EURL-PROFICIENCY TEST-FV-SC04, 2020-2021

Pesticide Residues in Sultana Raisins slurry

Final Report

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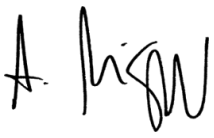

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EURL-EUROPEAN UNION PROFICIENCY TEST SC04
FOR THE DETERMINATION OF PESTICIDES IN SPECIAL COMMODITIES USING MULTIRESIDUE METHODS
2020-2021

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

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

Participation in European Proficiency Test FV-SC04 was on a voluntary basis. The invitation was sent to all National Reference Laboratories (NRLs), as well as all other EU official laboratories involved in the determination of pesticide residues in fruits and vegetables for the EU multi-annual coordinated control programme or for their own national monitoring programmes. Additionally, laboratories from non-EU/non-EFTA countries were invited to take part.

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

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

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

[3] The Community Reference Laboratory (CRL) changed its name to the European Union Reference Laboratory (EURL) on 1st December 2009 as a result of the Treaty of Lisbon. OJ of the EU C306 on 17.12.2007.

[4] Commission Regulation (EC) No 776/2006 of 23rd May 2006 - amending Annex VII to Regulation (EC) No 882/2004 of the European Parliament and of the Council as regards European Union Reference Laboratories.

1. INTRODUCTION

Forty-five laboratories agreed to participate in EUPT-FV-SC04.

The proficiency test was performed at the end of 2020 and beginning of 2021 using raisins slurry. Sultana raisins were purchased in the local market in Almería (Spain). They were analysed in the EURL-FV laboratory in Almería, and 23 pesticide residues were detected at concentrations above the Minimum Required Reporting Level (MRRL). Thus, all pesticide residues in the test item were incurred pesticide. Participating laboratories were not provided with a 'blank' of sultana raisins sample.

The test item, 100 g of sultana raisins slurry (sultana raisins and water in a proportion of 1:1.5 (w/w)) containing incurred pesticide residues, was shipped to participants on 23rd November 2020. The deadline for results submission to the Organiser was 15th January 2021. The participants were asked to determine the residue levels of all the pesticides that they detected in the raisins and to report the concentrations in mg/kg. The participants were provided with a target pesticide list, which contained 208 target pesticides. The pesticide target list is detailed in Annex A. The lists of target pesticides also contained the MRRL for each pesticide fixed at 0.01 mg/kg, except for the following pesticides which have lower MRRLs based on Regulation (EU) No. 396/2005 and EU Directive 2006/125/EC, or for which EFSA requested lower LOQs: aldrin (0.005 mg/kg), cadusafos (0.005 mg/kg), carbofuran (0.005 mg/kg), chlorpyrifos (0.005 mg/kg), demeton-S-methylsulfone (0.005 mg/kg), diazinon (0.005 mg/kg), dichlorvos (0.005 mg/kg), dieldrin (0.005 mg/kg), dimethoate (0.003 mg/kg), ethoprophos (0.005 mg/kg), fipronil (0.004 mg/kg), fipronil sulfone (0.004 mg/kg), monocrotophos (0.005 mg/kg), omethoate (0.003 mg/kg), oxydemeton-methyl (0.005 mg/kg) and triazophos (0.005 mg/kg).

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

For the assessment of overall laboratory performance, the Average of the squared z scores (AZ^2) was used. Laboratories that had 'sufficient scope' and were able to analyse at least 90 % of the compulsory pesticides in the target pesticides list, had correctly detected and quantified a sufficiently high percentage of the pesticides present in the Test Item (at least 90 %) and reported no false positives, were classified into Category A. Within this category, the laboratories were also subclassified as 'good', 'satisfactory' or 'unsatisfactory', in relation to the overall accuracy of the results that they reported.

All the other laboratories were classified into Category B. For laboratories in Category B, individual z scores were calculated but the overall accuracy of their results was not assessed.

Laboratories that did not report results have not been classified into any category and are listed in Annex B with the remainder of laboratories that participated in EUPT-FV-SC04.

2. TEST ITEM

2.1 Preparation of the treated test item

Sultana raisins were purchased in the local market in Almería. They were analysed in the EURL-FV laboratory in Almería for all the pesticides included in the target list. Twenty-three pesticide residues were detected at concentrations above the MRRL, so they were used for the preparation of the test item without the need to spike them.

Sultana raisins were thoroughly mixed, and they were frozen by inmersion in liquid nitrogen. Raisins were milled for 30 seconds. Afterwards, water (in a proportion 1 raisins: 1,5 water) was added to the processor, and the slurry was well mixed. All the processed material was homogeneised for 30 minutes in a big container by hand stirring. 100 g portions of the raisin's slurry were weighed out into screw-capped polyethylene plastic bottles, sealed and stored in a freezer at about - 20 °C prior to distribution to participants.

2.2 Homogeneity test

The homogeneity and stability tests were performed by the EURL-FV laboratory at the University of Almería. 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 raisins.

The statistical evaluation was performed according to the International Harmonized Protocol published by IUPAC, ISO and AOAC [1]. The individual residues data from the homogeneity tests are given in **Appendix 1**. The results of the statistical analyses are given in **Table 1**. The acceptance criteria for the test item to be sufficiently homogenous for the proficiency test were that: $Ss^2 < c$, where Ss is the between-bottle sampling standard deviation and $c = F_1\sigma^2_{all} + F_2S^2_{an}$; F_1 and F_2 being constant values of 1.88 and 1.01, respectively, from the ten samples taken, and σ^2_{all} = (0.3 x FFP-RSD(25 %) x mean concentration)^2. This was used to demonstrate that the between-bottle variance was not higher than the within-bottle variance.

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

Pesticide	Mean Conc. (mg/Kg)	Ss^2	c	$Ss^2 < c$ Pass/Fail
Boscalid	0.049	1.852E-05	5.00E-05	Pass
Carbendazim	0.237	2.951E-05	6.10E-04	Pass
ⓘCyflufenamid	0.020	2.506E-06	0.00E+00	Pass
Cyprodinil	1.049	7.597E-03	1.83E-02	Pass
ⓘEthirimol	0.009	6.643E-08	0.00E+00	Pass
ⓘFenvalerate	0.009	7.832E-08	0.00E+00	Pass
Flubendiamide	0.036	5.468E-06	2.00E-05	Pass
Fluxapyroxad	0.098	0.000E+00	8.00E-04	Pass

Pesticide	Mean Conc. (mg/Kg)	Ss^2	c	Ss^2 < c Pass/Fail
Indoxacarb	0.048	1.006E-05	3.00E-05	Pass
ⓘLambda-Cyhalothrin	0.020	2.350E-06	1.00E-05	Pass
Mandipropamid	0.039	0.000E+00	1.60E-04	Pass
Metalaxyl	0.229	3.174E-04	8.92E-04	Pass
Methoxyfenozide	0.194	0.000E+00	2.43E-03	Pass
ⓘMetrafenone	0.021	2.801E-06	1.00E-05	Pass
ⓘPenconazole	0.010	6.752E-07	0.00E+00	Pass
ⓘProquinazid	0.018	0.000E+00	2.00E-05	Pass
Pyrimethanil	0.405	1.017E-03	2.81E-03	Pass
ⓘSpirotetramat-enol	0.267	2.575E-05	8.00E-04	Pass
Spirotetramat-enolglukoside	0.274	3.901E-05	8.40E-04	Pass
Spirotetramat-ketohydroxy	0.031	3.147E-06	1.15E-05	Pass
ⓘTebuconazole	0.013	1.783E-06	0.00E+00	Pass
Thiophanate-methyl	0.113	7.278E-05	1.80E-04	Pass
ⓘZoxamide	0.011	1.056E-06	0.00E+00	Pass

S_s: Between-Sampling Standard Deviation
ⓘOnly for informative purposes

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

2.3 Stability tests

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

A pesticide was considered to be adequately stable if $|x_1 - y_i| \leq 0.3 \times \sigma$, where x_1 is the mean value of the Day 1 stability test, yi the mean value of the Day 2 stability test and σ the standard deviation used for proficiency assessment (typically 25 % of the assigned value).

The individual results are given in **Table 2**. This test did not show any significant decrease in the pesticide concentrations with time. This demonstrates that, for the duration of the proficiency test, and provided that the storage conditions prescribed were followed, the time elapsed until the participants performed the analysis would not have influenced their results.

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. Results for this 48-hour stability test are indicated in **Table 3**.

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

(mg/Kg)	Day 1							Day 2							(M2 – M1)	M2-M1 ≤ 0.3*σ
	Sample 2_A	Sample 2_B	Sample 17_A	Sample 17_B	Sample 22_A	Sample 22_B	Mean 1	Sample 26_A	Sample 26_B	Sample 40_A	Sample 40_B	Sample 62_A	Sample 62_B	Mean2		
Boscalid	0.048	0.049	0.049	0.045	0.054	0.050	0.049	0.049	0.050	0.049	0.050	0.049	0.049	0.049	0.000	Pass
Carbendazim	0.250	0.250	0.220	0.250	0.230	0.240	0.243	0.240	0.240	0.250	0.240	0.230	0.240	0.240	0.000	Pass
ⓘCyflufenamid	0.019	0.019	0.022	0.024	0.022	0.025	0.022	0.020	0.021	0.019	0.020	0.021	0.022	0.021	-0.001	Pass
Cyprodinil	1.000	1.000	1.150	1.000	0.980	0.990	1.029	1.000	1.000	0.890	0.990	1.040	1.090	1.010	-0.015	Pass
ⓘEthirimol	0.012	0.010	0.009	0.009	0.011	0.012	0.010	0.009	0.012	0.009	0.011	0.011	0.012	0.011	0.000	Pass
ⓘFenvalerate	0.010	0.011	0.009	0.011	0.015	0.012	0.011	0.012	0.012	0.009	0.011	0.011	0.012	0.011	0.000	Pass
Flubendiamide	0.038	0.037	0.035	0.032	0.037	0.039	0.036	0.032	0.035	0.033	0.035	0.036	0.037	0.035	-0.002	Pass
Fluxapyroxad	0.120	0.099	0.110	0.100	0.099	0.096	0.104	0.130	0.110	0.098	0.097	0.099	0.098	0.100	0.001	Pass
Indoxacarb	0.050	0.051	0.050	0.059	0.040	0.045	0.049	0.050	0.052	0.044	0.040	0.042	0.049	0.046	-0.003	Pass
ⓘLambda-Cyhalothrin	0.019	0.018	0.021	0.022	0.020	0.021	0.020	0.022	0.021	0.020	0.019	0.019	0.018	0.020	0.000	Pass
Mandipropamid	0.041	0.040	0.039	0.039	0.041	0.039	0.040	0.035	0.036	0.040	0.039	0.040	0.041	0.039	-0.001	Pass
Metalaxyl	0.230	0.240	0.250	0.240	0.200	0.220	0.232	0.215	0.230	0.210	0.200	0.210	0.230	0.216	-0.016	Pass
Methoxyfenozide	0.200	0.210	0.190	0.180	0.190	0.200	0.198	0.220	0.230	0.187	0.192	0.200	0.210	0.207	0.009	Pass
ⓘMetrafenone	0.018	0.019	0.022	0.019	0.022	0.025	0.021	0.021	0.019	0.018	0.019	0.020	0.021	0.020	-0.001	Pass
ⓘPenconazole	0.010	0.012	0.009	0.011	0.012	0.010	0.011	0.011	0.010	0.009	0.009	0.014	0.012	0.011	0.000	Pass
ⓘProquinazid	0.022	0.021	0.021	0.019	0.019	0.018	0.020	0.019	0.018	0.021	0.022	0.022	0.021	0.021	0.000	Pass
Pyrimethanil	0.370	0.390	0.410	0.390	0.410	0.440	0.403	0.400	0.410	0.380	0.390	0.420	0.410	0.400	0.000	Pass
ⓘSpirotetramat-enol	0.210	0.230	0.210	0.220	0.210	0.230	0.219	0.230	0.240	0.220	0.240	0.200	0.220	0.230	0.007	Pass
Spirotetramat-enolglukoside	0.230	0.240	0.240	0.260	0.200	0.220	0.233	0.210	0.230	0.260	0.250	0.210	0.230	0.230	0.000	Pass
Spirotetramat-ketohydroxy	0.035	0.036	0.039	0.039	0.040	0.038	0.038	0.041	0.040	0.040	0.039	0.041	0.039	0.040	0.002	Pass
ⓘTebuconazole	0.011	0.012	0.009	0.010	0.012	0.013	0.011	0.010	0.010	0.009	0.011	0.013	0.012	0.011	0.000	Pass
Thiophanate-methyl	0.110	0.100	0.110	0.120	0.150	0.120	0.121	0.110	0.120	0.110	0.140	0.100	0.120	0.120	-0.004	Pass
ⓘZoxamide	0.011	0.010	0.012	0.011	0.011	0.012	0.011	0.011	0.010	0.009	0.010	0.011	0.013	0.011	0.000	Pass

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

(mg/Kg)	Day 1							Day 3							(M3 – M1)	M3-M1 ≤ 0.3*σ
	Sample 2_A	Sample 2_B	Sample 17_A	Sample 17_B	Sample 22_A	Sample 22_B	Mean 1	Sample 55_A	Sample 55_B	Sample 63_A	Sample 63_B	Sample 10_A	Sample 10_B	Mean3		
Boscalid	0.048	0.049	0.049	0.045	0.054	0.050	0.049	0.048	0.049	0.049	0.045	0.053	0.050	0.049	0.000	Pass
Carbendazim	0.250	0.250	0.220	0.250	0.230	0.240	0.243	0.220	0.250	0.240	0.230	0.230	0.240	0.239	-0.004	Pass
ⓘCyflufenamid	0.019	0.019	0.022	0.024	0.022	0.025	0.022	0.020	0.022	0.020	0.024	0.025	0.023	0.022	0.001	Pass
Cyprodinil	1.000	1.000	1.150	1.000	0.980	0.990	1.029	1.120	1.110	0.950	0.990	1.120	1.050	1.059	0.029	Pass
ⓘEthirimol	0.012	0.010	0.009	0.009	0.011	0.012	0.010	0.010	0.009	0.010	0.010	0.011	0.011	0.010	0.000	Pass
ⓘFenvalerate	0.010	0.011	0.009	0.011	0.015	0.012	0.011	0.010	0.012	0.011	0.010	0.010	0.011	0.011	-0.001	Pass
Flubendiamide	0.038	0.037	0.035	0.032	0.037	0.039	0.036	0.032	0.033	0.033	0.034	0.035	0.035	0.034	-0.003	Pass
Fluxapyroxad	0.120	0.099	0.110	0.100	0.099	0.096	0.104	0.120	0.100	0.120	0.110	0.098	0.099	0.109	0.005	Pass
Indoxacarb	0.050	0.051	0.050	0.059	0.040	0.045	0.049	0.048	0.051	0.045	0.042	0.044	0.048	0.046	-0.003	Pass
ⓘLambda-Cyhalothrin	0.019	0.018	0.021	0.022	0.020	0.021	0.020	0.021	0.020	0.022	0.022	0.020	0.019	0.021	0.001	Pass
Mandipropamid	0.041	0.040	0.039	0.039	0.041	0.039	0.040	0.040	0.038	0.039	0.041	0.039	0.041	0.040	0.000	Pass
Metalaxyl	0.230	0.240	0.250	0.240	0.200	0.220	0.232	0.220	0.240	0.220	0.210	0.240	0.230	0.231	-0.001	Pass
Methoxyfenozide	0.200	0.210	0.190	0.180	0.190	0.200	0.198	0.210	0.220	0.200	0.200	0.210	0.200	0.205	0.008	Pass

(mg/Kg)	Day 1							Day 3								
	Sample 2_A	Sample 2_B	Sample 17_A	Sample 17_B	Sample 22_A	Sample 22_B	Mean 1	Sample 55_A	Sample 55_B	Sample 63_A	Sample 63_B	Sample 10_A	Sample 10_B	Mean3	(M3 – M1)	M3-M1 ≤ 0.3*σ
ⓘMetrafenone	0.018	0.019	0.022	0.019	0.022	0.025	0.021	0.019	0.020	0.020	0.019	0.020	0.019	0.020	-0.001	Pass
ⓘPenconazole	0.010	0.012	0.009	0.011	0.012	0.010	0.011	0.012	0.010	0.009	0.010	0.010	0.010	0.010	0.000	Pass
ⓘProquinazid	0.022	0.021	0.021	0.019	0.019	0.018	0.020	0.018	0.019	0.020	0.021	0.020	0.021	0.020	0.000	Pass
Pyrimethanil	0.370	0.390	0.410	0.390	0.410	0.440	0.403	0.420	0.420	0.390	0.410	0.410	0.420	0.413	0.010	Pass
ⓘSpirotetramat-enol	0.210	0.230	0.210	0.220	0.210	0.230	0.219	0.240	0.230	0.230	0.230	0.210	0.220	0.230	0.011	Pass
Spirotetramat-enolglukoside	0.230	0.240	0.240	0.260	0.200	0.220	0.233	0.220	0.230	0.250	0.250	0.220	0.240	0.239	0.006	Pass
Spirotetramat-ketohydroxy	0.035	0.036	0.039	0.039	0.040	0.038	0.038	0.035	0.034	0.042	0.038	0.041	0.042	0.039	0.001	Pass
ⓘTebuconazole	0.011	0.012	0.009	0.010	0.012	0.013	0.011	0.010	0.011	0.009	0.009	0.012	0.011	0.010	-0.001	Pass
Thiophanate-methyl	0.110	0.100	0.110	0.120	0.150	0.120	0.121	0.120	0.150	0.120	0.130	0.110	0.110	0.126	0.004	Pass
ⓘZoxamide	0.011	0.010	0.012	0.011	0.011	0.012	0.011	0.013	0.014	0.012	0.010	0.011	0.011	0.012	0.001	Pass

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2.4 Distribution of test items and protocol to participants

One bottle of frozen treated test item was shipped to each participant in boxes containing dry ice. The test items were sent out on 23rd November 2020. All the shipments to EU/EFTA countries arrived within the first 48 hours.

Before sample shipment, the laboratories received full instructions (Annex A) for the receipt and storage of the test item and they were encouraged to use their normal sample receipt procedure and method(s) of analysis. These instructions were uploaded onto the open site of the EURL-FV webpage as part of the Specific Protocol. The Application Form was also available as an on-line form. After applying for the test, each participant laboratory received their Lab Code. This ensured that confidentiality was maintained throughout the duration of Proficiency Test SC04. The Target Pesticide List was the same as for EUPT-FV22, and it was uploaded onto the EURL-FV open website at least three months before the shipment of the test item to allow laboratories enough time to purchase standards and to validate their methods.

3. STATISTICAL METHODS

3.1 False positives and negatives

3.1.1 False positives

These are results of pesticides from the Target Pesticides List, that are reported at, or above, their respective MRRLs although they were: (i) not detected by the Organiser, even after repeated analyses, and/or (ii) not detected by the overwhelming majority (e.g. > 95 %) of the participating laboratories that had targeted the specific pesticides. In certain instances, case-by-case decisions by the EUPT-Panel may be necessary.

Any results reported lower than the MRRL will not be considered as false positives, even though these results should not have been reported.

No z score values have been calculated for false positive results. Any laboratory reporting a false positive, even when reporting the necessary number of pesticides to obtain sufficient scope, has been classified into Category B.

3.1.2 False negatives

These are results for pesticides reported by the laboratories as 'analysed' but without reporting numerical values although they were: a) used by the Organiser to treat the Test Item and b) detected by the Organiser as well as the majority of the participants that had targeted these specific pesticides at or above the respective MRRLs. Results reported as '< RL' (RL= Reporting Limit of the laboratory) will be considered as not detected and will be judged as false negatives. In certain instances, case-by-case decisions by the EUPT-Panel may be necessary.

In cases of the assigned value being less than a factor of 3 times the MRRL, false negatives will typically not be assigned. The EUPT-Panel may decide to take case-by-case decisions in this respect after considering all relevant factors such as the result distribution and the reporting limits of the affected labs.

z scores have also been calculated for false negatives. However, these z scores were not taken into account in assessing the 90 %, or more, of pesticides present in the sample needed to be classified into Category A.

3.2 Estimation of the assigned values (x_{pt})

In order to minimise the influence of out-lying results on the statistical evaluation, the assigned value (= consensus concentration) was estimated using robust statistics as described in ISO 13528:2015, considering the results reported by EU and EFTA countries laboratories only. Individual results without any numerical values reported, such as detected (D), were not considered. The spread of results for each pesticide was tested for multimodality. In special justifiable cases, the EUPT-Panel may decide to eliminate certain results traceably associated with gross errors or to use only the results of a subgroup consisting of laboratories that have repeatedly demonstrated good performance for the specific compound in the past.

Considering the normative for robust analysis in ISO 13528:2015, the uncertainty accompanying the assigned value for each pesticide was calculated according to the following equation:

$$u(x_{pt}) = 1.25\frac{s^*}{\sqrt{p}}$$

Where:

- $u(x_{pt})$ is the uncertainty in mg/Kg.
- s^* is the robust standard deviation of the results.
- p is the total number of results.

3.3 Fixed target standard deviations

Based on the experience gained from previous EU proficiency tests and recommendations from the EURL Advisory Group, a fixed relative standard deviation (FFP-RSD) of 25 % was chosen [3]. This is in line with the internationally accepted target Measurement Uncertainty of 50 % for multiresidue analysis of pesticides [4], which is derived from, and linked to, the EUPTs. The same target RSD has been applied to all the pesticides, independent of concentration. For informative purposes the robust relative standard deviation (CVs*) is calculated according to ISO 13528:2015 Chapter 7.7 (Consensus value from participant results) following Algorithm A in Annex C, and it can be compared to the FFP-RSD in **Table 7**.

3.4 z scores

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

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

Where:

- x_i is the result reported by the participant, or the MRRL or the reporting limit (RL) (whichever one is lower) for those labs that have not detected the presence of the pesticide in the sample.
- X_{pt} is the assigned value.
- σ_{pt} is the target standard deviation (the FFP-RSD of 25 % multiplied by the assigned value).

z score classification is as follows:

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

- Any z score value of $\lvert z \rvert > 5$ has been reported as '>5' and a value of '5' has been used to calculate combined z scores.
- No z score calculations have been performed for false positive results.
- For false negative results, the MRRL (or RL) has been used to calculate the z score. These z scores have also been included in the graphical representation and are marked with an asterisk.

3.5 Combined z scores

In order to evaluate each laboratory's overall performance according to the quality of its results and its scope, two classifications - Category A and B - were used. To be classified into Category A, laboratories had to be able to analyse at least 90 % of the compulsory pesticides in the target pesticides list, to correctly identify and report quantitative results (that is *sought and detected*) for 90 % or more of the total number of pesticides evaluated in the test item and report

no false positives (for the 90 % criterion the number of pesticides needed to be correctly analysed to have sufficient scope will be calculated by multiplying the number of compulsory pesticides from the Target Pesticides List by 0.9 and rounded to the nearest full number with 0.5 decimals being rounded downwards). If these three requirements were met, then the combined z scores were calculated as the 'Average of the Squared z scores' (AZ^2) [5].

3.5.1 The Average of the Squared z scores (AZ^2)

The 'Average of the Squared z scores' was introduced for the first time in EUPT-FV12. The AZ^2 is calculated as follows:

$$AZ^2 = \frac{\sum_{i=1}^{n} Z_i^2}{n}$$

The resultant formula is the sum of the z scores value, multiplied by itself and divided by the number of z scores (n) detected by each laboratory, including those from false negatives.

This formula is subsequently used to produce an overall classification of laboratories with three sub-classifications: 'good', 'satisfactory' and 'unsatisfactory'.

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

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

Laboratories that did not detect and quantify sufficient pesticides, that were not able to analyse at least 90 % of the compulsory pesticides or reported a false positive, have been placed in Category B and no combined z score has been calculated.
In **Appendices 5 and 6**, only results of laboratories in Category A have been presented, along with their graphical representations.

4. RESULTS

4.1 Summary of reported results

The laboratories that agreed to participate are listed in Annex B. All results reported by the participants are given in **Appendix 2**.

Forty-five 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. Only one laboratory from a non-EU/EFTA country (Peru) participated in EUPT-SC04. The results from that laboratory in Peru have not been included for the calculation of the assigned value.

Up to 28 pesticide residues from the pesticide target list were detected in the test item. Five of them, bromuconazole (sum of diastereoisomers), chlorantraniliprole, famoxadone and spirotetramat metabolite BYI08330-monohydroxy were present at concentrations below the MRRL, and 23 of them were present at concentrations above the MRRL. However, cyflufenamid (sum of cyflufenamid (z-isomer) and its e-isomer), ethirimol, fenvalerate (any ratio of constituent isomers (RR, SS, RS & SR) including esfenvalerate), lambda-cyhalothrin, metrafenone, penconazole, proquinazid, spirotetramat metabolite BYI08330-enol, tebuconazole and zoxamide had assigned values below three times their MRRL (0.01 mg/kg in all cases). For this reason, the SC agreed that they should not be considered for the evaluation of the participants. Information about them will be displayed only for informative purposes.

A summary of the reported results for both the pesticides evaluated and those informative can be seen below in **Table 4.a** and **Table 4.b**.

Table 4.a. 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 44)
Boscalid	43	0	1	98
Carbendazim	42	1	1	95
Cyprodinil	42	2	0	95
Flubendiamide	31	6	7	70
Fluxapyroxad	41	0	3	93
Indoxacarb	43	0	1	98
Mandipropamid	43	0	1	98
Metalaxyl and metalaxyl-M	43	0	1	98
Methoxyfenozide	42	1	1	95
Pyrimethanil	44	0	0	100
Spirotetramat metabolite BYI08330 enol-glucoside	35	0	9	80
Spirotetramat metabolite BYI08330-ketohydroxy	34	2	8	77
Thiophanate-methyl	40	2	2	91

[a] The percentage of Reported Results comes from 44 laboratories. It does not take into account the laboratory from Peru.

