EURL-PROFICIENCY TEST-FV-BF01, 2016

Pesticide Residues in Peach Baby Food

Final Report
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EURL-EUROPEAN UNION PROFICIENCY TEST
FOR THE DETERMINATION OF PESTICIDES IN BABY FOOD
USING MULTIRESIDUE METHODS
2016

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 (EC) No 882/2004[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 BF01 has been organised by the EURL in Fruit and Vegetables at the University of Almería, Spain[4].

All National Reference Laboratories (NRLs), as well as all other EU official laboratories involved in the determination of pesticide residues in fruit and vegetables were invited to participate in this voluntary European Proficiency Test BF01.

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 (EC) No 882/2004 of the European Parliament and of the Council on official controls performed to ensure compliance verification with feed and food law, animal health and animal welfare rules. Published in the OJ of the EU L191 on 28.05.2004.
[3] The Community Reference Laboratory (CRL) changed its name to the European Union Reference Laboratory (EURL) on 1st December 2009 as a result of the Treaty of Lisbon. OJ of the EU C306 on 17.12.2007.
[4] Commission Regulation (EC) No 776/2006 of 23rd May 2006 - amending Annex VII to Regulation (EC) No 882/2004 of the European Parliament and of the Council as regards European Union Reference Laboratories.

1. INTRODUCTION

Fifty-seven laboratories agreed to participate in European Union Proficiency Test BF01.

The proficiency test was performed in 2016 using a baby food based on peach purée, which was supplied by a Spanish producer. The baby food was spiked with a mixture of 17 pesticides. Participating laboratories were not provided with a 'blank' baby food sample.

The test item, 200 g of peach baby food containing pesticide residues, was shipped to participants on 7th November 2016. The deadline for results submission to the Organiser was 28th November 2016. The participants were asked to determine the residue levels of all the pesticides that they detected and to report the concentrations in mg/kg. The participants were provided with a target pesticide list, which is detailed in Annex 1. The list of target pesticides also contained the Minimum Required Reporting Levels (MRRL) for each pesticide fixed at 0.01 mg/kg, except for those pesticides included in EU Directive 2006/125/EC.

The robust mean values of the analytical data submitted 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.

2. TEST ITEMS

2.1 Preparation of the treated test item

The baby food based on peach purée was supplied by a Spanish baby food producer.
Before preparation of the test item, the pesticides and target residue levels were selected, following recommendations made by the Quality Control Group (QCG). Approximately 25 kg of baby food were spiked with the selected pesticides. All the pesticides were used as analytical standards dissolved in ethyl acetate. The spiked material was mixed manually during 60 minutes until an homogeneous material was obtained. 200 g portions of the well-mixed homogenate were weighed out into screw-capped polyethylene plastic bottles, sealed and stored in a freezer at about - 20 °C prior to distribution to participants.

2.2 Homogeneity test

For the homogeneity tests ten bottles of the treated test item were randomly chosen and analyses were performed on duplicate portions taken from each bottle. The sequence of analyses was determined using a table of randomly-generated numbers. 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 calibration curves constructed from matrix-matched standards prepared from the 'blank' baby food test item.

The statistical evaluation was performed according to the International Harmonized Protocol published by IUPAC, ISO and AOAC [1]. The individual concentrations from the homogeneity tests are given in Appendix 1. The results of the statistical analyses are given in Table 2.1. 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 the analytical sampling mean for all the pesticides. This was used to demonstrate that the between-bottle variance was not higher than the within-bottle variance.

Table 2.1 Statistical evaluation of the homogeneity test data (*n* = 20 analyses)

Pesticide	Mean Conc. (mg/Kg)	Ss^2	c	$Ss^2 < c$ Pass/Fail
Boscalid	0.014	2.31E-07	3.09E-06	Pass
Chlorpropham	0.017	2.41E-06	4.69E-06	Pass
Chlorpyrifos	0.012	3.55E-07	2.56E-06	Pass
Cypermethrin	0.008	4.38E-07	8.59E-07	Pass
Cyprodinil	0.033	5.18E-07	1.21E-05	Pass
Deltamethrin	0.041	5.28E-06	2.89E-05	Pass
Dimethoate	0.011	1.69E-08	1.38E-06	Pass
Etofenprox	0.024	0	1.01E-05	Pass

Pesticide	Mean Conc. (mg/Kg)	Ss^2	c	Ss^2 < c Pass/Fail
Fipronil	0.011	0	2.04E-06	Pass
Heptachlor	0.007	1.11E-06	1.39E-06	Pass
Hexachlorobenzene (HCB)	0.008	5.06E-07	1.35E-06	Pass
Imidacloprid	0.015	1.64E-06	2.42E-06	Pass
Iprodione	0.039	4.55e-06	2.68E-05	Pass
Malathion	0.027	3.20E-06	1.11E-05	Pass
Omethoate	0.016	0	3.02E-06	Pass
Spinosad	0.011	6.94E-09	1.61E-06	Pass
Tebuconazole	0.026	6.00E-07	8.8E-06	Pass

S_s: Between-Sampling Standard Deviation

As can be seen from Table 2.1, all the pesticides in the baby food matrix passed the homogeneity test.

2.3 Stability tests

The stability tests were performed according to ISO 13528:2015, Annex B [2]. Three bottles that were stored in the freezer at -20°C were chosen randomly and duplicate analyses were performed for each one of them. This procedure was repeated on two different occasions:

-Day 1: shortly before the test item shipment, which took place on November 7th 2016.
-Day 2: shortly after the deadline for reporting results, on November 28th 2016.

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 first stability test, yi the mean value of the last stability test and σ the standard deviation used for proficiency assessment (typically 25 % of the assigned value).

The individual results are given in Table 2.2. The tests 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 arriving not completely frozen, a duplicate analysis of a bottle reproducing the delivery conditions that the samples experienced during 48 hours was performed. Laboratories could therefore be sufficiently confident in accepting the treated test item even if it was not completely frozen. Results for this 48 hours' stability test are indicated in Table 2.3.

Table 2.2 Statistical test for analytical precision and to demonstrate results stability after a time-elapse interval

(mg/Kg)	Day 1 (Sample 017_A)	Day 1 (Sample 017_B)	Day 1 (Sample 064_A)	Day 1 (Sample 064_B)	Day 1 (Sample 089_A)	Day 1 (Sample 089_B)	Mean 1	Day 3 (Sample 047_A)	Day 3 (Sample 047_B)	Day 3 (Sample 061_A)	Day 3 (Sample 061_B)	Day 3 (Sample 090_A)	Day 3 (Sample 090_B)	Mean3	(M3 – M1)	M3-M1 ≤ 0.3*σ
Boscalid	0.013	0.012	0.013	0.013	0.014	0.013	0.013	0.012	0.013	0.013	0.013	0.013	0.013	0.013	0.000	Pass
Chlorpropham	0.018	0.016	0.017	0.017	0.019	0.017	0.017	0.016	0.018	0.018	0.018	0.018	0.018	0.018	0.000	Pass
Chlorpyrifos	0.012	0.012	0.012	0.012	0.013	0.012	0.012	0.011	0.012	0.012	0.012	0.012	0.013	0.012	0.000	Pass
Cypermethrin	0.008	0.007	0.007	0.008	0.008	0.008	0.008	0.008	0.008	0.008	0.008	0.009	0.008	0.008	0.001	Pass
Cyprodinil	0.031	0.031	0.032	0.031	0.032	0.030	0.031	0.030	0.031	0.030	0.031	0.031	0.030	0.031	-0.001	Pass
Deltamethrin	0.039	0.034	0.037	0.037	0.040	0.037	0.037	0.034	0.036	0.034	0.035	0.036	0.037	0.035	-0.002	Pass
Dimethoate	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.011	0.010	0.011	0.010	0.001	Pass
Etofenprox	0.022	0.020	0.022	0.021	0.023	0.022	0.021	0.020	0.022	0.022	0.023	0.022	0.023	0.022	0.001	Pass
Fipronil	0.011	0.010	0.010	0.011	0.011	0.011	0.011	0.009	0.009	0.010	0.011	0.012	0.010	0.010	0.000	Pass
Heptachlor	0.008	0.007	0.007	0.007	0.007	0.007	0.007	0.006	0.007	0.007	0.007	0.007	0.007	0.007	0.000	Pass
HCB	0.009	0.008	0.008	0.009	0.009	0.008	0.008	0.007	0.008	0.008	0.008	0.008	0.008	0.008	-0.001	Pass
Imidacloprid	0.014	0.014	0.014	0.014	0.014	0.014	0.014	0.013	0.013	0.013	0.014	0.013	0.014	0.013	-0.001	Pass
Iprodione	0.035	0.031	0.035	0.035	0.038	0.036	0.035	0.030	0.036	0.035	0.034	0.036	0.035	0.034	-0.001	Pass
Malathion	0.025	0.023	0.025	0.025	0.027	0.024	0.025	0.023	0.027	0.026	0.026	0.026	0.026	0.025	0.000	Pass
Omethoate	0.016	0.016	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.000	Pass
Spinosad	0.011	0.010	0.011	0.010	0.011	0.010	0.011	0.011	0.011	0.010	0.011	0.011	0.010	0.011	0.000	Pass
Tebuconazole	0.021	0.024	0.024	0.024	0.024	0.025	0.024	0.021	0.024	0.024	0.024	0.024	0.025	0.024	0.000	Pass

