0,092	0,085	0,090	0,091	0,085	0,065	0,090	0,093	0,101	0,088	-0,003	Pass
Thiacloprid	0,035	0,041	0,039	0,038	0,043	0,038	0,039	0,035	0,038	0,034	0,037	0,039	0,040	0,037	-0,002	Pass
Thiamethoxam	0,066	0,090	0,081	0,076	0,081	0,084	0,080	0,074	0,068	0,070	0,075	0,085	0,085	0,076	-0,003	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 18th November 2024.

Before sample shipment, participant 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 SC08. The Target Pesticide List was the same as for EUPT-FV26, 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:

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

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'.

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

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

Laboratories that did not detect and quantify sufficient pesticides, that were not able to analyse at least 90 % of the compulsory pesticides or reported a false positive, have been placed in Category B and no combined z score has been calculated.

In **Appendices 4 and 5**, 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**.

Fifty-three 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. Two laboratories from non-EU/EFTA countries (Serbia and Ivory Coast) participated in EUPT-SC08. Their results have not been included for the calculation of the assigned value.

Fifteen pesticides from the pesticide target list were present in the test item at concentrations above the MRRL.

A summary of the reported results for the pesticides evaluated 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 51)
Acetamiprid	46	3	2	90
Bifenthrin	48	0	3	94
Biphenyl	38	5	8	75
Chlorfenapyr	46	0	5	90
Chlorpyrifos	49	1	1	96
Clothianidin	42	4	5	82
Cypermethrin	44	2	5	86
Deltamethrin	45	2	4	88
Difenoconazole	49	1	1	96
Flufenoxuron	48	1	2	94
Hexythiazox	48	1	2	94
Indoxacarb	47	2	2	92
Lambda-cyhalothrin	46	2	3	90
Thiacloprid	45	3	3	88
Thiamethoxam	39	5	7	76

^a The percentage of Reported Results comes from 51 laboratories. It does not take into account results reported by laboratories in Serbia and Ivory Coast.

4.1.1 False positives

Two laboratories reported two 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
Lab 02	Fenpropathrin	0,05	0,0793	GC-MS/MS QQQ
Lab 51	Orthophenylphenol	0,01	0,0161	GC-MS (tQ)

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).

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

Laboratory	Acetamiprid	Biphenyl	Chlorpyrifos	Clothianidin	Cypermethrin	Deltamethrin	Difenoconazole	Flufenoxuron	Hexythiazox	Indoxacarb	Lambda-Cyhalothrin	Thiacloprid	Thiamethoxam
Lab 01	ND												ND
Lab 02					ND								
Lab 03	ND		ND	ND	ND	ND				ND	ND	ND	ND
Lab 07	ND	ND		ND			ND			ND		ND	ND
Lab 09		ND											
Lab 17								ND					
Lab 18									ND				ND
Lab 28		ND											
Lab 30				ND									
Lab 33		ND											
Lab 35												ND	
Lab 36											ND		
Lab 37			ND										
Lab 40		ND											
Lab 48				ND									ND
Lab 51						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. The assigned values for the 15 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	46	0,043	19,7	0,01	0,002
Bifenthrin	48	0,264	27,1	0,01	0,013
Biphenyl	38	0,087	32,5	0,01	0,006
Chlorfenapyr	46	0,172	21,0	0,01	0,007
chlorpyrifos	49	0,061	21,4	0,01	0,002
Clothianidin	42	0,112	26,5	0,01	0,006

Pesticides	n	Robust mean (mg/kg)	CV (%)	MRRL (mg/kg)	Uncertainty (mg/kg)
Cypermethrin	44	0,093	20,8	0,01	0,004
Deltamethrin	45	0,07	25,4	0,01	0,003
Difenoconazole	49	0,075	16,6	0,01	0,002
Flufenoxuron	48	0,282	28,7	0,01	0,015
Hexythiazox	48	0,068	18,8	0,01	0,002
Indoxacarb	47	0,067	13,0	0,01	0,002
Lambda-cyhalothrin	46	0,079	21,5	0,01	0,003
Thiacloprid	45	0,047	20,4	0,01	0,002
Thiamethoxam	39	0,082	21,6	0,01	0,004

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	0,043	94	0	6
Bifenthrin	0,264	90	6	4
Biphenyl	0,087	77	5	18
Chlorfenapyr	0,172	98	2	0
chlorpyrifos	0,061	92	6	2
Clothianidin	0,112	83	4	13
Cypermethrin	0,093	94	4	2
Deltamethrin	0,07	92	2	6
Difenoconazole	0,075	96	2	2
Flufenoxuron	0,282	88	4	8
Hexythiazox	0,068	92	4	4
Indoxacarb	0,067	94	0	6
Lambda-cyhalothrin	0,079	96	0	4
Thiacloprid	0,047	90	4	6

Pesticide	Robust Mean (mg/kg)	Acceptable %	Questionable %	Unacceptable %
Thiamethoxam	0,082	87	2	11

z scores for false negative results have been assigned the fixed value of -4.0.

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 (13), 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**.

Thirty-nine of the 51 EU and EFTA laboratories were classified into Category A (76 %).

From the AZ^2 , 87 % were classed as 'good', 8 % as 'satisfactory' and 5 % as 'unsatisfactory' (Only considering EU and EFTA laboratories).

Of the 12 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 \ominus .

Table 9. Performance and Classification of laboratories in Category A using the AZ^2 formula

Lab Code	No. of pesticides detected (max.15)	% of pesticides analysed from target list	AZ^2	Classification
Lab 01 \ominus	15	99,1	4,2	Unsatisfactory
Lab 04	15	100,0	0,2	Good
Lab 05	15	99,5	0,7	Good
Lab 06	14	99,1	0,1	Good
Lab 08	15	100,0	0,3	Good
Lab 09 \ominus	15	100,0	1,3	Good

Lab Code	No. of pesticides detected (max.15)	% of pesticides analysed from target list	AZ ²	Classification
Lab 10	15	100,0	0,5	Good
Lab 11	15	90,6	0,6	Good
Lab 12	15	98,1	0,2	Good
Lab 13	14	97,7	1,6	Good
Lab 14	15	98,1	0,5	Good
Lab 15	15	100,0	0,3	Good
Lab 17 ⊖	15	95,3	1,8	Good
Lab 19	15	100,0	0,5	Good
Lab 21	15	100,0	0,4	Good
Lab 22	13	98,6	0,7	Good
Lab 23	15	99,5	0,6	Good
Lab 25	15	99,5	0,1	Good
Lab 26	15	98,6	1,7	Good
Lab 27	15	99,1	1,4	Good
Lab 29	15	100,0	0,4	Good
Lab 30 ⊖	15	98,6	1,6	Good
Lab 31	13	93,4	0,1	Good
Lab 32	15	94,4	4,4	Unsatisfactory
Lab 33 ⊖	15	100,0	1,9	Good
Lab 34	15	98,6	2,3	Satisfactory
Lab 36 ⊖	15	99,1	2,9	Satisfactory
Lab 38	15	100,0	0,4	Good
Lab 39	15	90,6	0,7	Good
Lab 40 ⊖	15	99,5	1,7	Good
Lab 41	15	99,5	1,0	Good
Lab 42	15	100,0	0,2	Good
Lab 44	14	95,8	0,7	Good
Lab 46	15	91,5	1,2	Good
Lab 47	15	98,1	0,3	Good
Lab 48 ⊖	15	100,0	2,4	Satisfactory
Lab 49	15	100,0	0,8	Good
Lab 52	15	96,2	1,4	Good
Lab 53	14	98,1	0,2	Good
Lab 54	15	92,0	0,2	Good

⊖ 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 AZ² 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)
Lab 02 ⊖ +	13	87	78	14	13
Lab 03 ⊖	9	60	89	15	5
Lab 07 ⊖	8	53	84	15	8
Lab 16	8	53	53	8	8
Lab 18 ⊖	9	60	62	11	9
Lab 20	0	0	2	0	0
Lab 24	9	60	73	9	9
Lab 28 ⊖	13	87	73	14	13
Lab 35 ⊖	12	80	82	13	12
Lab 37 ⊖	9	60	50	10	9
Lab 43	9	60	78	9	9
Lab 45	12	80	76	12	12
Lab 51 ⊖ +	14	93	100	15	13

⊖ Laboratories reporting a false negative result.

+ Laboratories reporting a false positive result.

5. CONCLUSIONS

Fifty-three laboratories agreed to participate in EUPT-FV-SC08. Two of them did not belong to EU nor EFTA countries, so their results were not considered for the estimation of the assigned value.

Fifteen pesticides were present in EUPT-FV-SC08 test item at concentrations above the MRRL (seven of them were incurred and eight were spiked as analytical standards).

Of a total number of 765 possible determinations from EU/EFTA laboratories (51 laboratories by 15 evaluated pesticides), 89 % were reported, 7 % were not analysed and 4 % were not detected (false negative results).

76 % of the EU and EFTA laboratories that submitted results were classified into Category A. Of them, 87 % were classed as 'good', 6 % as 'satisfactory' and 5 % as 'unsatisfactory'.

Participation in this EUPT-FV-SC08 involved laboratories from 18 EU Member States and 3 EFTA countries (Iceland, Norway and Switzerland). 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. Two non-European laboratories (from Serbia and Ivory Coast) participated in EUPT-FV-SC08.

6. REFERENCES

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APPENDIX 1. Homogeneity data.

Acetamiprid (mg/kg)		Bifenthrin (mg/kg)		Biphenyl (mg/kg)		Chlorfenapyr (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.040	0.035	0.292	0.268	0.091	0.086	0.217	0.185
0.041	0.038	0.255	0.284	0.091	0.084	0.205	0.195
0.036	0.036	0.241	0.269	0.078	0.091	0.185	0.205
0.038	0.035	0.258	0.283	0.083	0.091	0.204	0.215
0.034	0.037	0.256	0.270	0.089	0.089	0.200	0.204
0.037	0.036	0.272	0.264	0.084	0.085	0.182	0.197
0.035	0.036	0.197	0.244	0.064	0.078	0.150	0.186
0.037	0.034	0.263	0.259	0.081	0.089	0.206	0.187
0.035	0.036	0.239	0.254	0.083	0.085	0.178	0.190
0.035	0.035	0.234	0.242	0.081	0.083	0.179	0.181

Chlorpyrifos (mg/kg)		Clothianidin (mg/kg)		Cypermethrin (mg/kg)		Deltamethrin (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.080	0.073	0.101	0.096	0.110	0.099	0.064	0.066
0.069	0.071	0.103	0.093	0.096	0.094	0.062	0.060
0.068	0.071	0.086	0.093	0.085	0.093	0.057	0.058
0.068	0.073	0.101	0.096	0.095	0.095	0.054	0.064
0.070	0.075	0.088	0.091	0.091	0.094	0.060	0.058
0.074	0.069	0.099	0.094	0.093	0.090	0.056	0.054
0.053	0.068	0.093	0.091	0.070	0.089	0.045	0.057
0.071	0.072	0.091	0.100	0.090	0.093	0.062	0.058
0.062	0.070	0.095	0.094	0.084	0.087	0.053	0.054
0.066	0.070	0.086	0.085	0.081	0.086	0.053	0.053

Difenoconazole (mg/kg)		Flufenoxuron (mg/kg)		Hexythiazox (mg/kg)		Indoxacarb (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.076	0.071	0.154	0.228	0.062	0.067	0.074	0.083
0.081	0.065	0.250	0.248	0.069	0.071	0.079	0.081
0.071	0.061	0.325	0.223	0.071	0.069	0.085	0.076
0.071	0.066	0.285	0.291	0.063	0.077	0.086	0.075
0.071	0.056	0.287	0.204	0.055	0.071	0.073	0.073
0.066	0.064	0.357	0.214	0.068	0.068	0.083	0.081
0.067	0.062	0.298	0.325	0.081	0.059	0.087	0.076
0.061	0.058	0.249	0.470	0.067	0.057	0.063	0.074
0.067	0.052	0.165	0.346	0.057	0.066	0.067	0.064
0.061	0.060	0.247	0.432	0.067	0.056	0.091	0.071

APPENDIX 1. Homogeneity data.

Lambda-cyhalothrin (mg/kg)		Thiacloprid (mg/kg)		Thiamethoxam (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 1	Replicate 1	Replicate 2
0.092	0.082	0.035	0.035	0.071	0.070
0.077	0.082	0.042	0.037	0.068	0.066
0.075	0.080	0.035	0.038	0.065	0.076
0.077	0.081	0.038	0.039	0.071	0.075
0.080	0.080	0.038	0.035	0.066	0.065
0.078	0.080	0.037	0.033	0.074	0.067
0.059	0.073	0.038	0.034	0.070	0.070
0.080	0.078	0.036	0.038	0.065	0.076
0.071	0.076	0.033	0.035	0.069	0.069
0.071	0.073	0.036	0.036	0.063	0.071

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	Bifenthrin		Biphenyl		Chlorfenvinphos		Chlorpyrifos		Clothianidin		Cypermethrin		
	MRRL (mg/kg)	z score (FFP-RSD 25 %)	0.01											
	Robust mean (mg/kg)		0.264		0.087		0.172		0.061		0.112		0.093	
Lab 01	ND	-4.0	0.257	-0.1	0.051	-1.7	0.161	-0.3	0.061	0.0	0.023	-3.2	0.082	-0.5
Lab 02	0.028	-1.4	0.257	-0.1	0.139	2.4	0.184	0.3	0.058	-0.2	0.015	-3.5	ND	-4.0
Lab 03	ND	-4.0	0.664	6.1	0.170	3.8	0.200	0.7	ND	-4.0	ND	-4.0	ND	-4.0
Lab 04	0.039	-0.4	0.267	0.0	0.094	0.3	0.155	-0.4	0.061	0.0	0.106	-0.2	0.099	0.3
Lab 05	0.041	-0.2	0.300	0.5	0.043	-2.0	0.200	0.7	0.056	-0.3	0.089	-0.8	0.100	0.3
Lab 06	0.044	0.1	0.300	0.5	0.085	-0.1	0.170	0.0	0.070	0.6	0.130	0.6	0.083	-0.4
Lab 07	ND	-4.0	0.078	-2.8	ND	-4.0	0.178	0.1	0.068	0.5	ND	-4.0	0.187	4.0
Lab 08	0.041	-0.2	0.299	0.5	0.083	-0.2	0.197	0.6	0.070	0.6	0.110	-0.1	0.086	-0.3
Lab 09	0.048	0.4	0.301	0.6	ND	-4.0	0.196	0.6	0.065	0.3	0.130	0.6	0.103	0.4
Lab 10	0.038	-0.5	0.191	-1.1	0.079	-0.4	0.144	-0.7	0.059	-0.1	0.095	-0.6	0.069	-1.0
Lab 11	0.045	0.2	0.275	0.2	0.083	-0.2	0.205	0.8	0.067	0.4	0.104	-0.3	0.097	0.2
Lab 12	0.052	0.8	0.270	0.1	0.070	-0.8	0.180	0.2	0.056	-0.3	0.120	0.3	0.094	0.0
Lab 13	0.036	-0.7	0.149	-1.7	0.097	0.5	0.111	-1.4	0.038	-1.5	0.083	-1.0	NA	
Lab 14	0.050	0.6	0.330	1.0	0.103	0.7	0.181	0.2	0.072	0.7	0.136	0.8	0.106	0.6
Lab 15	0.049	0.5	0.225	-0.6	0.103	0.7	0.162	-0.2	0.053	-0.5	0.119	0.2	0.084	-0.4
Lab 16	0.041	-0.2	NA		NA		NA		0.032	-1.9	0.036	-2.7	NA	
Lab 17	0.032	-1.0	0.205	-0.9	0.043	-2.0	0.137	-0.8	0.053	-0.5	0.073	-1.4	0.079	-0.6
Lab 18	0.048	0.4	0.130	-2.0	NA		NA		0.027	-2.2	NA		0.050	-1.8
Lab 19	0.040	-0.4	0.200	-1.0	0.094	0.3	0.133	-0.9	0.050	-0.7	0.082	-1.1	0.073	-0.9
Lab 20	NA		NA		NA		NA		NA		NA		NA	
Lab 21	0.042	-0.1	0.340	1.1	0.098	0.5	0.200	0.7	0.072	0.7	0.130	0.6	0.110	0.7
Lab 22	0.033	-1.0	0.190	-1.1	0.071	-0.8	0.130	-1.0	0.051	-0.6	NA		0.063	-1.3
Lab 23	0.039	-0.4	0.274	0.1	0.062	-1.2	0.159	-0.3	0.061	0.0	0.096	-0.6	0.081	-0.5
Lab 24	0.045	0.2	NA		NA		NA		0.073	0.8	0.099	-0.5	NA	
Lab 25	0.042	-0.1	0.273	0.1	0.087	0.0	0.180	0.2	0.066	0.3	0.106	-0.2	0.105	0.5
Lab 26	0.050	0.6	0.472	3.1	0.067	-0.9	0.259	2.0	0.081	1.3	0.125	0.5	0.120	1.2
Lab 27	0.030	-1.3	0.163	-1.5	0.061	-1.2	0.166	-0.1	0.044	-1.1	0.062	-1.8	0.054	-1.7
Lab 28	0.029	-1.3	0.276	0.2	ND	-4.0	0.140	-0.7	0.063	0.2	0.100	-0.4	0.117	1.0
Lab 29	0.038	-0.5	0.132	-2.0	0.091	0.2	0.152	-0.5	0.062	0.1	0.132	0.7	0.109	0.7
Lab 30	0.044	0.0	0.320	0.8	0.121	1.5	0.152	-0.5	0.071	0.7	ND	-4.0	0.095	0.1
Lab 31	0.043	-0.1	0.290	0.4	NA		NA		0.069	0.5	0.129	0.6	0.093	0.0
Lab 32	0.061	1.6	0.219	-0.7	0.054	-1.5	0.294	2.8	0.050	-0.7	0.175	2.2	0.082	-0.5
Lab 33	0.057	1.3	0.259	-0.1	ND	-4.0	0.183	0.3	0.084	1.5	0.116	0.1	0.059	-1.5
Lab 34	0.044	0.1	0.430	2.5	0.160	3.3	0.223	1.2	0.078	1.1	0.110	-0.1	0.137	1.9