Table 4.b. Summary of Reported Results for informative purposes.

Pesticides (only for informative purposes)	No. of Reported Results	No. of False Negative Results	No. of Not Analysed Results	Percentage of Reported Results[a] (out of 44)
Cyflufenamid	38	1	5	86
Ethirimol	27	15	2	61
Fenvalerate	38	4	2	86
Lambda-Cyhalothrin	41	2	1	93
Metrafenone	40	1	3	91
Penconazole	33	11	0	75
Proquinazid	37	4	3	84
Spirotetramat metabolite BYI08330-enol	30	6	8	68
Tebuconazole	40	4	0	91
Zoxamide	31	11	2	70

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. Two additional laboratories reported spirotetramat (present in the test item at concentrations below the MRRL) at concentrations above the MRRL. However, the Scientific Committee decided that it would not be considered as a false positive result.

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
Lab029	Cypermethrin	0.01	0.024	GC-MS/MS (QQQ) LC-MS (QTOF)
Lab013	Prothiofos	0.04	0.0475	LC-MS/MS (QQQ)

4.1.2 False negatives

Table 6 summarises the results from laboratories (including non-EU/EFTA laboratories) that reported false negatives, presented as 'Not Detected' (ND). Those pesticides for which their assigned value is below three times the MRRL are not included in this table, as in that case, false negatives can not be assigned.

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

Laboratory	Carbendazim	Cyprodinil	Flubendiamide	Spirotetramat metabolite BYI08330– ketohydroxy	Thiophanate-methyl
Lab014			ND		
Lab016			ND	ND	
Lab025		ND			
Lab029	ND				
Lab039		ND			
Lab040			ND		
Lab041			ND		
Lab042			ND	ND	
Lab043					ND

ND: Not detected

4.2 Assigned values and target standard deviations

The assigned values are based on the robust mean values calculated using all the results reported by laboratories from EU and EFTA countries. The assigned values for the 23 pesticides and their uncertainties are presented in **Table 7.a** and **Table 7.b**.

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.a** and **Table 7.b**.

The assigned value for cyflufenamid (sum of cyflufenamid (z-isomer) and its e-isomer), ethirimol, fenvalerate (any ratio of constituent isomers (RR, SS, RS & SR) including esfenvalerate), lambda-cyhalothrin, metrafenone, penconazole, proquinazid, spirotetramat metabolite BYI08330-enol, tebuconazole and zoxamide was below three times their MRRL. For this reason, the SC agreed that they should not be considered for the evaluation of the participants. Information for those pesticides is displayed only for informative purposes in **Table 7.b**.

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

Pesticides	n	Robust mean	CV(%)	MRRL	Uncertainty
Boscalid	43	0.052	11.26	0.01	0.001
Carbendazim	42	0.229	22.7	0.01	0.010
Cyprodinil	42	1.073	9.2	0.01	0.019

Pesticides	n	Robust mean	CV(%)	MRRL	Uncertainty
Flubendiamide	31	0.035	13.6	0.01	0.001
Fluxapyroxad	41	0.105	15.1	0.01	0.003
Indoxacarb	43	0.050	17.6	0.01	0.002
Mandipropamid	43	0.047	13.9	0.01	0.001
Metalaxyl and metalaxyl-M	43	0.242	11.7	0.01	0.005
Methoxyfenozide	42	0.229	13.3	0.01	0.006
Pyrimethanil	44	0.401	10.8	0.01	0.008
Spirotetramat metabolite BYI08330 enol-glucoside	35	0.271	29.1	0.01	0.017
Spirotetramat metabolite BYI08330-ketohydroxy	34	0.032	22.2	0.01	0.002
Thiophanate-methyl	40	0.075	35.5	0.01	0.005

Table 7.b.
Robust mean values, uncertainty and % RSDs for pesticides shown for informative purposes.

Pesticides	n	Robust mean	CV(%)	MRRL	Uncertainty
Cyflufenamid	38	0.020	16.9	0.01	0.001
Ethirimol	27	0.011	18.3	0.01	0.0005
Fenvalerate	38	0.022	23.8	0.01	0.001
Lambda-Cyhalothrin	41	0.025	23.0	0.01	0.001
Metrafenone	40	0.022	15.9	0.01	0.001
Penconazole	33	0.011	10.5	0.01	0.0002
Proquinazid	37	0.018	23.8	0.01	0.001
Spirotetramat metabolite BYI08330-enol	30	0.016	31.9	0.01	0.001
Tebuconazole	40	0.015	11.4	0.01	0.0003
Zoxamide	31	0.011	9.2	0.01	0.0002

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 laboratory from Peru has been included in **Appendix 2** but has not been considered in **Table 8.a** or **Table 8.b**, where the classification of z scores reported by EU/EFTA laboratories is shown.

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

%	Robust Mean	Acceptable	Questionable	Unacceptable
Boscalid	0.052	95.3	4.7	0.0
Carbendazim	0.229	88.4	7.0	4.7
Cyprodinil	1.073	93.2	2.3	4.5
Flubendiamide	0.035	81.1	0.0	18.9
Fluxapyroxad	0.105	100.0	0.0	0.0
Indoxacarb	0.050	97.7	2.3	0.0
Mandipropamid	0.047	100.0	0.0	0.0
Metalaxyl and metalaxyl-M	0.242	97.7	2.3	0.0
Methoxyfenozide	0.229	97.7	0.0	2.3
Pyrimethanil	0.401	100.0	0.0	0.0
Spirotetramat metabolite BYI08330 enol-glucoside	0.271	100.0	0.0	0.0
Spirotetramat metabolite BYI08330-ketohydroxy	0.032	91.7	2.8	5.6
Thiophanate-methyl	0.075	83.3	7.1	9.5

Table 8.b.
Classification of z scores for the pesticides shown only for informative purposes (only EU/EFTA participants)

%	Robust Mean	Acceptable	Questionable	Unacceptable
Cyflufenamid	0.020	97.4	0.0	2.6
Ethirimol	0.011	95.2	0.0	4.8
Fenvalerate	0.022	90.5	2.4	7.1
Lambda-Cyhalothrin	0.025	95.3	2.3	2.3
Metrafenone	0.022	97.6	2.4	0.0
Penconazole	0.011	100.0	0.0	0.0
Proquinazid	0.018	100.0	0.0	0.0
Spirotetramat metabolite BYI08330-enol	0.016	86.1	5.6	8.3

%	Robust Mean	Acceptable	Questionable	Unacceptable
Tebuconazole	0.015	100.0	0.0	0.0
Zoxamide	0.011	100.0	0.0	0.0

z scores for false negative results have been calculated using the MRRL value given in the Target Pesticide List (Annex A) or the RL value from the laboratory (whichever was lower).

In **Appendix 3**, graphical representations of the z scores of EU/EFTA laboratories are presented. No z scores have been calculated for false positive results; z scores for false negative results have been included on the chart and are indicated by an asterisk. The charts have been constructed using different colour bars according to the determination technique used for each pesticide.

4.3.2 Combined z scores

As previously mentioned in Section 3.5., the AZ^2 formula has only been applied to those participants categorised into Category A and considering only compulsory pesticides.

The table in **Appendix 4** shows the values of individual z scores for each evaluated pesticide and the combined 'Average of the Squared z scores' (AZ^2) for all EU/EFTA laboratories in Category A (including non-EU/EFTA countries), which were those laboratories that were able to analyse at least 90 % of the pesticides in the target pesticides list (187), to detect and quantify at least 90 % of the pesticides present in the Test Item (12), 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 of the 44 EU and EFTA laboratories that submitted results were classified into Category A (68 %).

From the AZ^2, 93 % were classed as 'good', 7 % as 'satisfactory' and 0 % as 'unsatisfactory' (Only considering EU and EFTA laboratories).

Of the 14 EU and EFTA laboratories in Category B, two had reported a false positive result. One of them would have been classified into Category A if not for that false positive result.

Table 9 shows all the laboratories in Category A, the number of evaluated pesticides reported, the percentage of pesticides analysed from the target list, the AZ^2 values and their subclassifications. Laboratories that reported false negative results in Category A are marked with the symbol Θ.

Table 10 shows all the laboratories in Category B, the number of results reported, the percentage of pesticides analysed from the target list and the number of acceptable z scores. Laboratories

reporting a false negative are marked with the symbol ⊖ and laboratories reporting a false positive are marked with a '+'.

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

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

Lab Code	No. of pesticides detected (max.13)	% of pesticides analysed from target list	AZ^2	Classification
Lab003	12	96%	2.8	Satisfactory
Lab004	13	100%	0.4	Good
Lab005	13	100%	0.3	Good
Lab006	13	100%	0.3	Good
Lab007	13	100%	0.4	Good
Lab008 ⊖	12	94%	2.9	Satisfactory
Lab010 ⊖	12	100%	1.5	Good
Lab011	13	100%	1.9	Good
Lab014 ⊖	12	93%	1.9	Good
Lab015	13	100%	0.2	Good
Lab018	13	99%	0.6	Good
Lab019	13	98%	1.9	Good
Lab020	13	100%	0.2	Good
Lab021	13	100%	0.2	Good
Lab022	13	100%	0.8	Good
Lab023	13	100%	0.5	Good
Lab024	13	100%	1.9	Good
Lab026	13	100%	0.2	Good
Lab027	12	98%	0.7	Good
Lab028	12	99%	0.9	Good
Lab030	13	100%	0.2	Good
Lab031	13	92%	0.9	Good
Lab034	13	100%	0.4	Good
Lab036	13	100%	0.4	Good
Lab037	13	100%	1.6	Good
Lab038	13	100%	0.9	Good
Lab040 ⊖	12	100%	1.5	Good
Lab041 ⊖	12	100%	1.1	Good
Lab043 ⊖	12	100%	1.1	Good
Lab044	13	100%	0.6	Good
Lab046	13	100%	2.5	Satisfactory

⊖ Laboratories reporting a false negative result.

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)
Lab002	11	85%	93%	11	10
Lab009	9	69%	60%	9	9
Lab012 Θ	7	54%	87%	8	6
Lab013 +	9	69%	82%	9	9
Lab016 Θ	11	85%	100%	13	11
Lab017	13	100%	86%	13	13
Lab025 Θ	11	85%	90%	12	10
Lab029 Θ +	12	92%	100%	13	12
Lab032	10	77%	88%	10	10
Lab033	6	46%	58%	6	6
Lab035	11	85%	97%	11	11
Lab039 Θ	11	85%	99%	12	10
Lab042 Θ	10	77%	100%	12	9
Lab045	10	77%	87%	10	8

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

5. CONCLUSIONS

Forty-five laboratories agreed to participate in EUPT-FV-SC04. One of them did not belong to EU nor EFTA countries, so their results were not considered for the estimation of the assigned value.

From the total 28 pesticides present in EUPT-FV-SC04 test item, four of them were at concentrations below the MRRL, and 10 had an assigned value below three times its MRRL. Therefore, they were excluded from the data treatment (information is shown for informative purposes).

Of a total number of 572 possible determinations from EU/EFTA laboratories (44 laboratories by 13 evaluated pesticides), 91.4 % were reported, 6.1 % were not analysed and 2.4 % were not detected (false negative results).

68 % of the EU and EFTA laboratories that submitted results were classified into Category A. Of them, 93 % were classed as 'good', 7 % as 'satisfactory' and 0 % as 'unsatisfactory'.

The robust standard deviation (CV*) was below 25 % for most of the pesticides, except for spirotetramat metabolite BYI08330-enol, which had a CV* of 34.5 %. The average value of CV* was 17.4 % for the 13 pesticides evaluated.

Participation in this EUPT-FV-SC04 involved laboratories from 20 EU Member States and 2 EFTA countries (Iceland and Norwey). As laid down in paragraph 2 (h) of Article 94 of Regulation (EU) 2017/625, one of the EURL's duties is to collaborate with non-EU laboratories that are responsible for analysing food and feed samples and to help them improve the quality of their analyses. A non-European laboratory from Peru participated in EUPT-FV-SC04.

6. REFERENCES

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Boscalid (mg/kg)		Carbendazim (mg/kg)		Cyprodinil (mg/kg)		Flubendiamide (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.055	0.064	0.239	0.243	1.194	1.300	0.038	0.037
0.062	0.048	0.235	0.244	1.285	1.034	0.040	0.039
0.054	0.055	0.242	0.239	1.145	1.177	0.037	0.041
0.051	0.044	0.241	0.240	1.093	0.942	0.034	0.034
0.048	0.045	0.243	0.238	1.007	0.992	0.034	0.035
0.050	0.048	0.226	0.227	1.070	1.035	0.032	0.034
0.046	0.042	0.233	0.225	1.004	0.918	0.033	0.033
0.049	0.039	0.243	0.248	1.040	0.894	0.039	0.038
0.047	0.045	0.236	0.236	1.002	0.959	0.033	0.036
0.042	0.044	0.233	0.234	0.920	0.962	0.040	0.036

Fluxapyroxad (mg/kg)		Indoxacarb (mg/kg)		Mandipropamid (mg/kg)		Metalaxyl (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.100	0.105	0.052	0.054	0.039	0.042	0.253	0.286
0.100	0.102	0.051	0.053	0.040	0.041	0.285	0.235
0.100	0.104	0.053	0.052	0.040	0.041	0.242	0.253
0.124	0.069	0.046	0.046	0.051	0.026	0.237	0.212
0.125	0.073	0.045	0.046	0.050	0.028	0.222	0.212
0.118	0.068	0.044	0.045	0.047	0.025	0.235	0.218
0.119	0.067	0.045	0.042	0.048	0.025	0.220	0.202
0.102	0.103	0.053	0.049	0.040	0.040	0.231	0.192
0.122	0.069	0.048	0.045	0.050	0.026	0.222	0.217
0.097	0.097	0.047	0.051	0.039	0.040	0.205	0.209

Methoxyfenozide (mg/kg)		Pyrimethanil (mg/kg)		Spirotetramat-enolglukoside (mg/kg)		Spirotetramat-ketohydroxy (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.198	0.207	0.453	0.510	0.278	0.286	0.029	0.031
0.199	0.201	0.497	0.405	0.270	0.282	0.028	0.029
0.204	0.203	0.432	0.444	0.276	0.291	0.033	0.032
0.241	0.144	0.424	0.368	0.263	0.270	0.031	0.032
0.237	0.151	0.402	0.380	0.267	0.280	0.033	0.034
0.225	0.138	0.414	0.395	0.258	0.259	0.032	0.029
0.226	0.142	0.390	0.354	0.273	0.265	0.034	0.032
0.200	0.201	0.396	0.336	0.282	0.280	0.027	0.028
0.235	0.142	0.395	0.376	0.272	0.281	0.033	0.031
0.191	0.193	0.355	0.375	0.276	0.280	0.030	0.031

APPENDIX 1. Homogeneity data.

Thiophanate-methyl (mg/kg)	
Replicate 1	Replicate 2
0.125	0.119
0.120	0.121
0.117	0.120
0.102	0.109
0.091	0.114
0.096	0.098
0.109	0.104
0.121	0.108
0.111	0.114
0.130	0.126

ⓘFor informative purposes only

The sample numbers used for this test were: 4, 12, 16, 23, 27, 56, 70, 72, 74, 79.

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

Lab Code	Boscalid	z score (FFP-RSD 25 %)	Carbendazim	z score (FFP-RSD 25 %)	Cyprodinil	z score (FFP-RSD 25 %)	Flubendiamide	z score (FFP-RSD 25 %)	Fluxapyroxad	z score (FFP-RSD 25 %)	Indoxacarb	z score (FFP-RSD 25 %)	Mandipropamid	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)	**0.052**		**0.229**		**1.073**		**0.035**		**0.105**		**0.05**		**0.047**	
Lab002	0.0497	-0.2	0.229	0.0	0.994	-0.3	0.0216	-1.5	0.0887	-0.6	0.045	-0.4	0.0357	-1.0
Lab003	0.053	0.0	0.38	2.6	0.95	-0.5	0.035	0.0	0.095	-0.4	0.049	-0.1	0.05	0.2
Lab004	0.059	0.5	0.263	0.6	1.146	0.3	0.037	0.2	0.118	0.5	0.057	0.5	0.051	0.3
Lab005	0.055	0.2	0.238	0.2	1.03	-0.2	0.03	-0.6	0.098	-0.3	0.045	-0.4	0.043	-0.4
Lab006	0.051	-0.1	0.212	-0.3	1.109	0.1	0.036	0.1	0.11	0.2	0.061	0.8	0.053	0.5
Lab007	0.055	0.2	0.27	0.7	1.07	0.0	0.038	0.3	0.099	-0.2	0.063	1.0	0.049	0.1
Lab008	0.045	-0.6	0.199	-0.5	1.242	0.6	ND	-3.5	0.07	-1.3	0.065	1.2	0.048	0.1
Lab009	0.062	0.7	0.197	-0.6	1.07	0.0	NA		NA		0.054	0.3	0.053	0.5
Lab010	0.06	0.6	0.192	-0.6	0.89	-0.7	0.031	-0.5	0.111	0.2	0.056	0.4	0.052	0.4
Lab011	0.048	-0.3	0.51	4.9	1	-0.3	0.037	0.2	0.11	0.2	0.048	-0.2	0.048	0.1
Lab012	0.041	-0.9	0.079	-2.6	0.98	-0.3	0.033	-0.2	NA		NA		0.041	-0.5
Lab013	0.0481	-0.3	0.134	-1.7	1.06	0.0	NA		0.113	0.3	0.0412	-0.7	0.0409	-0.5
Lab014	0.058	0.4	0.279	0.9	1.28	0.8	ND	-3.5	0.131	1.0	0.058	0.6	0.065	1.5
Lab015	0.052	0.0	0.278	0.9	1.074	0.0	0.03	-0.6	0.098	-0.3	0.048	-0.2	0.041	-0.5
Lab016	0.049	-0.3	0.24	0.2	0.95	-0.5	ND	-3.5	0.11	0.2	0.049	-0.1	0.052	0.4
Lab017	0.052	0.0	0.224	-0.1	1.093	0.1	0.0382	0.4	0.106	0.0	0.0429	-0.6	0.0515	0.4
Lab018	0.049	-0.3	0.223	-0.1	1.1	0.1	0.03	-0.6	0.1	-0.2	0.04	-0.8	0.048	0.1
Lab019	0.049	-0.3	0.226	-0.1	1.01	-0.2	0.034	-0.1	0.098	-0.3	0.046	-0.3	0.044	-0.3
Lab020	0.057	0.4	0.287	1.0	1.13	0.2	0.036	0.1	0.108	0.1	0.06	0.8	0.049	0.1
Lab021	0.058	0.4	0.243	0.2	0.97	-0.4	0.035	0.0	0.11	0.2	0.052	0.1	0.049	0.1
Lab022	0.054	0.1	0.25	0.4	1.3	0.8	0.04	0.6	0.129	0.9	0.051	0.0	0.066	1.6
Lab023	0.054	0.1	0.263	0.6	1.076	0.0	0.042	0.8	0.105	0.0	0.048	-0.2	0.044	-0.3
Lab024	0.04	-0.9	0.24	0.2	0.66	-1.5	0.03	-0.6	0.09	-0.6	0.04	-0.8	0.04	-0.6
Lab025	0.055	0.2	0.36	2.3	ND	-4.0	NA		0.13	0.9	0.042	-0.7	0.047	0.0
Lab026	0.04661576	-0.4	0.261	0.6	1.17	0.4	0.035	0.0	0.10035707	-0.2	0.0482864	-0.2	0.043	-0.4
Lab027	0.0479	-0.3	0.1674	-1.1	1.095	0.1	NA		0.1072	0.1	0.0444	-0.5	0.0411	-0.5
Lab028	0.045	-0.6	0.16	-1.2	0.95	-0.5	NA		0.082	-0.9	0.039	-0.9	0.033	-1.2
Lab029	0.05	-0.2	ND	-3.8	1.1	0.1	0.037	0.2	0.12	0.5	0.075	2.0	0.047	0.0
Lab030	0.052	0.0	0.26	0.5	1.14	0.2	0.034	-0.1	0.096	-0.4	0.05	0.0	0.051	0.3
Lab031	0.028	-1.9	0.187	-0.7	0.84	-0.9	0.035	0.0	0.088	-0.7	0.038	-1.0	0.045	-0.2

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

Lab Code	Boscalid	z score (FFP-RSD 25 %)	Carbendazim	z score (FFP-RSD 25 %)	Cyprodinil	z score (FFP-RSD 25 %)	Flubendiamide	z score (FFP-RSD 25 %)	Fluxapyroxad	z score (FFP-RSD 25 %)	Indoxacarb	z score (FFP-RSD 25 %)	Mandipropamid	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)	**0.052**		**0.229**		**1.073**		**0.035**		**0.105**		**0.05**		**0.047**	
Lab032	0.0528	0.0	0.201	-0.5	0.99	-0.3	0.0362	0.1	NA		0.0475	-0.2	0.0515	0.4
Lab033	0.05	-0.2	NA		1.105	0.1	NA		0.085	-0.8	0.05	0.0	NA	
Lab034	0.051	-0.1	0.18	-0.9	1	-0.3	0.028	-0.8	0.092	-0.5	0.047	-0.3	0.047	0.0
Lab035	0.0485	-0.3	0.23	0.0	1.07	0.0	0.0311	-0.5	0.0949	-0.4	0.0474	-0.2	0.0433	-0.3
Lab036	0.055	0.2	0.29	1.1	1.032	-0.2	0.038	0.3	0.109	0.1	0.053	0.2	0.051	0.3
Lab037	0.087	2.6	0.252	0.4	1.284	0.8	0.049	1.6	0.149	1.6	0.067	1.3	0.064	1.4
Lab038	0.049	-0.3	0.16	-1.2	1.1	0.1	0.039	0.4	0.14	1.3	0.059	0.7	0.058	0.9
Lab039	0.08	2.1	0.153	-1.3	ND	-4.0	NA		0.103	-0.1	0.065	1.2	0.058	0.9
Lab040	0.06	0.6	0.239	0.2	1.08	0.0	ND	-3.5	0.108	0.1	0.021	-2.3	0.047	0.0
Lab041	0.053	0.0	0.225	-0.1	1.029	-0.2	ND	-3.5	0.097	-0.3	0.043	-0.6	0.04	-0.6
Lab042	0.049	-0.3	0.317	1.5	1.04	-0.1	ND	-3.5	0.101	-0.2	0.049	-0.1	0.039	-0.7
Lab043	0.054	0.1	0.25	0.4	1.1	0.1	0.031	-0.5	0.11	0.2	0.052	0.1	0.044	-0.3
Lab044	0.056	0.3	0.21	-0.3	1.3	0.8	0.041	0.7	0.12	0.5	0.06	0.8	0.05	0.2
Lab045	NA		0.093	-2.4	1.66	2.2	0.033	-0.2	0.07	-1.3	0.045	-0.4	0.038	-0.8
Lab046	0.063	0.8	0.27	0.7	1.1	0.1	0.083	5.0	0.13	0.9	0.056	0.4	0.051	0.3

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

Lab Code	Metalaxyl and metalaxyl-M	z score (FFP-RSD 25 %)	Methoxyfenozide	z score (FFP-RSD 25 %)	Pyrimethanil	z score (FFP-RSD 25 %)	Spirotetramat metabolite BYI08330 enol-glucoside	z score (FFP-RSD 25 %)	Spirotetramat metabolite BYI08330-ketohydroxy	z score (FFP-RSD 25 %)	Thiophanate-methyl	z score (FFP-RSD 25 %)
MRRL (mg/kg)	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Robust mean (mg/kg)	**0.242**		**0.229**		**0.401**		**0.271**		**0.032**		**0.075**	
Lab002	0.221	-0.4	0.186	-0.7	0.4	0.0	NA		NA		0.028	-2.5
Lab003	0.207	-0.6	0.27	0.7	0.357	-0.4	0.787	5.0	0.03	-0.2	NA	
Lab004	0.241	0.0	0.234	0.1	0.428	0.3	0.365	1.4	0.042	1.3	0.075	0.0
Lab005	0.24	0.0	0.243	0.2	0.373	-0.3	0.338	1.0	0.035	0.4	0.098	1.2
Lab006	0.223	-0.3	0.218	-0.2	0.439	0.4	0.323	0.8	0.029	-0.3	0.103	1.5
Lab007	0.23	-0.2	0.21	-0.3	0.36	-0.4	0.33	0.9	0.031	-0.1	0.047	-1.5
Lab008	0.25	0.1	0.236	0.1	0.458	0.6	0.161	-1.6	0.023	-1.1	0.153	4.2
Lab009	0.31	1.1	0.25	0.4	0.46	0.6	NA		NA		0.085	0.5
Lab010	0.2	-0.7	ND	-3.8	0.35	-0.5	0.282	0.2	0.026	-0.7	0.101	1.4