Table 2.3 Statistical test for analytical precision and to demonstrate stability for the 48-hour time-elapse interval.

(mg/Kg)	Day 1 (Sample 017_A)	Day 1 (Sample 017_B)	Day 1 (Sample 064_A)	Day 1 (Sample 064_B)	Day 1 (Sample 089_A)	Day 1 (Sample 089_B)	Mean 1	Day 2 (Sample 008_A)	Day 2 (Sample 008_B)	Day 2 (Sample 045_A)	Day 2 (Sample 045_B)	Day 2 (Sample 071_A)	Day 2 (Sample 071_B)	Mean2	(M2 – M1)	M2-M1 ≤ 0.3*σ
Boscalid	0.013	0.012	0.013	0.013	0.014	0.013	0.013	0.014	0.014	0.014	0.015	0.013	0.014	0.014	0.001	Pass
Chlorpropham	0.018	0.016	0.017	0.017	0.019	0.017	0.017	0.018	0.017	0.018	0.018	0.016	0.018	0.018	0.000	Pass
Chlorpyrifos	0.012	0.012	0.012	0.012	0.013	0.012	0.012	0.013	0.012	0.013	0.013	0.011	0.013	0.013	0.001	Pass
Cypermethrin	0.008	0.007	0.007	0.008	0.008	0.008	0.008	0.009	0.008	0.008	0.008	0.008	0.008	0.008	0.001	Pass
Cyprodinil	0.031	0.031	0.032	0.031	0.032	0.030	0.031	0.031	0.029	0.026	0.030	0.031	0.029	0.029	-0.002	Pass
Deltamethrin	0.039	0.034	0.037	0.037	0.040	0.037	0.037	0.040	0.037	0.041	0.041	0.036	0.041	0.039	0.002	Pass
Dimethoate	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.000	Pass
Etofenprox	0.022	0.020	0.022	0.021	0.023	0.022	0.021	0.023	0.021	0.023	0.023	0.020	0.023	0.022	0.001	Pass
Fipronil	0.011	0.010	0.010	0.011	0.011	0.011	0.011	0.010	0.010	0.009	0.011	0.011	0.010	0.010	-0.001	Pass
Heptachlor	0.008	0.007	0.007	0.007	0.007	0.007	0.007	0.008	0.008	0.008	0.008	0.007	0.008	0.008	0.001	Pass
HCB	0.009	0.008	0.008	0.009	0.009	0.008	0.008	0.009	0.009	0.009	0.009	0.008	0.008	0.008	0.000	Pass
Imidacloprid	0.014	0.014	0.014	0.014	0.014	0.014	0.014	0.015	0.014	0.014	0.014	0.014	0.014	0.014	0.000	Pass

(mg/Kg)	Day 1 (Sample 017_A)	Day 1 (Sample 017_B)	Day 1 (Sample 064_A)	Day 1 (Sample 064_B)	Day 1 (Sample 089_A)	Day 1 (Sample 089_B)	Mean 1	Day 2 (Sample 008_A)	Day 2 (Sample 008_B)	Day 2 (Sample 045_A)	Day 2 (Sample 045_B)	Day 2 (Sample 071_A)	Day 2 (Sample 071_B)	Mean2	(M2 – M1)	M2-M1 ≤ 0.3*σ
Iprodione	0.035	0.031	0.035	0.035	0.038	0.036	0.035	0.036	0.034	0.038	0.036	0.032	0.038	0.036	0.001	Pass
Malathion	0.025	0.023	0.025	0.025	0.027	0.024	0.025	0.028	0.025	0.028	0.027	0.023	0.027	0.026	0.001	Pass
Omethoate	0.016	0.016	0.015	0.015	0.015	0.015	0.015	0.015	0.014	0.014	0.014	0.014	0.014	0.014	-0.001	Pass
Spinosad	0.011	0.010	0.011	0.010	0.011	0.010	0.011	0.010	0.010	0.010	0.010	0.011	0.010	0.010	0.000	Pass
Tebuconazole	0.025	0.024	0.024	0.026	0.025	0.023	0.024	0.029	0.024	0.023	0.023	0.024	0.023	0.024	0.000	Pass

2.4 Distribution of test items and protocol to participants

One bottle of frozen treated test item was shipped to each participant in boxes containing dry ice. The test items were sent out on 7th November 2016. Ninety-eight percent of the shipments to EU/EFTA countries arrived within the first 48 hours.

Before sample shipment, the laboratories received full instructions (Annex 1) for the receipt and storage of the test items 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. This information was made available when laboratories received an e-mail from the Organiser confirming their acceptance along with their Lab Code and thus allowing them to participate. This ensured that confidentiality was maintained throughout the duration of Proficiency Test BF01.

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.

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 four 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 been calculated for all pesticides that were detected and reported at levels at, or above, the MRRL. They have also been calculated for false negatives.

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 [2], taking into account the results reported by EU and EFTA 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.

Taking into account the normative for robust analysis in ISO 13528:2015 [2], the uncertainty of the assigned value for each pesticide was calculated according to the following equation:

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

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, independently of their 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 4.4.

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 level (RL) (whichever one is lower) for those labs that have not detected the presence of the pesticide in the sample.
- X_{pt} is the assigned value.
- σ_{pt} is the target standard deviation (the FFP-RSD of 25 % multiplied by the assigned value).

z score classification is as follows:

$\|z\| \leq 2.0$	Acceptable
$2.0 < \|z\| < 3.0$	Questionable
$\|z\| \geq 3.0$	Unacceptable

- Any z score values of $|z| > 5$ have been reported as '>5'.
- 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.

4. RESULTS

4.1 Summary of reported results

Fifty-seven laboratories agreed to participate in this proficiency test. One cancelled their participation because the test item was retained at customs for two weeks. The total number of laboratories submitting results was 56. The results reported by all the laboratories are presented in this report. However, only results reported by laboratories from EU-countries and EFTA-countries have been included in the statistical treatment. The results from the laboratories in Egypt, India, Kenya, Serbia and Taiwan have not been included. This last group totals four laboratories that reported results.

Seventeen pesticides from the pesticide target list were used to treat the sample. The assigned values of all the pesticides except omethoate were below than a factor of four times the MRRL. For this reason, all the data shown in this report except for omethoate will be considerer only as informative purposes, and false negatives will be calculated only in the case of omethoate. A summary of the reported results can be seen below in Table 4.1.