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 %)	Biphenyl	z score (FFP-RSD 25 %)	Chlorfenapyr	z score (FFP-RSD 25 %)	Chlorpyrifos	z score (FFP-RSD 25 %)	Clothianidin	z score (FFP-RSD 25 %)	Cypermethrin	z score (FFP-RSD 25 %)	
MRRL (mg/kg)	0.01		0.01		0.01		0.01		0.01		0.01		0.01		0.01
Robust mean (mg/kg)	0.043		0.264		0.087		0.172		0.061		0.112		0.093		
Lab 35	0.032	-1.0	0.215	-0.7	0.074	-0.6	0.149	-0.5	0.028	-2.2	NA		0.070	-1.0	
Lab 36	0.034	-0.8	0.326	0.9	0.127	1.8	0.153	-0.4	0.072	0.7	0.079	-1.2	0.108	0.6	
Lab 37	0.038	-0.5	0.200	-1.0	NA		NA		ND	-4.0	0.068	-1.6	NA		
Lab 38	0.051	0.7	0.286	0.3	0.106	0.8	0.163	-0.2	0.067	0.4	0.157	1.6	0.114	0.9	
Lab 39	0.039	-0.4	0.246	-0.3	0.052	-1.6	0.157	-0.3	0.068	0.5	0.097	-0.5	0.077	-0.7	
Lab 40	0.049	0.5	0.353	1.3	ND	-4.0	0.227	1.3	0.077	1.1	0.129	0.6	0.111	0.8	
Lab 41	0.036	-0.7	0.173	-1.4	0.062	-1.2	0.121	-1.2	0.048	-0.9	0.105	-0.3	0.066	-1.1	
Lab 42	0.048	0.4	0.263	0.0	0.071	-0.8	0.168	-0.1	0.063	0.2	0.121	0.3	0.096	0.1	
Lab 43	NA		0.205	-0.9	NA		0.122	-1.2	0.042	-1.2	NA		NA		
Lab 44	0.051	0.7	0.252	-0.2	NA		0.158	-0.3	0.053	-0.5	0.110	-0.1	0.083	-0.4	
Lab 45	0.035	-0.8	0.221	-0.7	NA		0.123	-1.1	0.057	-0.3	0.105	-0.3	0.080	-0.6	
Lab 46	0.057	1.3	0.240	-0.4	0.041	-2.1	0.140	-0.7	0.048	-0.8	0.160	1.7	0.093	0.0	
Lab 47	0.048	0.4	0.251	-0.2	0.097	0.4	0.161	-0.3	0.069	0.5	0.154	1.5	0.105	0.5	
Lab 48	0.047	0.3	0.302	0.6	0.089	0.1	0.164	-0.2	0.068	0.5	ND	-4.0	0.095	0.1	
Lab 49	0.058	1.3	0.323	0.9	0.107	0.9	0.200	0.7	0.042	-1.2	0.158	1.6	0.101	0.3	
Lab 51	0.055	1.1	0.359	1.4	0.158	3.2	0.221	1.1	0.096	2.3	0.151	1.4	0.107	0.6	
Lab 52	0.048	0.4	0.407	2.2	0.094	0.3	0.233	1.4	0.062	0.1	0.115	0.1	0.111	0.8	
Lab 53	0.038	-0.5	0.270	0.1	0.101	0.6	0.217	1.0	0.064	0.2	0.106	-0.2	0.097	0.2	
Lab 54	0.044	0.1	0.250	-0.2	0.088	0.0	0.160	-0.3	0.065	0.3	0.120	0.3	0.085	-0.3	

NA: Not analysed

ND: Not detected (False negative)

Lab Code	Deltamethrin	z score (FFP-RSD 25 %)	Difenoconazole	z score (FFP-RSD 25 %)	Flufenoxuron	z score (FFP-RSD 25 %)	Hexythiazox	z score (FFP-RSD 25 %)	Indoxacarb	z score (FFP-RSD 25 %)	Lambda-cyhalothrin	z score (FFP-RSD 25 %)	Thiacloprid	z score (FFP-RSD 25 %)
MRRL (mg/kg)	0.01		0.01		0.01		0.01		0.01		0.01		0.01	
Robust mean (mg/kg)	0.070		0.075		0.282		0.068		0.067		0.079		0.047	
Lab 01	0.087	0.9	0.091	0.8	0.472	2.7	0.045	-1.4	0.069	0.1	0.061	-0.9	0.019	-2.4
Lab 02	0.048	-1.3	0.051	-1.3	0.237	-0.6	0.055	-0.8	0.061	-0.4	0.081	0.1	0.029	-1.5
Lab 03	ND	-4.0	0.060	-0.8	0.320	0.5	0.060	-0.5	ND	-4.0	ND	-4.0	ND	-4.0
Lab 04	0.060	-0.6	0.089	0.7	0.261	-0.3	0.074	0.3	0.069	0.1	0.080	0.0	0.044	-0.3
Lab 05	0.047	-1.3	0.078	0.1	0.260	-0.3	0.055	-0.8	0.063	-0.3	0.088	0.5	0.030	-1.5
Lab 06	0.071	0.0	0.076	0.0	0.300	0.3	0.077	0.5	0.063	-0.3	0.081	0.1	0.049	0.2

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

Lab Code	Deltamethrin	z score (FFP-RSD 25 %)	Difenoconazole	z score (FFP-RSD 25 %)	Flufenoxuron	z score (FFP-RSD 25 %)	Hexythiazox	z score (FFP-RSD 25 %)	Indoxacarb	z score (FFP-RSD 25 %)	Lambda-cyhalothrin	z score (FFP-RSD 25 %)	Thiacloprid	z score (FFP-RSD 25 %)	
MRRL (mg/kg)	0.01		0.01		0.01		0.01		0.01		0.01		0.01		0.01
Robust mean (mg/kg)	0.070		0.075		0.282		0.068		0.067		0.079		0.047		
Lab 07	0.039	-1.8	ND	-4.0	0.124	-2.2	0.034	-2.0	ND	-4.0	0.050	-1.5	ND	-4.0	
Lab 08	0.074	0.2	0.085	0.5	0.233	-0.7	0.070	0.1	0.077	0.5	0.107	1.4	0.053	0.5	
Lab 09	0.076	0.3	0.077	0.1	0.327	0.6	0.084	0.9	0.065	-0.1	0.081	0.1	0.051	0.3	
Lab 10	0.077	0.4	0.087	0.6	0.243	-0.6	0.064	-0.3	0.076	0.5	0.051	-1.4	0.050	0.2	
Lab 11	0.101	1.7	0.100	1.3	0.365	1.2	0.073	0.3	0.077	0.6	0.098	1.0	0.046	-0.1	
Lab 12	0.064	-0.4	0.068	-0.4	0.240	-0.6	0.067	-0.1	0.065	-0.1	0.072	-0.4	0.045	-0.2	
Lab 13	0.045	-1.5	0.055	-1.1	0.140	-2.0	0.048	-1.2	0.042	-1.5	0.067	-0.6	0.034	-1.1	
Lab 14	0.092	1.3	0.078	0.1	0.280	0.0	0.078	0.6	0.063	-0.3	0.102	1.2	0.055	0.7	
Lab 15	0.067	-0.2	0.080	0.3	0.207	-1.1	0.066	-0.1	0.070	0.2	0.065	-0.7	0.055	0.7	
Lab 16	NA		0.056	-1.0	0.300	0.3	0.055	-0.8	NA		NA		0.041	-0.5	
Lab 17	0.060	-0.6	0.070	-0.3	ND	-4.0	0.069	0.0	0.066	-0.1	0.076	-0.2	0.042	-0.4	
Lab 18	0.029	-2.4	0.081	0.3	0.212	-1.0	ND	-4.0	0.041	-1.6	0.043	-1.8	NA		
Lab 19	0.051	-1.1	0.068	-0.4	0.220	-0.9	0.069	0.0	0.063	-0.3	0.072	-0.4	0.045	-0.2	
Lab 20	NA		NA		NA		NA		NA		NA		NA		
Lab 21	0.078	0.4	0.069	-0.3	0.350	1.0	0.079	0.6	0.065	-0.1	0.085	0.3	0.051	0.3	
Lab 22	0.052	-1.0	0.080	0.3	0.260	-0.3	0.060	-0.5	0.072	0.3	0.063	-0.8	0.037	-0.9	
Lab 23	0.070	0.0	0.045	-1.6	0.168	-1.6	0.055	-0.8	0.051	-1.0	0.090	0.6	0.042	-0.4	
Lab 24	NA		0.076	0.0	0.325	0.6	0.053	-0.9	0.068	0.0	NA		0.052	0.4	
Lab 25	0.065	-0.3	0.074	-0.1	0.230	-0.7	0.064	-0.3	0.060	-0.4	0.086	0.4	0.041	-0.5	
Lab 26	0.098	1.6	0.074	-0.1	0.221	-0.9	0.063	-0.3	0.071	0.2	0.108	1.5	0.055	0.7	
Lab 27	0.066	-0.3	0.087	0.6	0.207	-1.1	0.053	-0.9	0.090	1.4	0.078	-0.1	0.034	-1.1	
Lab 28	0.081	0.6	0.042	-1.8	0.218	-0.9	0.056	-0.7	0.052	-0.9	0.097	0.9	0.034	-1.1	
Lab 29	0.064	-0.4	0.076	0.0	0.299	0.2	0.075	0.4	0.065	-0.1	0.066	-0.7	0.047	0.0	
Lab 30	0.087	1.0	0.076	0.0	0.311	0.4	0.091	1.4	0.064	-0.2	0.096	0.9	0.056	0.7	
Lab 31	0.072	0.1	0.074	-0.1	0.294	0.2	0.069	0.1	0.068	0.0	0.075	-0.2	0.049	0.2	
Lab 32	0.095	1.4	0.105	1.6	0.514	3.3	0.063	-0.3	0.148	4.8	0.073	-0.3	0.075	2.4	
Lab 33	0.084	0.8	0.099	1.3	0.254	-0.4	0.084	0.9	0.086	1.1	0.070	-0.5	0.061	1.2	
Lab 34	0.097	1.5	0.078	0.1	0.373	1.3	0.094	1.5	0.075	0.5	0.116	1.9	0.048	0.1	
Lab 35	0.043	-1.6	0.045	-1.6	0.422	2.0	0.078	0.6	0.065	-0.1	0.071	-0.4	ND	-4.0	
Lab 36	0.080	0.5	0.090	0.8	0.057	-3.2	0.018	-2.9	0.077	0.6	ND	-4.0	0.040	-0.6	
Lab 37	0.110	2.3	0.098	1.2	0.380	1.4	NA		0.073	0.3	0.094	0.8	0.065	1.5	
Lab 38	0.068	-0.1	0.071	-0.2	0.328	0.7	0.070	0.1	0.061	-0.4	0.073	-0.3	0.047	0.0	
Lab 39	0.063	-0.4	0.067	-0.4	0.227	-0.8	0.049	-1.1	0.055	-0.7	0.062	-0.9	0.037	-0.9	
Lab 40	0.085	0.8	0.081	0.3	0.293	0.2	0.076	0.4	0.067	0.0	0.107	1.4	0.051	0.4	
Lab 41	0.053	-1.0	0.062	-0.7	0.192	-1.3	0.060	-0.5	0.039	-1.7	0.058	-1.0	0.039	-0.7	
Lab 42	0.074	0.2	0.071	-0.2	0.325	0.6	0.076	0.5	0.059	-0.5	0.076	-0.1	0.054	0.6	
Lab 43	0.077	0.4	0.078	0.1	0.300	0.3	0.077	0.5	0.065	-0.1	0.042	-1.9	NA		

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

Lab Code	Deltamethrin	z score (FFP-RSD 25 %)		Difenoconazole	z score (FFP-RSD 25 %)		Flufenoxuron	z score (FFP-RSD 25 %)		Hexythiazox	z score (FFP-RSD 25 %)		Indoxacarb	z score (FFP-RSD 25 %)		Lambda-cyhalothrin	z score (FFP-RSD 25 %)		Thiacloprid	z score (FFP-RSD 25 %)	
MRRL (mg/kg)	0.01			0.01			0.01			0.01			0.01			0.01			0.01		
Robust mean (mg/kg)	0.070			0.075			0.282			0.068			0.067			0.079			0.047		
Lab 44	0.070	0.0	0.065	-0.5	0.384	1.4	0.104	2.1	0.084	1.0	0.089	0.5	0.053	0.5							
Lab 45	0.078	0.4	0.068	-0.4	NA		NA		0.069	0.1	0.075	-0.2	0.033	-1.2							
Lab 46	0.069	-0.1	0.074	-0.1	0.220	-0.9	0.064	-0.3	0.060	-0.4	0.074	-0.3	0.066	1.6							
Lab 47	0.061	-0.5	0.089	0.7	0.258	-0.3	0.075	0.4	0.070	0.2	0.088	0.5	0.054	0.6							
Lab 48	0.061	-0.5	0.085	0.5	0.393	1.6	0.076	0.5	0.057	-0.6	0.089	0.5	0.049	0.2							
Lab 49	0.075	0.3	0.076	0.0	0.224	-0.8	0.076	0.5	0.075	0.5	0.110	1.6	0.055	0.7							
Lab 51	ND	-4.0	0.121	2.4	0.549	3.8	0.156	5.1	0.070	0.2	0.087	0.4	0.065	1.5							
Lab 52	0.123	3.0	0.067	-0.4	0.371	1.3	0.072	0.2	0.073	0.3	0.072	-0.4	0.051	0.4							
Lab 53	NA		0.072	-0.2	0.286	0.1	0.071	0.2	0.077	0.6	0.079	0.0	0.045	-0.2							
Lab 54	0.061	-0.5	0.072	-0.2	0.350	1.0	0.073	0.3	0.074	0.4	0.066	-0.7	0.051	0.3							

NA: Not analysed

ND: Not detected (False negative)

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

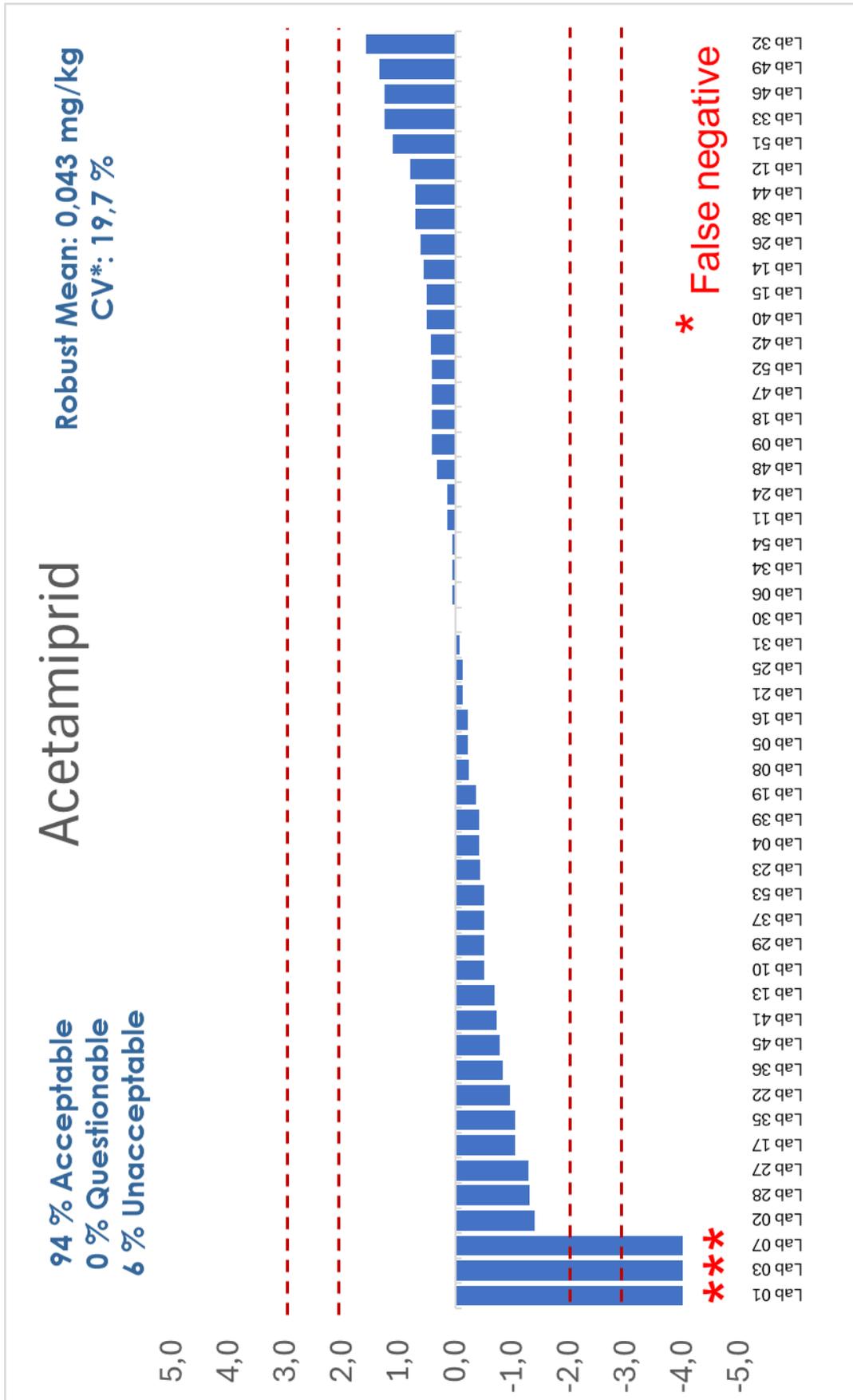
Lab Code	Thiamethoxam	z score (FFP-RSD 25 %)
MRRL (mg/kg)	0.01	
Robust mean (mg/kg)	0.082	
Lab 01	ND	-4.0
Lab 02	NA	
Lab 03	ND	-4.0
Lab 04	0.065	-0.8
Lab 05	0.067	-0.7
Lab 06	NA	
Lab 07	ND	-4.0
Lab 08	0.098	0.8
Lab 09	0.099	0.8
Lab 10	0.060	-1.1
Lab 11	0.078	-0.2
Lab 12	0.085	0.2
Lab 13	0.068	-0.7
Lab 14	0.089	0.3
Lab 15	0.079	-0.1
Lab 16	0.037	-2.2
Lab 17	0.061	-1.0
Lab 18	ND	-4.0
Lab 19	0.072	-0.5
Lab 20	NA	
Lab 21	0.083	0.1
Lab 22	NA	
Lab 23	0.089	0.4
Lab 24	0.071	-0.5
Lab 25	0.071	-0.5
Lab 26	0.092	0.5
Lab 27	0.047	-1.7