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

Lab Code	Metalaxyl and metalaxyl-M	z score (FFP-RSD 25 %)	Methoxyfenozide	z score (FFP-RSD 25 %)	Pyrimethanil	z score (FFP-RSD 25 %)	Spirotetramat metabolite BYI08330 enol-glucoside	z score (FFP-RSD 25 %)	Spirotetramat metabolite BYI08330-ketohydroxy	z score (FFP-RSD 25 %)	Thiophanate-methyl	z score (FFP-RSD 25 %)
MRRL (mg/kg)	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Robust mean (mg/kg)	**0.242**		**0.229**		**0.401**		**0.271**		**0.032**		**0.075**	
Lab011	0.22	-0.4	0.22	-0.2	0.39	-0.1	0.25	-0.3	0.033	0.2	0.064	-0.6
Lab012	NA		0.169	-1.0	0.38	-0.2	NA		NA		ND	-3.5
Lab013	0.235	-0.1	0.225	-0.1	0.368	-0.3	NA		NA		NA	
Lab014	0.25	0.1	0.27	0.7	0.33	-0.7	0.292	0.3	0.051	2.4	0.091	0.8
Lab015	0.247	0.1	0.252	0.4	0.437	0.4	0.222	-0.7	0.032	0.0	0.086	0.6
Lab016	0.26	0.3	0.24	0.2	0.36	-0.4	0.22	-0.8	ND	-3.5	0.085	0.5
Lab017	0.256	0.2	0.2527	0.4	0.401	0.0	0.287	0.2	0.0316	0.0	0.0885	0.7
Lab018	0.27	0.5	0.23	0.0	0.42	0.2	0.17	-1.5	0.016	-2.0	0.085	0.5
Lab019	0.237	-0.1	0.206	-0.4	0.402	0.0	0.323	0.8	0.034	0.3	0.167	4.9
Lab020	0.234	-0.1	0.231	0.0	0.397	0.0	0.282	0.2	0.035	0.4	0.074	-0.1
Lab021	0.28	0.6	0.236	0.1	0.47	0.7	0.345	1.1	0.035	0.4	0.073	-0.1
Lab022	0.24	0.0	0.29	1.1	0.4	0.0	0.35	1.2	0.037	0.7	0.104	1.5
Lab023	0.258	0.3	0.251	0.4	0.414	0.1	0.304	0.5	0.044	1.5	0.045	-1.6
Lab024	0.11	-2.2	0.2	-0.5	0.33	-0.7	0.16	-1.6	0.02	-1.5	0.02	-2.9
Lab025	0.22	-0.4	0.22	-0.2	0.35	-0.5	0.19	-1.2	0.033	0.2	0.05	-1.3
Lab026	0.2544241	0.2	0.246	0.3	0.42216549	0.2	0.27	0.0	0.032188	0.1	0.0487	-1.4
Lab027	0.202	-0.7	0.202	-0.5	0.454	0.5	0.14	-1.9	0.0286	-0.4	0.0526	-1.2
Lab028	0.21	-0.5	0.15	-1.4	0.32	-0.8	0.24	-0.5	0.02	-1.5	0.079	0.2
Lab029	0.28	0.6	0.23	0.0	0.39	-0.1	0.26	-0.2	0.028	-0.5	0.055	-1.1
Lab030	0.26	0.3	0.23	0.0	0.39	-0.1	0.35	1.2	0.034	0.3	0.06	-0.8
Lab031	0.214	-0.5	0.151	-1.4	0.372	-0.3	0.206	-1.0	0.022	-1.2	0.092	0.9
Lab032	0.263	0.3	0.227	0.0	0.417	0.2	NA		NA		0.0837	0.5
Lab033	0.21	-0.5	NA		0.395	-0.1	NA		NA		NA	
Lab034	0.22	-0.4	0.21	-0.3	0.37	-0.3	0.2	-1.0	0.035	0.4	0.097	1.2
Lab035	0.235	-0.1	0.218	-0.2	0.407	0.1	NA		NA		0.0492	-1.4
Lab036	0.222	-0.3	0.214	-0.3	0.351	-0.5	0.376	1.5	0.027	-0.6	0.059	-0.9
Lab037	0.325	1.4	0.266	0.6	0.46	0.6	0.252	-0.3	0.04	1.0	0.062	-0.7
Lab038	0.26	0.3	0.29	1.1	0.39	-0.1	0.38	1.6	0.031	-0.1	0.11	1.9
Lab039	0.27	0.5	0.258	0.5	0.44	0.4	0.303	0.5	0.03	-0.2	0.105	1.6
Lab040	0.248	0.1	0.184	-0.8	0.428	0.3	0.237	-0.5	0.039	0.9	0.073	-0.1
Lab041	0.233	-0.2	0.259	0.5	0.427	0.3	0.316	0.7	0.034	0.3	0.063	-0.6
Lab042	0.266	0.4	0.232	0.1	0.45	0.5	NA		ND	-3.5	0.028	-2.5
Lab043	0.2	-0.7	0.23	0.0	0.35	-0.5	0.21	-0.9	0.029	-0.3	ND	-3.5

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

Lab Code	Metalaxyl and metalaxyl-M	z score (FFP-RSD 25 %)	Methoxyfenozide	z score (FFP-RSD 25 %)	Pyrimethanil	z score (FFP-RSD 25 %)	Spirotetramat metabolite BYI08330 enol-glucoside	z score (FFP-RSD 25 %)	Spirotetramat metabolite BYI08330-ketohydroxy	z score (FFP-RSD 25 %)	Thiophanate-methyl	z score (FFP-RSD 25 %)
MRRL (mg/kg)	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Robust mean (mg/kg)	**0.242**		**0.229**		**0.401**		**0.271**		**0.032**		**0.075**	
Lab044	0.24	0.0	0.24	0.2	0.42	0.2	0.17	-1.5	0.024	-1.0	0.1	1.3
Lab045	0.35	1.8	0.15	-1.4	0.52	1.2	NA		NA		0.048	-1.4
Lab046	0.27	0.5	0.3	1.2	0.4	0.0	0.37	1.5	0.04	1.0	0.076	0.0

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

Lab Code	Cyflufenamid	z score (FFP-RSD 25 %)	Ethirimol	z score (FFP-RSD 25 %)	Fenvalerate	z score (FFP-RSD 25 %)	Lambda-Cyhalothrin	z score (FFP-RSD 25 %)	Metrafenone	z score (FFP-RSD 25 %)
MRRL (mg/kg)	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Robust mean (mg/kg)	**0.020**		**0.011**		**0.022**		**0.025**		**0.022**	
Lab002	NA		NR		0.022	0.0	0.0216	-0.6	0.0192	-0.5
Lab003	0.02	0.0	0.009	-0.8	0.016	-1.1	0.026	0.1	0.019	-0.5
Lab004	0.021	0.2	NR		0.022	0.0	0.022	-0.5	0.022	0.1
Lab005	0.015	-1.0	0.008	-1.2	0.015	-1.3	0.025	0.0	0.023	0.2
Lab006	0.015	-1.0	0.012	0.2	0.02	-0.4	0.022	-0.5	0.017	-0.9
Lab007	0.021	0.2	0.01	-0.5	0.02	-0.4	0.024	-0.2	0.02	-0.3
Lab008	NA		0.015	1.3	0.084	11.3	0.025	0.0	NR	
Lab009	NA		0.012	0.2	NA		0.026	0.1	NA	
Lab010	0.019	-0.2	0.009	-0.8	0.015	-1.3	0.017	-1.3	0.022	0.1
Lab011	0.021	0.2	NR		0.031	1.7	0.022	-0.5	0.023	0.2
Lab012	0.02	0.0	0.013	0.6	0.023	0.2	0.018	-1.1	0.018	-0.7
Lab013	NA		NA		NR		NR		NA	
Lab014	0.023	0.6	0.01	-0.5	NR		0.02	-0.8	0.024	0.4
Lab015	0.021	0.2	NR		0.025	0.6	0.033	1.3	0.023	0.2
Lab016	0.02	0.0	0.012	0.2	0.02	-0.4	0.0245	-0.1	0.019	-0.5
Lab017	0.016	-0.8	NR		NR		0.0268	0.3	0.0218	0.0
Lab018	0.017	-0.6	NR		0.014	-1.4	0.021	-0.7	0.017	-0.9
Lab019	0.018	-0.4	0.016	1.6	0.026	0.7	0.033	1.3	0.019	-0.5
Lab020	0.021	0.2	0.0093	-0.7	0.018	-0.7	0.021	-0.7	0.018	-0.7
Lab021	0.021	0.2	0.01	-0.5	0.022	0.0	0.032	1.1	0.021	-0.1

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

Lab Code	Cyflufenamid	z score (FFP-RSD 25 %)	Ethirimol	z score (FFP-RSD 25 %)	Fenvalerate	z score (FFP-RSD 25 %)	Lambda-Cyhalothrin	z score (FFP-RSD 25 %)	Metrafenone	z score (FFP-RSD 25 %)
MRRL (mg/kg)	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Robust mean (mg/kg)	**0.020**		**0.011**		**0.022**		**0.025**		**0.022**	
Lab022	0.02	0.0	0.012	0.2	0.022	0.0	0.019	-1.0	0.024	0.4
Lab023	0.02	0.0	0.01	-0.5	0.018	-0.7	0.016	-1.5	0.02	-0.3
Lab024	0.01	-2.0	NR		0.01	-2.2	0.02	-0.8	0.01	-2.2
Lab025	0.022	0.4	0.012	0.2	0.019	-0.5	0.058	5.2	NA	
Lab026	0.018604	-0.3	NR		0.025982	0.7	0.025043	0.0	0.020654	-0.2
Lab027	0.0148	-1.0	NR		0.025	0.6	0.033	1.3	0.025	0.6
Lab028	0.015	-1.0	NR		0.014	-1.4	0.018	-1.1	0.016	-1.0
Lab029	0.024	0.8	0.038	9.4	NR		0.024	-0.2	0.022	0.1
Lab030	0.023	0.6	0.01	-0.5	0.022	0.0	0.028	0.5	0.022	0.1
Lab031	0.02	0.0	0.009	-0.8	NA		NA		0.018	-0.7
Lab032	NA		NR		0.0193	-0.5	0.0184	-1.1	0.0207	-0.2
Lab033	0.021	0.2	NA		0.022	0.0	0.03	0.8	0.021	-0.1
Lab034	0.02	0.0	NR		0.022	0.0	0.028	0.5	0.023	0.2
Lab035	0.0193	-0.1	NR		0.0159	-1.1	0.0243	-0.1	0.0197	-0.4
Lab036	0.014	-1.2	0.01	-0.5	0.02	-0.4	0.029	0.6	0.02	-0.3
Lab037	0.04	4.0	0.011	-0.1	0.039	3.1	0.04	2.4	0.028	1.2
Lab038	0.025	1.0	0.011	-0.1	0.025	0.6	0.029	0.6	0.025	0.6
Lab039	0.021	0.2	0.011	-0.1	0.04	3.3	0.028	0.5	0.025	0.6
Lab040	0.022	0.4	0.03	6.6	0.024	0.4	0.021	-0.7	0.021	-0.1
Lab041	NR		NR		0.022	0.0	0.024	-0.2	0.032	1.9
Lab042	0.02	0.0	0.011	-0.1	0.028	1.1	0.037	1.9	0.027	1.0
Lab043	0.018	-0.4	0.011	-0.1	0.023	0.2	0.025	0.0	0.022	0.1
Lab044	0.022	0.4	0.011	-0.1	0.026	0.7	0.025	0.0	0.028	1.2
Lab045	0.025	1.0	NR		0.018	-0.7	NR		0.023	0.2
Lab046	0.023	0.6	0.014	0.9	0.027	0.9	0.03	0.8	0.028	1.2

NA: Not analysed NR: Not reported (when assigned value <3 x MRRL)

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

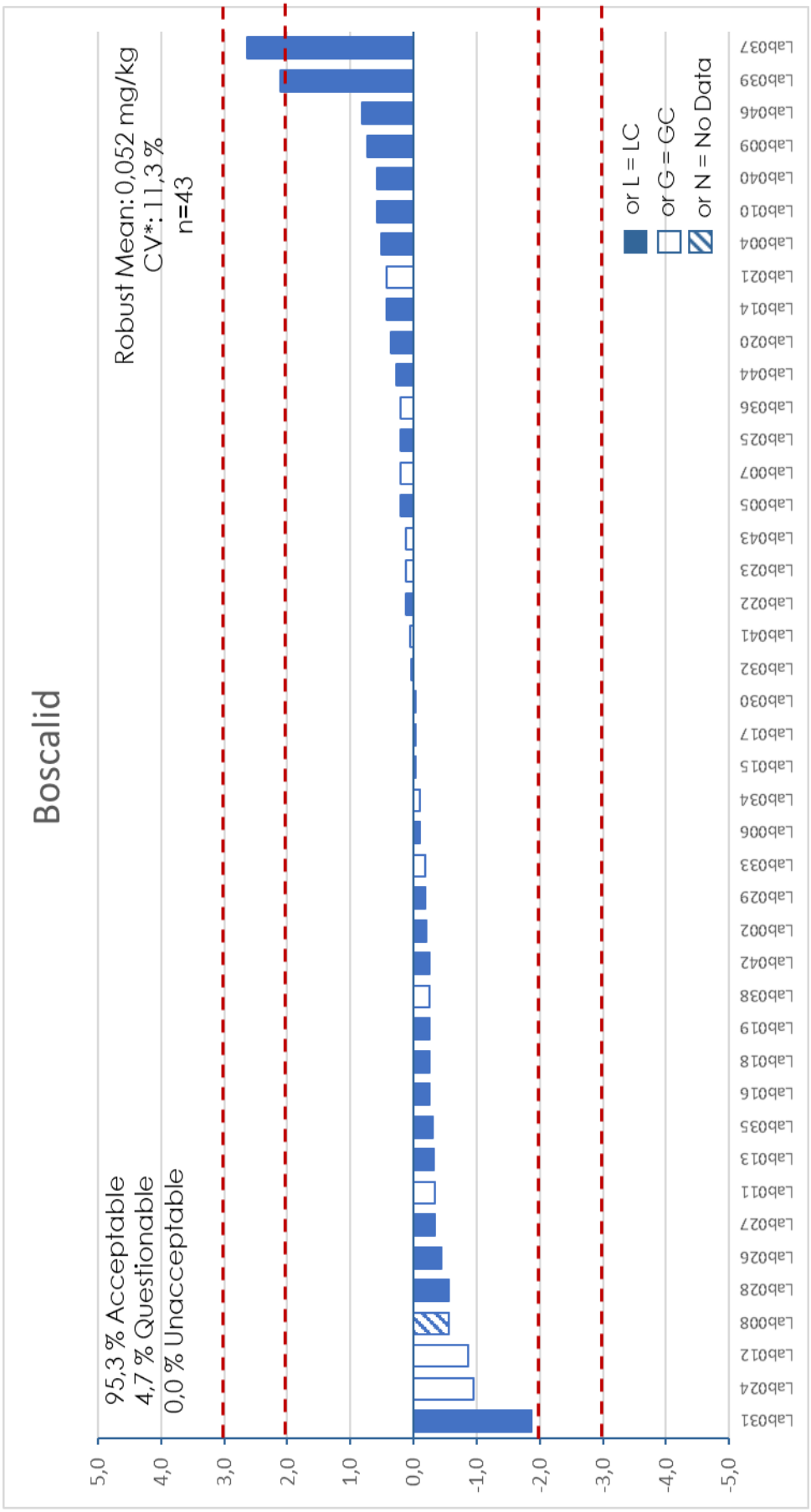
Lab Code	Penconazole	z score (FFP-RSD 25 %)	Proquinazid	z score (FFP-RSD 25 %)	Spirotetramat metabolite BYI08330-enol	z score (FFP-RSD 25 %)	Tebuconazole	z score (FFP-RSD 25 %)	Zoxamide	z score (FFP-RSD 25 %)
MRRL (mg/kg)	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Robust mean (mg/kg)	**0.011**		**0.018**		**0.016**		**0.015**		**0.011**	
Lab002	0.0094	-0.5	0.014	-0.9	NA		0.0125	-0.7	0.0102	-0.3
Lab003	0.011	0.1	0.02	0.4	0.013	-0.8	0.017	0.5	0.01	-0.4
Lab004	0.01	-0.3	0.019	0.2	0.017	0.2	0.015	0.0	0.011	-0.1
Lab005	0.01	-0.3	0.015	-0.7	0.013	-0.8	0.013	-0.5	0.01	-0.4
Lab006	NR		0.015	-0.7	0.021	1.2	0.017	0.5	NR	
Lab007	0.01	-0.3	0.018	-0.1	0.016	0.0	0.01	-1.3	0.011	-0.1
Lab008	0.01	-0.3	NR		NA		0.011	-1.1	0.012	0.3
Lab009	0.011	0.1	NA		NA		0.015	0.0	NA	
Lab010	0.011	0.1	0.021	0.6	0.004	-3.0	0.016	0.3	0.012	0.3
Lab011	NR		0.019	0.2	0.011	-1.3	0.015	0.0	0.011	-0.1
Lab012	0.012	0.4	NR		NA		0.017	0.5	NR	
Lab013	NR		NA		NA		NR		NR	
Lab014	0.01	-0.3	0.019	0.2	0.018	0.5	0.017	0.5	0.011	-0.1
Lab015	0.011	0.1	0.018	-0.1	0.024	1.9	0.014	-0.3	0.011	-0.1
Lab016	0.011	0.1	0.011	-1.6	0.014	-0.5	0.013	-0.5	NR	
Lab017	NR		0.0214	0.7	0.045	7.1	NR		NR	
Lab018	0.012	0.4	0.012	-1.4	0.015	-0.3	0.015	0.0	0.01	-0.4
Lab019	0.01	-0.3	0.018	-0.1	0.014	-0.5	0.014	-0.3	0.01	-0.4
Lab020	0.011	0.1	0.017	-0.3	0.014	-0.5	0.016	0.3	0.011	-0.1
Lab021	0.012	0.4	0.02	0.4	0.015	-0.3	0.017	0.5	0.01	-0.4
Lab022	0.01	-0.3	0.021	0.6	0.02	1.0	0.015	0.0	0.013	0.7
Lab023	0.01	-0.3	0.015	-0.7	0.018	0.5	0.014	-0.3	NR	
Lab024	NR		0.01	-1.8	0.01	-1.5	NR		0.01	-0.4
Lab025	0.012	0.4	0.019	0.2	0.031	3.7	0.016	0.3	0.012	0.3
Lab026	NR		0.020469	0.5	0.012556	-0.9	0.014215	-0.2	0.0106	-0.2
Lab027	NR		0.023	1.0	NR		0.0149	0.0	NR	
Lab028	0.012	0.4	0.012	-1.4	0.01	-1.5	0.013	-0.5	NR	
Lab029	0.011	0.1	0.024	1.3	0.01	-1.5	NR		0.011	-0.1
Lab030	0.011	0.1	0.02	0.4	0.018	0.5	0.016	0.3	0.011	-0.1
Lab031	NR		0.01	-1.8	0.014	-0.5	0.012	-0.8	NA	
Lab032	0.0102	-0.2	NA		NA		0.015	0.0	0.0108	-0.1
Lab033	0.014	1.2	0.022	0.8	NA		0.016	0.3	NR	
Lab034	NR		0.019	0.2	0.011	-1.3	0.015	0.0	0.011	-0.1

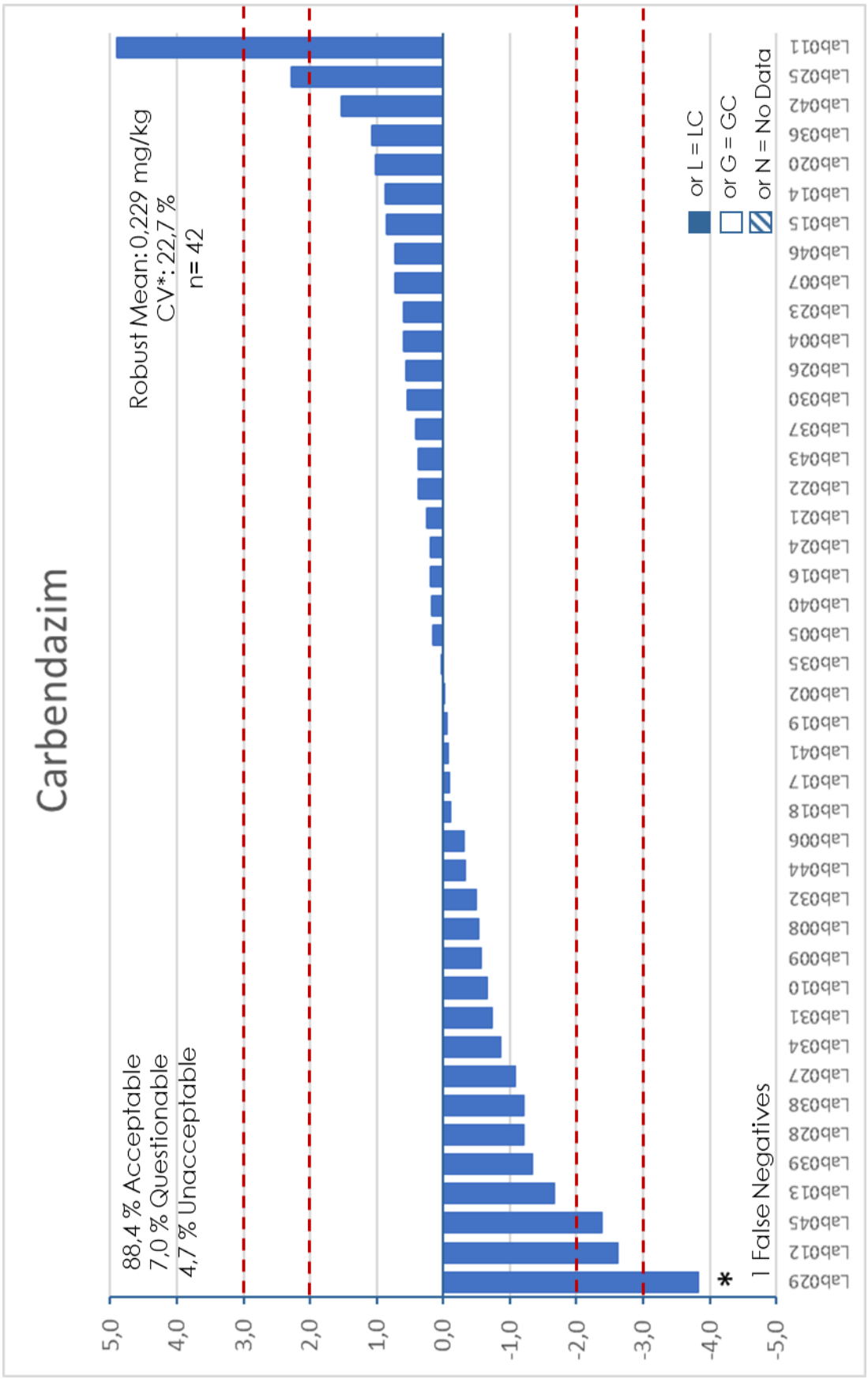
APPENDIX 2. Results (mg/Kg) and z scores for FFP-RSD (25 %).