Table 4.1 Summary of Reported Results

Pesticides	No. of Reported Results	No. of False Negative Results	No. of Not Analysed Results	Percentage of Reported Results* (out of 52)
Boscalid	48	-	2	92
Chlorpropham	47	-	4	90
Chlorpyrifos	51	-	1	98
Cypermethrin	44	-	3	85
Cyprodinil	50	-	2	96
Deltamethrin	52	-	0	100
Dimethoate	49	-	0	94
Etofenprox	49	-	2	94
Fipronil	47	-	4	90
Heptachlor	47	-	4	90
HCB	48	-	4	92
Imidacloprid	46	-	3	88
Iprodione	46	-	4	88
Malathion	48	-	2	92
Omethoate	48	3	1	92
Spinosad	47	-	4	90
Tebuconazole	50	-	1	96

* The percentage of Reported Results comes from 52 laboratories. It does not take into account the four laboratories from Egypt, Kenya, Serbia and Taiwan.

Annex 2 gathers all laboratories that agreed to participate. All results reported by the participants are given in Appendix 3.

4.1.1 False positives

No laboratories reported results for additional pesticides that were not present in the test item.

4.1.2 False negatives

Table 4.3 summarises the results from laboratories (including non-EU/EFTA laboratories) that reported false negatives, presented as 'Not Detected' (ND). As set in the general proctocol, in cases of the assigned value being less than a factor of four times the MRRL, false negatives will typically not be assigned. Therefore, only false negatives for omethoate were considered.

Table 4.3 Laboratories that failed to report pesticides that were present in the treated test item.

Laboratory Code	Omethoate
Lab015*	ND
Lab045	ND
Lab051	ND
Lab056	ND

*Non-EU/EFTA laboratories

4.1.3 Distribution of data

The distribution of the concentrations of the pesticides reported by the laboratories has been plotted as histograms with a bandwidth of $0.75 \cdot \sigma$ (σ is the target standard deviation (the FFP-RSD of 25 % multiplied by the assigned value) after removing outliers, if necessary. The histograms of both the compulsory and voluntary pesticides present in the test item are presented in Appendix 2.

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 seventeen pesticides and their uncertainties are presented in Table 4.4.

The target standard deviation was calculated using a fixed FFP-RSD value of 25 %. For comparison, a robust standard deviation (CV*) was also calculated for informative purposes, employing also this value for the calculation of the uncertainty. These RSDs can be seen in Table 4.4.

Table 4.4 Robust mean values, uncertainty and % RSDs for all pesticides in the test item.

Pesticides	MRRL (mg/kg)	Robust mean (mg/kg)	Uncertainty (mg/kg)	FFP-RSD (%)	CV* (%)
Boscalid	0.010	0.015	0.0004	25.0	14.9
Chlorpropham	0.010	0.020	0.0006	25.0	16.8
Chlorpyrifos	0.010	0.012	0.0003	25.0	14.6
Cypermethrin	0.010	0.010	0.0006	25.0	28.1
Cyprodinil	0.010	0.026	0.0007	25.0	14.8
Deltamethrin	0.010	0.035	0.0011	25.0	18.5
Dimethoate	0.003	0.009	0.0003	25.0	19.0
Etofenprox	0.010	0.024	0.0007	25.0	16.9
Fipronil	0.004	0.010	0.0004	25.0	21.7
Heptachlor	0.003	0.008	0.0003	25.0	21.6
HCB	0.003	0.009	0.0003	25.0	17.7
Imidacloprid	0.010	0.013	0.0005	25.0	19.5
Iprodione	0.010	0.037	0.0011	25.0	16.8
Malathion	0.010	0.030	0.0008	25.0	15.0
Omethoate	0.003	0.014	0.0007	25.0	25.4
Spinosad	0.010	0.012	0.0004	25.0	17.9
Tebuconazole	0.010	0.025	0.0005	25.0	11.6

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 in the test item. Z scores of false negative results were only calculated for omethoate.

In Appendix 3, the individual z scores are presented for each laboratory, together with the assigned values for each pesticide. The z scores of laboratories from non-EU countries have been included in Appendix 3 but have not been considered in the following table.

Table 4.5 Classification of z scores for the pesticides reported (Only EU/EFTA participants)

Pesticides	Acceptable (%)	Questionable (%)	Unacceptable (%)
Boscalid	97.9	0.0	2.1
Chlorpropham	100.0	0.0	0.0
Chlorpyrifos	96.1	2.0	2.0
Cypermethrin	83.7	2.3	14.0
Cyprodinil	98.0	0.0	2.0
Deltamethrin	96.2	1.9	1.9
Dimethoate	100.0	0.0	0.0
Etofenprox	95.9	2.0	2.0
Fipronil	97.9	2.1	0.0
Heptachlor	97.9	0.0	2.1
HCB	95.8	2.1	2.1
Imidacloprid	97.8	0.0	2.2
Iprodione	95.7	0.0	4.3
Malathion	97.9	0.0	2.1
Omethoate	86.3	5.9	7.8
Spinosad	89.4	4.3	6.4
Tebuconazole	98.0	2.0	0.0

In Appendix 4, graphical representations of the z scores of EU/EFTA laboratories are presented. No z scores have been calculated for false positive results. The charts have been constructed using different colour bars according to the determination technique used for each particular pesticide.

5. CONCLUSIONS

Fifty-seven laboratories agreed to participate in EUPT-FV-BF01. One of them cancelled its participation. From the remaining 56 laboratories that submitted results, four did not belong to EU nor EFTA countries, so their results were not considered for the estimation of the assigned value.

Seventeen pesticides were present in EUPT-FV-BF01 test item, based on the analysis of peach baby food. The assigned values of all the pesticides except omethoate were below than a factor of four times the MRRL. For this reason, all the data shown in this report except for omethoate was be considerer only as informative purposes, and false negatives were calculated only in the case of omethoate.

Of a total number of 884 possible determinations from EU/EFTA laboratories (52 laboratories by 17 pesticides), 92.4 % results were reported and 4.6 % were not analysed. The false positive rate was of 0 %.

The total number of z scores of laboratories from EU/EFTA countries was 820, with 95.6 % of them acceptable, 1.5 % questionable and 2.9 % unacceptable.

The robust standard deviation (CV*) was in all cases below 28.1 %, with an average value of 18.3 % for the 17 pesticides. These figures reveal the good performance of the participant laboratories, even at low concentration levels.

Participation in this first European Proficiency Test on baby food involved laboratories from 21 Member States. Additionally, laboratories from Norway and Switzerland participated as EFTA countries. Non-European laboratories from Egypt, Indonesia, Kenya, Serbia and Taiwan participated in EUPT-FV-BF01. These Non-EU laboratories, however, are official laboratories in their own countries.

6. REFERENCES

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

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

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

7. ACKNOWLEDGEMENTS

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The Organiser wishes to thank the members of the Quality Control Group and the Scientific Committee for their invaluable expert advice.

The Organiser wishes to give a special thank-you to the University of Almeria for the use of their facilities.