NA: Not analysed

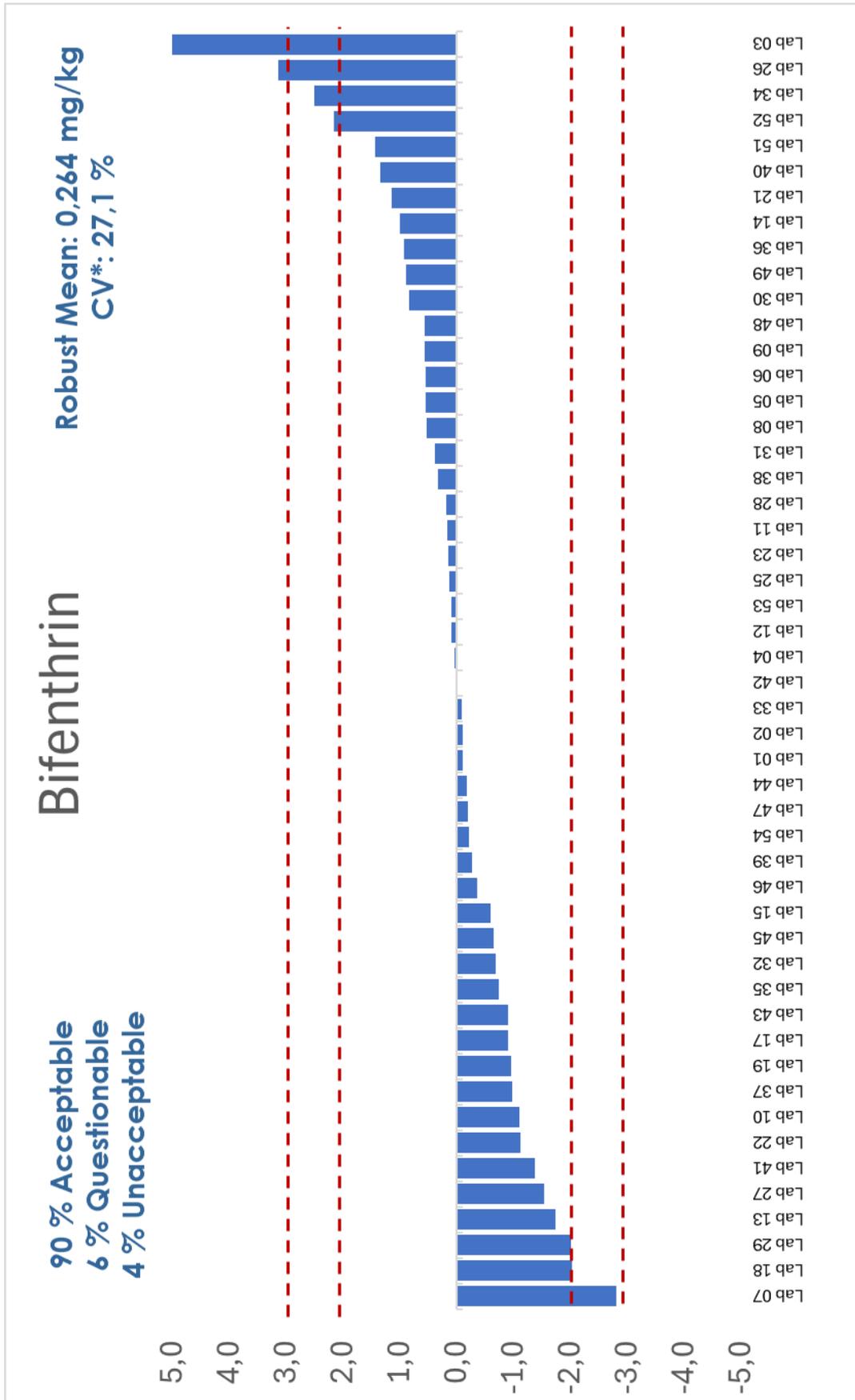
Lab Code	Thiamethoxam	z score (FFP-RSD 25 %)
MRRL (mg/kg)	0.01	
Robust mean (mg/kg)	0.082	
Lab 28	NA	
Lab 29	0.087	0.3
Lab 30	0.090	0.4
Lab 31	0.074	-0.4
Lab 32	0.119	1.8
Lab 33	0.075	-0.3
Lab 34	0.106	1.2
Lab 35	NA	
Lab 36	0.074	-0.4
Lab 37	NA	
Lab 38	0.101	0.9
Lab 39	0.053	-1.4
Lab 40	0.092	0.5
Lab 41	0.076	-0.3
Lab 42	0.092	0.5
Lab 43	NA	
Lab 44	0.074	-0.4
Lab 45	0.062	-1.0
Lab 46	0.120	1.9
Lab 47	0.081	0.0
Lab 48	ND	-4.0
Lab 49	0.083	0.1
Lab 51	0.116	1.7
Lab 52	0.108	1.3
Lab 53	0.063	-0.9
Lab 54	0.086	0.2

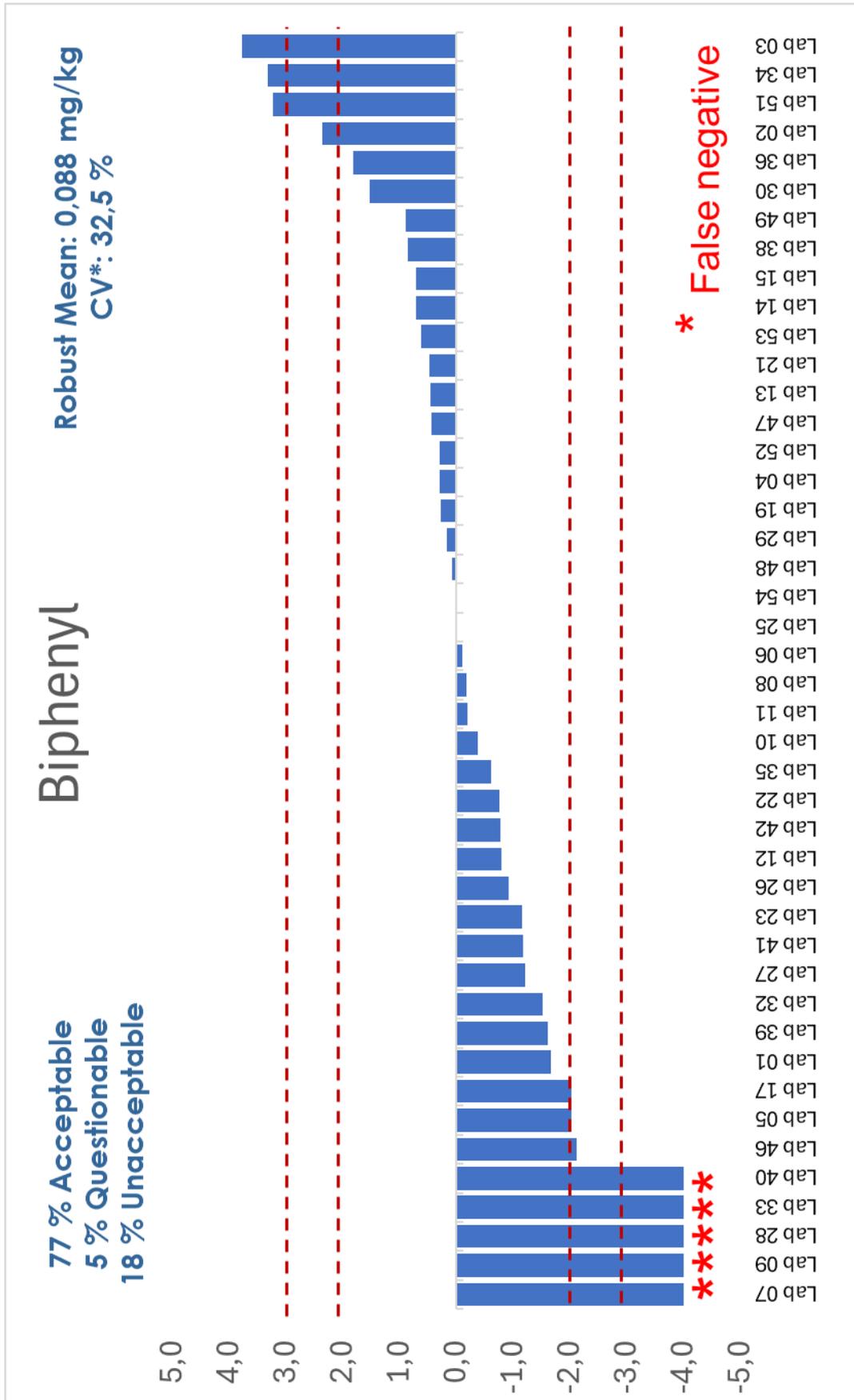
ND: Not detected (False negative)

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

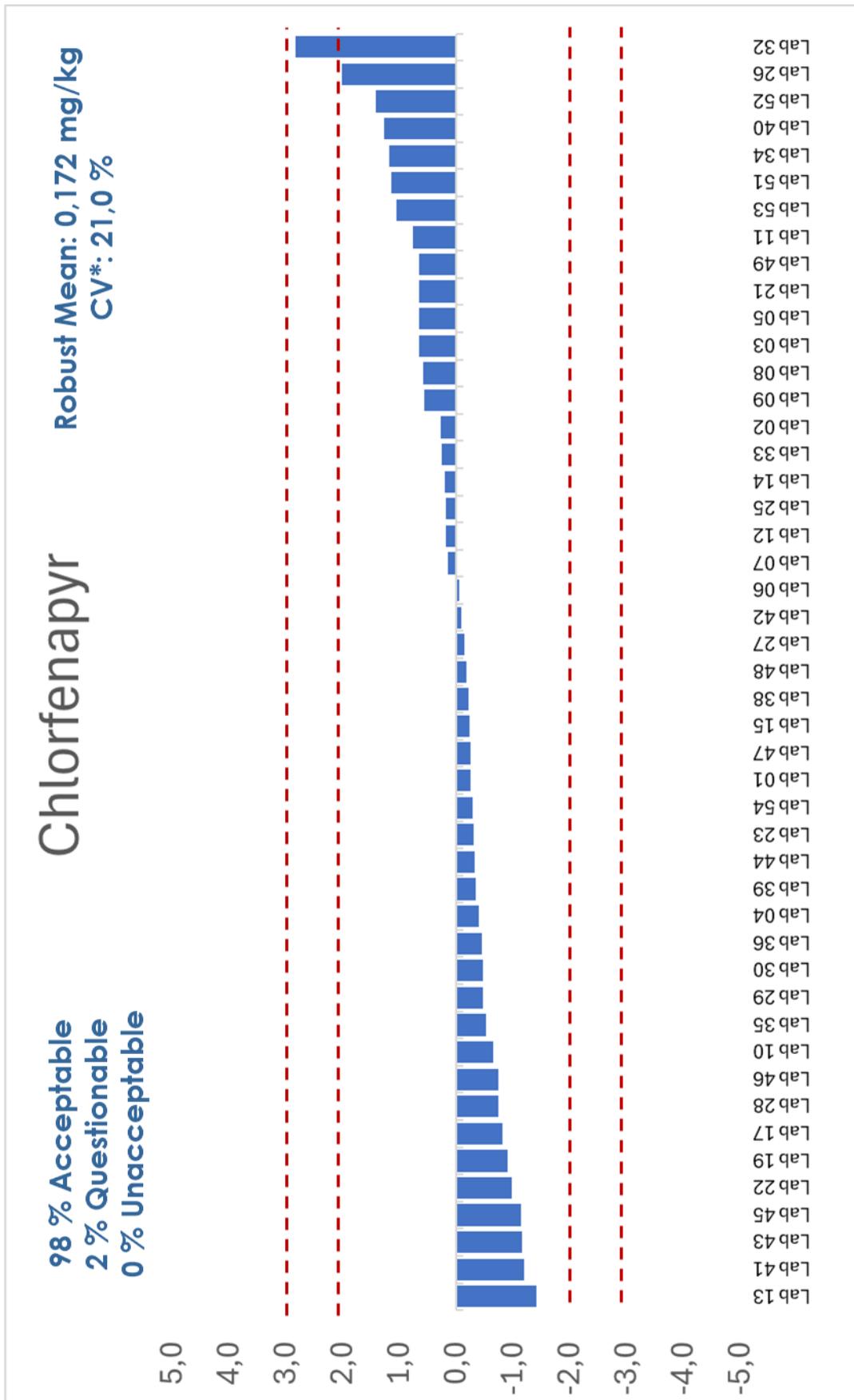


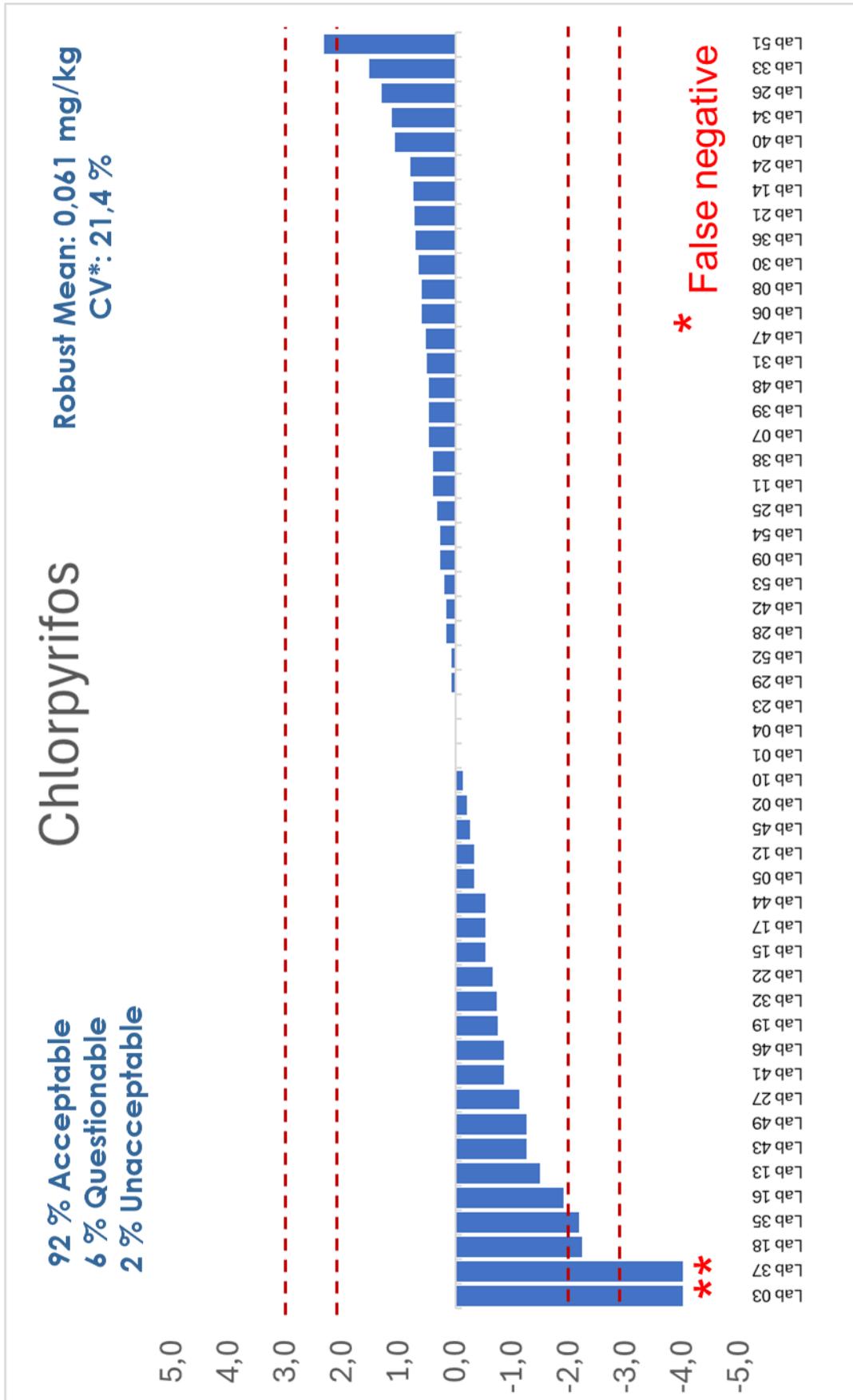
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %)