Lab Code	Penconazole	z score (FFP-RSD 25 %)	Proquinazid	z score (FFP-RSD 25 %)	Spirotetramat metabolite BYI08330-enol	z score (FFP-RSD 25 %)	Tebuconazole	z score (FFP-RSD 25 %)	Zoxamide	z score (FFP-RSD 25 %)
MRRL (mg/kg)	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Robust mean (mg/kg)	**0.011**		**0.018**		**0.016**		**0.015**		**0.011**	
Lab035	NR		0.0129	-1.2	NR		0.0133	-0.5	NR	
Lab036	0.01	-0.3	0.017	-0.3	0.02	1.0	0.016	0.3	0.011	-0.1
Lab037	0.013	0.8	0.025	1.5	NR		0.021	1.6	0.013	0.7
Lab038	0.012	0.4	0.019	0.2	0.014	-0.5	0.015	0.0	0.014	1.0
Lab039	0.01	-0.3	0.02	0.4	0.688	166.5	0.017	0.5	0.011	-0.1
Lab040	0.012	0.4	0.018	-0.1	NR		0.016	0.3	0.012	0.3
Lab041	0.01	-0.3	NR		0.016	0.0	0.015	0.0	NR	
Lab042	0.011	0.1	0.02	0.4	NR		0.014	-0.3	0.011	-0.1
Lab043	0.009	-0.7	0.018	-0.1	0.013	-0.8	0.015	0.0	0.01	-0.4
Lab044	0.01	-0.3	0.026	1.7	0.025	2.2	0.015	0.0	0.013	0.7
Lab045	0.015	1.5	NR		NA		0.018	0.8	0.013	0.7
Lab046	NR		0.026	1.7	NR		0.017	0.5	0.011	-0.1

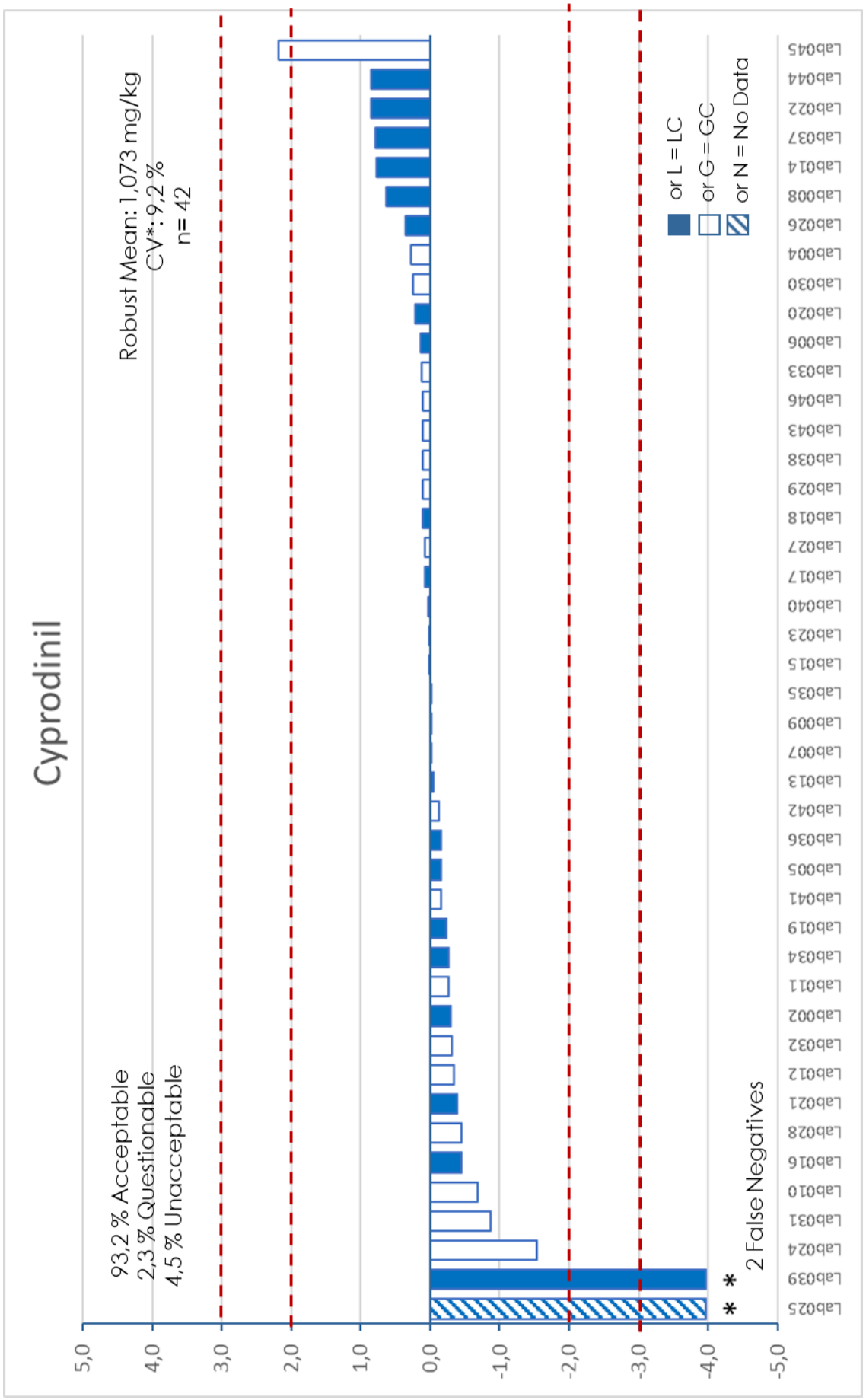
NA: Not analysed NR: Not reported (when assigned value <3 x MRRL)

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

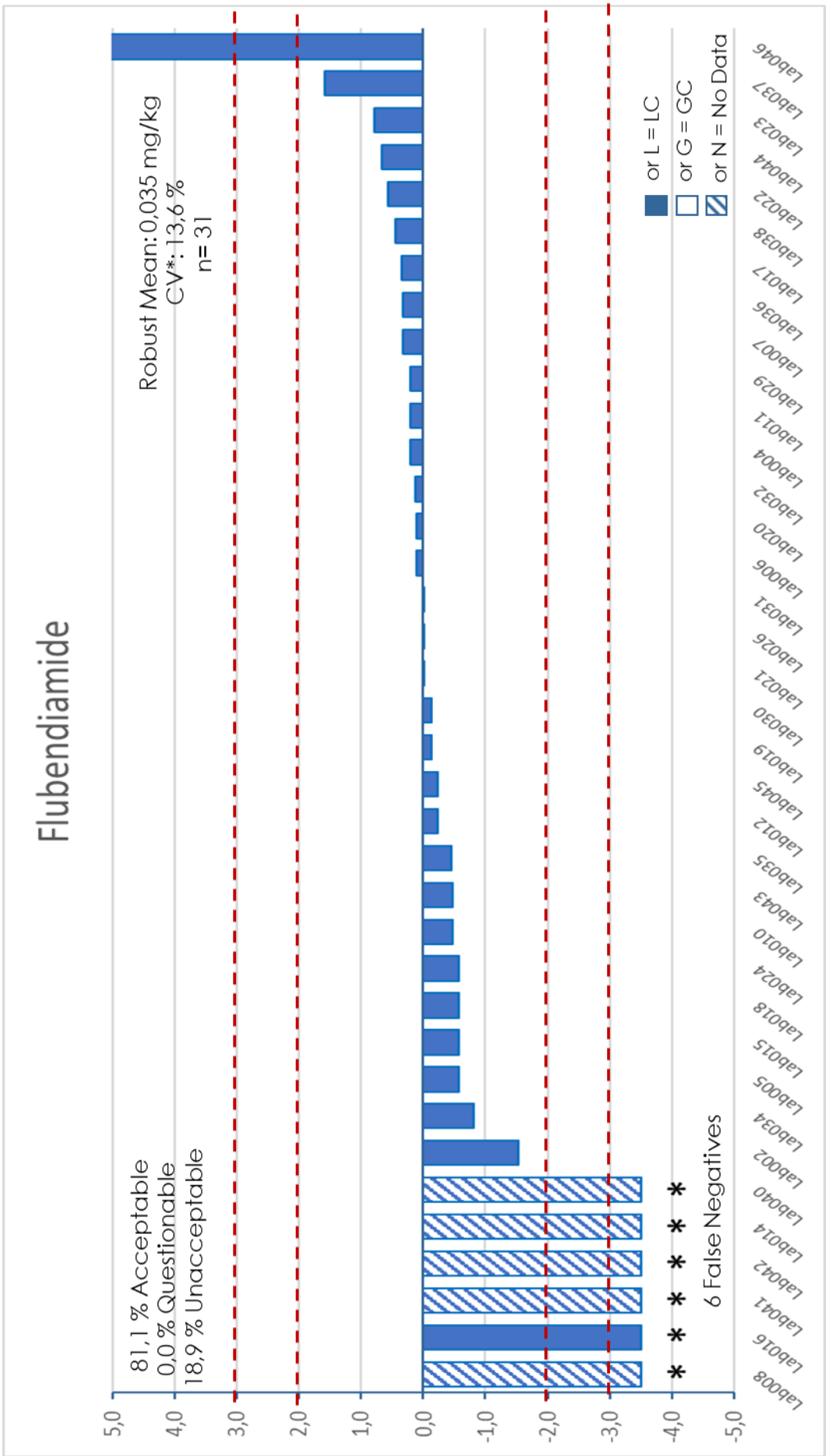



Carbendazim
Robust Mean:0,229 mg/kg
CV*:22,7 %
n= 42
88,4 % Acceptable
7,0 % Questionable
4,7 % Unacceptable
or L = LC
or G = GC
or N = No Data
1 False Negatives
*
5,0
4,0
3,0
2,0
1,0
0,0
-1,0
-2,0
-3,0
-4,0
-5,0
lab011
lab025
lab042
lab036
lab020
lab014
lab015
lab046
lab007
lab023
lab004
lab026
lab030
lab037
lab043
lab022
lab021
lab024
lab016
lab040
lab005
lab035
lab002
lab019
lab041
lab017
lab018
lab006
lab044
lab032
lab008
lab009
lab010
lab031
lab034
lab027
lab038
lab028
lab039
lab013
lab045
lab012
lab029

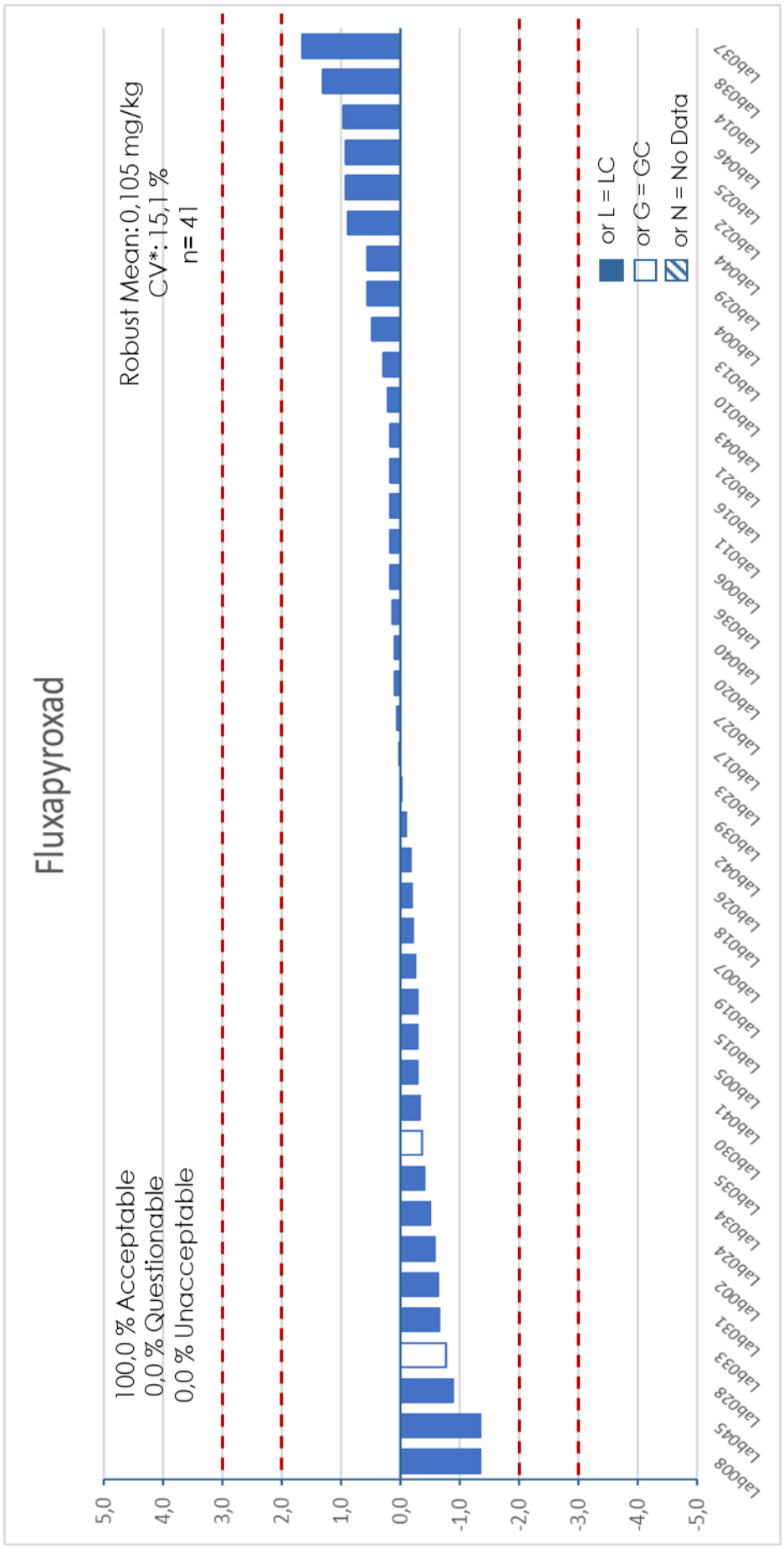
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



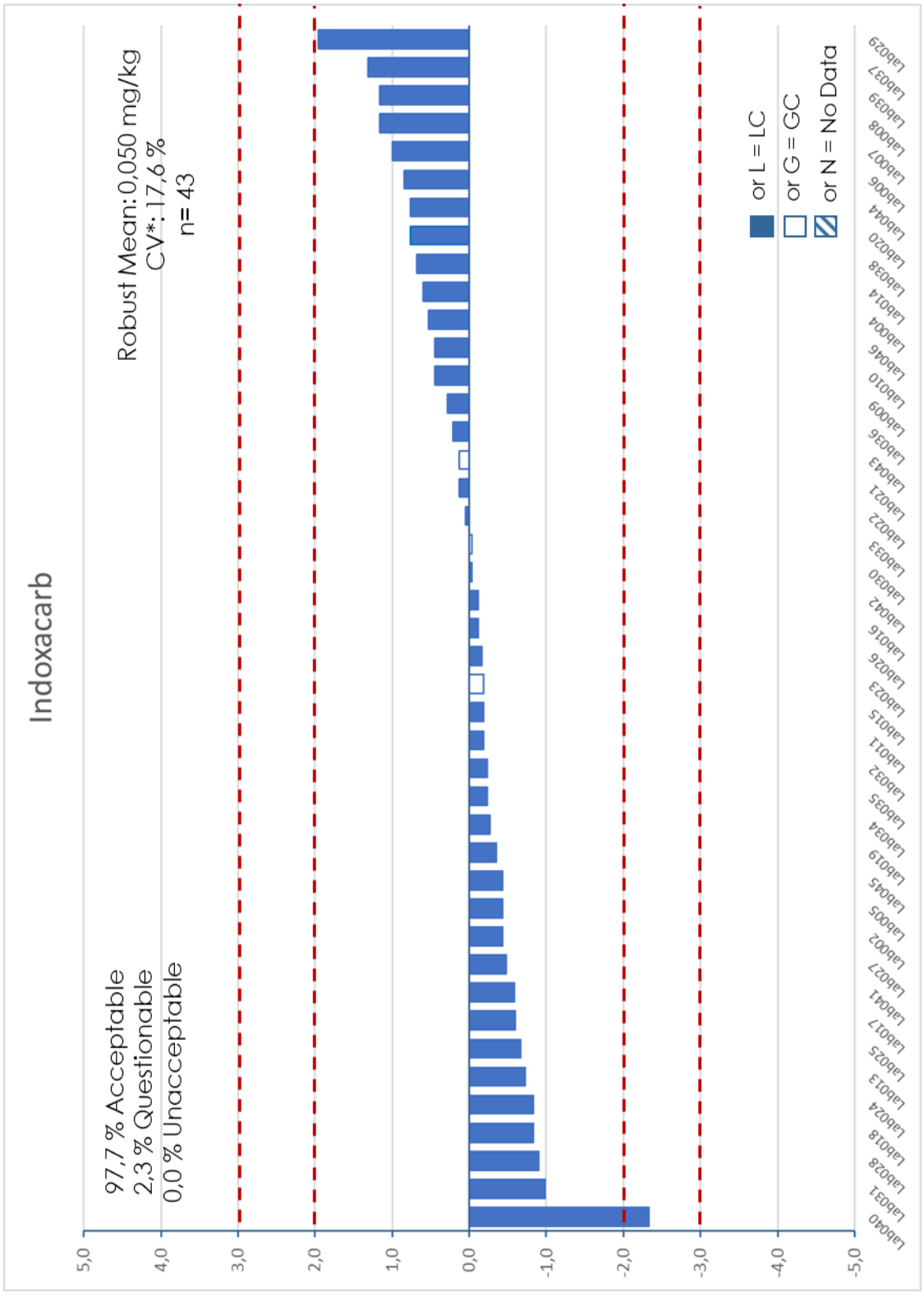
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



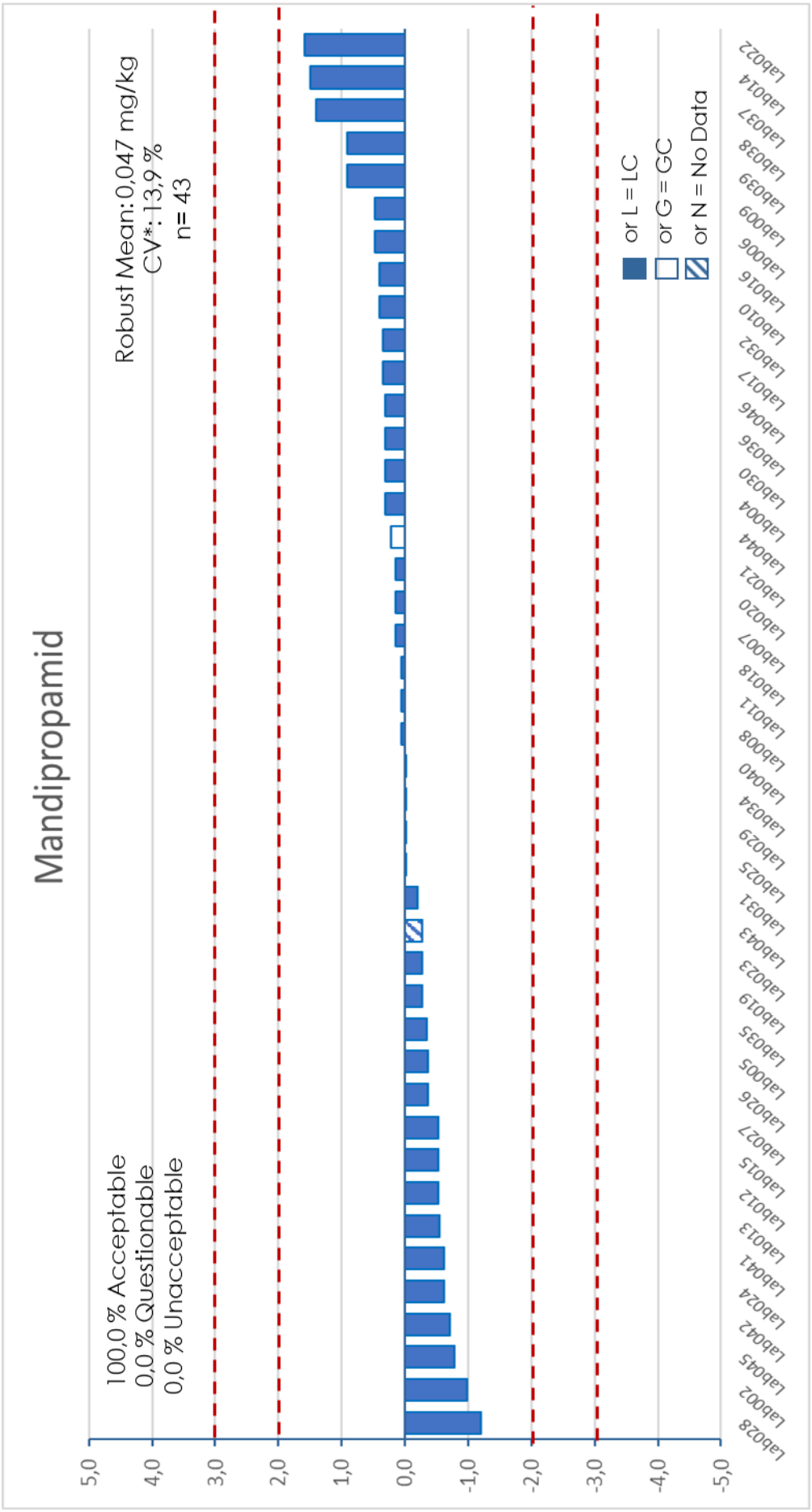
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



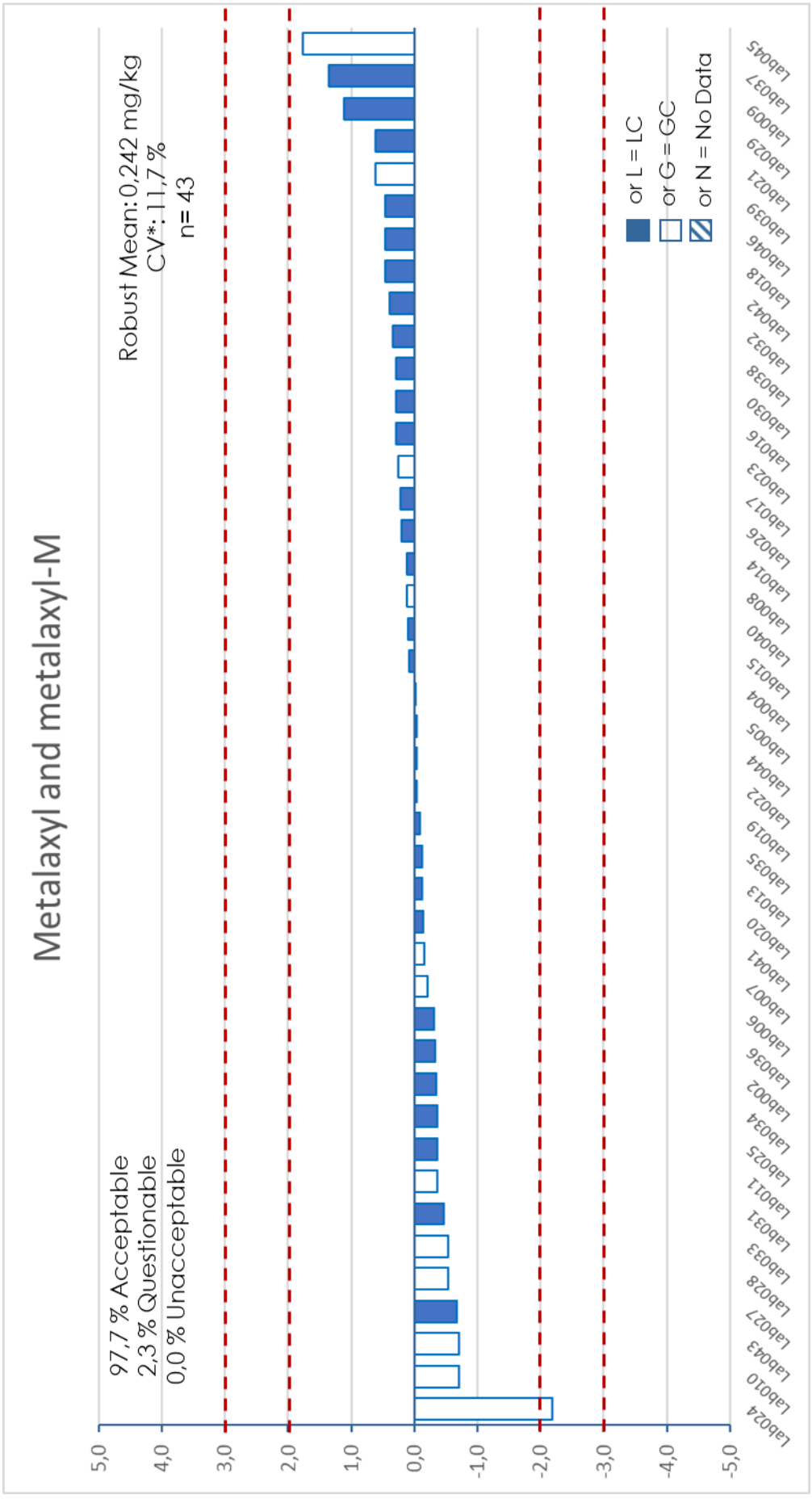
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



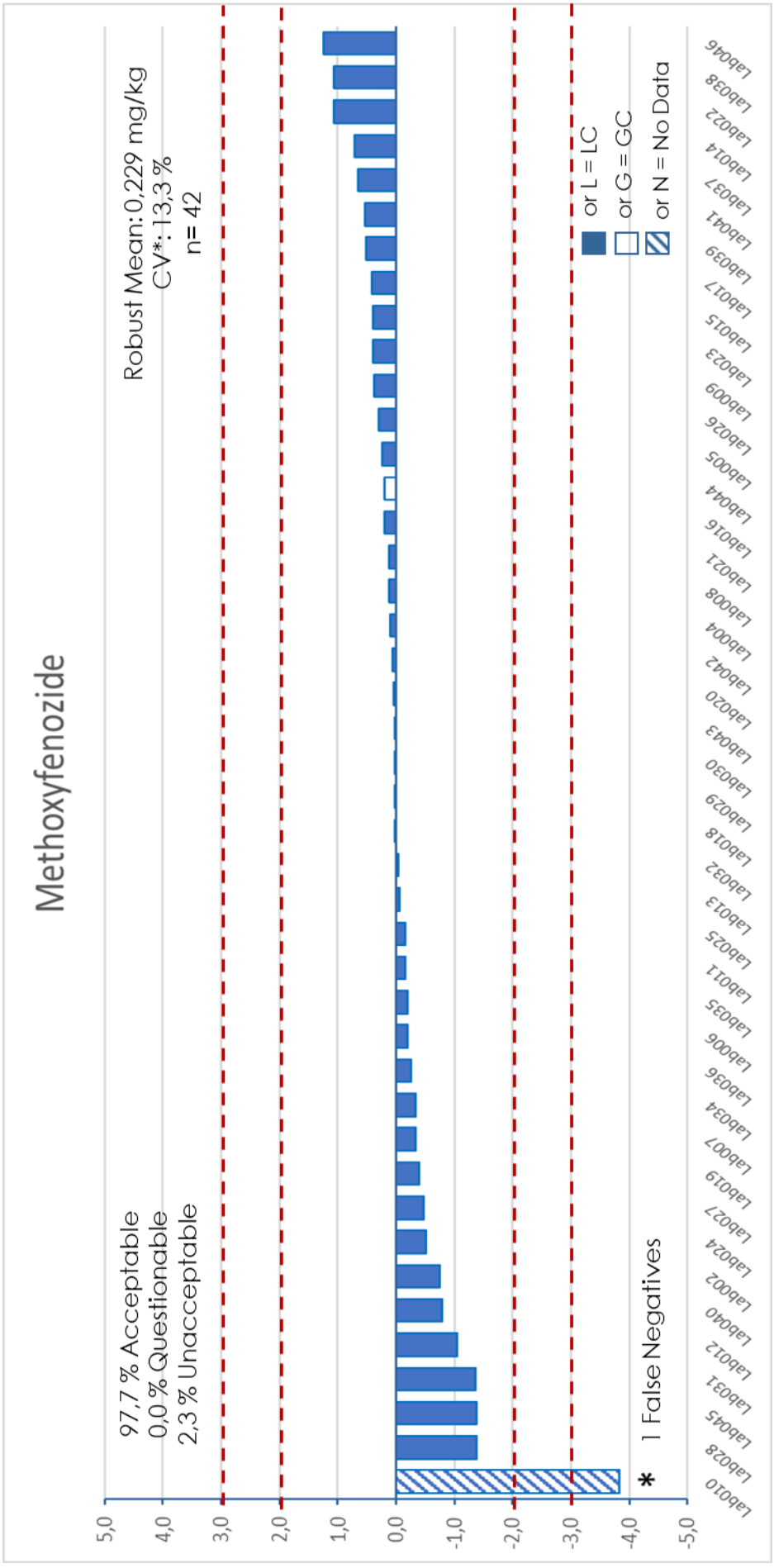
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