Boscalid (mg/kg)		Chlorpropham (mg/kg)		Chlorpyrifos (mg/kg)		Cypermethrin (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.013	0.015	0.016	0.018	0.012	0.013	0.007	0.008
0.015	0.015	0.019	0.018	0.012	0.013	0.008	0.008
0.014	0.015	0.018	0.019	0.012	0.013	0.008	0.008
0.015	0.016	0.018	0.019	0.013	0.013	0.008	0.008
0.015	0.016	0.018	0.019	0.013	0.015	0.008	0.009
0.016	0.014	0.019	0.017	0.012	0.013	0.008	0.007
0.013	0.015	0.016	0.018	0.011	0.013	0.007	0.008
0.013	0.014	0.016	0.018	0.011	0.012	0.007	0.008
0.014	0.013	0.015	0.014	0.011	0.010	0.007	0.007
0.015	0.012	0.015	0.012	0.012	0.010	0.008	0.007

Cyprodinil (mg/kg)		Deltamethrin (mg/kg)		Dimethoate (mg/kg)		Etofenprox (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.035	0.035	0.035	0.040	0.010	0.011	0.020	0.024
0.033	0.033	0.044	0.042	0.011	0.012	0.024	0.025
0.032	0.034	0.041	0.044	0.011	0.011	0.024	0.025
0.033	0.032	0.042	0.048	0.011	0.011	0.024	0.028
0.032	0.032	0.040	0.048	0.011	0.011	0.024	0.027
0.033	0.032	0.046	0.040	0.011	0.011	0.026	0.024
0.032	0.032	0.038	0.044	0.011	0.011	0.022	0.025
0.032	0.033	0.036	0.038	0.011	0.011	0.021	0.023
0.033	0.031	0.038	0.038	0.011	0.011	0.023	0.021
0.033	0.033	0.037	0.034	0.011	0.011	0.025	0.021

Fipronil (mg/kg)		Heptachlor (mg/kg)		Hexachlorobenzene (mg/kg)		Imidacloprid (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.013	0.011	0.007	0.008	0.008	0.008	0.015	0.014
0.011	0.011	0.009	0.008	0.010	0.008	0.016	0.015
0.010	0.011	0.009	0.009	0.010	0.009	0.015	0.015
0.011	0.012	0.008	0.009	0.008	0.009	0.015	0.015
0.011	0.011	0.008	0.008	0.008	0.007	0.015	0.015
0.010	0.012	0.008	0.007	0.007	0.006	0.015	0.015
0.010	0.012	0.006	0.008	0.006	0.007	0.016	0.015
0.012	0.012	0.007	0.008	0.007	0.008	0.015	0.015
0.011	0.011	0.005	0.004	0.008	0.008	0.015	0.014
0.011	0.011	0.007	0.005	0.008	0.006	0.014	0.014

APPENDIX 1. Homogeneity data.

Iprodione (mg/kg)		Malathion (mg/kg)		Omethoate (mg/kg)		Spinosad (mg/kg)	
Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
0.034	0.039	0.024	0.028	0.017	0.016	0.011	0.012
0.041	0.040	0.029	0.028	0.016	0.016	0.011	0.011
0.039	0.040	0.027	0.028	0.016	0.015	0.011	0.011
0.042	0.044	0.029	0.029	0.016	0.015	0.011	0.011
0.041	0.044	0.029	0.031	0.016	0.015	0.011	0.011
0.045	0.037	0.031	0.027	0.015	0.015	0.010	0.010
0.038	0.040	0.026	0.029	0.016	0.015	0.011	0.010
0.036	0.038	0.025	0.027	0.016	0.015	0.012	0.011
0.037	0.032	0.025	0.024	0.016	0.014	0.011	0.010
0.038	0.029	0.025	0.021	0.016	0.015	0.011	0.011

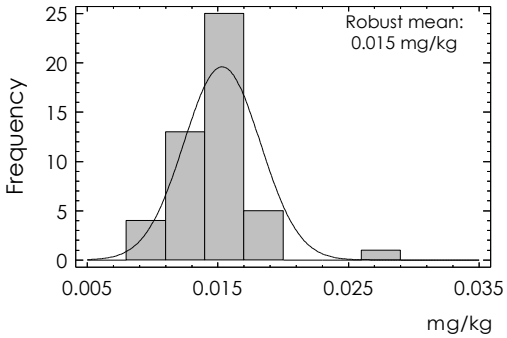
Tebuconazole (mg/kg)	
Replicate 1	Replicate 2
0.026	0.025
0.027	0.025
0.027	0.027
0.026	0.023
0.025	0.026
0.025	0.025
0.029	0.028
0.027	0.024
0.028	0.026
0.025	0.028

The sample numbers used for this test were: 1, 33, 35, 44, 50, 65, 70, 81, 91, and 101.

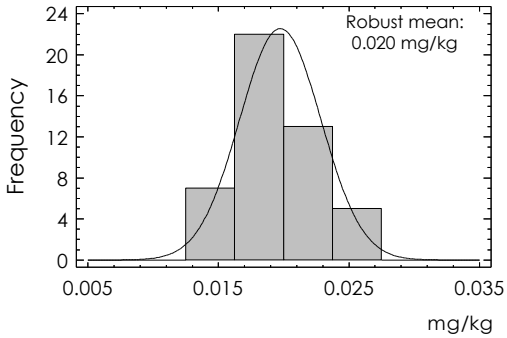
APPENDIX 2. Histograms of residue data for each pesticide from all the laboratories.

Results presented as histograms.

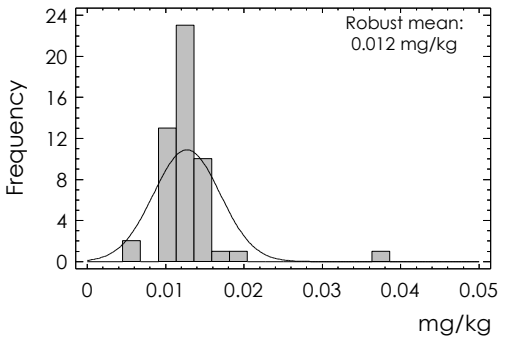
Boscalid



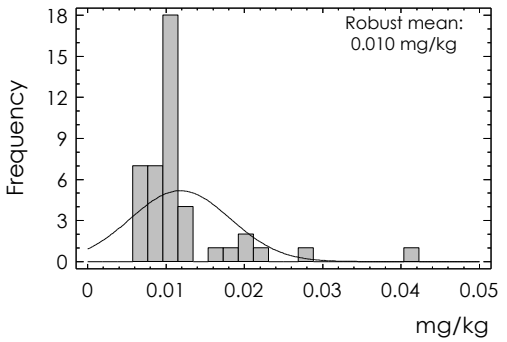
Chlorpropham



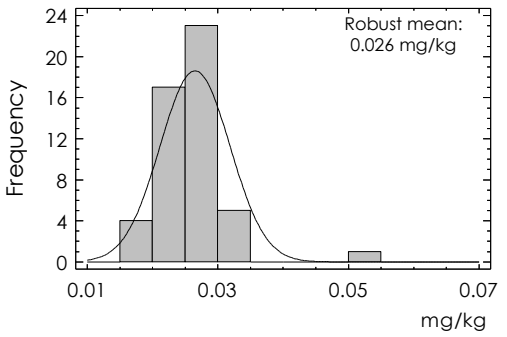
Chlorpyrifos



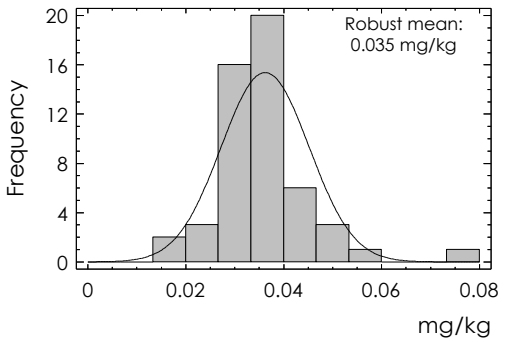
Cypermethrin (cypermethrin incl. other mixtures of constituent isomers (sum of isomers))



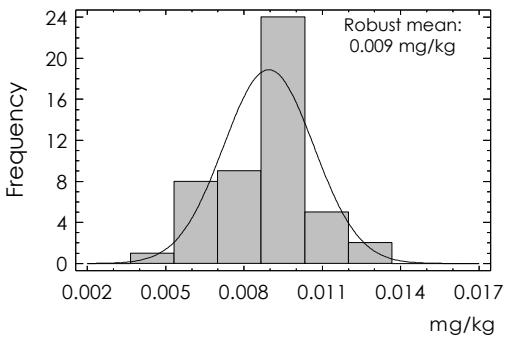
Cyprodinil



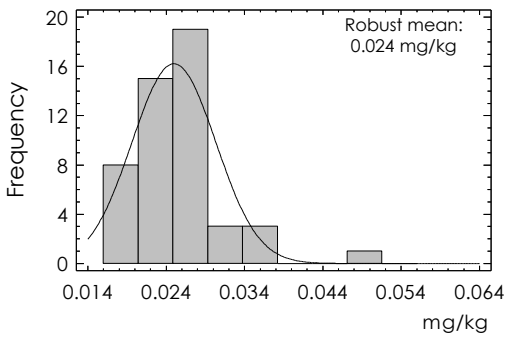
Deltamethrin (cis-deltamethrin)