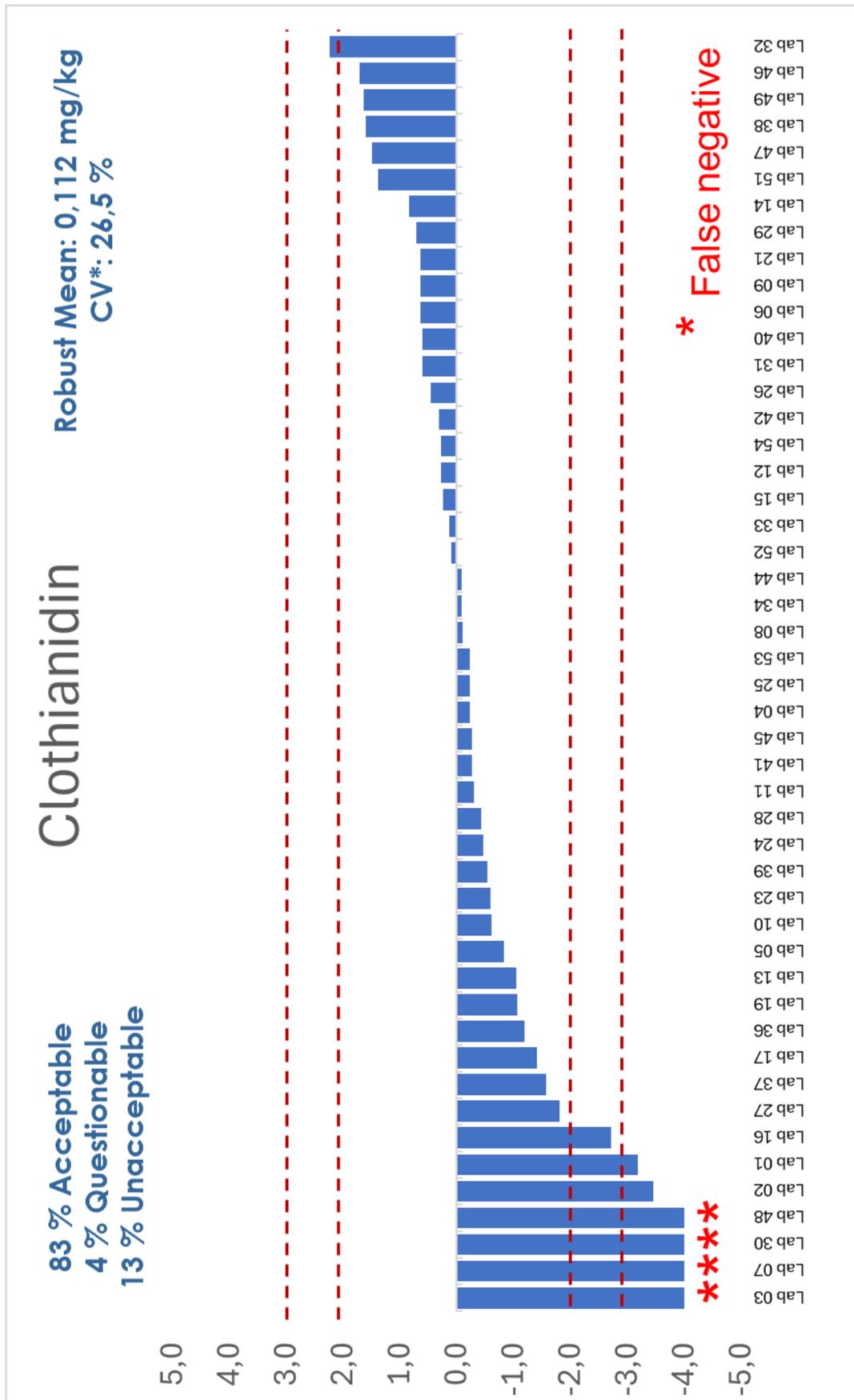




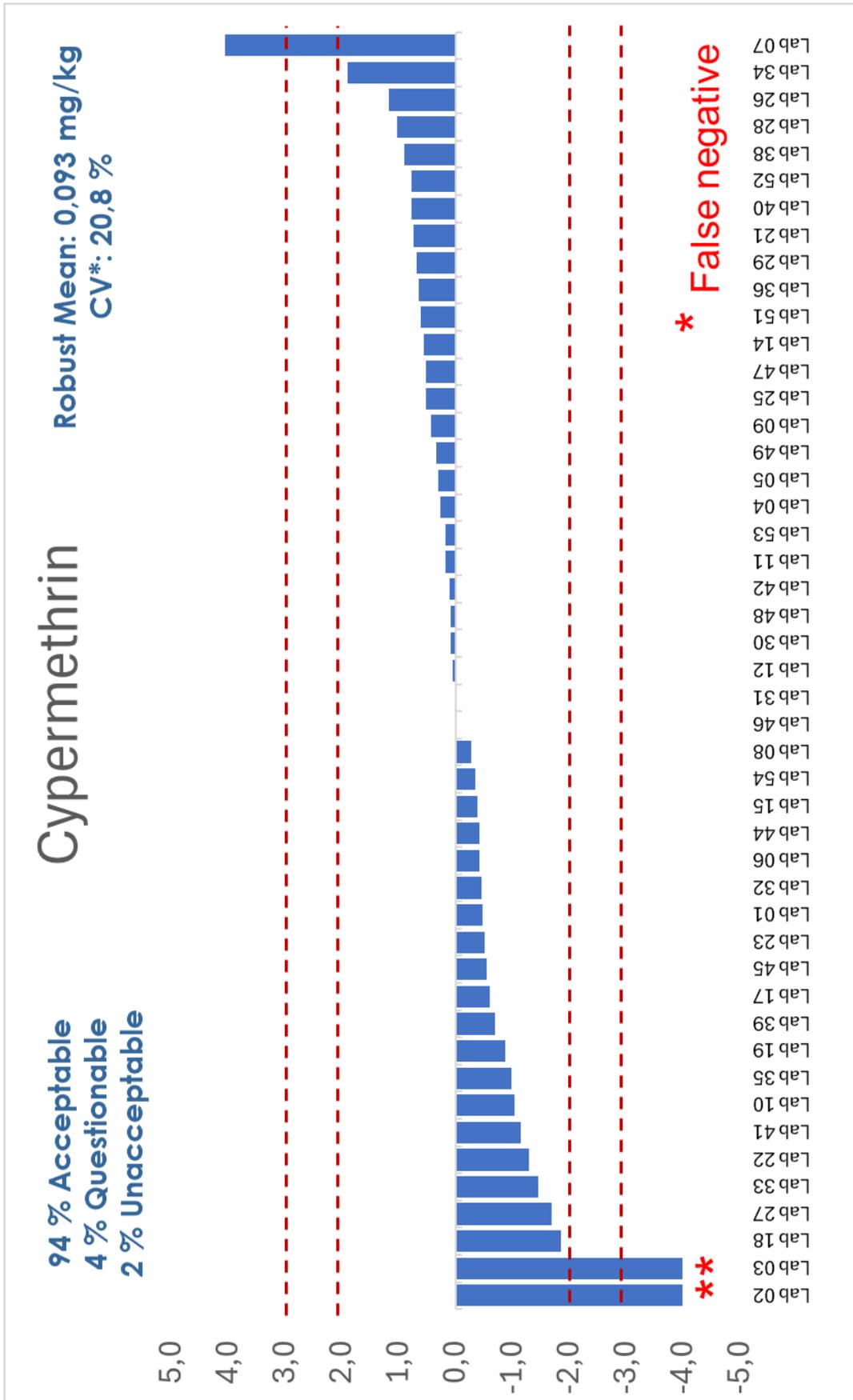
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %)

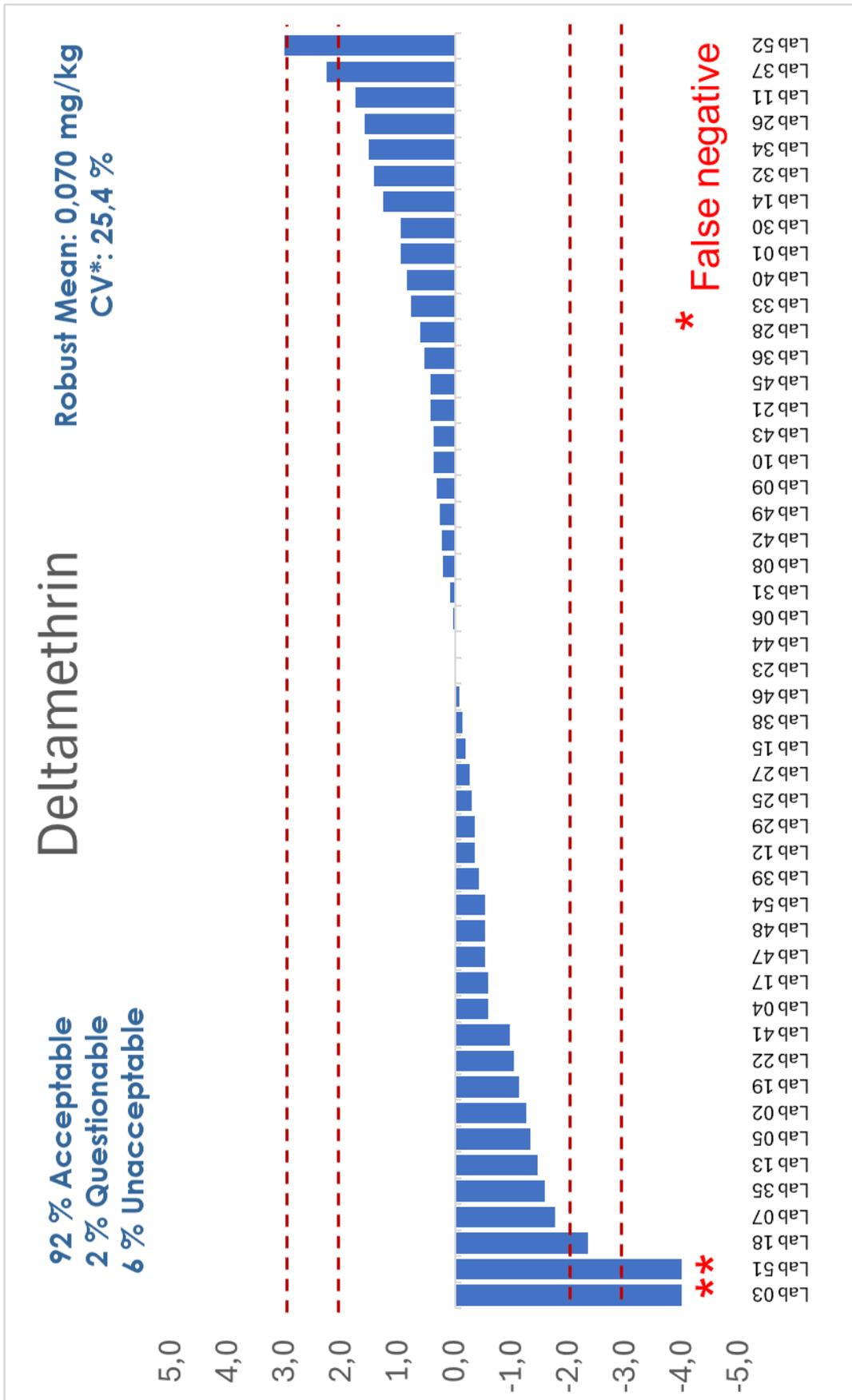




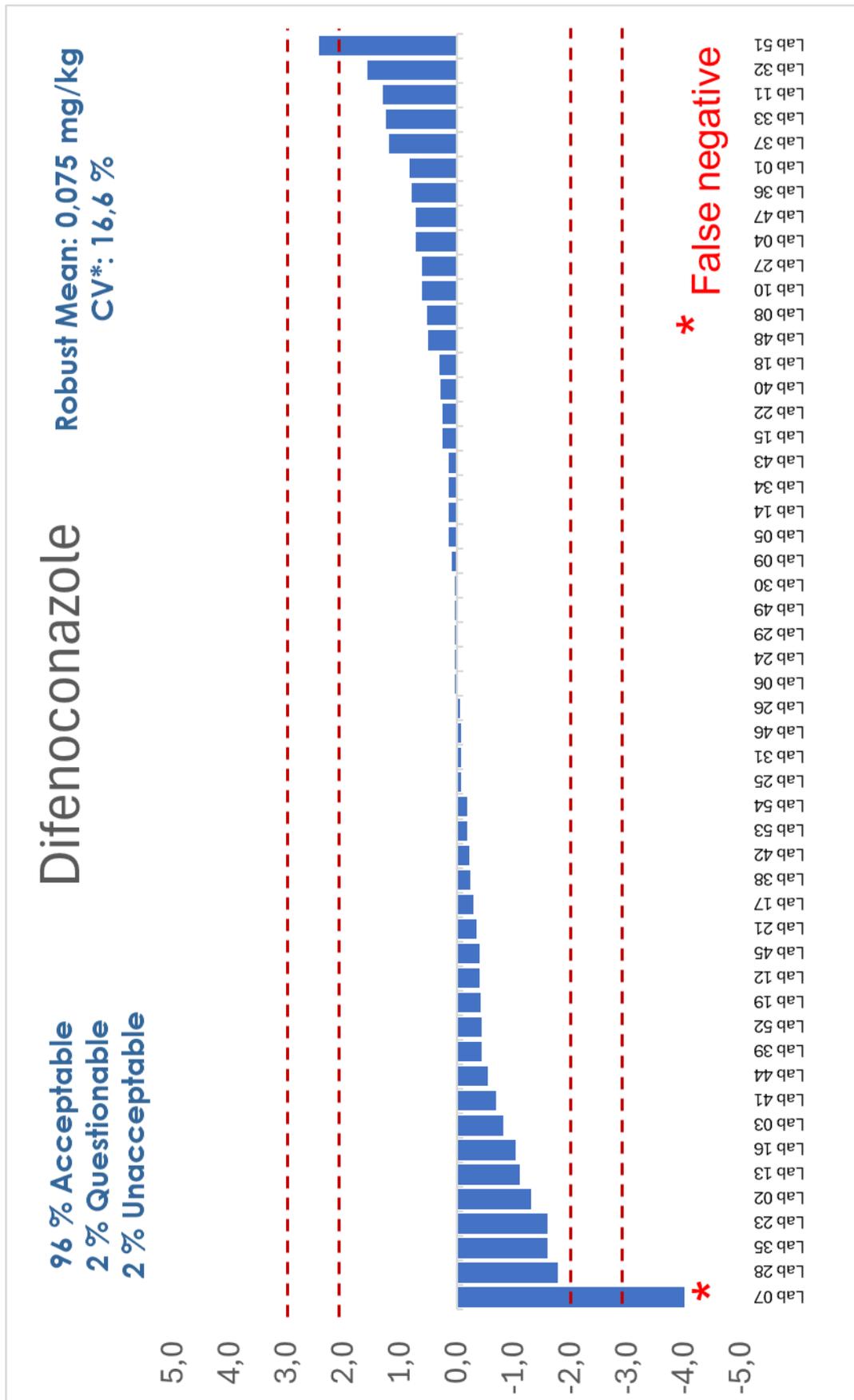


APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %)