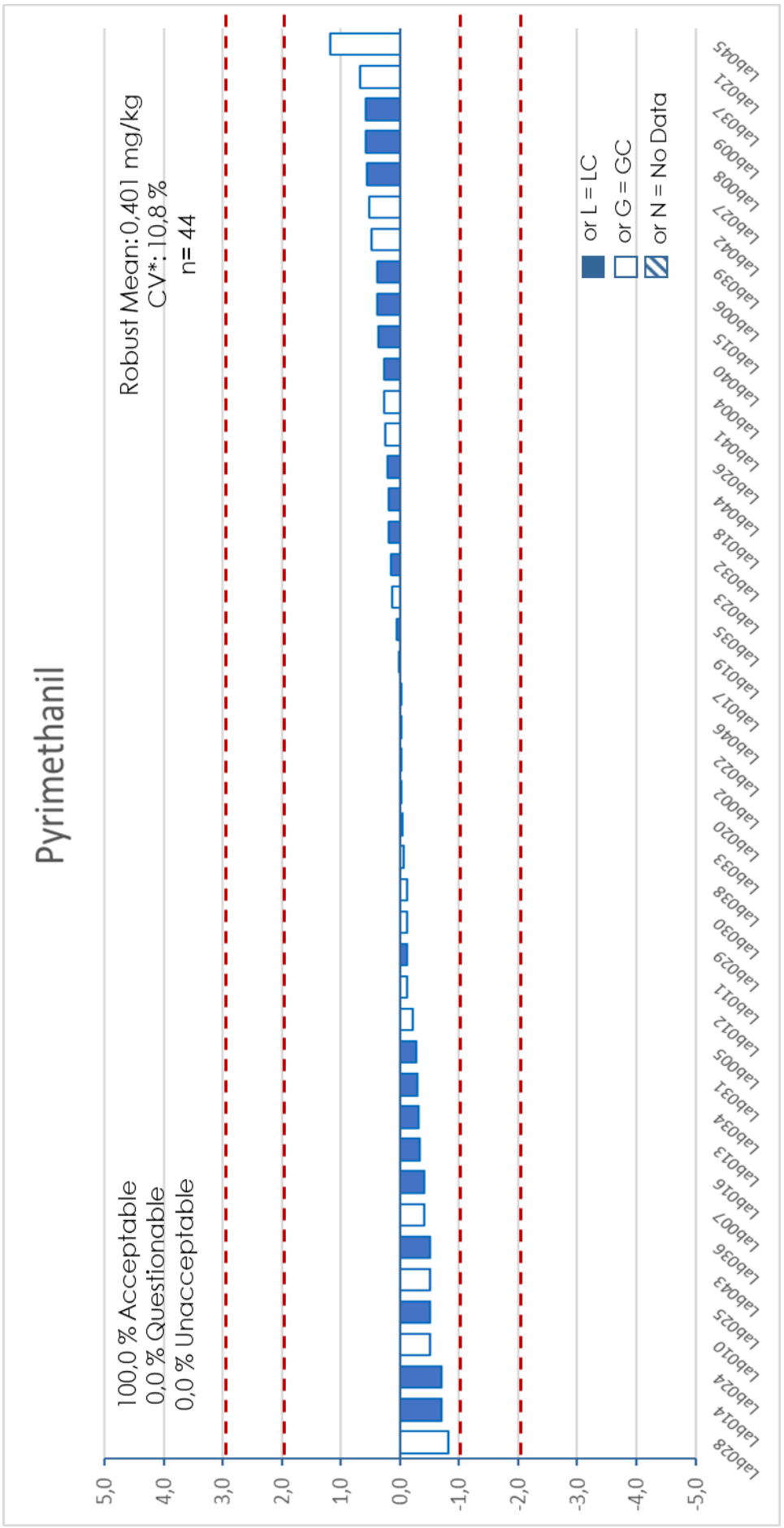
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



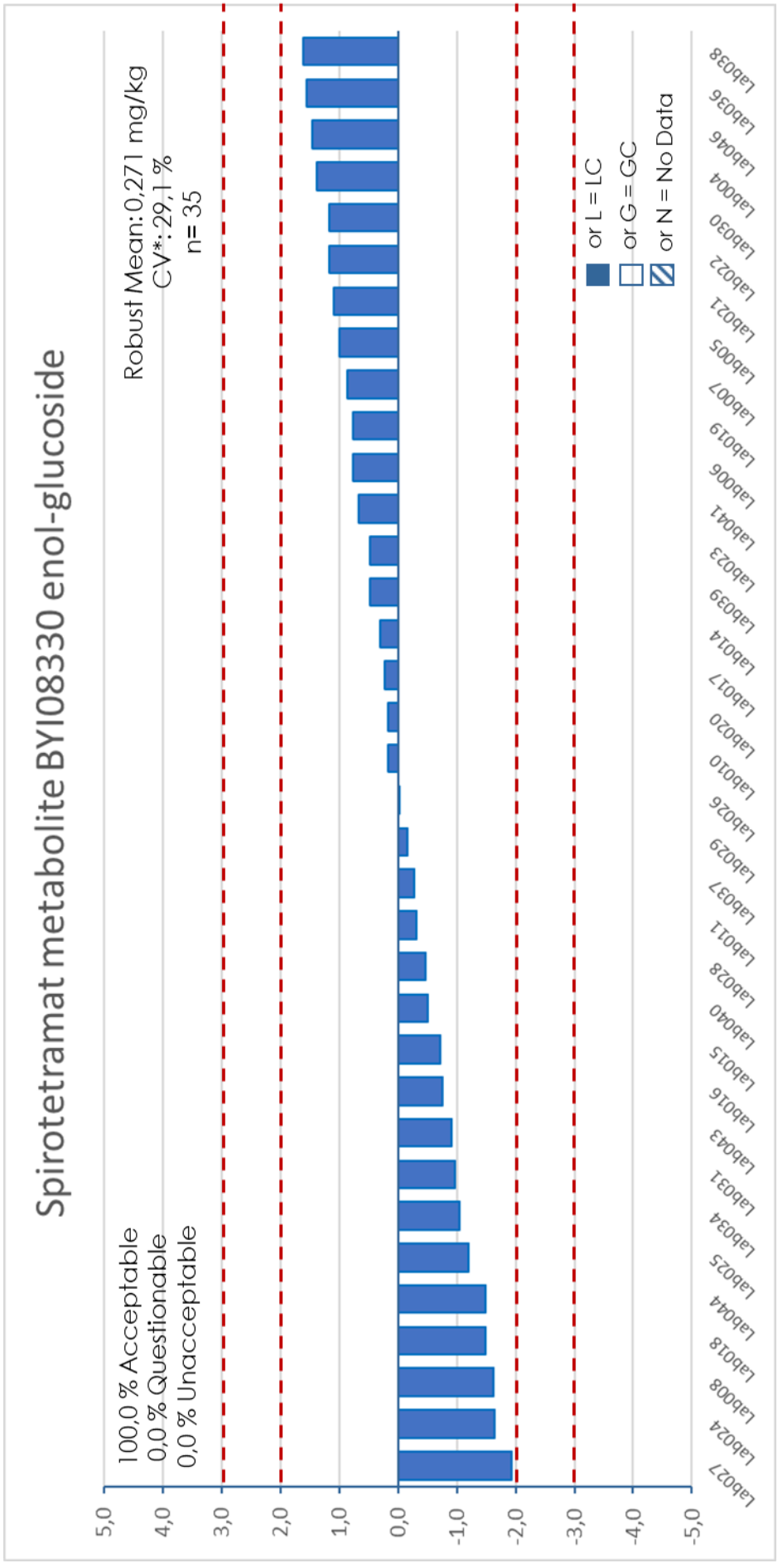
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



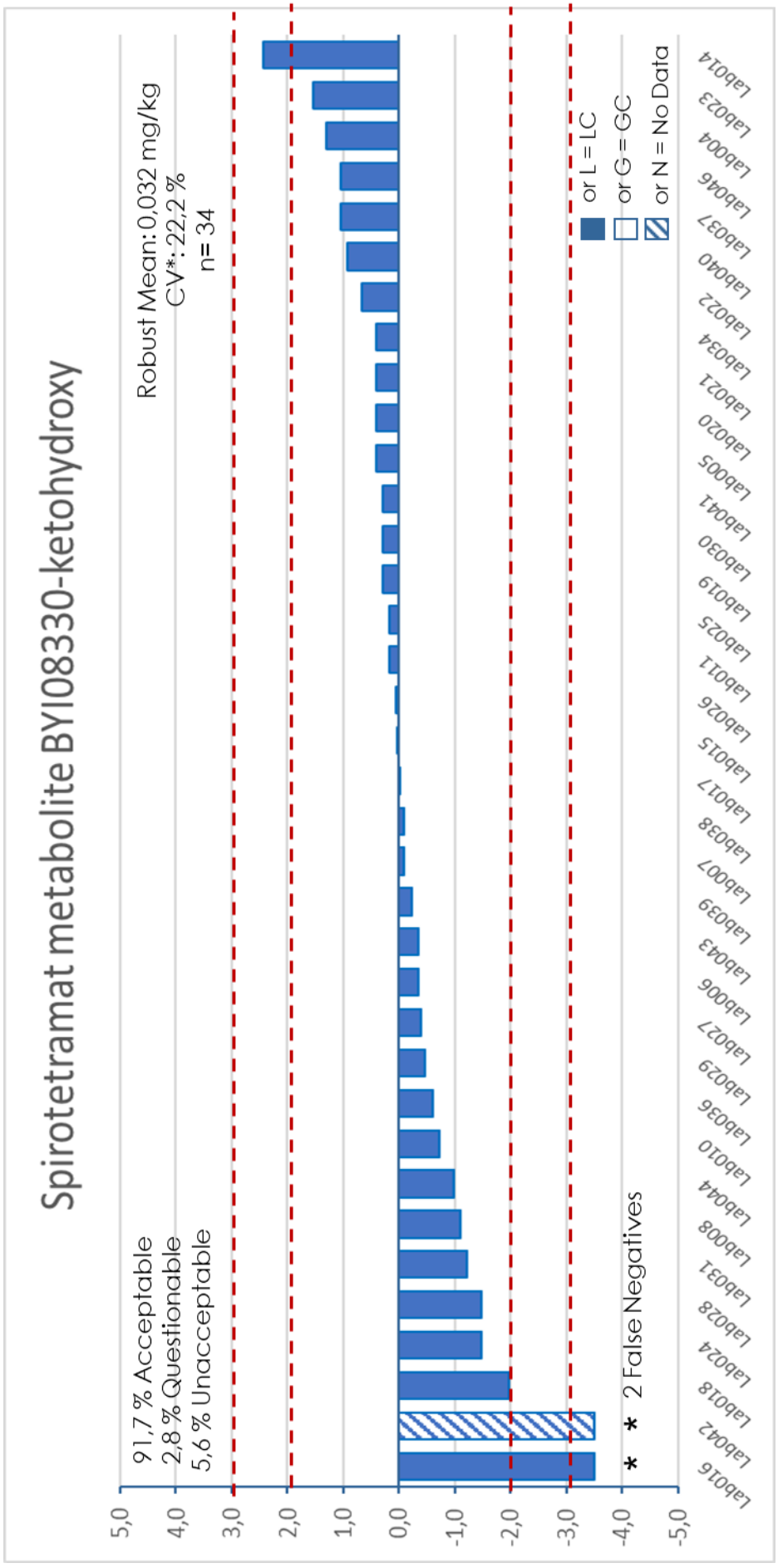
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



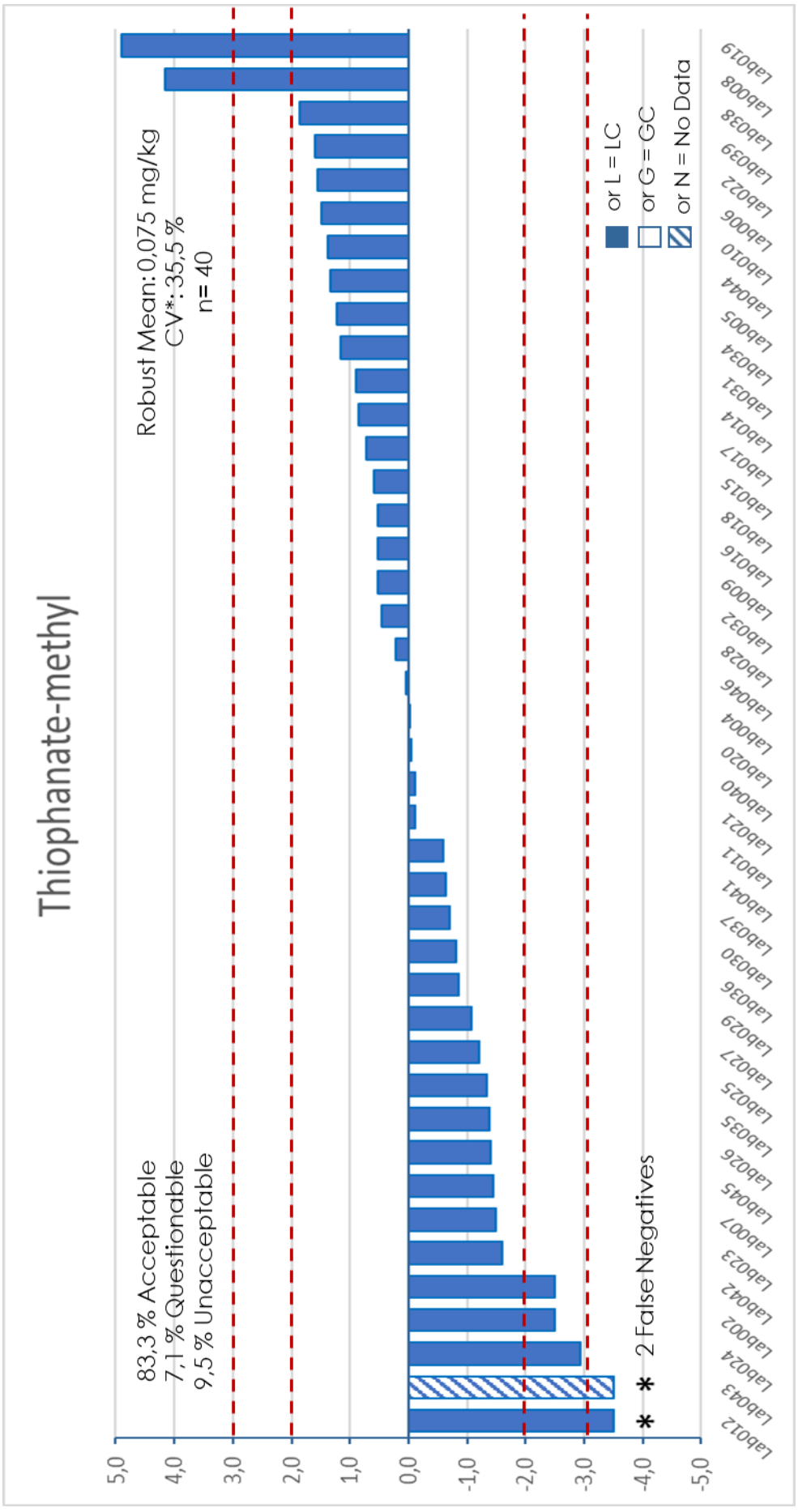
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).

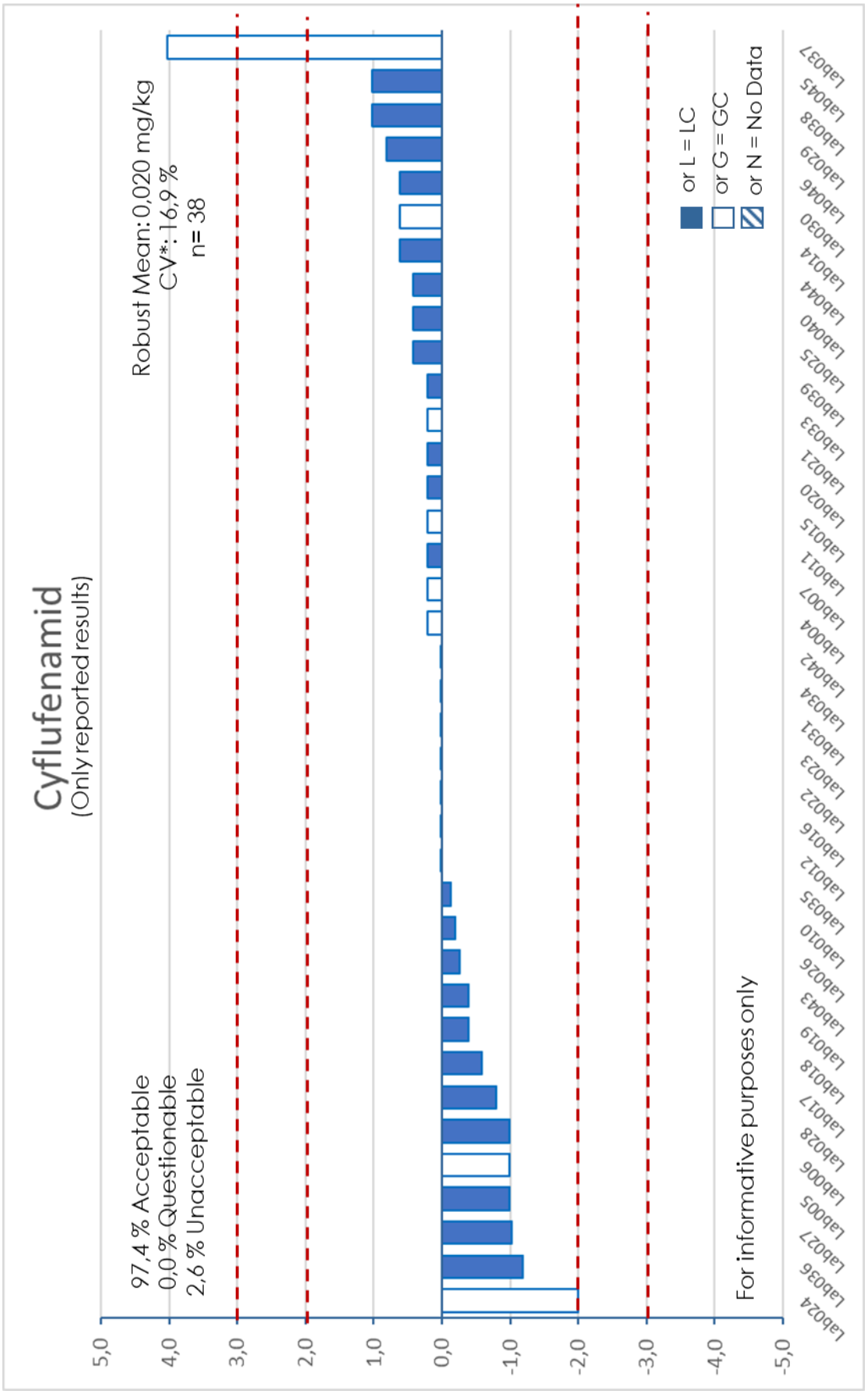


APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).

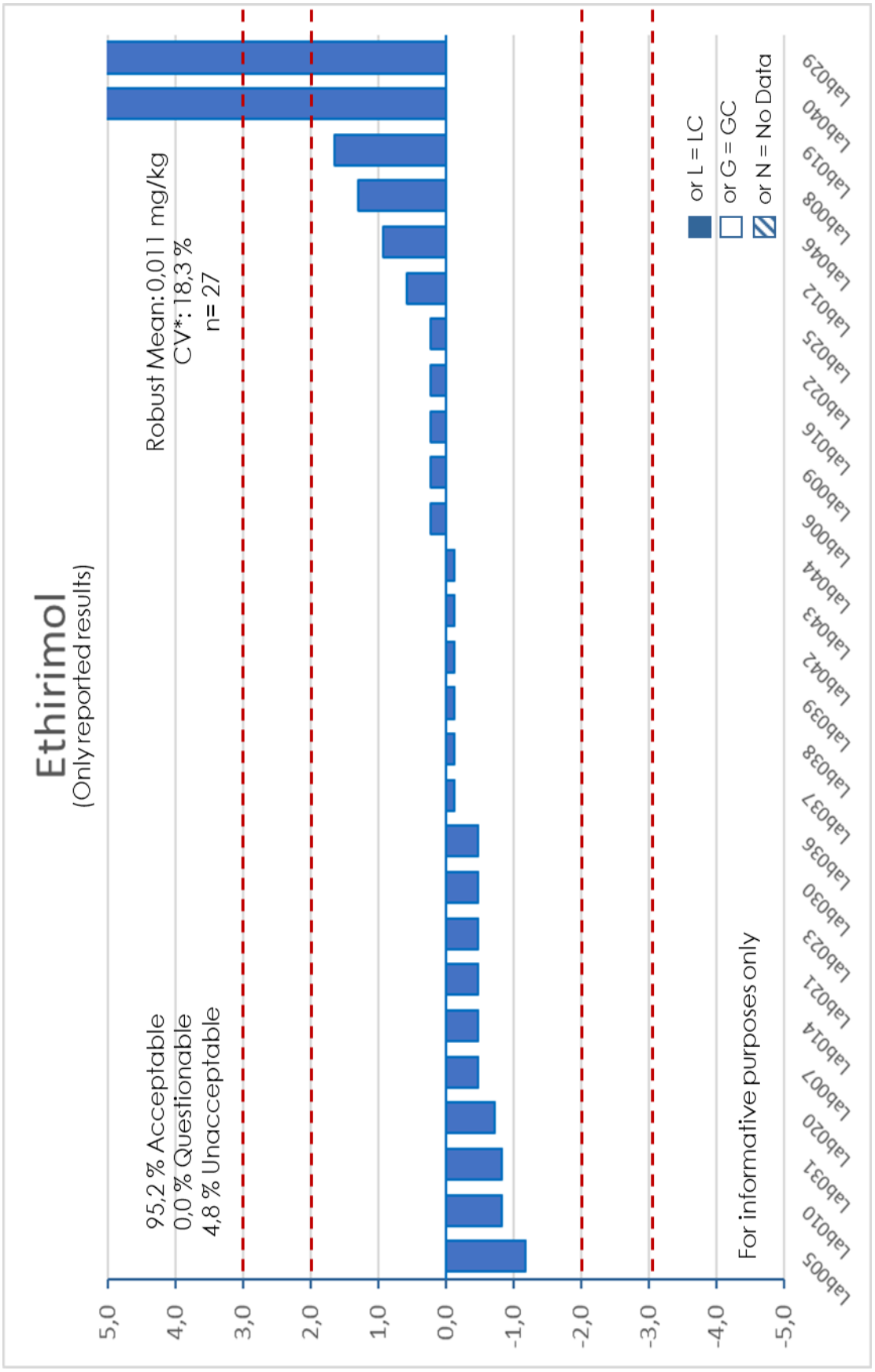


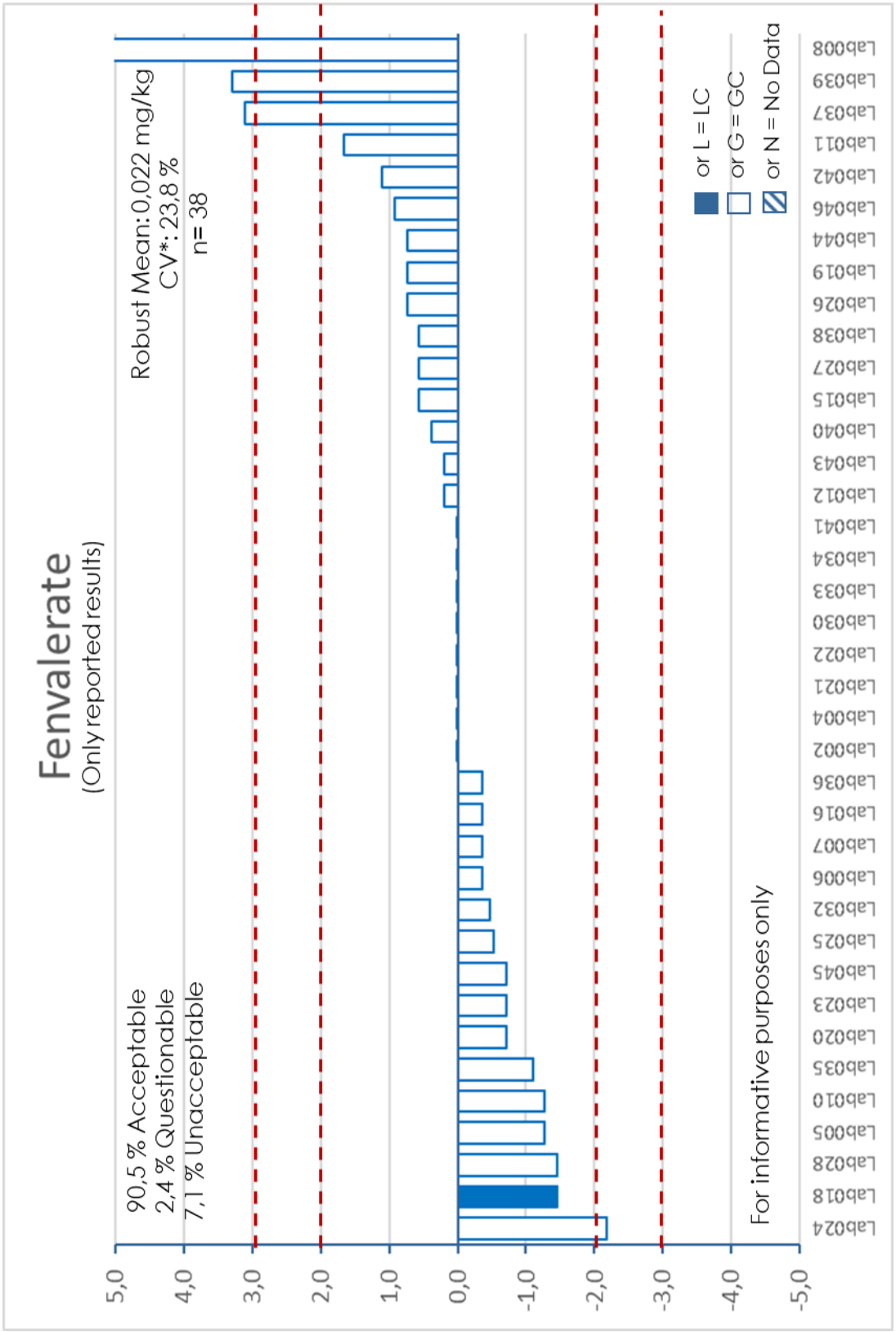
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