Dimethoate

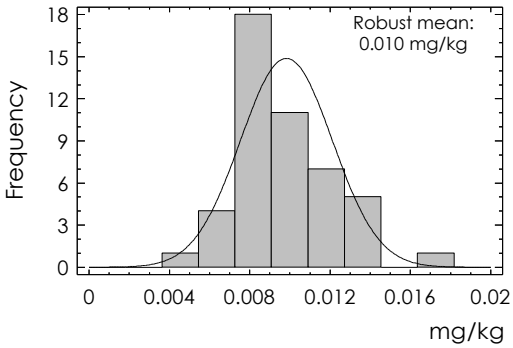


Etofenprox

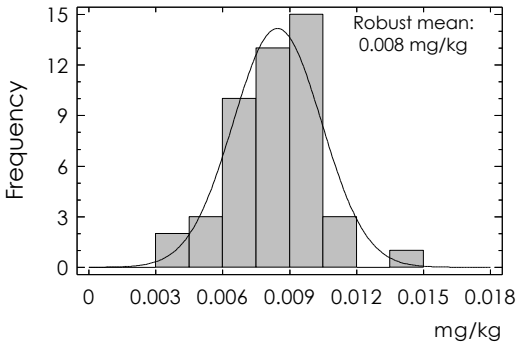


APPENDIX 2. Histograms of residue data for each pesticide from all the laboratories.

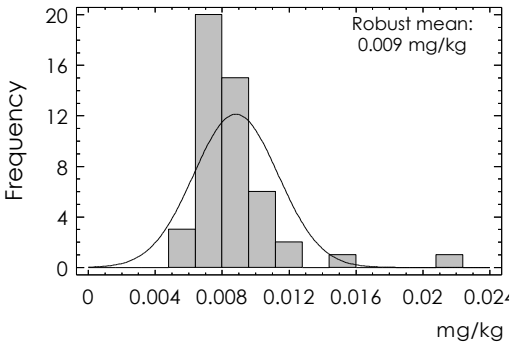
Fipronil



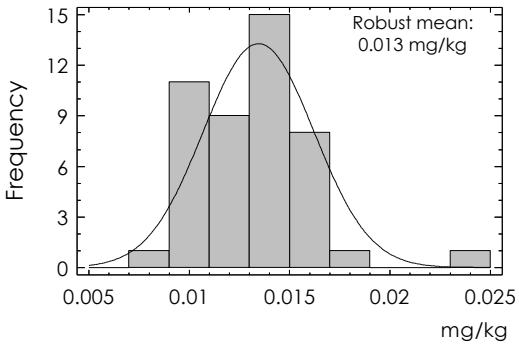
Heptachlor



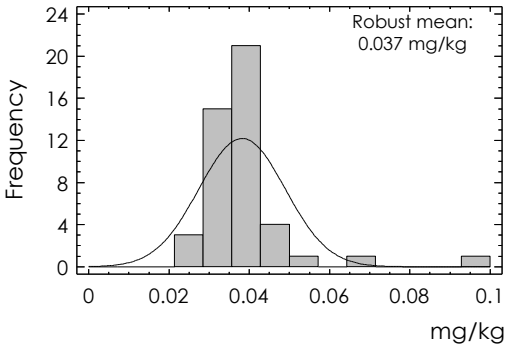
Hexachlorobenzene



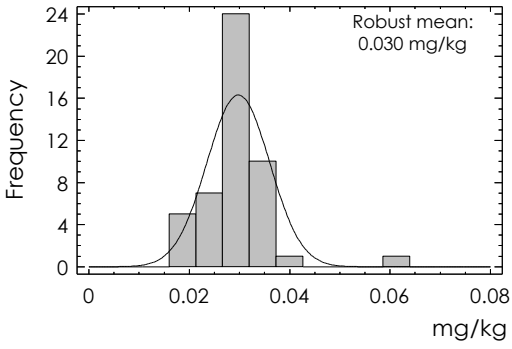
Imidacloprid



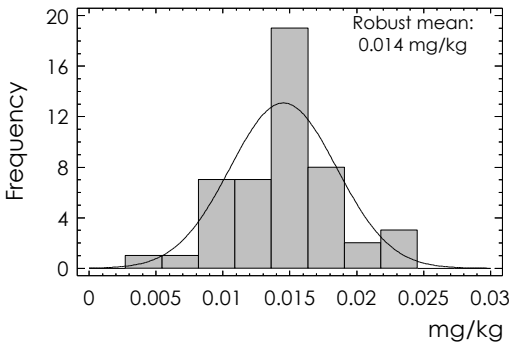
Iprodione



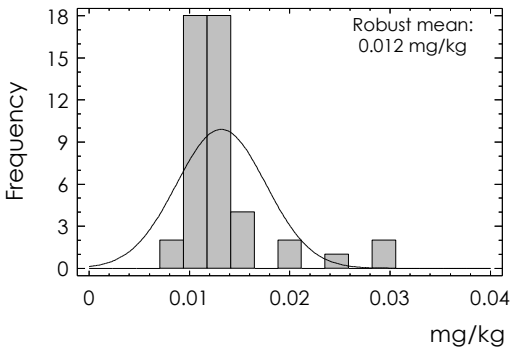
Malathion



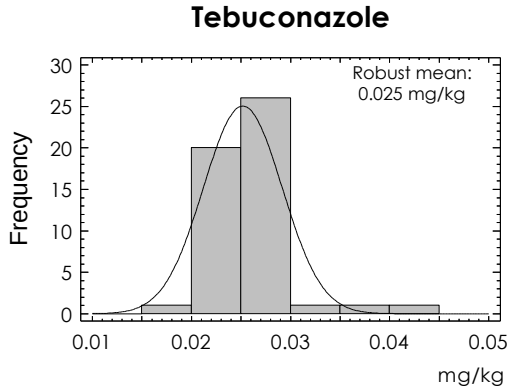
Omethoate



Spinosad (sum of spinosyn A and spinosyn D, expr. as spinosad)



APPENDIX 2. Histograms of residue data for each pesticide from all the laboratories.



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

Results reported by the laboratories for pesticides boscalid, chlorpropham, chlorpyrifos, cypermethrin, cyprodinil, deltamethrin, dimethoate, etofenprox, fipronil, heptachlor, hexachlorobenzene, imidacloprid, iprodione, malathion, omethoate, spinosad and tebuconazole (mg/kg) and their calculated z score value using FFP RSD 25 %