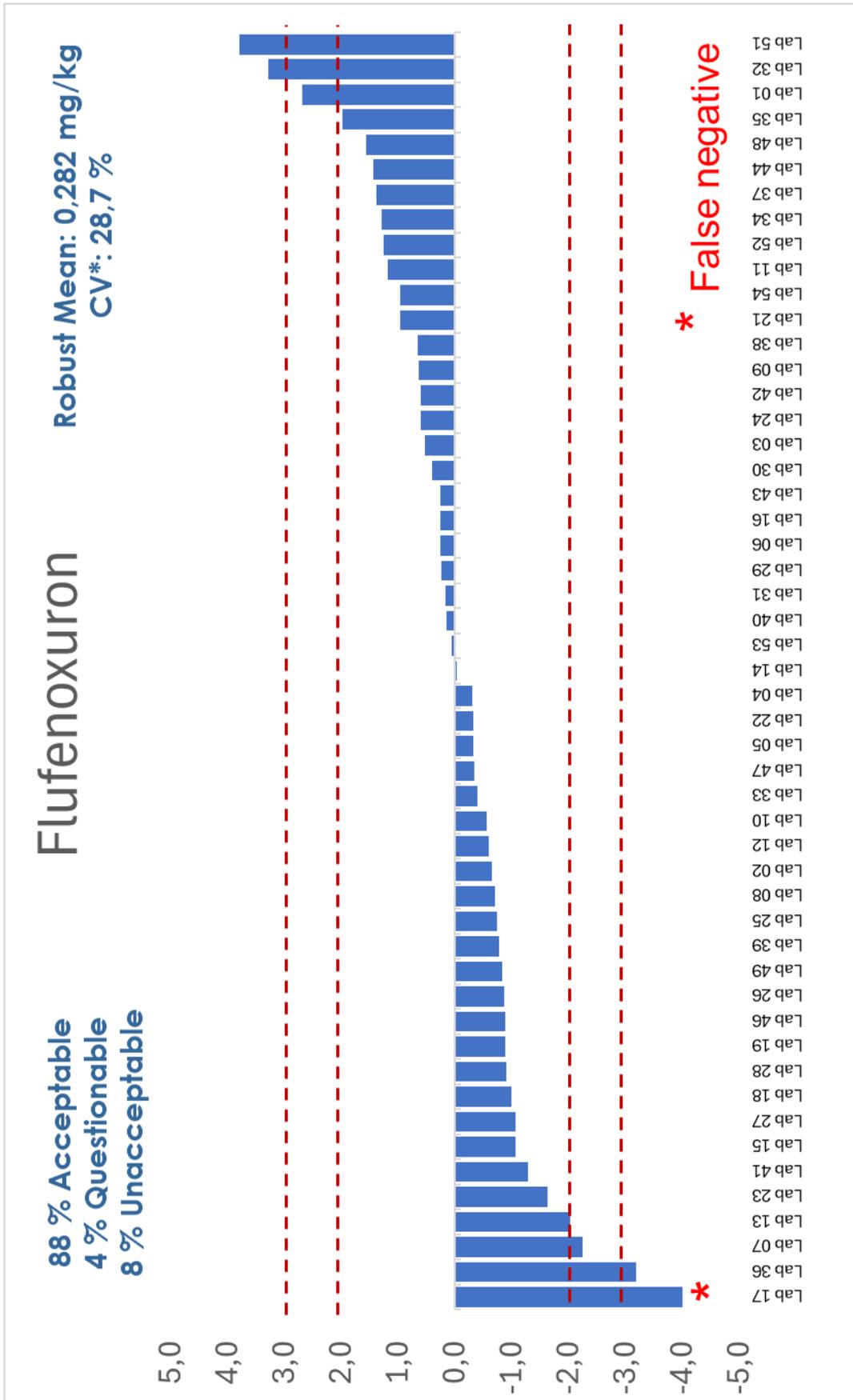




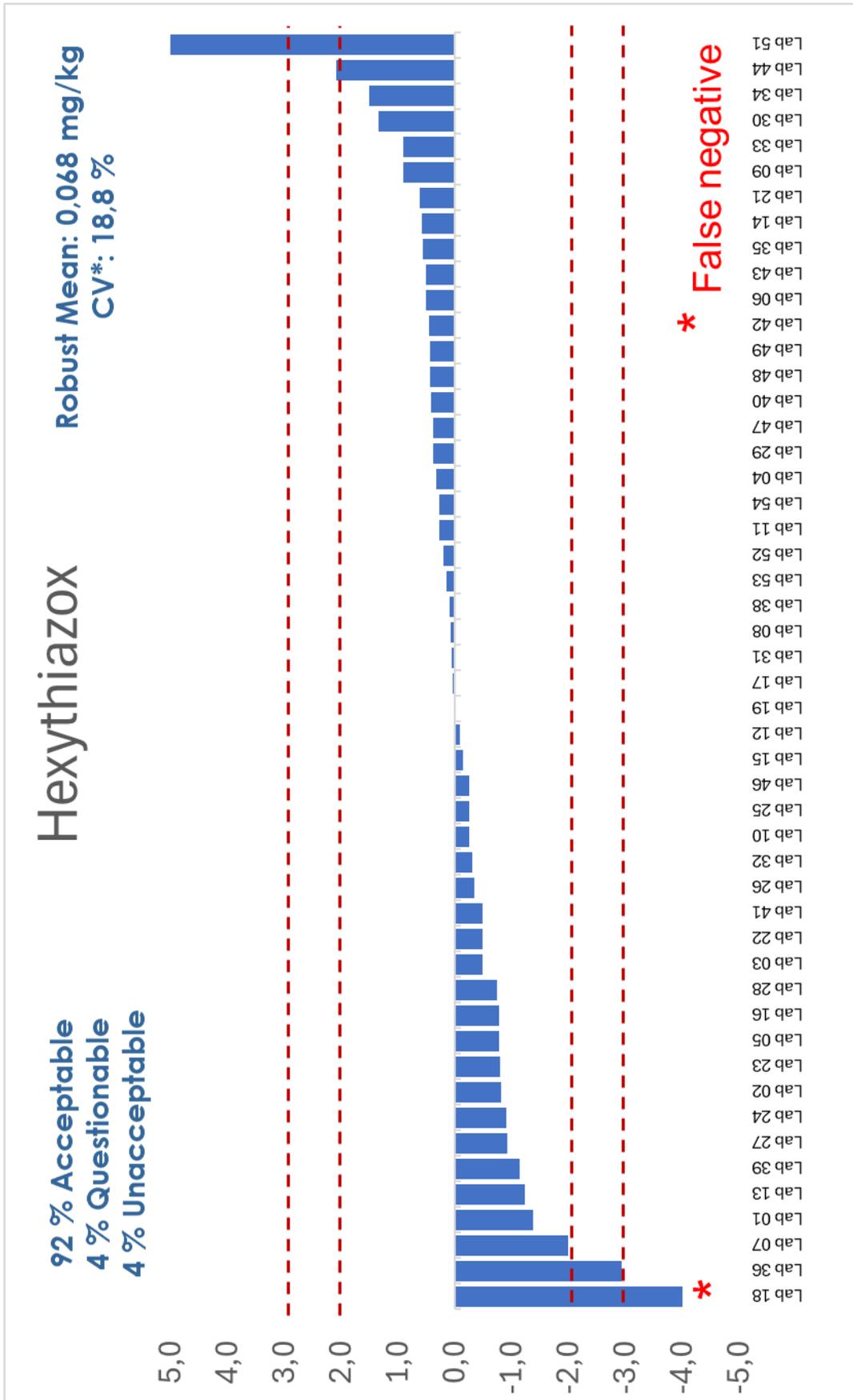
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %)



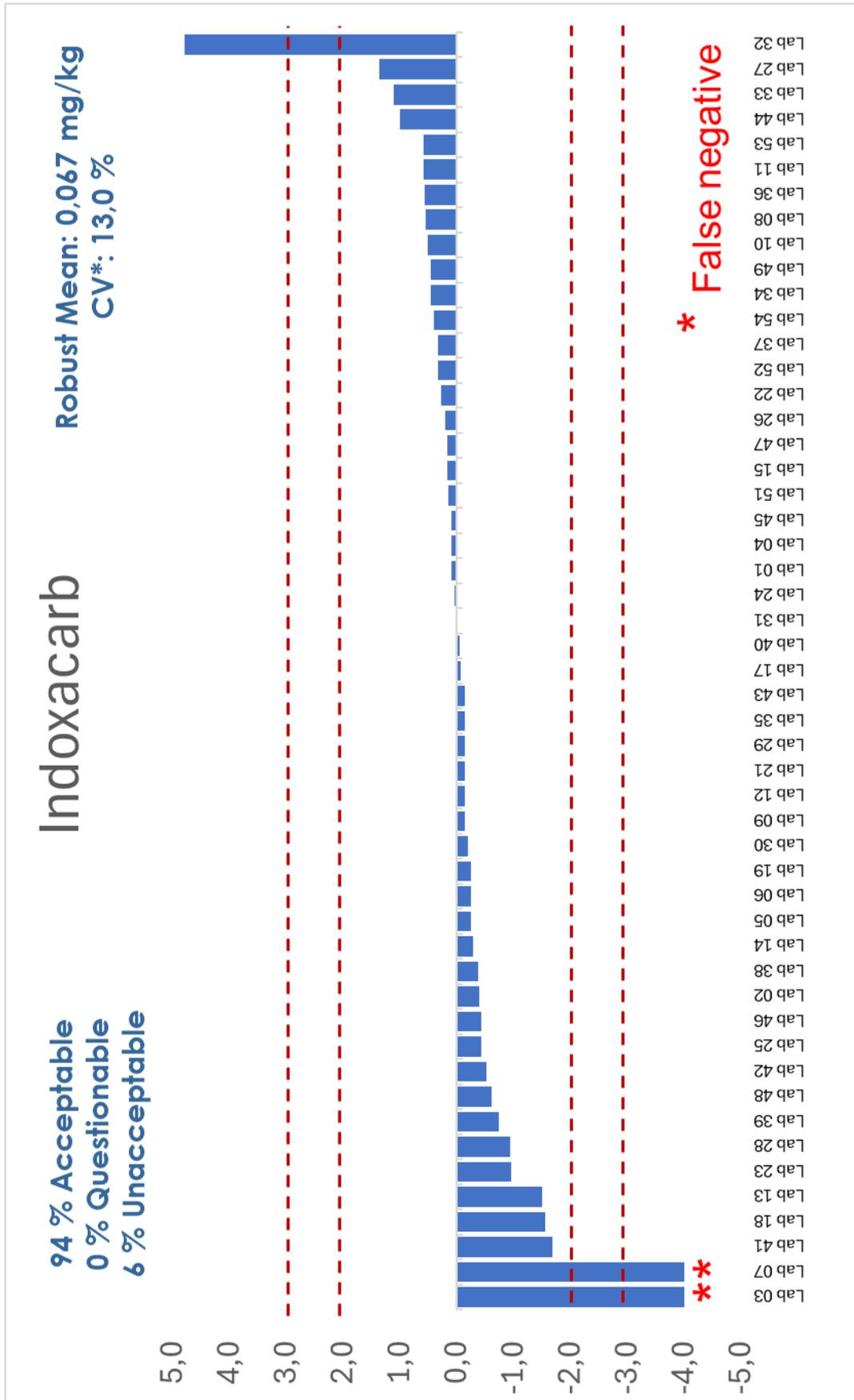
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %)



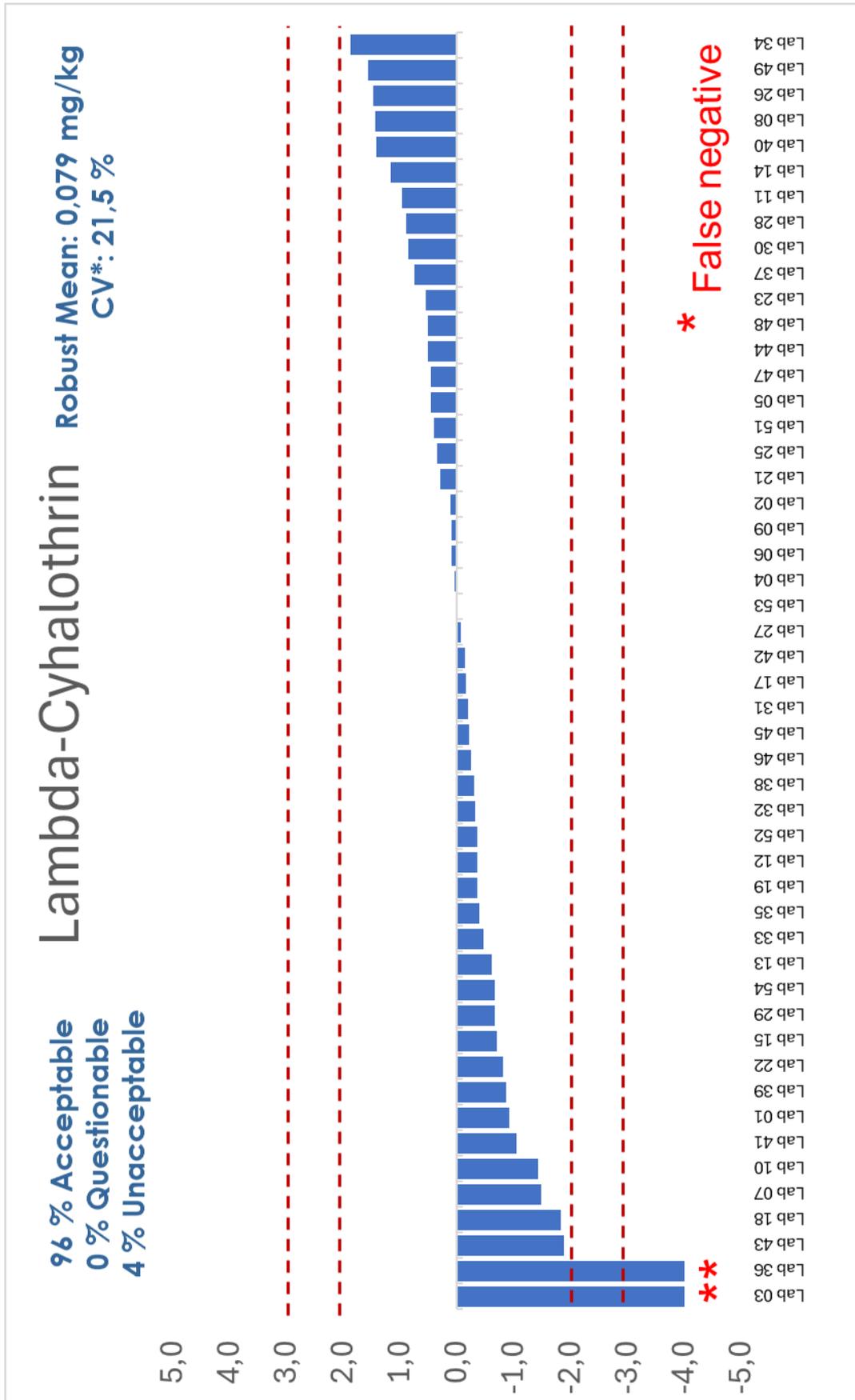
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %)



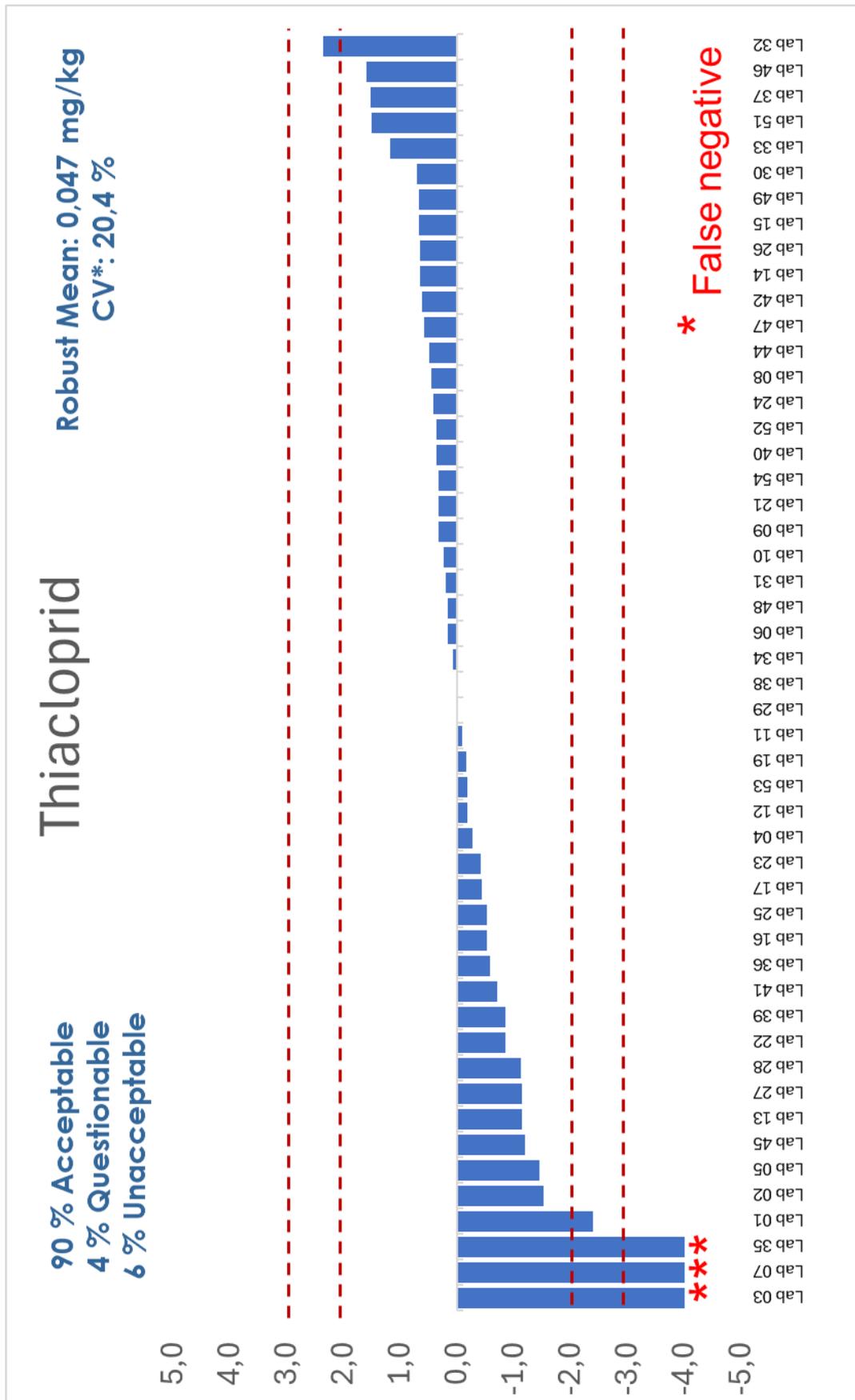
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %)