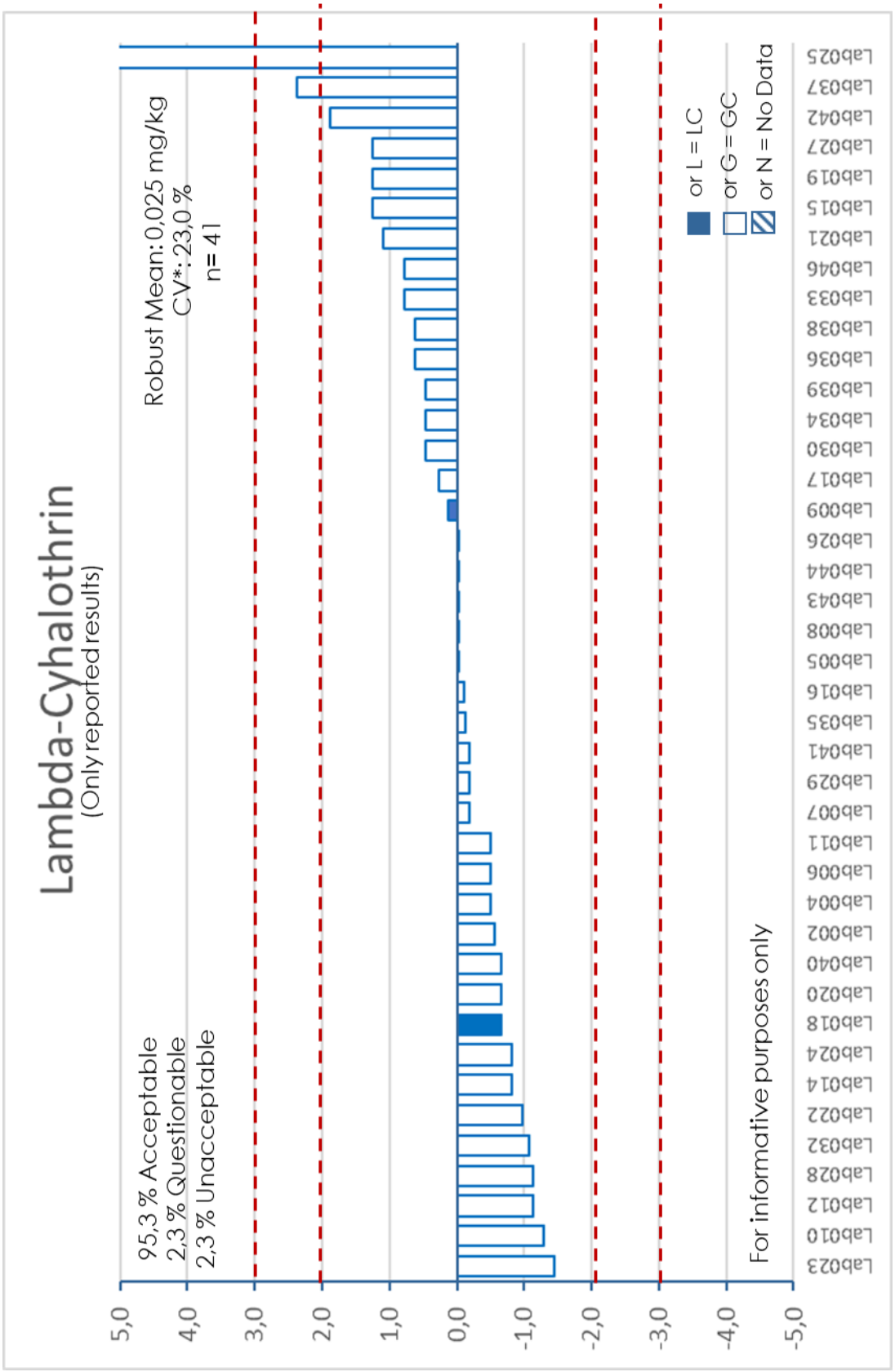

Cyflufenamid
(Only reported results)
Robust Mean: 0,020 mg/kg
CV*: 16,9 %
n= 38
97,4 % Acceptable
0,0 % Questionable
2,6 % Unacceptable
or L = LC
or G = GC
or N = No Data
For informative purposes only
5,0
4,0
3,0
2,0
1,0
0,0
-1,0
-2,0
-3,0
-4,0
-5,0
lab024
lab036
lab027
lab005
lab006
lab028
lab017
lab018
lab019
lab043
lab026
lab010
lab035
lab012
lab016
lab022
lab023
lab031
lab034
lab042
lab004
lab007
lab011
lab015
lab020
lab021
lab033
lab039
lab025
lab040
lab044
lab014
lab030
lab046
lab029
lab038
lab045
lab037

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

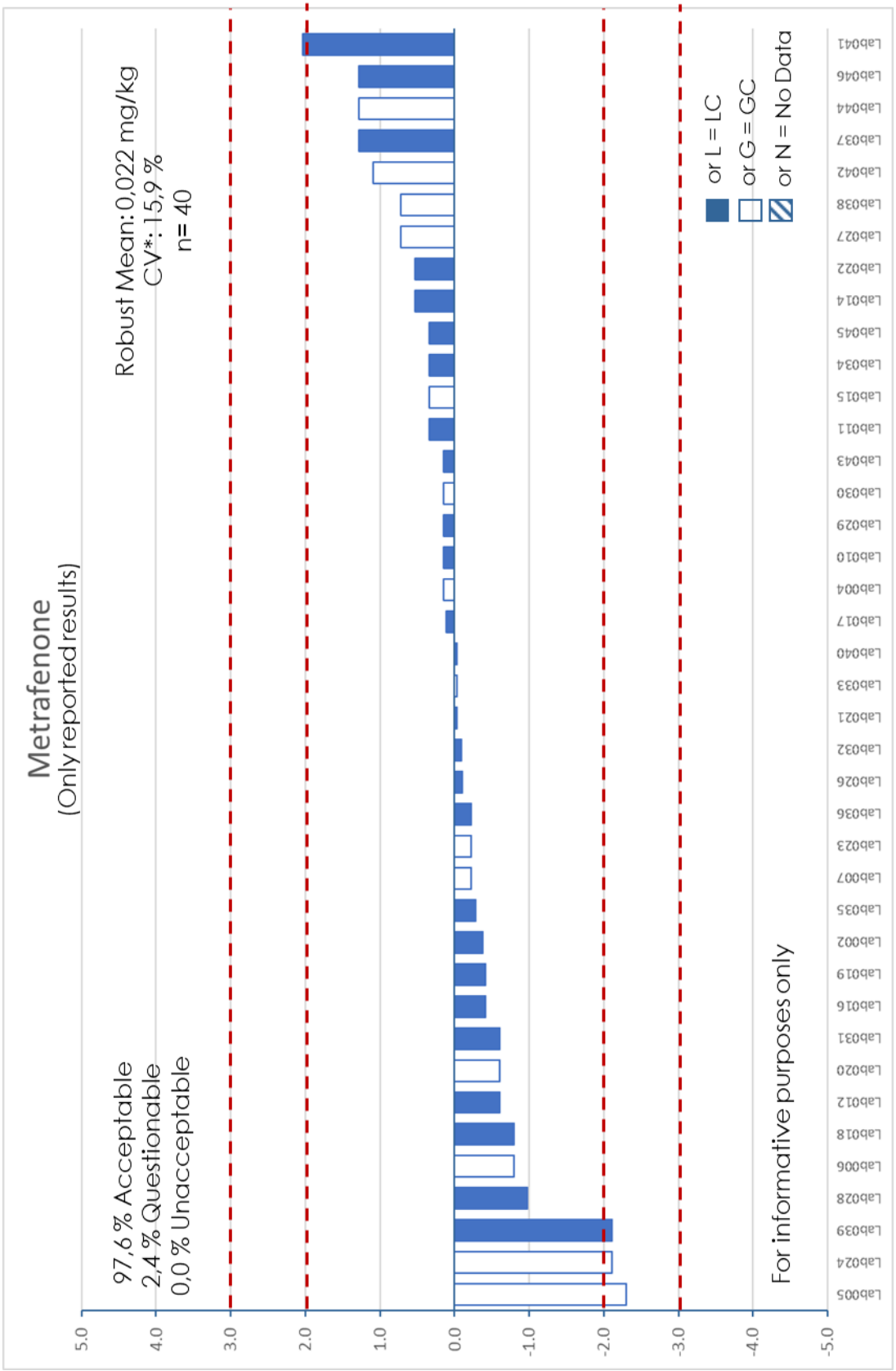



Fenvalerate
(Only reported results)
Robust Mean: 0,022 mg/kg
CV*: 23,8 %
n= 38
90,5 % Acceptable
2,4 % Questionable
7,1 % Unacceptable
For informative purposes only
or L = LC
or G = GC
or N = No Data
5,0
4,0
3,0
2,0
1,0
0,0
-1,0
-2,0
-3,0
-4,0
-5,0
lab008
lab039
lab037
lab011
lab042
lab046
lab044
lab019
lab026
lab038
lab027
lab015
lab040
lab043
lab012
lab041
lab034
lab033
lab030
lab022
lab021
lab004
lab002
lab036
lab016
lab007
lab006
lab032
lab025
lab045
lab023
lab020
lab035
lab010
lab005
lab028
lab018
lab024

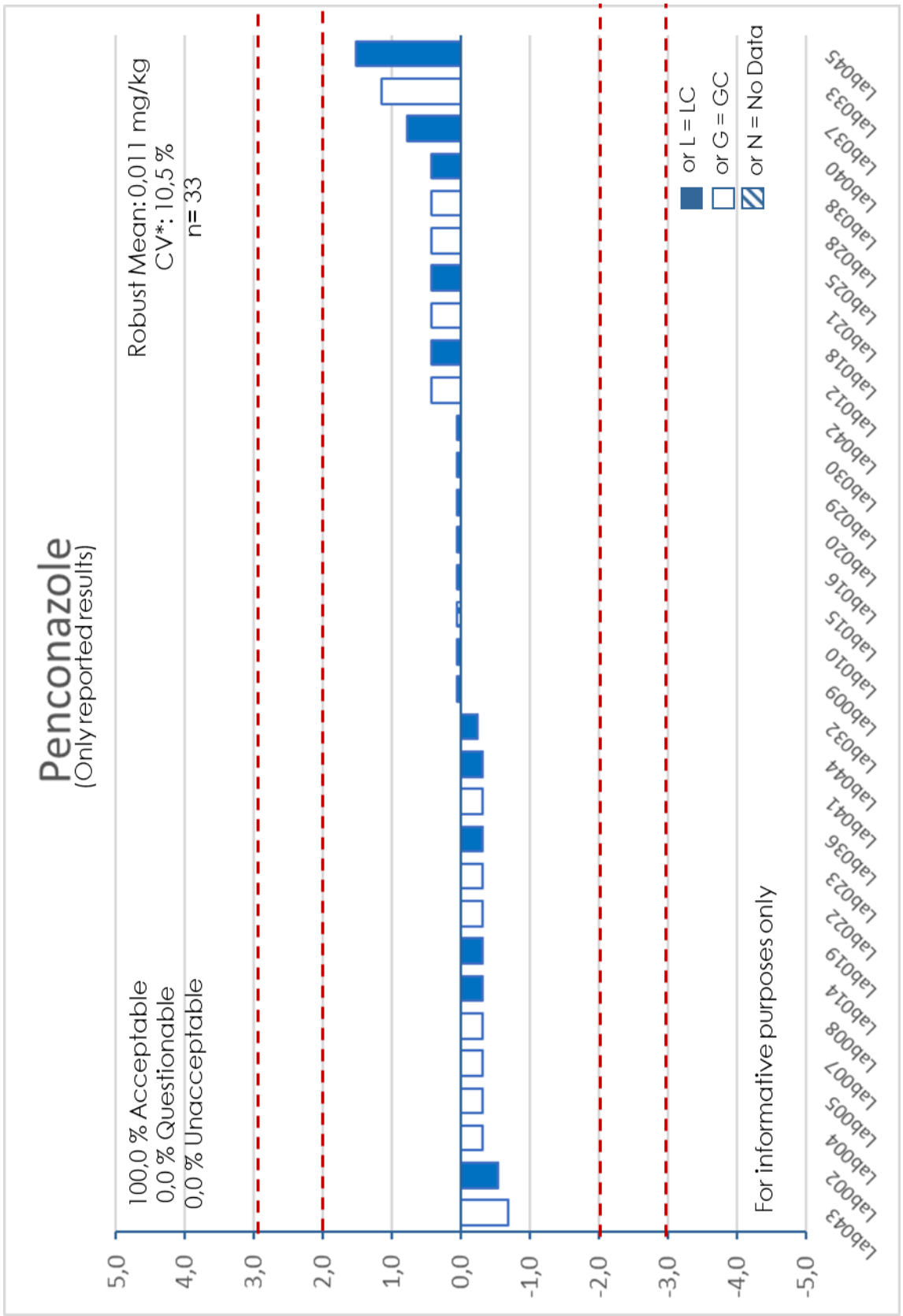
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



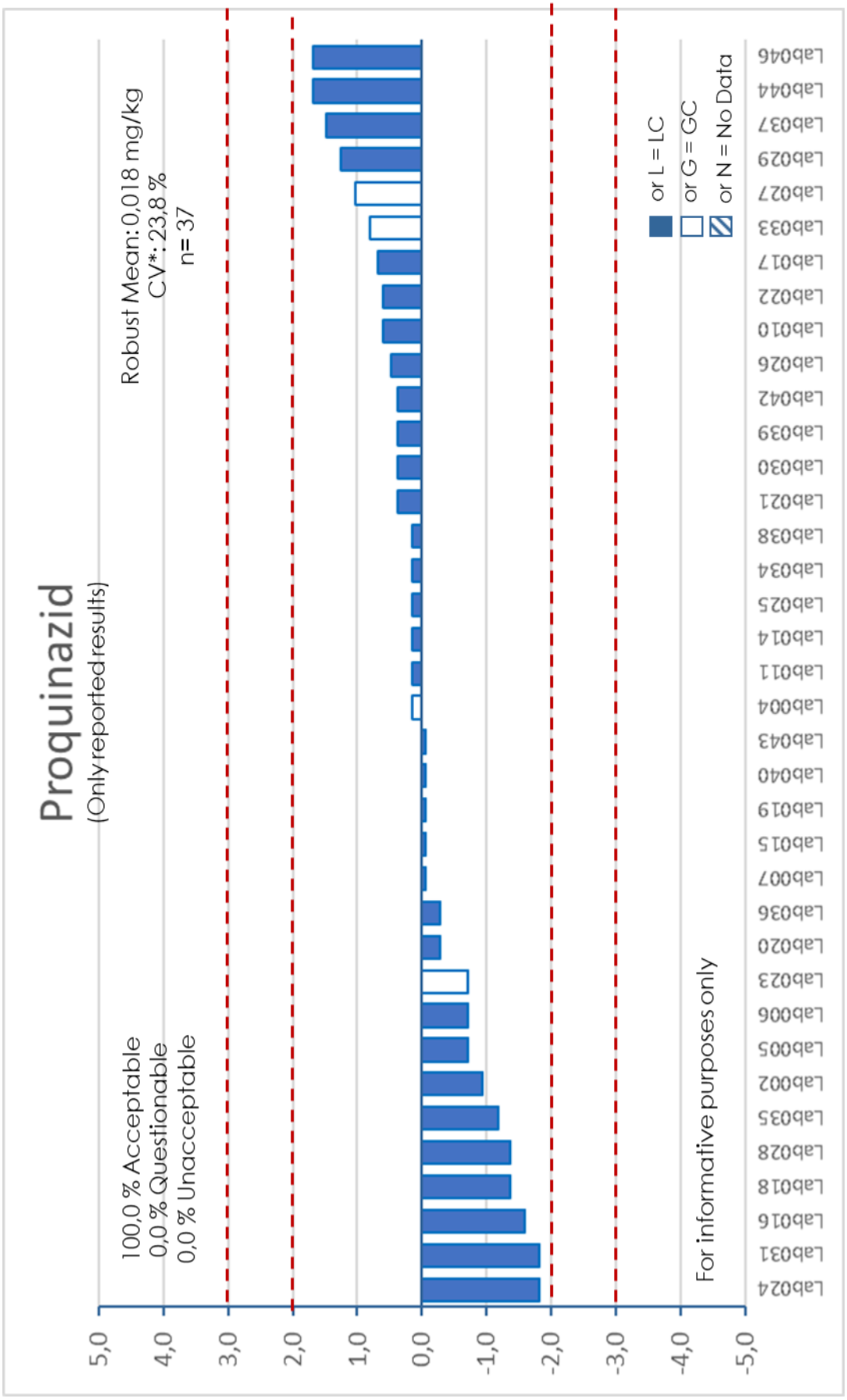
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



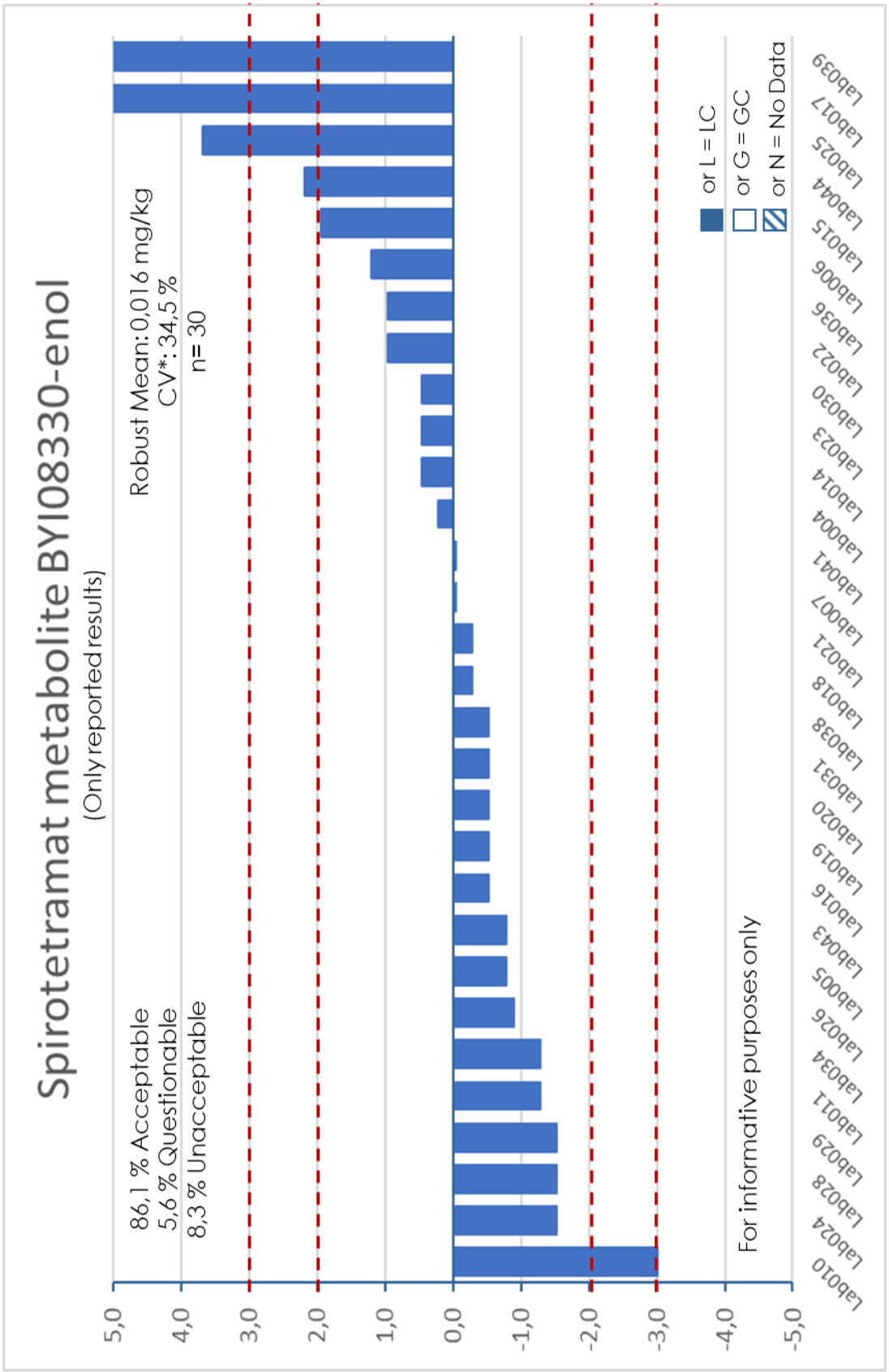
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).

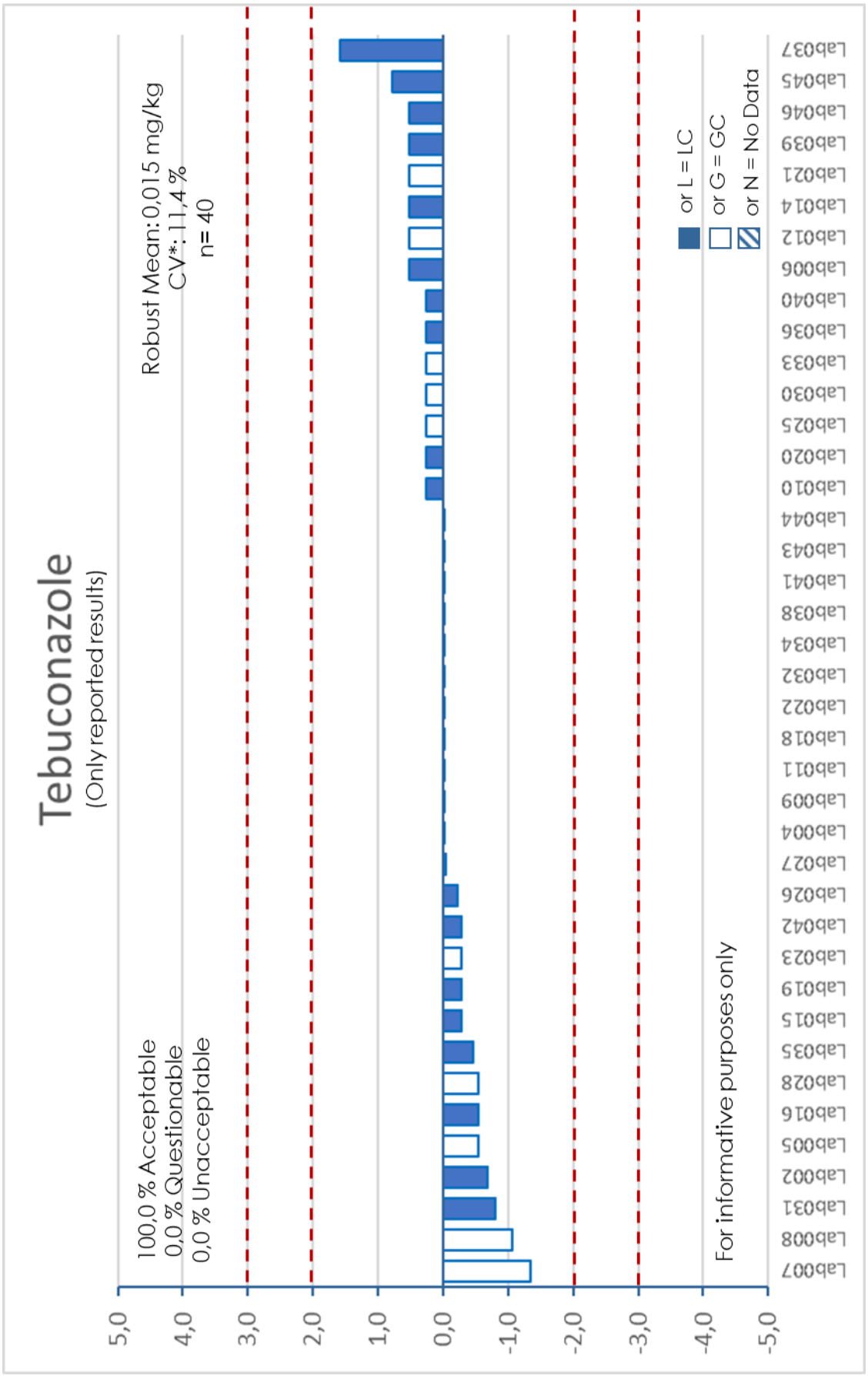


APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).