Lab Code	Boscalid	z score (FFP RSD 25 %)	Chlorpropham	z score (FFP RSD 25 %)	Chlorpyrifos	z score (FFP RSD 25 %)	Cypermethrin	z score (FFP RSD 25 %)	Cyprodinil	z score (FFP RSD 25 %)	Deltamethrin	z score (FFP RSD 25 %)	Dimethoate	z score (FFP RSD 25 %)	Etofenprox	z score (FFP RSD 25 %)	Fipronil	z score (FFP RSD 25 %)
MRRL	**0.01**		**0.01**		**0.01**		**0.003**		**0.01**		**0.01**		**0.003**		**0.01**		**0.004**	
Robust mean (mg/kg)	**0.015**		**0.020**		**0.012**		**0.010**		**0.026**		**0.035**		**0.009**		**0.024**		**0.010**	
Lab001	0.01	-1.3	NA	NA	0.01	-0.7	NA	NA	0.02	-0.9	0.04	0.6	NR	NR	0.02	-0.7	NR	NR
Lab002	0.0184	0.9	0.0185	-0.3	0.0113	-0.2	0.0095	-0.2	0.0309	0.8	0.0367	0.2	0.00932	0.1	0.0249	0.2	0.0122	0.9
Lab003	0.015	0.0	0.017	-0.6	0.012	0.0	0.027	>5	0.028	0.3	0.037	0.2	0.013	1.8	0.022	-0.3	0.009	-0.4
Lab004	0.016	0.3	0.024	0.8	0.013	0.3	0.01	0.0	0.03	0.6	0.037	0.2	0.01	0.4	0.023	-0.2	0.012	0.8
Lab005	0.018	0.8	0.027	1.4	0.019	2.3	0.016	2.4	0.028	0.3	0.048	1.5	0.011	0.9	0.037	2.2	0.013	1.2
Lab006	0.019	1.1	0.014	-1.2	0.011	-0.3	0.041	>5	0.017	-1.4	0.036	0.1	0.0091	0.0	0.024	0.0	0.0099	0.0
Lab007	0.015	0.0	0.016	-0.8	0.012	0.0	0.021	4.4	0.025	-0.2	0.036	0.1	0.008	-0.4	0.022	-0.3	0.008	-0.8
Lab008	Participation cancelled																	
Lab009	0.012	-0.8	0.021	0.2	0.012	0.0	0.007	-1.2	0.023	-0.5	0.028	-0.8	0.006	-1.3	0.018	-1.0	0.009	-0.4
Lab010	0.017	0.5	0.017	-0.6	0.012	0.0	0.01	0.0	0.022	-0.6	0.03	-0.6	0.007	-0.9	0.025	0.2	0.01	0.0
Lab011	0.015	0.0	0.023	0.6	0.014	0.7	0.011	0.4	0.024	-0.3	0.042	0.8	0.009	0.0	0.028	0.7	0.009	-0.4
Lab012	0.014	-0.3	0.019	-0.2	0.012	0.0	0.011	0.4	0.027	0.2	0.041	0.7	0.012	1.3	0.02	-0.7	0.007	-1.2
Lab013	0.011	-1.1	0.016	-0.8	0.01	-0.7	NR	NR	0.022	-0.6	0.026	-1.0	0.008	-0.4	0.018	-1.0	0.007	-1.2
Lab014	0.013	-0.5	0.019	-0.2	0.011	-0.3	0.011	0.4	0.027	0.2	0.037	0.2	0.0082	-0.4	0.025	0.2	0.0086	-0.6
Lab015	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	0.02	-1.7	0.018	4.0	0.03	1.0	NR	NR
Lab016	0.017	0.5	0.015	-1.0	0.01	-0.7	0.013	1.2	0.025	-0.2	0.032	-0.3	0.006	-1.3	0.025	0.2	0.009	-0.4
Lab017	NR	NR	0.022	0.4	0.012	0.0	0.012	0.8	0.024	-0.3	0.033	-0.2	NR	NR	0.022	-0.3	0.009	-0.4
Lab018	NA	NA	NA	NA	0.013	0.3	0.011	0.4	0.026	0.0	0.026	-1.0	0.01	0.4	0.032	1.3	NA	NA
Lab019	0.015	0.0	0.017	-0.6	0.012	0.0	0.008	-0.8	0.028	0.3	0.036	0.1	0.009	0.0	0.021	-0.5	0.009	-0.4
Lab020	0.014	-0.3	0.0235	0.7	0.0109	-0.4	0.0205	4.2	0.028	0.3	0.0368	0.2	0.0101	0.5	NR	NR	0.0094	-0.2
Lab021	0.0115	-0.9	0.023	0.6	0.01	-0.7	0.01	0.0	0.0275	0.2	0.0265	-1.0	0.0074	-0.7	0.0225	-0.3	0.0097	-0.1
Lab022	0.01	-1.3	0.017	-0.6	0.011	-0.3	0.01	0.0	0.019	-1.1	0.035	0.0	0.009	0.0	0.021	-0.5	0.008	-0.8
Lab023	0.015	0.0	0.017	-0.6	0.012	0.0	0.007	-1.2	0.028	0.3	0.04	0.6	0.01	0.4	0.027	0.5	0.01	0.0
Lab024	0.01	-1.3	0.015	-1.0	0.012	0.0	0.006	-1.6	0.022	-0.6	0.02	-1.7	0.005	-1.8	0.021	-0.5	0.005	-2.0
Lab025	0.016	0.3	0.022	0.4	0.013	0.3	0.01	0.0	0.03	0.6	0.045	1.1	0.008	-0.4	0.024	0.0	0.014	1.6
Lab026	0.017	0.5	0.021	0.2	0.014	0.7	0.013	1.2	0.029	0.5	0.038	0.3	0.009	0.0	0.028	0.7	0.009	-0.4
Lab027	0.0135	-0.4	0.019	-0.2	0.0112	-0.3	0.0101	0.0	0.0242	-0.3	0.0362	0.1	0.009	0.0	0.0222	-0.3	0.0085	-0.6
Lab028	0.017	0.5	0.018	-0.4	0.014	0.7	0.01	0.0	0.024	-0.3	0.03	-0.6	0.01	0.4	0.034	1.7	0.01	0.0
Lab029	0.015	0.0	0.015	-1.0	0.0068	-1.7	NR	NR	0.016	-1.5	0.02	-1.7	0.006	-1.3	0.025	0.2	0.0077	-0.9
Lab030	0.017	0.5	0.0192	-0.2	0.0118	-0.1	NR	NR	0.0294	0.5	0.0329	-0.2	0.01	0.4	0.0217	-0.4	0.01	0.0
Lab031	0.0157	0.2	NA	NA	0.012	0.0	0.006	-1.6	0.022	-0.6	0.032	-0.3	0.0076	-0.6	0.0251	0.2	NA	NA
Lab032	0.016	0.3	0.021	0.2	0.014	0.7	0.0008	-0.8	0.029	0.5	0.04	0.6	0.009	0.0	0.026	0.3	0.009	-0.4
Lab033	0.018	0.8	0.023	0.6	0.014	0.7	0.012	0.8	0.029	0.5	0.043	0.9	0.01	0.4	0.027	0.5	0.011	0.4
Lab034	0.014	-0.3	0.015	-1.0	0.012	0.0	0.01	0.0	0.022	-0.6	0.032	-0.3	0.01	0.4	0.027	0.5	0.007	-1.2
Lab035	0.0133	-0.5	0.02565	1.1	0.01295	0.3	NR	NR	0.0213	-0.7	0.0361	0.1	0.0083	-0.3	0.02625	0.4	0.00825	-0.7
Lab036	0.0278	3.4	0.024	0.8	0.0384	>5	0.0217	4.7	0.0531	4.2	0.0771	4.8	0.0126	1.6	0.0477	4.0	0.017	2.8
Lab037	0.0159	0.2	0.0183	-0.3	0.0137	0.6	0.0064	-1.4	0.0252	-0.1	0.0405	0.6	0.0099	0.4	0.0301	1.0	0.009	-0.4
Lab038	0.017	0.5	0.018	-0.4	0.01	-0.7	0.007	-1.2	0.028	0.3	0.034	-0.1	0.009	0.0	0.018	-1.0	0.008	-0.8
Lab039	0.016	0.3	0.023	0.6	0.013	0.3	NR	NR	0.027	0.2	0.028	-0.8	0.009	0.0	0.025	0.2	0.011	0.4
Lab040	0.017	0.5	0.022	0.4	0.015	1.0	0.01	0.0	0.03	0.6	0.03	-0.6	0.007	-0.9	0.025	0.2	0.011	0.4
Lab041	0.014	-0.3	0.019	-0.2	0.012	0.0	0.0097	-0.1	0.025	-0.2	0.033	-0.2	0.0083	-0.3	0.026	0.3	0.0085	-0.6
Lab042	0.02187	1.8	NR	NR	0.01496	1.0	NR	NR	0.03161	0.9	NR	NR	0.01197	1.3	0.03714	2.2	0.01164	0.7