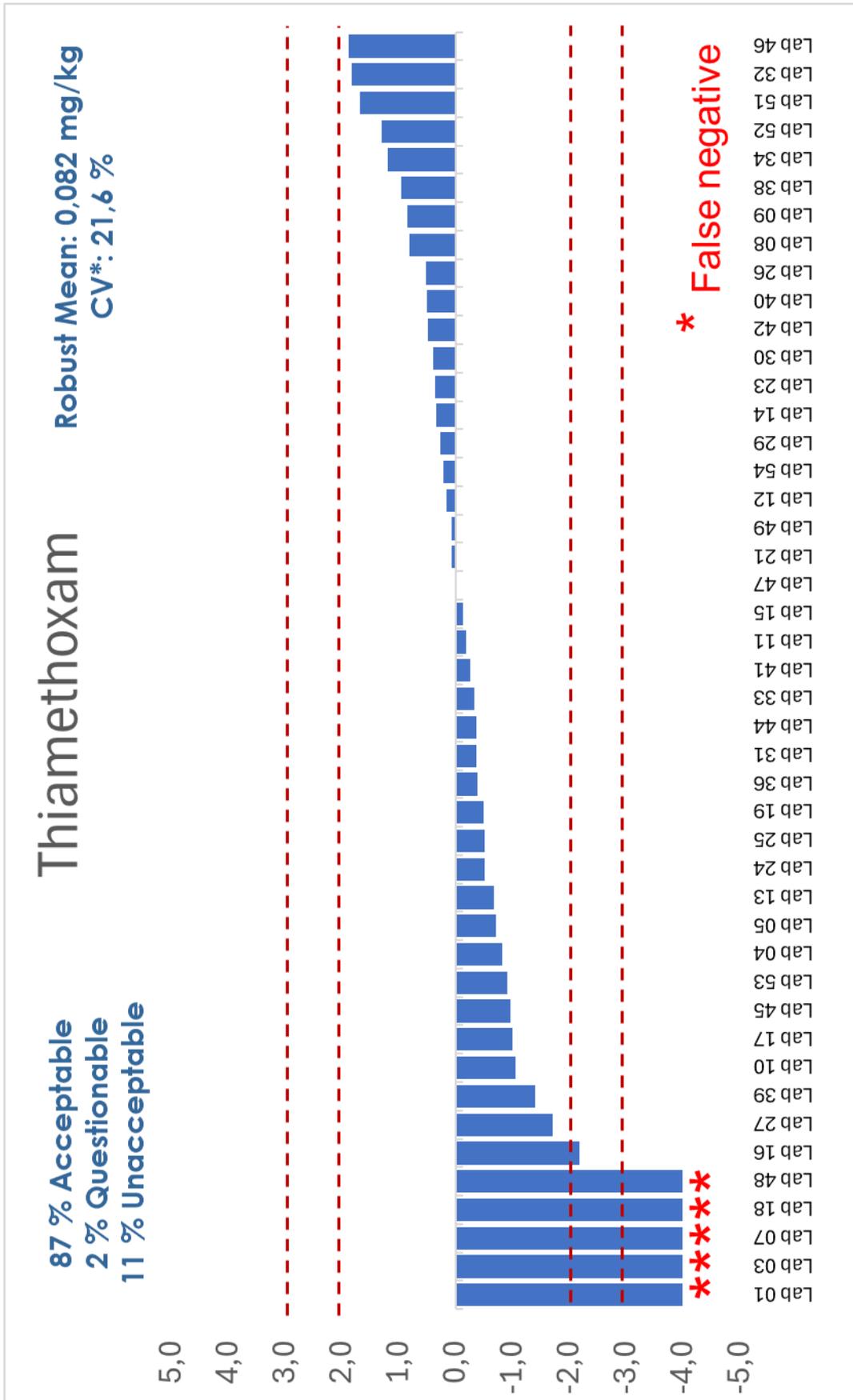
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %)



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

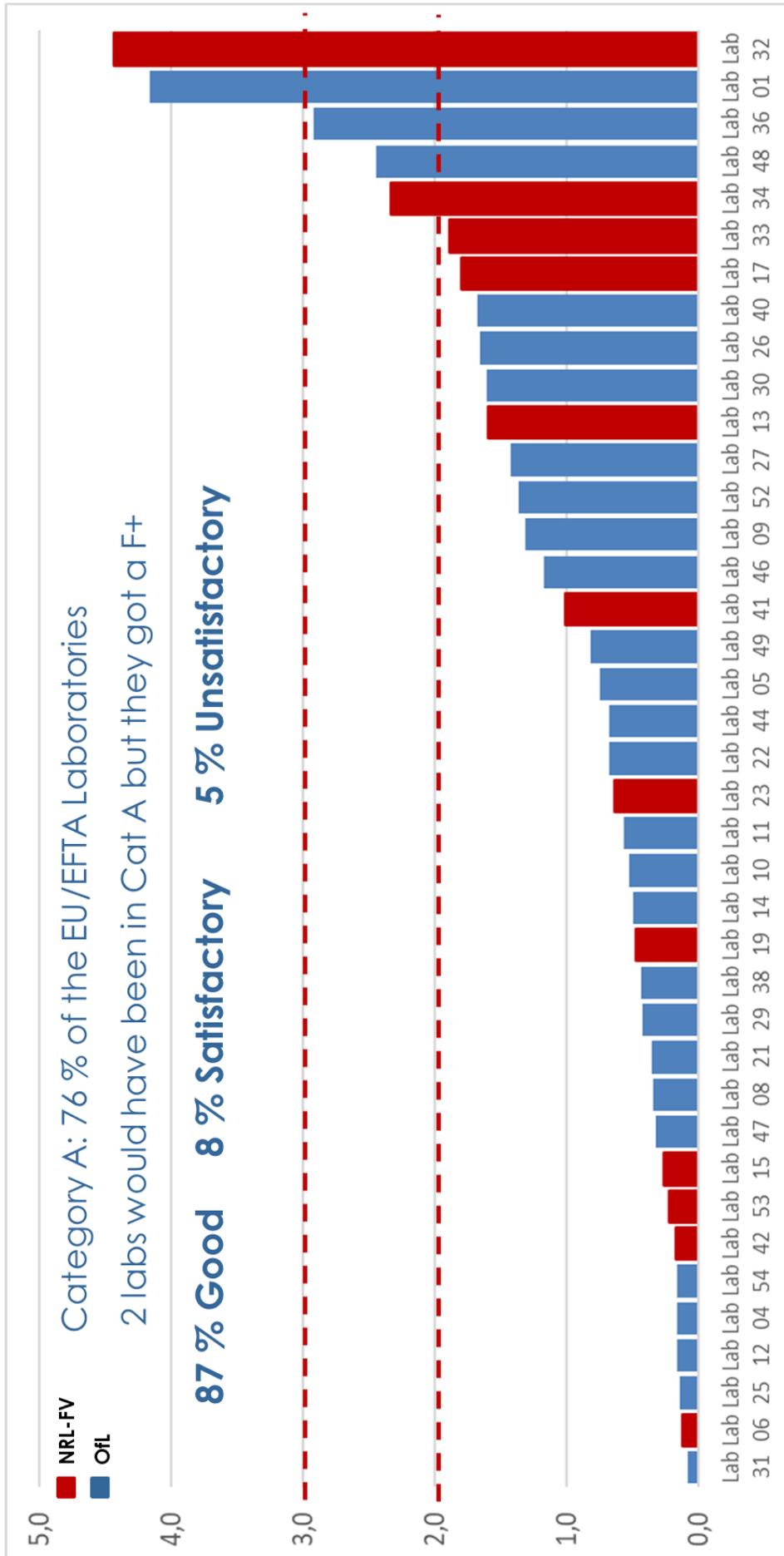


APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %)



APPENDIX 4. Average of the Squared z scores (AZ2) for laboratories in Category A.

Laboratory	Acetamidrid	Bifenthrin	Biphenyl	Chlorfenapyr	Chlorpyrifos	Clothianidin	Cypermethrin	Deltamethrin	Difenoconazole	Flufenoxuron	Hexythiazox	Indoxacarb	Lambda-Cyhalothrin	Thiacloprid	Thiamethoxam	No. of z scores	AZ ²
Lab 01	-4.0	-0.1	-1.7	-0.3	0.0	-3.2	-0.5	0.9	0.8	2.7	-1.4	0.1	-0.9	-2.4	-4.0	15	4.2
Lab 04	-0.4	0.0	0.3	-0.4	0.0	-0.2	0.3	-0.6	0.7	-0.3	0.3	0.1	0.0	-0.3	-0.8	15	0.2
Lab 05	-0.2	0.5	-2.0	0.7	-0.3	-0.8	0.3	-1.3	0.1	-0.3	-0.8	-0.3	0.5	-1.5	-0.7	15	0.7
Lab 06	0.1	0.5	-0.1	0.0	0.6	0.6	-0.4	0.0	0.0	0.3	0.5	-0.3	0.1	0.2		14	0.1
Lab 08	-0.2	0.5	-0.2	0.6	0.6	-0.1	-0.3	0.2	0.5	-0.7	0.1	0.5	1.4	0.5	0.8	15	0.3
Lab 09	0.4	0.6	-4.0	0.6	0.3	0.6	0.4	0.3	0.1	0.6	0.9	-0.1	0.1	0.3	0.8	15	1.3
Lab 10	-0.5	-1.1	-0.4	-0.7	-0.1	-0.6	-1.0	0.4	0.6	-0.6	-0.3	0.5	-1.4	0.2	-1.1	15	0.5
Lab 11	0.2	0.2	-0.2	0.8	0.4	-0.3	0.2	1.7	1.3	1.2	0.3	0.6	1.0	-0.1	-0.2	15	0.6
Lab 12	0.8	0.1	-0.8	0.2	-0.3	0.3	0.0	-0.4	-0.4	-0.6	-0.1	-0.1	-0.4	-0.2	0.2	15	0.2
Lab 13	-0.7	-1.7	0.5	-1.4	-1.5	-1.0		-1.5	-1.1	-2.0	-1.2	-1.5	-0.6	-1.1	-0.7	14	1.6
Lab 14	0.6	1.0	0.7	0.2	0.7	0.8	0.6	1.3	0.1	0.0	0.6	-0.3	1.2	0.7	0.3	15	0.5
Lab 15	0.5	-0.6	0.7	-0.2	-0.5	0.2	-0.4	-0.2	0.3	-1.1	-0.1	0.2	-0.7	0.7	-0.1	15	0.3
Lab 17	-1.0	-0.9	-2.0	-0.8	-0.5	-1.4	-0.6	-0.6	-0.3	-4.0	0.0	-0.1	-0.2	-0.4	-1.0	15	1.8
Lab 19	-0.4	-1.0	0.3	-0.9	-0.7	-1.1	-0.9	-1.1	-0.4	-0.9	0.0	-0.3	-0.4	-0.2	-0.5	15	0.5
Lab 21	-0.1	1.1	0.5	0.7	0.7	0.6	0.7	0.4	-0.3	1.0	0.6	-0.1	0.3	0.3	0.1	15	0.4
Lab 22	-1.0	-1.1	-0.8	-1.0	-0.6		-1.3	-1.0	0.3	-0.3	-0.5	0.3	-0.8	-0.9		13	0.7
Lab 23	-0.4	0.1	-1.2	-0.3	0.0	-0.6	-0.5	0.0	-1.6	-1.6	-0.8	-1.0	0.6	-0.4	0.4	15	0.6
Lab 25	-0.1	0.1	0.0	0.2	0.3	-0.2	0.5	-0.3	-0.1	-0.7	-0.3	-0.4	0.4	-0.5	-0.5	15	0.1
Lab 26	0.6	3.1	-0.9	2.0	1.3	0.5	1.2	1.6	-0.1	-0.9	-0.3	0.2	1.5	0.7	0.5	15	1.7
Lab 27	-1.3	-1.5	-1.2	-0.1	-1.1	-1.8	-1.7	-0.3	0.6	-1.1	-0.9	1.4	-0.1	-1.1	-1.7	15	1.4
Lab 29	-0.5	-2.0	0.2	-0.5	0.1	0.7	0.7	-0.4	0.0	0.2	0.4	-0.1	-0.7	0.0	0.3	15	0.4
Lab 30	0.0	0.8	1.5	-0.5	0.7	-4.0	0.1	1.0	0.0	0.4	1.4	-0.2	0.9	0.7	0.4	15	1.6
Lab 31	-0.1	0.4			0.5	0.6	0.0	0.1	-0.1	0.2	0.1	0.0	-0.2	0.2	-0.4	13	0.1
Lab 32	1.6	-0.7	-1.5	2.8	-0.7	2.2	-0.5	1.4	1.6	3.3	-0.3	4.8	-0.3	2.4	1.8	15	4.4
Lab 33	1.3	-0.1	-4.0	0.3	1.5	0.1	-1.5	0.8	1.3	-0.4	0.9	1.1	-0.5	1.2	-0.3	15	1.9
Lab 34	0.1	2.5	3.3	1.2	1.1	-0.1	1.9	1.5	0.1	1.3	1.5	0.5	1.9	0.1	1.2	15	2.3
Lab 36	-0.8	0.9	1.8	-0.4	0.7	-1.2	0.6	0.5	0.8	-3.2	-2.9	0.6	-4.0	-0.6	-0.4	15	2.9
Lab 38	0.7	0.3	0.8	-0.2	0.4	1.6	0.9	-0.1	-0.2	0.7	0.1	-0.4	-0.3	0.0	0.9	15	0.4
Lab 39	-0.4	-0.3	-1.6	-0.3	0.5	-0.5	-0.7	-0.4	-0.4	-0.8	-1.1	-0.7	-0.9	-0.9	-1.4	15	0.7
Lab 40	0.5	1.3	-4.0	1.3	1.1	0.6	0.8	0.8	0.3	0.2	0.4	0.0	1.4	0.4	0.5	15	1.7
Lab 41	-0.7	-1.4	-1.2	-1.2	-0.9	-0.3	-1.1	-1.0	-0.7	-1.3	-0.5	-1.7	-1.0	-0.7	-0.3	15	1.0
Lab 42	0.4	0.0	-0.8	-0.1	0.2	0.3	0.1	0.2	-0.2	0.6	0.5	-0.5	-0.1	0.6	0.5	15	0.2
Lab 44	0.7	-0.2		-0.3	-0.5	-0.1	-0.4	0.0	-0.5	1.4	2.1	1.0	0.5	0.5	-0.4	14	0.7
Lab 46	1.3	-0.4	-2.1	-0.7	-0.8	1.7	0.0	-0.1	-0.1	-0.9	-0.3	-0.4	-0.3	1.6	1.9	15	1.2
Lab 47	0.4	-0.2	0.4	-0.3	0.5	1.5	0.5	-0.5	0.7	-0.3	0.4	0.2	0.5	0.6	0.0	15	0.3
Lab 48	0.3	0.6	0.1	-0.2	0.5	-4.0	0.1	-0.5	0.5	1.6	0.5	-0.6	0.5	0.2	-4.0	15	2.4
Lab 49	1.3	0.9	0.9	0.7	-1.2	1.6	0.3	0.3	0.0	-0.8	0.5	0.5	1.6	0.7	0.1	15	0.8
Lab 52	0.4	2.2	0.3	1.4	0.1	0.1	0.8	3.0	-0.4	1.3	0.2	0.3	-0.4	0.4	1.3	15	1.4
Lab 53	-0.5	0.1	0.6	1.0	0.2	-0.2	0.2		-0.2	0.1	0.2	0.6	0.0	-0.2	-0.9	14	0.2
Lab 54	0.1	-0.2	0.0	-0.3	0.3	0.3	-0.3	-0.5	-0.2	1.0	0.3	0.4	-0.7	0.3	0.2	15	0.2



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 Testings (EUPTs) organised on behalf of the European Commission, DG-SANTE¹ by the four European Union Reference Laboratories (EURLs) responsible for the area of pesticide residues analysis in food and feed. These EUPTs are organised for laboratories belonging to the Network² 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. 2017/625³:

- 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 programme⁴. Participating laboratories will be provided with an assessment of their analytical performance that they can use to demonstrate their (ongoing) analytical proficiency and compare themselves with other participating laboratories. By pointing out areas of analytical deficiencies, EUPTs contribute to the continuous improvement of the analytical quality of OfLs, thus helping to increase the confidence on the results generated by them.

EUPT-Organisers and Scientific Committee

EUPTs are organised either by single EURLs, or collaboratively by more than one EURL.

An **organising team** (in the following named **organisers**⁵) is appointed by the EURL(s) in charge of a given PT. The organisers are in charge of all administrative and technical PT activities of a proficiency testing (PT) round. These tasks include the PT-announcement, the production of the proficiency testing item (PT-item), the undertaking of homogeneity and stability assessments, the portioning, packing and shipment of the PT-items, 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 the 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)⁶ 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 latest 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).

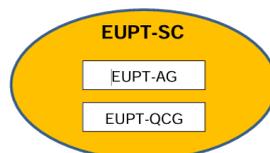


Figure 1: Composition of EUPT-Scientific Committee

The EUPT-SC's role is to assist the organisers during the planning and the data evaluation phase of a PT-round. Input from the EUPT-SC is requested, when it comes to e.g. selecting the commodities for the EUPTs of the following season, selecting the analytes to be included in the Target Pesticides List (p. 8), establishing the Minimum Required Reporting Levels (MRRLs) for each of the analytes, and statistically evaluating the participants' results (in anonymous form). The EUPT-SC is furthermore consulted when it comes to drafting and updating documents, such as the General and Specific PT Protocols and the Final EUPT-Reports.