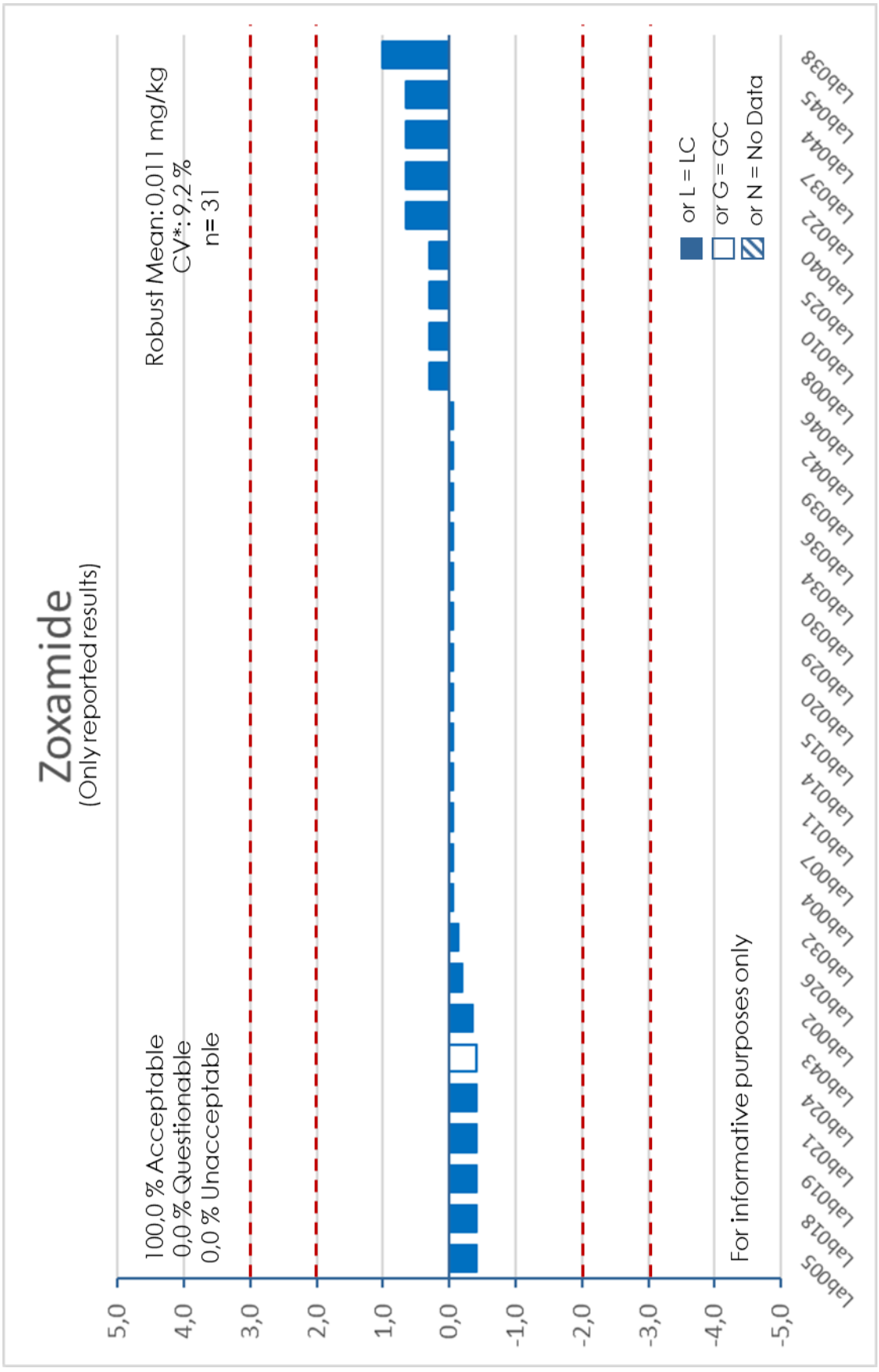


APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).




Tebuconazole
(Only reported results)
Robust Mean: 0,015 mg/kg
CV*: 11,4 %
n= 40
100,0 % Acceptable
0,0 % Questionable
0,0 % Unacceptable
or L = LC
or G = GC
or N = No Data
For informative purposes only
5,0
4,0
3,0
2,0
1,0
0,0
-1,0
-2,0
-3,0
-4,0
-5,0
lab007
lab008
lab031
lab002
lab005
lab016
lab028
lab035
lab015
lab019
lab023
lab042
lab026
lab027
lab004
lab009
lab011
lab018
lab022
lab032
lab034
lab038
lab041
lab043
lab044
lab010
lab020
lab025
lab030
lab033
lab036
lab040
lab006
lab012
lab014
lab021
lab039
lab046
lab045
lab037

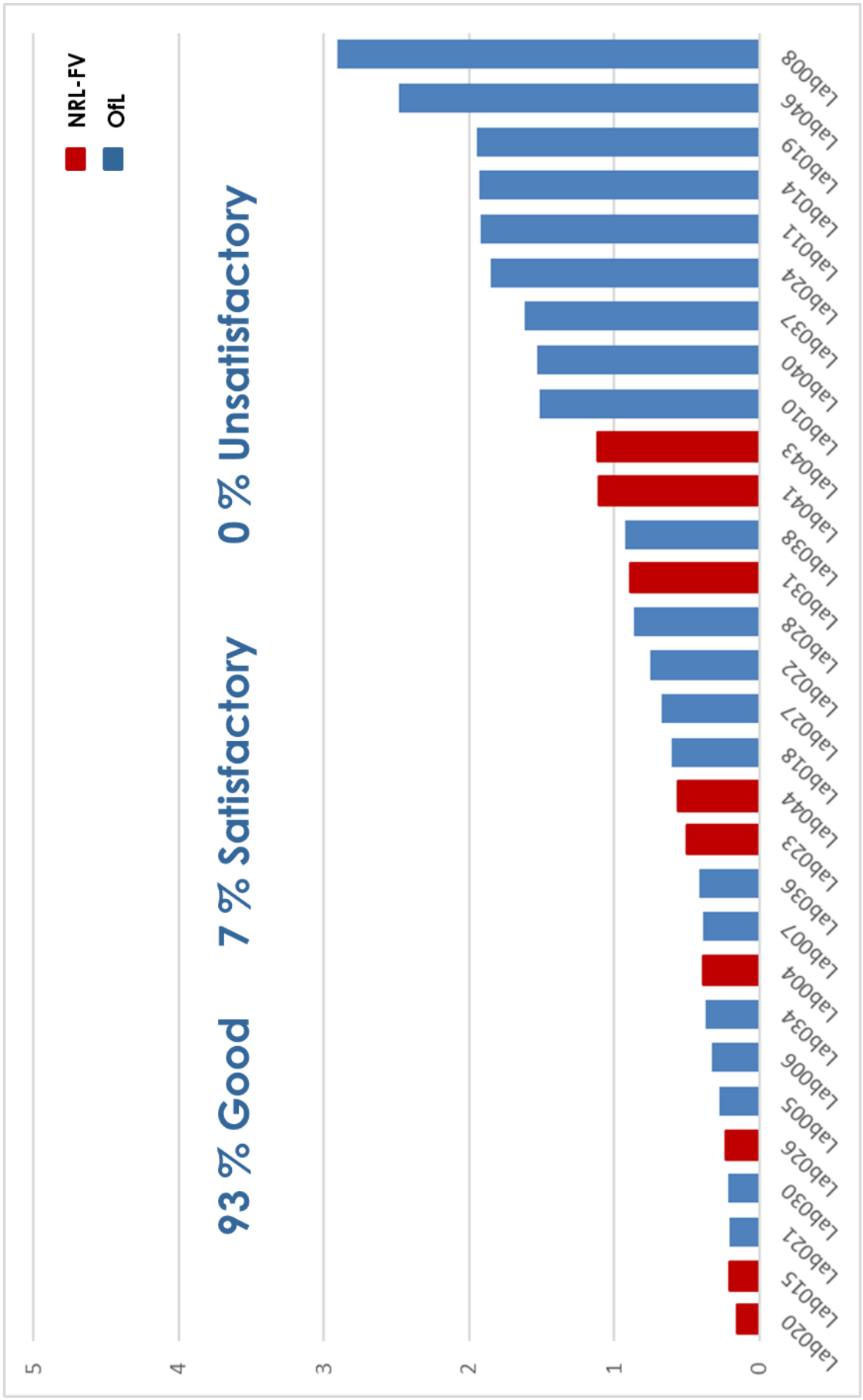
APPENDIX 3. Graphical representation of z scores for FFP-RSD (25 %).



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

Lab Code	Boscalid	Carbendazim	Cyprodinil	Flubendiamide	Fluxapyroxad	Indoxacarb (sum of indoxacarb and its R enantiomer)	Mandipropamid	Metalaxyl and metalaxyl-M	Methoxyfenozide	Pyrimethanil	Spirotetramat metabolite BYI08330 enol-glucoside	Spirotetramat metabolite BYI08330-ketohydroxy	Thiophanate-methyl	No. of z scores	AZ^2
	z scores														
Lab003	0.046	2.634	-0.46	-0.01	-0.4	-0.11	0.228	-0.58	0.719	-0.44	5	-0.22		12	2.79
Lab004	0.504	0.591	0.272	0.217	0.474	0.523	0.313	-0.02	0.089	0.265	1.386	1.297	-0	13	0.385
Lab005	0.198	0.155	-0.16	-0.58	-0.28	-0.43	-0.36	-0.04	0.247	-0.28	0.988	0.414	1.221	13	0.277
Lab006	-0.11	-0.3	0.134	0.103	0.171	0.84	0.482	-0.32	-0.19	0.375	0.767	-0.34	1.488	13	0.33
Lab007	0.198	0.714	-0.01	0.331	-0.25	0.999	0.144	-0.21	-0.33	-0.41	0.87	-0.09	-1.5	13	0.389
Lab008	-0.57	-0.53	0.63	-3.5	-1.35	1.158	0.059	0.125	0.124	0.564	-1.62	-1.1	4.151	13	2.91
Lab010	0.58	-0.65	-0.68	-0.47	0.209	0.444	0.397	-0.7	-3.83	-0.51	0.162	-0.72	1.381	13	1.514
Lab011	-0.34	4.904	-0.27	0.217	0.171	-0.19	0.059	-0.37	-0.16	-0.11	-0.31	0.162	-0.59	13	1.923
Lab014	0.427	0.871	0.772	-3.5	0.967	0.602	1.497	0.125	0.719	-0.71	0.309	2.432	0.848	13	1.93
Lab015	-0.03	0.853	0.004	-0.58	-0.28	-0.19	-0.53	0.075	0.404	0.355	-0.72	0.036	0.582	13	0.202
Lab018	-0.26	-0.11	0.101	-0.58	-0.21	-0.83	0.059	0.455	0.02	0.185	-1.49	-1.98	0.529	13	0.602
Lab019	-0.26	-0.05	-0.23	-0.12	-0.28	-0.35	-0.28	-0.09	-0.4	0.006	0.767	0.288	4.897	13	1.942
Lab020	0.351	1.01	0.213	0.103	0.095	0.761	0.144	-0.14	0.037	-0.04	0.162	0.414	-0.06	13	0.156
Lab021	0.427	0.242	-0.38	-0.01	0.171	0.126	0.144	0.62	0.124	0.683	1.091	0.414	-0.11	13	0.207
Lab022	0.122	0.364	0.846	0.559	0.891	0.047	1.581	-0.04	1.068	-0.01	1.165	0.666	1.541	13	0.753
Lab023	0.122	0.591	0.011	0.787	-0.02	-0.19	-0.28	0.257	0.387	0.125	0.486	1.549	-1.6	13	0.503
Lab024	-0.95	0.19	-1.54	-0.58	-0.59	-0.83	-0.62	-2.19	-0.5	-0.71	-1.64	-1.48	-2.93	13	1.851
Lab026	-0.44	0.557	0.362	-0.01	-0.19	-0.17	-0.36	0.198	0.299	0.207	-0.02	0.059	-1.41	13	0.23
Lab027	-0.34	-1.08	0.082		0.065	-0.48	-0.52	-0.67	-0.47	0.524	-1.93	-0.39	-1.2	12	0.672
Lab028	-0.57	-1.21	-0.46		-0.89	-0.91	-1.21	-0.54	-1.38	-0.81	-0.46	-1.48	0.209	12	0.862
Lab030	-0.03	0.539	0.25	-0.12	-0.36	-0.03	0.313	0.29	0.02	-0.11	1.165	0.288	-0.8	13	0.214
Lab031	-1.86	-0.74	-0.87	-0.01	-0.66	-0.98	-0.19	-0.47	-1.36	-0.29	-0.96	-1.23	0.901	13	0.893
Lab034	-0.11	-0.86	-0.27	-0.81	-0.51	-0.27	-0.03	-0.37	-0.33	-0.31	-1.05	0.414	1.168	13	0.368
Lab036	0.198	1.063	-0.15	0.331	0.133	0.205	0.313	-0.34	-0.26	-0.5	1.549	-0.6	-0.86	13	0.414
Lab037	2.641	0.399	0.787	1.585	1.65	1.316	1.412	1.362	0.649	0.584	-0.28	1.044	-0.7	13	1.614
Lab038	-0.26	-1.21	0.101	0.445	1.308	0.682	0.905	0.29	1.068	-0.11	1.608	-0.09	1.86	13	0.925
Lab040	0.58	0.172	0.026	-3.5	0.095	-2.33	-0.03	0.092	-0.78	0.265	-0.5	0.918	-0.11	13	1.529
Lab041	0.046	-0.07	-0.16	-3.5	-0.32	-0.59	-0.62	-0.16	0.526	0.255	0.663	0.288	-0.64	13	1.109
Lab043	0.122	0.364	0.101	-0.47	0.171	0.126	-0.28	-0.7	0.02	-0.51	-0.9	-0.34	-3.5	13	1.11
Lab044	0.275	-0.33	0.846	0.673	0.55	0.761	0.228	-0.04	0.194	0.185	-1.49	-0.97	1.328	13	0.561
Lab046	0.809	0.714	0.101	5	0.929	0.444	0.313	0.455	1.243	-0.01	1.46	1.044	0.049	13	2.485

APPENDIX 5. EUPT-FV-SC04-AZ²-Graphical representation for EU/EFTA laboratories in Category A.



9th Edition
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GENERAL PROTOCOL

for EU Proficiency Tests on Pesticide Residues in Food and Feed

Introduction

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

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

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

EUPT-Organisers and Scientific Committee

EUPTs are organised by individual EURLs, or by more than one EURL, in collaboration.
An **Organising Team** (in the following named Organisers) is appointed by the EURL(s) in charge. This team is responsible for all administrative and technical matters concerning the organisation of the PT, e.g. the PT-announcement, the production of the PT-material (Test Item), the undertaking of homogeneity and stability tests, the packing and shipment of the PT-materials, the handling and evaluation of the results and method information submitted by the participants, the drafting of the preliminary and final reports as well as generation and distribution of EUPT-participation certificates.
To complement the internal expertise of the EURLs, a group of external consultants forming the **EUPT-Scientific Committee** (EUPT-SC)[9] has been established and approved by DG-SANTE. The EUPT-SC consists of expert scientists with many years of experience in PTs and/or pesticide residue analysis. The actual composition of the EUPT-SC and the affiliation of each of its members is shown on the EURL-Website. The members of the EUPT-SC are also listed in the Specific Protocol and the Final Report of each EUPT.
The EUPT-SC is made up of the following two subgroups:

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

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

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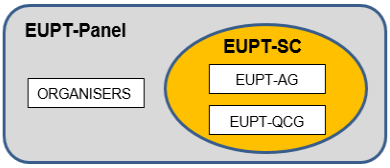
[5] DG-SANTE = European Commission, Health and Food Safety Directorate-General
[6] For more information about the EURL/NRL/OfL-Network please refer to the EURL-Web-portal under: "http://www.eurl-pesticides.eu"
[7] Regulation (EU) 2017/625 of the European Parliament and of the Council on official controls and other official activities performed to ensure the application of food and feed law, rules on animal health and welfare, plant health and plant protection products. Published at OJ of the EU L95 of 07.04.2017
[8] European Commission Proficiency Tests for Pesticide Residues in Fruits and Vegetables, Trends in Analytical Chemistry, 2010, 29 (1), 70 – 83.
[9] Link to the List of current members of the EUPT Scientific Committee:
http://www.eurl-pesticides.eu/library/docs/allcrl/EUPT-SC.pdf

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

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



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

EUPT Participants
Within the European Union all NRLs operating in the same area as the organising EURL, as well as all OfLs whose scope overlaps with that of the EUPT, are legally obliged to participate in EUPTs. The legal obligation of NRLs and OfLs to participate in EUPTs arises from:

- Art 38 (b) of Reg. 625/2017/EC and Art. 28 of Reg. 396/2005/EC[10] (for all OfLs analysing for pesticide residues within the framework of official controls[11] of food or feed)
- Art. 101 (1)(a) of Reg. 625/2017/EC (for all NRLs)

The four EURLs will annually issue and distribute, via the EURL-website, a joint list of all OfLs that must participate in each of the EUPTs to be conducted within a given year. The list of obliged labs will be updated every year to take account of any changes in the lab profiles. Interim updates will be issued to eliminate any possible errors.
NRLs are responsible for checking whether all relevant OfLs within their network are included in the list of obligated laboratories with their actual commodity-scopes and contact information.
OfLs are furthermore urged to keep their own profiles within the EURL-DataPool up-to-date, especially their commodity and pesticide scopes and their contact information.
Labs that are obliged to participate in a given EUPT, and that are not able to participate, must provide the reasons for their non-participation This also applies to any participating laboratories that fail to report results.
OfLs not paying the EUPT sample delivery fee will be initially warned that their participation in subsequent EUPTs could be denied. In case of a repetitive non-payment, the EUPT organisers will inform the corresponding NRL to take action.

Confidentiality and Communication
The proprietor of all EUPT data is DG-SANTE and as such has access to all information.
For each EUPT, the laboratories are given a unique code (lab code), initially only known to themselves and the Organisers. In the final EUPT-Report, the names of participating laboratories will not be linked to their laboratory codes. It should be noted, however, that the Organisers, at the request by DG-SANTE, may present the EUPT-results on a country-by-country basis. It may therefore be possible that a link between codes and laboratories could be made, especially for those countries where only one laboratory has participated. Furthermore, the EURLs reserve the right to share EUPT results and codes amongst themselves: for example, for the purpose of evaluating overall lab or country performance as requested by DG-SANTE.
As laid down in Regulation 625/2017/EC, NRLs are responsible for evaluating and improving their own OfL-Network. On request from the NRLs, the EURLs will provide them with the PT-codes of the participating OfLs belonging to their OfL-Network. This will allow NRLs to follow the participation and performance of the laboratories within their network.
Communication between participating laboratories during the test, on matters concerning a PT exercise, is not permitted from the start of the PT exercise until the distribution of the preliminary report.
For each EUPT the organising EURL prepares a specific EUPT-Website where all PT-relevant documents in their latest version are linked. In case of important modifications on any of these documents, the participating laboratories will be informed via e-mail. In any case, as soon as the PT-period starts the participants are encouraged to visit the particular EUPT-Website, to make sure that they are using the latest versions of all PT-relevant documents.
The official language used in all EUPTs is English.

Announcement / Invitation Letter
At least 3 months before the distribution of the Test Item the EURLs will publish an Announcement/Invitation letter on the EURL-web-portal and distribute it via e-mail to the NRL/OfL mailing list available to the EURLs. This letter will inform about the commodity to be used as Test Item, as well as links to the tentative EUPT-Target Pesticide List and the tentative EUPT-Calendar.

Target Pesticide List
This list contains all analytes (pesticides and metabolites) to be sought for, along with the Minimum Required Reporting Levels (MRRLs) valid for the specific EUPT. The MRRLs are typically based upon the lowest MRLs found either in Regulation 396/2005/EC or Commission Directive 2006/125/EC (Baby Food Directive).

[10] Regulation (EC) No 396/2005, published at OJ of the EU L70 of 16.03.2005, as last amended by Regulation 839/2008 published at OJ of the EU L234 of 30.08.2008.
[11] Official controls in the sense of Reg. 625/2017/EC. This includes labs involved in controls within the framework of national and/or EU-controlled programmes as well as labs involved in import controls according to Regulation 669/2009/EC.

Labs must express their results as stated in the Target Pesticides List.

Specific Protocol
For each EUPT the organizing EURL will publish a Specific Protocol at least 2 weeks before the Test Item is distributed to the participating laboratories. The Specific Protocol will contain all the information previously included in the Invitation Letter but in its final version, information on payment and delivery, instructions on how to handle the Test Item upon receipt and on how to submit results, as well as any other relevant information.

Homogeneity of the Test Item
The Test Item will be tested for homogeneity typically before distribution to participants. The homogeneity tests usually involve the analysis of two replicate analytical portions, taken from at least ten randomly chosen units of treated Test Item. Both, sample preparation and measurements should be conducted in random order.
The homogeneity test data are statistically evaluated according to ISO 13528, Annex B or to the International Harmonized Protocols jointly published by ISO, AOAC and IUPAC. The results of all homogeneity tests are presented to the EUPT-SC. In special cases, where the above homogeneity test criteria are not met, the EUPT-Panel, considering all relevant aspects (e.g. the homogeneity results of other pesticides spiked at the same time, the overall distribution of the participants' results (CV*), the analytical difficulties faced during the test, knowledge of the analytical behaviour of the pesticide question), may decide to overrule the test. The reasons of this overruling have to be transparently explained in the Final EUPT-Report. For certain analytes with comparable properties, an equivalent distribution within the sample can be expected if they were spiked/used at simultaneously. The homogeneity test, of one or more of these analytes, may thus be skipped or simplified. If, however, the distribution of participants' results for an analyte that was not or not fully tested for homogeneity, is found to be atypically broad, compared to the tested analytes, the EUPT-SC may decide that a homogeneity test should be performed *a posteriori* by the EURL.

Stability of the analytes contained in the Test Item
The Test Items will also be tested for stability - according to ISO 13528, Annex B. The time delay between the first and the last stability test must exceed the period of the EUPT-exercise. Typically the first analysis is carried out shortly before the shipment of the Test Items and the last one shortly after the deadline for submission of results. To better recognise trends and gain additional certainty one or more additional tests may be conducted by the Organisers. At least 6 sub-samples (analytical portions) should be analysed on each test day (e.g. 2 analytical portions withdrawn from three randomly chosen containers OR 6 portions withdrawn from a single container). In principle all pesticides contained in the Test Item should be checked for stability. However, in individual cases, where sufficient knowledge exists that the stability of a certain analyte is very unlikely to be significantly affected during storage (e.g. based on experience from past stability tests or knowledge of its physicochemical properties), the Organisers, after consultation with the EUPT-QCG, may decide to omit a specific stability test. The EUPT-Panel will finally decide whether analytes for which the stability test was not undertaken will be included in the Final EUPT-Report, considering all relevant aspects such as the distribution of the participant's results (CV*).
A pesticide is considered to be adequately stable if $|y_i - y| \leq 0.3 \times \sigma_{pt}$, with y_i being the mean value of the results of the last phase of the stability test, y being the mean value of the results of the first phase of the stability test and σ_{pt} being the standard deviation used for proficiency assessment (typically 25 % of the assigned value).
The results of all stability tests are presented to the EUPT-SC. In special cases where the above stability test criteria are not met, the EUPT-SC considering all relevant aspects (e.g. the past experience with the stability of the compound, the overall distribution the participants' results, the measurement variability, analytical difficulties faced during the test and knowledge about the analytical behaviour of the pesticide question) may decide to overrule the test. The reasons of this overruling will be transparently explained in the Final EUPT-Report.
The Organisers may also decide to conduct additional stability tests at different storage conditions than those recommended to the participants e.g. at ambient temperature.

Stability during shipment: Considering knowledge about the expected susceptibility of pesticides in the Test Item to possible losses, the Organisers will choose the shipment conditions to be such that pesticide losses are minimised (e.g. shipment of frozen samples, addition of dry ice). As shipment time can differ between labs/countries it is recommended that the Organisers keep track of the shipment duration and then decide whether it is reasonable to conduct additional stability tests at conditions simulating shipment. Should critical losses be detected for certain pesticides, the EUPT-SC will be informed (or the EUPT-QCG before or during the test). Case-by-case decisions may be taken by the EUPT-Panel considering all relevant aspects including the duration and conditions of the shipment to the laboratory as well as the feedback by the laboratory.

Methodologies to be used by the participants
Participating laboratories are instructed to use the analytical procedure(s) that they would routinely employ in official control activities (monitoring etc.). Where an analytical method has not yet been established routinely this should be stated.

General procedures for reporting results
Participating laboratories are responsible for reporting their own quantitative results to the Organiser within the stipulated deadline. Any pesticide that was targeted by a participating laboratory should be reported as

"analysed". Each laboratory will be able to report only one result for each analyte detected in the Test Item. The concentrations of the pesticides detected should be expressed in 'mg/kg' unless indicated otherwise in the specific protocol. Laboratories should not report results below their reporting limits.

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

a) the use of recovery correction factors,
b) the use of stable isotope labelled analogues of the target analytes as Internal Standards (ILISs),
c) the 'procedural calibration' approach as well as
d) the approach of 'standard addition' with additions of analyte(s) being made to analytical portions.

Results may be corrected for recovery only in cases where this correction is applied in routine practice (including cases of MRL-violations). Laboratories are required to report whether their results were adjusted for recovery and, if a recovery factor was used, the recovery rate (in percentage) must also be reported. If one or more of the approaches b), c) and d) were employed, in which correction for recovery is inherent to the procedures, the apparent recovery figures obtained during validation experiments are not mandatory, and the approached followed are to be reported in the appropriate fields within the data submission tool.