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

Lab Code	Boscalid	z score (FFP RSD 25 %)	Chlorpropham	z score (FFP RSD 25 %)	Chlorpyrifos	z score (FFP RSD 25 %)	Cypermethrin	z score (FFP RSD 25 %)	Cyprodinil	z score (FFP RSD 25 %)	Deltamethrin	z score (FFP RSD 25 %)	Dimethoate	z score (FFP RSD 25 %)	Etofenprox	z score (FFP RSD 25 %)	Fipronil	z score (FFP RSD 25 %)
MRRL	**0.01**		**0.01**		**0.01**		**0.003**		**0.01**		**0.01**		**0.003**		**0.01**		**0.004**	
Robust mean (mg/kg)	**0.015**		**0.020**		**0.012**		**0.010**		**0.026**		**0.035**		**0.009**		**0.024**		**0.010**	
Lab043	0.014	-0.3	0.02	0.0	0.012	0.0	0.007	-1.2	0.025	-0.2	0.034	-0.1	0.006	-1.3	0.021	-0.5	0.01	0.0
Lab044	0.017	0.5	0.019	-0.2	0.012	0.0	0.007	-1.2	0.022	-0.6	0.047	1.4	0.007	-0.9	NA	NA	0.008	-0.8
Lab045	NR	NR	NR	NR	0.0059	-2.0	0.01	0.0	NA	NA	0.028	-0.8	0.011	0.9	NA	NA	NA	NA
Lab046	0.02	1.3	0.026	1.2	0.015	1.0	0.018	3.2	0.031	0.8	0.054	2.2	0.0089	0.0	0.034	1.7	0.014	1.6
Lab047	0.017	0.5	0.025	1.0	0.017	1.7	0.011	0.4	0.031	0.8	0.052	1.9	0.011	0.9	0.027	0.5	0.014	1.6
Lab048	0.0142	-0.2	0.0196	-0.1	0.0116	-0.1	0.0083	-0.7	0.0264	0.1	0.0301	-0.6	0.0091	0.0	0.0226	-0.2	0.0099	0.0
Lab049	0.0136	-0.4	0.0203	0.1	0.0135	0.5	NR	NR	0.027	0.2	0.033	-0.2	0.0112	1.0	0.0257	0.3	0.0131	1.2
Lab050	0.012	-0.8	0.02	0.0	0.013	0.3	NA	NA	0.023	-0.5	0.029	-0.7	0.0064	-1.2	0.019	-0.8	0.0098	-0.1
Lab051	0.014	-0.3	0.0175	-0.5	0.01	-0.7	0.0084	-0.6	0.0245	-0.2	0.037	0.2	0.0086	-0.2	0.025	0.2	NA	NA
Lab052	0.016	0.3	0.0217	0.3	0.014	0.7	0.011	0.4	0.0307	0.7	0.0397	0.5	0.01	0.4	0.0297	1.0	0.0125	1.0
Lab053	0.015	0.0	0.02	0.0	0.014	0.7	0.009	-0.4	0.03	0.6	0.043	0.9	0.01	0.4	0.028	0.7	0.009	-0.4
Lab054	0.015	0.0	0.022	0.4	0.012	0.0	0.009	-0.4	0.029	0.5	0.035	0.0	0.0067	-1.0	0.021	-0.5	0.0094	-0.2
Lab055	0.013	-0.5	0.019	-0.2	0.013	0.3	0.01	0.0	0.022	-0.6	0.034	-0.1	0.0079	-0.5	0.02	-0.7	0.0066	-1.4
Lab056	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.032	-0.3	NR	NR	0.02	-0.7	0.012	0.8
Lab057	0.014	-0.3	0.02	0.0	0.01	-0.7	0.009	-0.4	0.032	0.9	0.031	-0.5	0.009	0.0	0.019	-0.8	0.008	-0.8

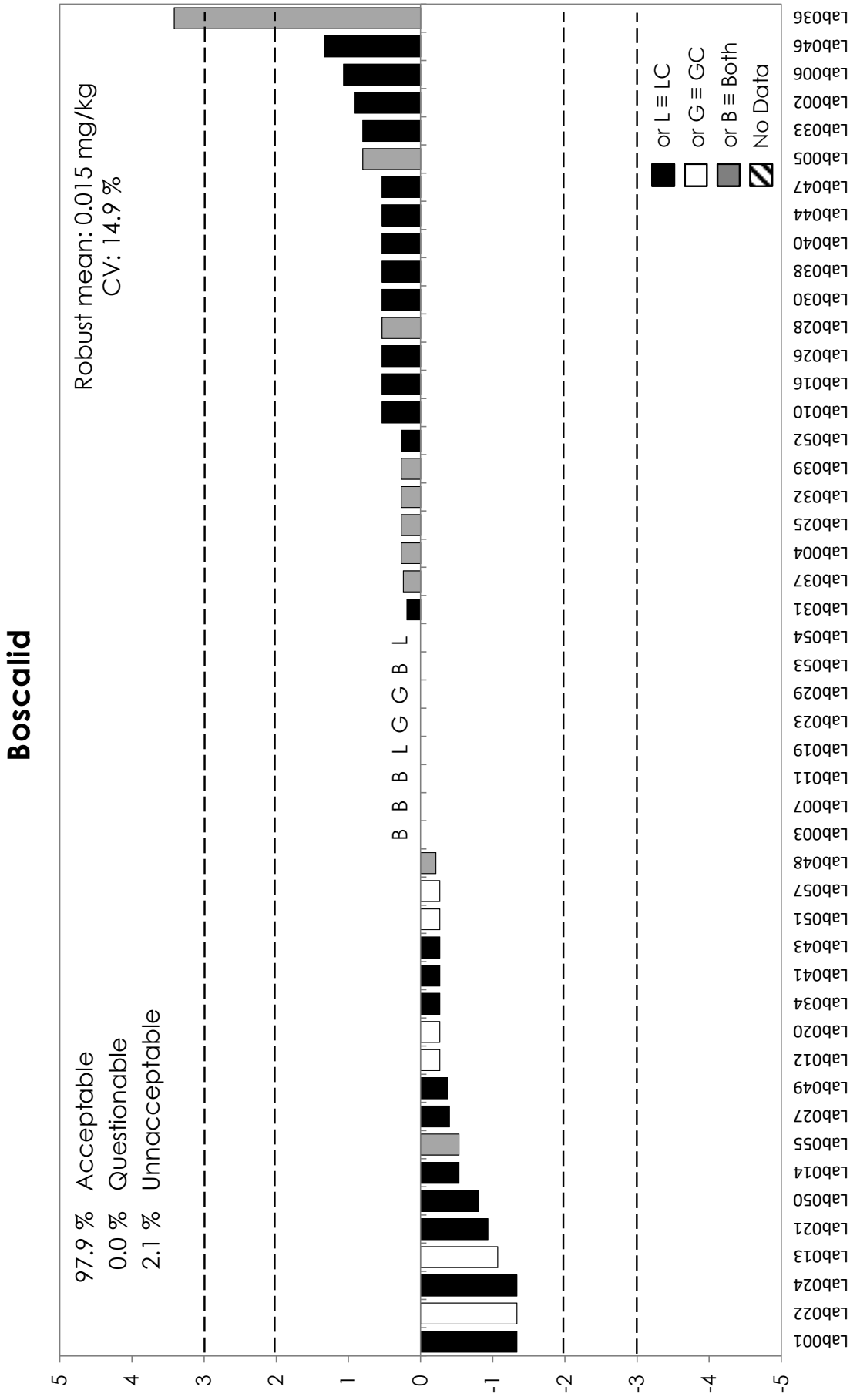
NA: Not analysed; NR: Not reported; ND: Not detected (False negative)