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 Testings for Pesticide Residues in Fruits and Vegetables, Trends in Analytical Chemistry, 2010, 29 (1), 70 – 83.

⁵ The term organisers is to be considered equivalent to the term PT-provider in ISO 17043:2023

⁶ Link to the List of current members of the EUPT Scientific Committee: <http://www.eurl-pesticides.eu/library/docs/allcrl/EUPT-SC.pdf>

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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 PT 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 preliminary evaluation of 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.

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 EUPT-GP versions](#). The latest version of the EUPT-GP is highlighted.

EUPT Participants – Eligibility and Obligation for Participation

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 (2) 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 feed⁷)
- 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 to 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 obliged laboratories with their current 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, but are not able to participate, must provide the reasons for their non-participation. This also applies to any participating laboratories failing to report results.

EUPTs are furthermore open to the following laboratories as long as sufficient material is available:

- a) any other OfLs from EU countries that are not covered by the above obligations to participate;
- b) NRLs and OfLs from EU-candidate countries and EFTA countries;
- c) other laboratories from EU or EFTA countries analysing official organic samples within the frame of Reg. 889/2008/EC;
- d) governmental laboratories from Third Countries (countries outside EU)
- e) other laboratories from Third Countries as long as they are involved in controls of products destined for export to the EU.

Note on a): Laboratories having been designated as OfLs, according to Art. 37(2)(b) of Regulation (EU) No. 2017/625³ by a Competent Authority of an EU Member State (MS1) will normally also need to be commissioned with OfL activities in a different EU Member State (MS2) for being eligible for participation. Scan-copies of documents giving information about the period and scope of these OfL activities for MS2 may be requested by the EUPT organizers. The responsible NRL and/or Competent Authority of MS2 may be contacted before deciding whether the laboratory in question is eligible or even obliged to participate in a certain PT. A laboratory whose OfL-appointment in the area of pesticide residue analysis has ceased, will normally lose its eligibility (and obligation) to participate in EUPTs, but participation may be allowed if the responsible NRL and/or Competent Authority of MS1 or MS2 considers its participation essential for judging the proficiency in view of a planned or potential OfL activity in the future.

Laboratories of groups c) and e) will be requested to provide a proof of their function (e.g. scan copy of a document stating official appointment).

Obligation of OfLs and NRLs to double-check Status of EUPT-Participation:

Based on the latest information within the DataPool and considering the selected commodities of the upcoming EUPTs, the OfLs (including the NRLs) are grouped into those for which participation in a given EUPT is obligatory and those for which participation is voluntary ("OV-grouping").

Upon accessing the EUPT Registration Form within the EURL-DataPool, laboratories can choose the EUPTs they would like to participate and view their OV-grouping status for each of the selected PTs. If a laboratory does not agree with its OV-Grouping, it should promptly contact the corresponding NRL and the EUPT-organisers and give the reasons why it believes it should be grouped differently. The reasons provided by the laboratories will be noted by the organisers and if indicated, the DataPool will be updated accordingly. In any case, the OV-grouping prepared by the EURLs is indicative only, as the real obligation to participate in a given EUPT arises from the abovementioned EU-regulations, not the DataPool entries or any lab's claims. Additional requirements may arise from accreditation bodies or local rules and regulations.

Within the DataPool, NRLs have the possibility to view data relevant to OfLs within their network (OV- grouping, registration progress) and are responsible for checking whether the OV- grouping of all relevant OfLs within their network is correct.

⁷ 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).

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

OfLs that are obliged but not able to participate in a given EUPT must provide the reasons for their non-participation. This also applies to any participating laboratories that fail to submit PT-results.

Participation fee and Invoicing

By completing the registration for participation in a given EUPT, a laboratory agrees to proceed with a timely payment of the participation fee after being accepted for participation and after the invoice issued by the organiser is received. The invoice fee covers the costs of production, handling and delivery of the PT-materials. The organisers will issue digital invoices in PDF format only, and without any electronic signature. By registering to an EUPT the laboratories also accept that the pdf invoice, issued by the organisers and sent via e-mail to the participant, is sufficient for triggering the payment of the participation fee. The EURLs retain the right to decline any request for supplementary forms or additional paperwork in connection to the payment. The laboratories should note that additional costs may incur if such extra services are requested, depending on the incurring extra workload. Extra costs may also incur if new modified invoice is requested, e.g. because of missing or erroneous information caused by errors or omissions by the registered laboratory during registration. OfLs not paying the EUPT participation fee will be initially reminded, and then warned that information concerning their laboratory may be blacked out in the final report of the concerned EUPT and the certificate of participation may not be issued to them, and that their participation in subsequent EUPTs could be denied. In case of a repetitive non-payment, the EUPT organisers may inform the corresponding NRL and/or the competent authority responsible for the OfL.

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 laboratory 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

Approximately 3 months before the distribution of the PT items to the participants 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 for preparing the PT item, as well as links to the tentative EUPT-Target Pesticides List and the tentative EUPT-Calendar.

Target Pesticides List and PT-Residue Definitions

The Target Pesticides List contains all analytes (pesticides and metabolites) to be sought for, along with the Minimum Required Reporting Levels (MRRs) valid for the specific EUPT. The MRRs are typically based upon the lowest MRLs found either in Regulation (EC) No. 2005/396 or in Regulation (EU) No. 2016/128 (Baby Food Directive).

The residue definition in an EUPT may differ from the legal one if this is deemed necessary by the organisers for ensuring a better evaluation of the results. Participants must express their results as defined in the Target Pesticides List of the respective EUPT. Separately quantifiable analytes are typically listed separately unless stated otherwise.

Specific Protocol

For each EUPT, the organising EURL will publish a Specific Protocol at least 2 weeks before the PT 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 PT item upon receipt and on how to submit results, as well as other relevant information.

Assessing the Homogeneity of the PT Item

A suitable homogeneity of the EUPT item is of high importance as it ensures that portion-to-portion variability has only a negligible impact on the evaluation of the participant's performance. The PT item is tested for homogeneity, typically after bottling and before distribution to participants, but in justifiable cases the tests for homogeneity assessment may also be conducted after the distribution of the material to the participants⁸. The homogeneity assessment usually involves analysis of two replicate analytical portions, taken from at least ten randomly chosen units (bottled portions) of treated PT item. Measurements should be conducted in random

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

order with the aim of minimizing the risk of misinterpreting signal drifts within a measurement sequence as concentration shifts linked to the bottle numbering, i.e. the order of the bottle filling. The homogeneity test data are statistically evaluated according to ISO 13528:2022, Annex B⁹ or to the International Harmonized Protocols jointly published by ISO, AOAC and IUPAC¹⁰. The results of all homogeneity assessment are presented to the EUPT-SC. In special cases, where the above 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 tests, and 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 simultaneously. The homogeneity test of one or more of these analytes may thus be skipped or simplified. The organisers should keep an eye on the participants' results of such analytes not tested for homogeneity in order to detect at an early stage any signs that could raise doubts about the homogeneity of the material (e.g. an atypically broad distribution of the results compared to other analytes). In such a case, the EUPT-SC may decide that a proper homogeneity assessment should still be performed to clarify the situation.

Assessing the Stability of the Analytes Contained in the PT Item

The PT item will also be tested for stability - according to ISO 13528:2022, Annex B⁹. The time delay between the first and the last stability test (stability assessment period) must exceed the period of the EUPT-exercise. Typically, the first analysis is carried out shortly before the shipment of the PT items and the last one shortly after the deadline for submission of results. If justifiable, the stability assessment period may precede the PT period, partly overlap with it or postdate it. Close proximity to the PT-period is to be favoured, however, to minimize the risk that matrix properties alter in a way that will affect analyte stability. To better recognise trends and gain additional certainty, one or more additional tests may be conducted by the organisers in the interim. At least 6 sub-samples (analytical portions) should be analysed on each test day (e.g. 2 analytical portions withdrawn from three randomly chosen containers OR 6 portions withdrawn from a single container). In principle, all analytes contained in the PT item should be checked for stability. However, in individual cases, where sufficient knowledge exists that the stability of a certain analyte is very unlikely to be significantly affected during storage (e.g. based on experience from past stability tests or knowledge of its physicochemical properties), the organisers, after consultation with the EUPT-QCG, may decide to omit a specific stability test. The EUPT-SC will finally decide whether analytes for which the stability test was not undertaken will be included in the Final 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 stability test, y being the mean value of the results of the first stability test and σ_{pt} being the standard deviation used for proficiency assessment (typically 25 % of the assigned value by default).

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 storage conditions other than those recommended to the participants e.g. at ambient temperature.

If insight about insufficient analyte stability is gained before the end of the PT-period, the EUPTQCG will be contacted in order to decide whether the EUPT-SC should be involved in the discussion (as confidential information is involved), whether the PT-participants should be informed about this insight and whether the affected analytes should be removed from the target list.

Stability during shipment: Considering knowledge about the expected susceptibility of analytes in the PT item to possible losses, the organisers will choose suitable shipping conditions to minimize such losses, e.g. shipment of frozen samples, addition of dry ice. As shipment duration can vary from labs/countries to 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 made 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. Follow-up measures in case of instability during shipment may include the exclusion of the affected results from the population used for establishing the assigned value (x_{pt}) and the non-calculation of z scores for the affected analytes in order to avoid unfair penalization of the laboratories involved.

⁸ To minimize the risk of PT item not being acceptably homogeneous, the organisers may opt to conduct a small-scale preliminary homogeneity test prior to bottling the PT item for shipment. The pre-tests may focus on a selected fraction of the analytes, and may also serve for verifying the presence and the approximate levels of the analytes spiked.

⁹ ISO 13528:2022: "Statistical methods for use in proficiency testing by interlaboratory comparisons", International Organization for Standardization.
¹⁰ 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

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If the PT entails analytes that are expected to have a high risk of degradation within the PT item, the organisers should conduct model tests prior to the final preparation of the test item in order to gain insight about the stability behavior of the analytes intended to be spiked during homogenization, transport and storage of the samples. Based on the results of these experiments measures should be taken to minimize the risk of certain analytes failing to meet the stability criteria, which may include adjusting the conditions of homogenization and/or storage and/or shipment or even deciding not to spike the material with certain analytes.

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. This can be done via the EURL data submission tool (in the following named Webtool) by answering the question whether the concerned analyte is included within the routine scope of the laboratory and the question about the analytical experience with the compound.

General Procedures for Reporting Results

Participating laboratories are responsible for reporting their own quantitative results to the organiser within the stipulated deadline. Any analyte targeted by a participating laboratory should be reported as "analysed" in the Webtool. In EUPTs by EURLs responsible for MRM compounds (FV, CF, AO) this is done before shipment of the PT test item. In EUPT-SRMs this is done in the period during which the platform is open for result submission. Each laboratory will be able to report only one result for each analyte detected in the PT item. The concentrations of the analytes detected should be expressed in 'mg/kg' unless indicated otherwise in the specific protocol of the respective EUPT.

For reporting, concentration values ≤ 0.01 mg/kg are recommended being rounded to two significant figures (e.g. 0.0078; 0.010) and values > 0.01 mg/kg to three significant figures (e.g. 0.123; 1.23; 12.3 mg/kg). No penalties will apply where a laboratory reports deviating numbers of significant figures, but in case of less significant figures, zeros will be assumed after the last significant figure (e.g. 0.1 = 0.100 and 0.11 = 0.110). For the calculation of z scores the values will be used as reported. In the preliminary and final report the results will be shown with up to three significant figures.

Laboratories should not report results below their own reporting limits (RLs). Any reported numerical result that is lower than the RL will be marked as a 'False Reporting' (FR) but it will be allocated a z score as any other numerical result. Such results will be, furthermore, included in the results population for establishing the assigned value (x_{pt}), unless they are eliminated for other reasons (e.g. laboratory status, use of biased methodology).

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 the method used 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 bias 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 Webtool. In such cases, reporting of the recovery rate is not mandatory.

- 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
- 'procedural calibration' approach
- '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 Webtool, which serves for submitting analytical results, is typically also 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. $CV^* > 35\%$).

Where certain methodologies or analytical steps are suspected to lead to biased or otherwise erroneous results, the PT-organisers will substantiate this suspicion by own experiments and discuss the issue with the EUPT-SC. Laboratories affected will be informed, e.g. via direct contact and/or via EURL-workshops or trainings and/or through the inclusion of recommendations within the Final EUPT Report.

Cases where reporting limits (RL) of laboratories exceed the MRRL indicate insufficient sensitivity and may be highlighted in the final report as with "PS" for poor sensitivity.

Results evaluation

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

False Positive (FP) results

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These are results of analytes on the Target Pesticides List that are reported at or above their respective MRRL although they were: (i) "not detected"¹¹ 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. If these results are additionally lower than the lab's reporting limit, they will be attributed with FR ('False Reporting').

False Negative (FN) results

These are results of analytes reported by the laboratories as 'analysed' but without reporting numerical values although they were: a) used by the organiser to treat the PT 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. Numerical results < RL (RL= Reporting Limit of the laboratory) may be judged as false negatives and may be also regarded as "not correctly found" when it comes to categorization in A and B based on scope. Such results wouldn't be reported in a routine laboratory environment. Case-by-case decisions by the EUPT-Panel will be taken by the EUPT-SC in such cases.

Where the RL of a laboratory for a certain analyte present in the PT item exceeds the assigned value, with the laboratory not reporting a numerical value, the result may still be judged as a false negative, despite this reporting being unobjectionable in a routine working environment. The FN judgement should in this case penalize the laboratory for not being able to achieve sufficient sensitivity for the analyte in question.

In cases of the robust mean of the participant results being less than 3 times higher than the MRRL, false negatives will typically not be assigned. The EUPT-SC may decide to make case-by-case decisions in this respect after considering all relevant factors such as the result distribution and the RLs of the affected labs. In case where the not fixing a valid assigned value is due to other reasons, e.g. because the uncertainty of the assigned value (UAV) criteria were not met and/or because of a bimodal distribution of the participant results, the EUPT-SC will decide on case-by-case basis whether FNs should be assigned for the respective analyte or not.

Estimation of the assigned value (x_{pt})

To minimise the influence of out-lying results on the statistical evaluation, the assigned value x_{pt} (= consensus concentration) will typically be estimated using the robust mean estimate of the participant results (x^*) as described in ISO 13528:2022¹², taking into account the results reported by EU and EFTA countries laboratories only. In special justifiable cases, the EUPT-Panel may decide to include results submitted by laboratories not belonging to the EU-/EFTA-OfLs network or even to use only the results of a subgroup of ('expert') laboratories that have previously repeatedly demonstrated good performance for the specific or similar compounds.

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).

In special justifiable cases, the EUPT-Panel may furthermore decide to use the spiked concentration of an analyte as the best estimate of the assigned value. In such cases, a detailed explanation of the reasons behind this decision will be given and a comparison with calculations involving robust statistics will be undertaken.

In reports, assigned values will be rounded to 3 significant figures if ≥ 0.01 mg/kg and to 2 significant figures if < 0.01 mg/kg (i.e. 0.0078; 0,123; 1.23; 12.3 mg/kg). For the calculation of z scores, the organisers may opt to use assigned values rounded to more significant figures than those stated above.

Since the assigned values of the EUPT analytes are typically generated using robust mean concentrations of participant results, 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

Results reported by laboratories from non-EU and non-EFTA Member States are typically excluded from the population used to derive the assigned value (for exceptions see 'Estimation of the assigned value').

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 that certain participant's results are associated with gross errors, 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.

¹¹ The term "not detected" is also used in the Webtool. In this context this term entails also all cases where no numerical result were reported (e.g. because the level determined was < MRRL and/or < RL)

¹² 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)

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In case of traceable calculation errors by the participants (e.g. use of wrong factors to express the result as required by the PT's residue definition¹³), and in case of non-reporting results that can be calculated from reported values (e.g. summed result not calculated and not reported), the EUPT Panel may decide to correct or complement results within the population by applying (the correct) factors. The new population of results may then be used for establishing the assigned values. The z score of the concerned results will, however, be calculated using the originally reported values. Although robust statistics are applied for estimating assigned values and robust standard deviations, certain results showing a strong bias compared to the rest of the population may be, in certain cases, eliminated before applying robust statistics¹⁴. To identify such strongly biased results, a preliminary consensus calculation of the robust mean (prelim- x^*) may be conducted and any results being ≥ 3 fold the prelim- x^* ¹⁵ may be potentially eliminated. This approach may need to be iterated if the population still entails obvious outliers.

The result population remaining after the elimination of certain results as described above may be then used to establish the actual assigned value (x_{pt}) and the robust standard deviation (s^*) according to the consensus approach described above. The z scores of all results, including those corrected or removed, are to be recalculated using the new assigned value.

All decisions to omit/exclude results will be discussed with the EUPT-SC and the reasoning for the omission of each result clearly stated in the Final EUPT-Report. However, z scores will be calculated for all results irrespective of the fact that they were omitted from the calculation of the assigned value.