Methodology information
All laboratories are requested to provide information on the analytical method(s) they have used. A compilation of the methodology information submitted by all participants is presented in an Annex of the Final EUPT-Report or in a separate report. Where necessary the methods are evaluated and discussed, especially in those cases where the result distribution is not unimodal or very broad (e.g. CV* > 35 %). If no sufficient information on the methodology used is provided, the Organisers reserve the right not to accept the analytical results reported by the participants concerned or even refuse participation in the following PT.

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

- ***False Positive results***

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

- ***False Negative results***

These are results for pesticides reported by the laboratories as 'analysed' but without reporting numerical values although they were: a) used by the Organiser to treat the Test Item and b) detected by the Organiser as well as the majority of the participants that had targeted these specific pesticides at or above the respective MRRLs. Results reported as '< RL' (RL= Reporting Limit of the laboratory) will be considered as not detected and will be judged as false negatives. In certain instances, case-by-case decisions by the EUPT-Panel may be necessary.
In cases of the assigned value being less than a factor of 3 times the MRRL, false negatives will typically not be assigned. The EUPT-Panel may decide to take case-by-case decisions in this respect after considering all relevant factors such as the result distribution and the reporting limits of the affected labs.

- ***Estimation of the assigned value (x_{pt})***

In order to minimise the influence of out-lying results on the statistical evaluation, the assigned value x_{pt} (= consensus concentration) will typically be estimated using the robust estimate of the participant's mean (x*) as described in ISO 13528:2015[12], taking into account the results reported by EU and EFTA countries laboratories only. In special justifiable cases, the EUPT-Panel may decide to eliminate certain results traceably associated with gross errors (see "Omission or Exclusion of results" below) or to use only the results of a subgroup consisting of laboratories that have repeatedly demonstrated good performance for the specific or similar compounds in the past.

- ***Omission or Exclusion of results***

Before estimating the assigned value, results associated with obvious mistakes have to be examined to decide whether they should be removed from the population. Such gross errors may include incorrect recording (e.g. due to transcription errors by the participant, decimal point faults or transposed digits, incorrect unit), calculation errors (e.g. missing factors), analysis of a wrong sample/extract (e.g. a spiked blank), use of wrong concentrations of standard solutions, incorrect data processing (e.g. integration of wrong peak), inappropriate storage or transport conditions (in case of susceptible compounds), and the use

[12] DIN ISO 13528:2015, Statistical methods for use in proficiency testing by interlaboratory comparisons, International Organization for Standardization. Therein a specific robust method for determination of the consensus mean and standard deviation without the need for removal of deviating results is described (Algorithm A in Annex C).

of inappropriate analytical steps or procedures that demonstrably lead to significantly biased results (e.g. employing inappropriate internal standards or analytical steps or conditions leading to considerable losses, due to degradations, adsorptions, incomplete extractions, partitioning etc.). Where the Organisers (e.g. after the publication of the preliminary report) receive information of such gross errors, having a significant impact on a generated result, the affected results will be examined on a case-by-case basis to decide whether, or not, they should be excluded from the population used for robust statistics. Results may also be omitted e.g. if an inappropriate method has been used even if they are not outliers. All decisions to omit/exclude results will be discussed with the EUPT-SC and the reasoning for the omission of each result clearly stated in the Final EUPT-Report. However, z scores will be calculated for all results irrespective of the fact that they were omitted from the calculation of the assigned value.
Omitted results might be interesting as they might give indications about possible source(s) of errors. The Organisers will thus ask the relevant lab(s) to provide feedback on possible sources of errors (see also "follow-up activities").
Results reported by laboratories from non EU member states are typically excluded from the population that is used to derive the assigned value (see also "Estimation of the assigned value").

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

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

where s* is the robust standard deviation and p is the number of results.
In certain cases, and considering all relevant factors (e.g. the result distribution, multimodality, the number of submitted results, information regarding analyte homogeneity/stability, information regarding the use of methodologies that might produce a bias that were used by the participants), the EUPT-Panel may consider the assigned value of a specific analyte to be too uncertain and decide that the results should not be evaluated, or only evaluated for informative purposes. The provisions of ISO 13528:2015 concerning the uncertainty of the assigned value will be taken into account.

- ***Standard deviation of the assigned value (target standard deviation)***

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

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

The EUPT-Panel reserves the right to also employ other FFP-RSDs or other approaches for setting the assigned value on a case-by-case basis, considering analytical difficulties and experience gained from previous proficiency tests.
For informative purposes the robust relative standard deviation (CV*) of the participants results is calculated according to ISO 13528:2015; Chapter 7.7 following Algorithm A in Annex C (so called "consensus approach").

- ***z scores***

This parameter is calculated using the following formula:

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

where x_i is the value reported by the laboratory, x_{pt} is the assigned value, and FFP-σ_{pt} is the standard deviation using the FFP approach. Z scores will be rounded to one decimal place. For the calculation of combined z scores (see below) the original z scores will be used and the combined z-scores will be rounded to one decimal place after calculation.
Any z scores > 5 will be typically reported as '> 5' and a value of '5' will be used to calculate combined z scores (see below).

Z scores will be interpreted in the following way, as is set in the ISO 17043:2010[14]:

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

For results considered as false negatives, z scores will be calculated using the MRRL or RL (the laboratory's Reporting Limit) if RL < MRRL. Where, using this approach, the calculated z scores for false negatives are > −3 (still questionable), they will be fixed at −3.5 to underline that these are unacceptable results. These z-scores will typically appear in the z-score histograms and used in the calculation of combined z-scores.

[13] Comparative Study of the Main Top-down Approaches for the Estimation of Measurement Uncertainty in Multiresidue Analysis of Pesticides in Fruits and Vegetables. J. Agric. Food Chem., 2011, 59(14), 7609-7619.
[14] ISO/IEC 17043:2010. Conformity assessment – General requirements for proficiency testing

- **Collection of measurement uncertainty (MU) figures**

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

- ***Category classification***

The EUPT-Panel will decide if and how to classify the laboratories into categories based on their scope and/or performance. Currently a scope-based classification into Category A and Category B is employed. Laboratories that a) are able to analyse at least 90% of the compulsory pesticides in the target pesticides list, b) have correctly detected and quantified a sufficiently high percentage of the pesticides present in the Test Item (at least 90 %) and c) reported no false positives, will have demonstrated 'sufficient scope' and will be therefore classified into Category A. For the 90% criterion the number of pesticides needed to be correctly analysed to have sufficient scope will be calculated by multiplying the number of compulsory pesticides from the Target Pesticides List by 0.9 and rounding to the nearest full number with 0.5 decimals being rounded downwards (see some examples in Table 1).

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

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

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

- ***Overall performance of laboratories - combined z scores***

For evaluation of the overall performance of laboratories within Category A, the Average of the Squared z score (AZ^2)[15,16] (see below) will be used. The AZ^2 is calculated as follows:

$$AZ^2 = \frac{\sum_{i=1}^{n} z_i^2}{n}$$

Where n is the number of z scores to be considered in the calculation. In the calculation of the AZ^2, z scores higher than 5 will be set as 5. Based on the AZ^2 achieved, the laboratories are classified as follows:

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

Combined z scores are considered to be of lesser importance than individual z scores. The EUPT-Panel retains the right not to calculate AZ^2 if it is considered as not being useful or if the number of results reported by any participant is considered to be too low.

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

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

In the case of EUPT-SRMs, where only a few results per lab may be available, the Average of the Absolute z scores (AAZ) may be calculated for informative purposes, but only for labs that have reported enough results to obtain 5 or more z scores. For the calculation of the AAZ, z scores higher than 5 will also be set as 5. The z-scores appointed to false negatives will be also included in the calculation of the combined z-scores.
Laboratories within Category B will be typically ranked according to the total number of pesticides they correctly reported to be present in the Test Item. The number of acceptable z scores achieved will be presented, too. The EURL-Panel retains the right to calculate combined z scores (see above) also for labs within Category B, e.g. for informative purposes, provided that a minimum number of results (z scores) have been reported.

Publication of results
The EURLs will publish a preliminary report, containing tentative assigned values and z score values for all pesticides present in the Test Item, within 2 months of the deadline for result submission.
The Final EUPT-Report will be published after the EUPT-Panel has discussed the results. Taking into account that the EUPT-Panel meets normally only once a year (typically in late summer or autumn) to discuss the results of all EUPTs organised by the EURLs earlier in the year, the Final EUPT-Report may be published up to 10 months after the deadline for results submission. Results submitted by non-EU/EFTA laboratories might not always be used in the tables or figures in the Final EUPT-Report.

Certificates of participation
Together with the Final EUPT-Report, the EURL Organiser will deliver a Certificate of Participation to each participating laboratory showing the z scores achieved for each individual pesticide, the combined z scores calculated (if any), and the classification into Categories.

Feedback
At any time before, during or after the PT participants have the possibility to contact the Organisers and make suggestions or indicate errors. After the distribution of the Final EUPT-Report, participating laboratories will be given the opportunity to give their feedback to the Organisers and make suggestions for future improvements.

Correction of errors
Should errors be discovered in any of the documents issued prior to the EUPT (Calendar, Target Pesticides List, Specific Protocol, General Protocol) the corrected documents will be uploaded onto the website and in the case of substantial errors the participants will be informed. **Before starting the exercise, participants should make sure to download the latest version of these documents.**
If substantial errors are discovered in the Preliminary EUPT-Report the Organisers will distribute a new corrected version, where it will be stated that the previous version is no longer valid.
Where substantial errors are discovered in the Final EUPT-Report the EUPT-Panel will decide whether a corrigendum will be issued and how this should look like. The online version of the Final EUPT report will be replaced by the new one and all affected labs will be contacted.
Where errors are discovered in EUPT-Certificates the relevant laboratories will be sent new corrected ones. Where necessary the laboratories will be asked to return the old ones.

Follow-up activities
Laboratories are expected to undertake follow-up activities to trace back the sources of erroneous or strongly deviating results (typically those with $|z| > 2.0$) - including all false positives. In exceptional cases, follow-up activities may even be indicated for results within $|z| \leq 2.0$ (e.g. where two errors with opposed tendency cancel each other leading to acceptable results).
Upon request, the laboratory's corresponding NRL and EURL are to be informed of the outcome of any investigative activities for false positives, false negatives and for results with $|z| \geq 3.0$. Concerning z scores between 2.0 and 3.0 the communication of the outcome of follow-up activities is optional but highly encouraged where the source of deviation could be identified and could be of interest to other labs.
According to instructions from DG-SANTE, the "Protocol for management of underperformance in comparative testing and/or lack of collaboration of National Reference Laboratories (NRLs) with EU Reference Laboratories (EURLs) activities" is to be followed.
NRLs will be considered as **underperforming in relation to scope** if in at least two of the last four EUPTs falling within their responsibility area they: a) haven't participated, or b) targeted less than 90% of the compulsory pesticides in the target lists (80% for SRM-compounds), or c) detected less than 90% of the compulsory compounds present in the test items (80% for SRM-compounds). Additionally, NRLs that obtained AZ^2 higher than 3 (AAZ higher than 1.3 for SRM-compounds) in two consecutive EUPTs of the last four EUPTs, will be considered as **underperforming in accuracy**. A two-step protocol established by DG-SANTE will be applied as soon as underperformance of an NRL is detected[17]:

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

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

Phase 2:

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

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

Disclaimer

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

EUPT-FV-SC04 SPECIFIC PROTOCOL

European Union Proficiency Test for Pesticide Residues in sultana raisins homogenate (2020)

Introduction

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

Test item

This proficiency test is based on the analysis of **sultana raisins** containing incurred pesticide residues. The test item will consist of a **slurry of sultana raisins and water** in a proportion 1:1.5 (w/w). That way, 12.5 g of test item will contain 5 g of sultana raisins.

The test item will be homogenised and sub-sampled into coded bottles. Ten of those bottles containing the test item will be chosen randomly and analysed to check for homogeneity.

The test item will be stored frozen (–20ºC) prior to shipment to participants.

Three bottles, again chosen randomly, will be analysed by the Organiser over a period of time to confirm the stability of the pesticides in the test item (firstly, when the test items are shipped, then a few days after the receipt deadline for participants´ results).

No blank material will be provided.

Steps to follow

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

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

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

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

4.The sample will be delivered to the participant laboratories on November 23rd 2020. 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 15th January 2021.

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 200 g of sultana raisins and water homogenate.

Shipment of Test item

All Test Items will be frozen and packed in polystyrene boxes surrounded in dry ice and packed into cardboard boxes.

The shipment of the test item will be on 23rd November 2020. The Organiser will try to ensure that all the packages arrive on the same day at each laboratory. An information message will be sent out by e-mail before shipment. Laboratories must make their own arrangements for the receipt of the package. They must inform the Organiser of any public holidays in their country/city during the delivery period given in the calendar, as well as making the necessary arrangements for receiving the shipment, even if the laboratory is closed.

Advice on Test item Handling

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

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

Test item Receipt

Once the laboratory has received the test item, its arrival must be reported to the Organiser by e-mail. The deadline for acceptance (or non-acceptance) is 27th November 2020. If the laboratory does not respond by this date, the Organiser will assume that the test item has been received and accepted.

If any laboratory has not received the test item by 27th November, they must inform the Organiser by e-mail (cferrer@ual.es)

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

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

- Residue levels above the reporting level and < 10 mg/kg should be rounded to two significant figures.
- Residue levels ≥ 10 mg/kg may be rounded to three significant figures or to a whole number.

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

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

Calendar

ACTIVITY	DATE
Opening Registration period	23rd October 2020
Deadline for receiving Application Form from laboratories.	16th November 2020
Sample distribution	23rd November 2020
Deadline for receiving results	15th January 2021
Preliminary Report with statistical treatment	February 2021
Final Report	August 2021

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

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

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

Contact information
The official organising group details are as follows:
Universidad de Almería. Edificio Químicas CITE I
Ctra. Sacramento s/n
04120 La Cañada de San Urbano Almería - Spain
Fax No.: +34 950015008

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

TARGET PESTICIDE LIST FOR THE EUPT-FV-SC04

Pesticide	MRRL (mg/Kg)
Acephate	0.01
Acetamiprid	0.01
Acrinathrin	0.01
Aldicarb	0.01
Aldicarb Sulfone	0.01
Aldicarb Sulfoxide	0.01
Aldrin	**0.005**
Ametoctradin	0.01
Azinphos-methyl	0.01
Azoxystrobin	0.01
Bifenthrin (sum of isomers)	0.01
Biphenyl	0.01
Bitertanol (sum of isomers)	0.01
Boscalid	0.01
Bromopropylate	0.01
Bromuconazole (sum of diastereoisomers)	0.01
Bupirimate	0.01
Buprofezin	0.01
Cadusafos	**0.005**
Carbaryl	0.01
Carbendazim	0.01
Carbofuran	**0.005**
Carbofuran-3-hydroxy	0.01
Chlorantraniliprole	0.01
Chlorfenapyr	0.01
Chlorfenvinphos	0.01
Chlorobenzilate	0.01
Chlorothalonil	0.01
Chlorpropham	0.01
Chlorpyrifos	**0.005**
Chlorpyrifos-methyl	0.01
Clofentezine	0.01
Clothianidin	0.01
Cyazofamid	0.01
Cyfluthrin (cyfluthrin incl. other mixtures of constituent isomers (sum of isomers))	0.01
Cyflufenamid: sum of cyflufenamid (Z-isomer) and its E-isomer	0.01
Cymoxanil	0.01
Cypermethrin (cypermethrin incl. other mixtures of constituent isomers (sum of isomers))	0.01
Cyproconazole	0.01
Cyprodinil	0.01
Deltamethrin (cis-deltamethrin)	0.01
Demeton-S-methylsulfone	**0.005**
Diazinon	**0.005**
Dichlofluanid	0.01
Dichlorvos	**0.005**
Dicloran	0.01
Dicofol (sum of p, p´ and o,p´ isomers)	0.01
Dieldrin	**0.005**
Diethofencarb	0.01
Difenoconazole	0.01
Diflubenzuron	0.01
Dimethoate	**0.003**
Dimethomorph (sum of isomers)	0.01
Dimethylaminosulfotoluidide (DMST)	0.01
Diniconazole (sum of isomers)	0.01
Diphenylamine	0.01
Endosulfan alpha	0.01
Endosulfan beta	0.01
Endosulfan sulfate	0.01
EPN	0.01
Epoxiconazole	0.01
Ethion	0.01

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

Pesticide	MRRL (mg/Kg)
Ethirimol	0.01
Ethoprophos	**0.005**
Etofenprox	0.01
Etoxazole	0.01
Famoxadone	0.01
Fenamidone	0.01
Fenamiphos	0.01
Fenamiphos sulfone	0.01
Fenamiphos sulfoxide	0.01
Fenarimol	0.01
Fenazaquin	0.01
Fenbuconazole	0.01
Fenhexamid	0.01
Fenitrothion	0.01
Fenoxycarb	0.01
Fenpropathrin	0.01
Fenpropidin	0.01
Fenpropimorph (sum of isomers)	0.01
Fenpyrazamine	0.01
Fenpyroximate	0.01
Fenthion	0.01
Fenthion oxon	0.01
Fenthion oxon sulfone	0.01
Fenthion oxon sulfoxide	0.01
Fenthion sulfone	0.01
Fenthion sulfoxide	0.01
Fenvalerate (any ratio of constituent isomers (RR, SS, RS & SR) including esfenvalerate)	0.01
Fipronil	**0.004**
Fipronil sulfone	**0.004**
Flonicamid	0.01
Flubendiamide	0.01
Fludioxonil	0.01
Flufenoxuron	0.01
Fluopicolide	0.01
Fluopyram	0.01
Fluquinconazole	0.01
Flusilazole	0.01
Flutolanil	0.01
Flutriafol	0.01
Fluxapyroxad	0.01
Formetanate (expressed as formetanate (hydrochloride))	0.01
Fosthiazate	0.01
Hexaconazole	0.01
Hexythiazox	0.01
Imazalil	0.01
Imidacloprid	0.01
Indoxacarb (sum of indoxacarb and its R enantiomer)	0.01
Iprodione	0.01
Iprovalicarb	0.01
Isocarbophos	0.01
Isofenphos-methyl	0.01
Isoprothiolane	0.01
Kresoxim-methyl	0.01
Lambda-Cyhalothrin	0.01
Linuron	0.01
Lufenuron (any proportion of constituent isomers)	0.01
Malaoxon	0.01
Malathion	0.01
Mandipropamid	0.01
Mepanipyrim	0.01
Metaflumizone (sum of E- and Z- isomers)	0.01
Metalaxyl and metalaxyl-M	0.01
Methamidophos	0.01
Methidathion	0.01

Pesticide	MRRL (mg/Kg)
Methiocarb	0.01
Methiocarb sulfone	0.01
Methiocarb sulfoxide	0.01
Methomyl	0.01
Methoxyfenozide	0.01
Metrafenone	0.01
Monocrotophos	**0.005**
Myclobutanyl	0.01
Omethoate	**0.003**
Orthophenylphenol (Free compound only)	0.01
Oxadixyl	0.01
Oxamyl	0.01
Oxydemeton-methyl	**0.005**
Paclobutrazole	0.01
Paraoxon-methyl	0.01
Parathion-ethyl	0.01
Parathion-methyl	0.01
Penconazole	0.01
Pencycuron	0.01
Pendimethalin	0.01
Permethrin (sum of isomers)	0.01
Phenthoate	0.01
Phosalone	0.01
Phosmet	0.01
Phosmet oxon	0.01
Phoxim	0.01
Pirimicarb	0.01
Pirimicarb-desmethyl	0.01
Pirimiphos-methyl	0.01
Prochloraz (only parent compound)	0.01
Procymidone	0.01
Profenofos	0.01
Propamocarb (only parent compound)	0.01
Propargite	0.01
Propiconazole (sum of isomers)	0.01
Propyzamide	0.01
Proquinazid	0.01
Prosulfocarb	0.01
Prothioconazole (Prothioconazole-desthio) (sum of isomers)	0.01
Prothiofos	0.01
Pymetrozine	0.01
Pyraclostrobin	0.01
Pyridaben	0.01
Pyrimethanil	0.01
Pyriproxyfen	0.01
Quinoxyfen	0.01
Spinosad (sum of spinosyn A and spinosyn D, expr. as spinosad)	0.01
Spirodiclofen	0.01
Spiromesifen	0.01
Spirotetramat	0.01
Spirotetramat metabolite BYI08330-enol	0.01
Spirotetramat metabolite BYI08330-ketohydroxy	0.01
Spirotetramat metabolite BYI08330-monohydroxy	0.01
Spirotetramat metabolite BYI08330 enol-glucoside	0.01
Spiroxamine (sum of isomers)	0.01
Tau-Fluvalinate	0.01
Tebuconazole	0.01
Tebufenozide	0.01
Tebufenpyrad	0.01
Teflubenzuron	0.01
Tefluthrin	0.01
Terbuthylazine	0.01
Tetraconazole	0.01
Tetradifon	0.01
Thiabendazole	0.01

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

Pesticide	MRRL (mg/Kg)
Thiacloprid	0.01
Thiamethoxam	0.01
Thiodicarb	0.01
Thiophanate-methyl	0.01
Tolclofos-methyl	0.01
Tolylfluanid	0.01
Triadimefon	0.01
Triadimenol (any proportion of constituent isomers)	0.01
Triazophos	**0.005**
Trichlorfon	0.01
Tricyclazole	0.01
Trifloxystrobin	0.01
Triflumuron	0.01
Trifluralin	0.01
Triticonazole	0.01
Vinclozolin (only parent compound)	0.01
Zoxamide	0.01

In bold new pesticides this year
MRRLs are based on Regulation (EC) No. 396/2005 and Commission Directive 2006/125/EC.

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
Austria	AGES - Innsbruck (Austrian Agency for Health and Food Safety), Institute for Food Safety	Innsbruck	Yes
Belgium	Groen Agro Control	Delfgauw	Yes
Belgium, France, Luxemburg	Primoris Belgium	Zwijnaarde	Yes
Bulgaria	Central Laboratory for Chemical Testing and Control	Sofia	Yes
Croatia	Sample Control d.o.o.	Lučko-Zagreb	Yes
Croatia	Inspecto laboratorij	Osijek	Yes
Cyprus	Pesticide Residues Laboratory of the State General Laboratory of Cyprus	Nicosia	Yes
Estonia	Laboratory for Residues and Contaminants	Saku, Saku vald	Yes
Finland	Finnish Customs Laboratory	Espoo	Yes
France	Cereco	Garons	Yes
France	Scl Paris	Massy	Yes
France	SCL Montpellier	Montpellier	Yes
France, Belgium, Luxemburg	Primoris Belgium	Zwijnaarde	Yes
Germany	CVUA RRW	Krefeld	Yes
Germany	Landesuntersuchungsamt - Institut für Lebensmittelchemie Speyer	Speyer	Yes
Germany	Zentrales Institut des Sanitätsdienstes der Bundeswehr Kiel	Kronshagen	Yes
Germany	GALAB Laboratories GmbH	Hamburg	Yes
Germany	Agrolab Lufa GmbH	Kiel	Yes
Germany	Landesuntersuchungsanstalt für das Gesundheits- und Veterinärwesen Sachsen	Dresden	Yes
Germany	Analytica Alimentaria GmbH	Kleinmachnow	Yes
Germany	Bavarian Health and Food Safety Authority	Erlangen	yes
Greece	Laboratory of Pesticide residues, Benaki Phytopathological Institute	Kifissia, Athens	Yes
Greece	General Chemical State Laboratory	Athens	Yes
Iceland	Matis	Reykjavik	Yes
Ireland	Pesticide Residue Laboratory	Kildare	Yes

ANNEX B. List of laboratories that agreed to participate in EUPT-FV-SC04

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
Italy	Laboratorio Sanità Pubblica USL Toscana Centro	Florence	Yes
Italy	Laboratory Pesticide - Istituto Zooprofilattico Sperimentale Lombardia Emilia Romagna - IZSLER	Brescia	Yes
Italy	Laboratorio di Prevenzione ATS Città Metropolitana di Milano	Milano	Yes
Lithuania	National Food and Veterinary Risk Assessment Institute	Vilnius	Yes
Luxemburg, Belgium, France,	Primoris Belgium	Zwijnaarde	Yes
Norway	NIBIO, Department of Pesticides and Natural Products Chemistry	Aas	Yes
Perú	Inspectorate Services Perú S.A - Chorrillos Laboratory	Lima	Yes
Poland	Fertico Sp. z o. o.	Grójec	Yes
Poland	Hamilton UO-Technologia	Grójec	Yes
Poland	Food Safety Laboratory, Research Institute of Horticulture	Skierniewice	Yes
Slovakia	Veterinary and Food Institute in Bratislava	Bratislava	Yes
Spain	AINIA	Valencia	Yes
Spain	Analytica Alimentaria	Almería	Yes
Spain	Eurofins sica agriq, s.l.u.	Almeria	Yes
Spain	Laboratorio Analitico Bioclinico, Slu	Almeria	Yes
Spain	Laboratorio Agroambiental de Zaragoza (Gobierno de Aragón)	Zaragoza	Yes
Spain	Laboratorio de Residuos. Departamento de Análisis Ambiental. Instituto Tecnológico de Canarias, S. A.	Agüimes, Gran Canaria	Yes
Spain	Centro Nacional De Alimentación - Aesan	Majadahonda-Madrid	Yes
Spain	Laboratorio de Salud Pública del Ayuntamiento de Madrid. MadridSalud	Madrid	Yes
Sweden	Eurofins Food & Feed Testing Sweden AB	Lidköping	Yes
The Netherlands	Wageningen Food Safety Research (WFSR)	Wageningen	Yes
United Kingdom	Fera Science Ltd.	York	Yes