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

Uncertainty of the assigned value

The uncertainty of the robust mean values (x_{pt}) is calculated according to ISO 13528:2022 as:

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

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

A broad results distribution (high s^*) and/or a limited number of results (p) will increase the uncertainty of the robust mean $u(x_{pt})$ values exceeding $0.3 \times \sigma_{pt}$ (see ISO 13528:2022) will typically mean that the robust mean is too uncertain for the purpose and cannot be straightforwardly taken up as the assigned value. In each of these cases, investigations for elucidating the reasons behind the high uncertainty should be undertaken. Taking into account all relevant aspects¹⁶ the EUPT-SC may decide that the analyte results should be re-evaluated based on a refined or extended result population or an alternative approach. If, despite these considerations and irrespective of the outcome of the UAV test the EUPT-SC concludes that, the assigned value of a specific analyte is too uncertain for a valid evaluation, it may decide that the results for the analyte in question should not be evaluated or only evaluated for informative purposes.

Considering the UAV when Calculating z Scores

Where the vast majority of the results is close to the robust mean and narrowly distributed but the UAV-test is still marginally failing¹⁷ (e.g. where $u(x_{pt})$ is up to $0.4 \times \sigma_{pt} = 10\%$ in absolute terms), the EUPT-Panel may consider to calculate z' scores using the following formula, which considers the uncertainty of the assigned value:

$$z' = \frac{x_i - x_{pt}}{\sqrt{\sigma_{pt}^2 + u^2(x_{pt})}}$$

where $u(x_{pt})$ being the uncertainty of the assigned value and σ_{pt} being the standard deviation of the assigned value that may be set equal to $FFP-\sigma_{pt}$ (see below). z' scores will be shown for Informative purposes only.

In special cases¹⁸, the EUPT-SC may consider useful to proceed with the calculation of z scores for both extremes of the assigned value as derived by applying the UAV (i.e. $x_{pt} \pm u(x_{pt})$). This upper and lower bound calculation of the z scores will also be for informative purposes only. The aim of this calculation is to help laboratories having performed well in a PT demonstrate their good performance even in cases where the UAV-test has not passed the criteria. Example: $x_{pt} = 1.0$ mg/kg, $u(x_{pt}) = 0.1$. Taking into account the calculated uncertainty, the AV should range between 0.9 and 1.1 mg/kg. If the result of a laboratory is 0.7 mg/kg, the z score calculates to -1.2 using $x_{pt} = 1.0$ mg/kg, For the upper limit of $x_{pt} = 1.1$ the z score calculates to -1.76 and for the lower limit of $x_{pt} = 0.9$ the z score calculates to -0.72. This means that, even at worst-case scenario, the laboratory's result remains within the acceptable range.

Standard deviation of the assigned value (target standard deviation)

The standard deviation for proficiency assessment (σ_{pt}) will be calculated using a Fit-For-Purpose approach with a fixed Relative Standard Deviation (FFP-RSD). Based on experience from previous EUPTs¹⁹, a percentage of 25 % is currently used as FFP-RSD for all analyte-matrix combination, and the Fit-For-Purpose target standard deviation ($FFP-\sigma_{pt}$) is calculated as follows:

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

¹³ irrespective of who is accounted responsible for the confusion

¹⁴ Please see ISO 13528:2022 Chapter 6.6, "Outlier techniques for individual results", therein 6.6.3, Note 3.

¹⁵ Corresponds to preliminary z scores ≥ 8 using the FFP-approach

¹⁶ e.g. information about methodologies used by the participants (especially if these are likely to produce biased results), multimodality, number of submitted results, homogeneity data, stability data

¹⁷ e.g. due to a combination of few results, and sporadic biased results.

¹⁸ E.g. where the population of results is narrow, but the UAV tests fails due to a few deviating results in combination with a relatively small number of results, e.g. <20

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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 testings.

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 from participant results").

z scores

This parameter is calculated using the following formula:

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

where x_i is the value reported by the laboratory, x_{pt} is the assigned value, and $FFP-\sigma_{pt}$ is the standard deviation using the FFP approach. Z scores shown in the preliminary and Final EUPT-Report 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.

For practical reasons, any z scores > 5 will be typically reported as '> 5' and a value of '5' will be used to calculate combined z scores (p. 19). Following ISO 17043:2023²⁰, z scores will be classified as follows:

$$\begin{aligned} |z| \leq 2.0 & \text{ Acceptable} \\ 2.0 < |z| < 3.0 & \text{ Questionable} \\ |z| \geq 3.0 & \text{ Unacceptable} \end{aligned}$$

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

For each EUPT the participating labs are asked to voluntarily report the MU figure they would report in routine analyses. The EUPT-SC will decide how to evaluate these figures and whether indications will be made to the laboratories in this regard.

Categorization of Laboratories

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 have:

- analysed at least 90% of the compulsory analytes in the target pesticides list,
- reported numerical results for at least 90 % of the compulsory analytes present in the PT item
- reported no false positives

are considered to 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: Number of analytes from the Target Pesticides List needed to be targeted or analytes present in the PT 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	
6	5.4	5	N - 1
7	6.3	6	
8	7.2	7	
9	8.1	8	
10	9.0	9	
11	9.9	10	
12	10.8	11	
13	11.7	12	N - 2
14	12.6	13	
15	13.5	13	
16	14.4	14	
17	15.3	15	
18	16.2	16	
19	17.1	17	
20	18	18	
21	18.9	19	
22	19.8	20	
23	20.7	21	N - 3
24	21.6	22	
25	22.5	22	
26	23.4	23	

¹⁹ 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

²⁰ ISO/IEC 17043:2023. Conformity assessment – General requirements for the competence of proficiency testing providers

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Overall Performance of Laboratories - Combined z Scores

For evaluation of the overall performance of laboratories the average of the squared z scores (AZ^2)²¹ and/or the average of the absolute z scores (AAZ) can be calculated for informative purposes. To minimize the influence of outlying results, the calculation of AZ^2 and AAZ will not be conducted in the case of < 10 and < 5 results, respectively, and z scores higher than 5 will be set as 5. Combined z scores are typically only calculated for laboratories within Category A and considering results of compulsory analytes, but the organisers may deviate from this if considered reasonable, provided that a minimum number of results (z scores) have been reported. Combined z scores may be also calculated using results of across PTs. Considering the cut-off of high z scores at 5, 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.
Based on the AZ^2 achieved, the laboratories are classified as follows:

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

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 being too low.

In the case of EUPT-SRMs, where only a few results per laboratory 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. In general, laboratories should aim to achieve AAZ scores < 0.9, which corresponds to an average bias of 22.5 %²².

Laboratories within Category B will be typically ranked according to the total number of analytes they correctly reported to be present in the PT item. The number of acceptable z scores achieved may be presented, too.

Publication of results

The EURLs will publish a preliminary report, containing tentative assigned values and z score values for all analytes present in the PT item, within 2 months of the deadline for result submission. An early distribution of the preliminary report, entailing preliminary assigned values (prAV), will allow an early investigation of possible errors by the participants.

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 included in all tables or figures in the Final EUPTReport.

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 and can be accessed by the concerned laboratories only.

Feedback and Complaints

Participants have the right to complain about any aspect concerning the PT (e.g. about the on-line tools used for registration and data submission, the organisation and communication with the participants, the timing of the PT, transcription errors and the result evaluation if it is not compliant with the provisions of the general protocol). Complaints about a non-arrival of a PT item or about the bad condition of the PT item upon arrival should be done through the Webtool shortly as indicated in the specific protocols. The EURLs will track the complaints and will try to accommodate all substantiated complaints in due time. After the publication of the final EUPT report, the organizers reserve the right not to consider any complaints arriving more than two months after its publication.

Appeals and complaints concerning the principles of organisation and statistical analysis of the results according to the General Protocol should be made prior to the start of a PT. By signing up to an EUPT, the participant agrees with the provisions of the General Protocol valid for the PT-season in question.

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

²¹ 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. DOI:10.1007/s00216-010-3877-3

²² At 22.5% average bias (i.e. $AAZ=0.9$) and assuming a precision of 10%, the uncertainty calculates to 24.6% (error propagation formula), which is just acceptable. At a precision of 15%, the maximally tolerable average bias calculates to 20%, which translates to an AAZ of 0.8. The uncertainty of the bias was not considered in these calculations.

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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 and carefully study the latest version of these documents.**

If substantial errors are discovered in the Preliminary EUPT-Report the organisers will distribute a new corrected version, therein it will be stated that the previous version is no longer valid. The online version on the PT website will be replaced.

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.

If a new version of any EUPT document is released, each page of the new version must be marked in a way distinguishing it from previous versions, e.g. with the version number.

Where errors are discovered in EUPT-Certificates, the revised certificates will be issued and uploaded to the DataPool. The concerned laboratories will be informed and asked to download the corrected 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, or where the procedure used turns out being significantly biased.

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 PT items (80% for SRM-compounds). Additionally, NRLs that obtained AZ^2 higher than 3 (AAZ higher than 1.3 for SRM-compounds) in two consecutive EUPTs of the last four EUPTs, will be considered as underperforming in accuracy. As soon as underperformance of an NRL is detected, a two-step protocol established by DG-SANTE will be applied²³:

Phase 1:

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

Phase 2:

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

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

Disclaimer

The EURLs retain 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.

²³ Article 101 of Regulation (EU) 2017/625

EUPT-FV-SC08 SPECIFIC PROTOCOL

European Union Proficiency Test for Pesticide Residues in green tea (2024)

Introduction

This protocol is complementary to the General Protocol of EU Proficiency Tests (EUPTs) for Pesticide Residues in Food and Feed (11th 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 tea** containing both incurred and spiked pesticide residues. The test item will consist of **ground green tea**.

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 (MRRs). 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 18th, 2024. 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 18th December 2024.
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 30 g of green tea containing pesticide residues.

No blank material will be provided.

Shipment of Test item

The shipment of the test item will be on 18th November 2024, 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 22nd November 2024, 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.

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

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	8 th October - 28 th October 2024
Specific Protocol published on the Web site.	4 th November 2024 at the latest
Sample distribution.	18 th November 2024
Deadline for receiving results	18 th December 2024
Preliminary Report: (containing preliminary assigned values and z scores)	January 2025
Final Report distributed to the Laboratories.	September 2025

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 Almería
BANK ADDRESS: Office Number 990, Universidad de Almería, 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.
Paula Medina, Senior Chemist (QCG).	European Food Safety Authority, Italy.
Michelangelo Anastassiades, Senior Chemist (AG).	EURL-SRM, CVUA Stuttgart, Fellbach, Germany.
Björn Hardebusch, Senior Chemist (AG).	EURL-AO, CVUA Freiburg, Freiburg, Germany.
Magnus Jezussek, Senior Chemist (AG).	LGL, Erlangen, Germany.
André de Kok, Senior Chemist (AG).	Formerly Wageningen Food Safety Research, Wageningen, The Netherlands.
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 EUPF-FV-SC08

Pestide no.	Pesticides	MRRL (mg/kg)
1	Acephate	0.01
2	Acetamidrid	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	0.01
9	Ametoctradin	0.01
10	Azinphos-methyl	0.01
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	0.01
21	Carbaryl	0.01
22	Carbendazim	0.01
23	Carbofuran	0.01
24	Carbofuran-3-hydroxy	0.01
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	0.01
32	Chlorpyrifos-methyl	0.01
33	Clofentezine	0.01
34	Clothianidin	0.01
35	Cyantraniliprole	0.01
36	Cyazofamid	0.01
37	Cyflufenamid	0.01
38	Cyflumetofen	0.01
39	Cyfluthrin	0.01
40	Cymoxanil	0.01
41	Cypermethrin	0.01
42	Cyproconazole	0.01
43	Cyprodinil	0.01
44	Deltamethrin	0.01
45	Demeton-S-methylsulfone	0.01
46	Diazinon	0.01
47	Dichlofluanid	0.01
48	Dichlorvos	0.01
49	Dicloran	0.01
50	Dicofol	0.01
51	Dieldrin	0.01
52	Diethofencarb	0.01
53	Difenoconazole	0.01
54	Diflubenzuron	0.01
55	Dimethoate	0.01
56	Dimethomorph	0.01
57	Dimethylaminosulfotoluidide (DMST)	0.01
58	Diniconazole	0.01
59	Diphenylamine	0.01
60	Endosulfan alpha	0.01
61	Endosulfan beta	0.01
62	Endosulfan sulfate	0.01
63	EPN	0.01
64	Epoxiconazole	0.01
65	Ethion	0.01
66	Ethirimol	0.01
67	Ethoprophos	0.01
68	Etofenprox	0.01
69	Etoxazole	0.01
70	Famoxadone	0.01
71	Fenamidone	0.01
72	Fenamiphos	0.01

Pestide no.	Pesticides	MRRL (mg/kg)
73	Fenamiphos sulfone	0.01
74	Fenamiphos sulfoxide	0.01
75	Fenarimol	0.01
76	Fenazaquin	0.01
77	Fenbuconazole	0.01
78	Fenhexamid	0.01
79	Fenitrothion	0.01
80	Fenoxycarb	0.01
81	Fenpropathrin	0.01
82	Fenpropidin	0.01
83	Fenpropimorph	0.01
84	Fenpyrazamine	0.01
85	Fenpyroximate	0.01
86	Fenthion	0.01
87	Fenthion oxon	0.01
88	Fenthion oxon sulfone	0.01
89	Fenthion oxon sulfoxide	0.01
90	Fenthion sulfone	0.01
91	Fenthion sulfoxide	0.01
92	Fenvalerate	0.01
93	Fipronil	0.01
94	Fipronil sulfone	0.01
95	Fonicamid	0.01
96	Flubendiamide	0.01
97	Fludioxonil	0.01
98	Flufenoxuron	0.01
99	Fluopicolide	0.01
100	Fluopyram	0.01
101	Flupyradifurone	0.01
102	Fluquinconazole	0.01
103	Flusilazole	0.01
104	Flutolanil	0.01
105	Flutriafol	0.01
106	Fluxapyroxad	0.01
107	Formetanate	0.01
108	Fosthiazate	0.01
109	Hexaconazole	0.01
110	Hexythiazox	0.01
111	Imazalil	0.01
112	Imidacloprid	0.01
113	Indoxacarb	0.01
114	Iprodione	0.01
115	Iprovalicarb	0.01
116	Isocarbophos	0.01
117	Isofenphos-methyl	0.01
118	Isoprothiolane	0.01
119	Kresoxim-methyl	0.01
120	Lambda-Cyhalothrin	0.01
121	Linuron	0.01
122	Lufenuron	0.01
123	Malaoxon	0.01
124	Malathion	0.01
125	Mandipropamid	0.01
126	Mepanipyrim	0.01
127	Metaflumizone	0.01
128	Metalaxyl	0.01
129	Methamidophos	0.01
130	Methidathion	0.01
131	Methiocarb	0.01
132	Methiocarb sulfone	0.01
133	Methiocarb sulfoxide	0.01
134	Methomyl	0.01
135	Methoxyfenozide	0.01
136	Metrafenone	0.01
137	Monocrotophos	0.01
138	Myclobutanil	0.01
139	Omethoate	0.01
140	Orthophenylphenol	0.01
141	Oxadixyl	0.01
142	Oxamyl	0.01
143	Oxydemeton-methyl	0.01
144	Pacllobutrazole	0.01
145	Paraoxon-methyl	0.01

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

Pestide no.	Pesticides	MRRL (mg/kg)
146	Parathion	0.01
147	Parathion-methyl	0.01
148	Penconazole	0.01
149	Pencycuron	0.01
150	Pendimethalin	0.01
151	Permethrin	0.01
152	Phenthoate	0.01
153	Phosalone	0.01
154	Phosmet	0.01
155	Phosmet oxon	0.01
156	Phoxim	0.01
157	Pirimicarb	0.01
158	Pirimiphos-methyl	0.01
159	Prochloraz	0.01
160	Procymidone	0.01
161	Profenofos	0.01
162	Propamocarb	0.01
163	Propargite	0.01
164	Propiconazole	0.01
165	Propyzamide	0.01
166	Proquinazid	0.01
167	Prosulfocarb	0.01
168	Prothioconazole	0.01
169	Prothiofos	0.01
170	Pymetrozine	0.01
171	Pyraclostrobin	0.01
172	Pyridaben	0.01
173	Pyridalyl	0.01
174	Pyrimethanil	0.01
175	Pyriproxyfen	0.01
176	Quinoxifen	0.01
177	Spinetoram	0.01
178	Spinosad	0.01
179	Spirodiclofen	0.01
180	Spiromesifen	0.01

Pestide no.	Pesticides	MRRL (mg/kg)
181	Spirotetramat	0.01
182	Spirotetramat-enol	0.01
183	Spiroxamine	0.01
184	Sulfoxaflor	0.01
185	Tau-Fluvalinate	0.01
186	Tebuconazole	0.01
187	Tebufenozide	0.01
188	Tebufenpyrad	0.01
189	Teflubenzuron	0.01
190	Tefluthrin	0.01
191	Terbutylazine	0.01
192	Tetraconazole	0.01
193	Tetradifon	0.01
194	Thiabendazole	0.01
195	Thiacloprid	0.01
196	Thiamethoxam	0.01
197	Thiodicarb	0.01
198	Thiophanate-methyl	0.01
199	Tolclofos-methyl	0.01
200	Tolyfluanid	0.01
201	Triadimefon	0.01
202	Triadimenol	0.01
203	Triazophos	0.01
204	Trichlorfon	0.01
205	Tricyclazole	0.01
206	Trifloxystrobin	0.01
207	Triflumizole	0.01
208	Triflumizole metabolite (FM-6-1)	0.01
209	Triflumuron	0.01
210	Trifluralin	0.01
211	Triticonazole	0.01
212	Vinclozolin	0.01
213	Zoxamide	0.01

MRRL: Minimum Required Reporting Level

This list is based on Commission Implementing Regulation (EU) 2023/731 of 3 April 2023

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.