Lab Code	Heptachlor	z score (FFP RSD 25 %)	Hexachlorobenzene	z score (FFP RSD 25 %)	Imidacloprid	z score (FFP RSD 25 %)	Iprodione	z score (FFP RSD 25 %)	Malathion	z score (FFP RSD 25 %)	Omethoate	z score (FFP RSD 25 %)	Spinosad	z score (FFP RSD 25 %)	Tebuconazole	z score (FFP RSD 25 %)
MRRL	**0.003**		**0.003**		**0.01**		**0.003**		**0.01**		**0.003**		**0.01**		**0.01**	
Robust mean (mg/kg)	**0.008**		**0.009**		**0.013**		**0.037**		**0.030**		**0.014**		**0.012**		**0.025**	
Lab001	NA	NA	NA	NA	0.01	-0.9	NA	NA	NA	NA	0.01	-1.1	NA	NA	0.02	-0.8
Lab002	0.0096	0.8	0.0108	0.8	0.0163	1.0	NR	NR	0.0348	0.6	0.0109	-0.9	0.012	0.0	0.0336	1.4
Lab003	0.01	1.0	0.011	0.9	0.015	0.6	0.041	0.4	0.029	-0.1	0.014	0.0	0.01	-0.7	0.023	-0.3
Lab004	0.008	0.0	0.008	-0.4	0.011	-0.6	0.036	-0.1	0.03	0.0	0.011	-0.9	0.009	-1.0	0.026	0.2
Lab005	0.014	3.0	0.012	1.3	0.017	1.2	0.044	0.8	0.035	0.7	0.017	0.9	0.03	>5	0.029	0.6
Lab006	0.0085	0.3	0.0085	-0.2	NR	NR	0.093	>5	0.033	0.4	0.022	2.3	0.015	1.0	0.0264	0.2
Lab007	0.008	0.0	0.007	-0.9	0.011	-0.6	0.026	-1.2	0.024	-0.8	0.019	1.4	0.01	-0.7	0.027	0.3
Lab008	Participation cancelled															
Lab009	0.011	1.5	0.006	-1.3	0.009	-1.2	0.022	-1.6	0.029	-0.1	0.009	-1.4	0.011	-0.3	0.021	-0.6
Lab010	0.009	0.5	0.008	-0.4	0.017	1.2	0.039	0.2	0.029	-0.1	0.015	0.3	0.012	0.0	0.022	-0.5
Lab011	0.009	0.5	0.008	-0.4	0.014	0.3	0.041	0.4	0.033	0.4	0.015	0.3	0.012	0.0	0.025	0.0
Lab012	0.01	1.0	0.008	-0.4	0.019	1.8	0.039	0.2	0.03	0.0	0.024	2.9	0.019	2.3	0.037	1.9
Lab013	0.007	-0.5	0.007	-0.9	0.014	0.3	0.029	-0.9	0.025	-0.7	0.015	0.3	0.012	0.0	0.025	0.0
Lab014	0.0091	0.6	0.0089	0.0	0.012	-0.3	0.035	-0.2	0.032	0.3	0.016	0.6	0.012	0.0	0.023	-0.3
Lab015	NR	NR	NR	NR	0.043	>5	NR	NR	NA	NA	ND	ND	NR	NR	0.035	1.6
Lab016	0.005	-1.5	0.006	-1.3	0.014	0.3	0.032	-0.5	0.02	-1.3	0.015	0.3	0.009	-1.0	0.029	0.6
Lab017	0.009	0.5	0.009	0.0	NR	NR	0.034	-0.3	0.026	-0.5	0.014	0.0	0.013	0.3	0.017	-1.3
Lab018	0.012	2.0	0.009	0.0	NA	NA	0.037	0.0	NA	NA	NA	NA	NA	NA	0.025	0.0
Lab019	0.006	-1.0	0.005	-1.8	0.015	0.6	0.039	0.2	0.027	-0.4	0.016	0.6	0.013	0.3	0.022	-0.5
Lab020	0.0095	0.8	0.0094	0.2	0.0139	0.3	0.0364	-0.1	0.0284	-0.2	0.0156	0.2	0.0109	-0.4	0.0201	-0.8

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

Lab Code	Heptachlor	z score (FFP RSD 25 %)	Hexachlorobenzene	z score (FFP RSD 25 %)	Imidacloprid	z score (FFP RSD 25 %)	Iprodione	z score (FFP RSD 25 %)	Malathion	z score (FFP RSD 25 %)	Omethoate	z score (FFP RSD 25 %)	Spinosad	z score (FFP RSD 25 %)	Tebuconazole	z score (FFP RSD 25 %)
MRRL	**0.003**		**0.003**		**0.01**		**0.003**		**0.01**		**0.003**		**0.01**		**0.01**	
Robust mean (mg/kg)	**0.008**		**0.009**		**0.013**		**0.037**		**0.030**		**0.014**		**0.012**		**0.025**	
Lab021	0.0071	-0.5	0.01	0.4	0.01	-0.9	0.0275	-1.0	0.0295	-0.1	0.016	0.3	0.01	-0.7	0.0225	-0.4
Lab022	0.008	0.0	0.008	-0.4	0.015	0.6	0.03	-0.8	0.021	-1.2	0.012	-0.8	0.012	0.0	0.022	-0.5
Lab023	0.01	1.0	0.009	0.0	0.012	-0.3	0.038	0.1	0.032	0.3	0.012	-0.8	0.011	-0.3	0.027	0.3
Lab024	0.007	-0.5	0.009	0.0	0.013	0.0	0.029	-0.9	0.02	-1.3	0.01	-1.3	0.011	-0.3	0.025	0.0
Lab025	0.01	1.0	0.009	0.0	0.009	-1.2	0.035	-0.2	0.03	0.0	0.017	0.5	0.01	-0.7	0.023	-0.3
Lab026	0.008	0.0	0.008	-0.4	0.014	0.3	0.041	0.4	0.031	0.1	0.017	0.5	0.014	0.7	0.028	0.5
Lab027	0.008	0.0	0.008	-0.4	0.0133	0.1	0.0288	-0.9	0.0275	-0.3	0.013	-0.5	0.0108	-0.4	0.0243	-0.1
Lab028	0.008	0.0	0.007	-0.9	0.016	0.9	0.045	0.9	0.031	0.1	0.018	0.8	0.01	-0.7	0.027	0.3
Lab029	0.0041	-2.0	0.009	0.0	NA	NA	0.035	-0.2	0.017	-1.7	0.0032	-3.1	NA	NA	0.022	-0.5
Lab030	0.0078	-0.1	0.0076	-0.6	0.016	0.9	0.0363	-0.1	0.0322	0.3	0.014	-0.3	0.012	0.0	0.028	0.5
Lab031	0.004	-2.0	0.007	-0.9	0.0106	-0.7	0.036	-0.1	NR	ND	0.0091	-1.6	0.0135	0.5	0.0269	0.3
Lab032	0.009	0.5	0.009	0.0	0.016	0.9	0.04	0.3	0.032	0.3	0.018	0.8	0.012	0.0	0.025	0.0
Lab033	0.0091	0.6	0.0087	-0.1	0.012	-0.3	0.042	0.5	0.03	0.0	0.017	0.5	0.011	-0.3	0.028	0.5
Lab034	0.012	2.0	0.009	0.0	0.01	-0.9	0.026	-1.2	0.027	-0.4	0.01	-1.3	0.013	0.3	0.024	-0.2
Lab035	0.00915	0.6	NA	NA	0.01275	-0.1	0.0457	0.9	0.02785	-0.3	0.01175	-0.9	0.011475	-0.2	0.02555	0.1
Lab036	NR	NR	0.021	>5	0.0235	3.2	0.045	0.9	0.0588	3.8	0.0238	2.3	0.024	4.0	0.0404	2.5
Lab037	0.0088	0.4	0.0086	-0.2	0.0158	0.9	0.0361	-0.1	0.0385	1.1	0.0132	-0.2	0.0151	1.0	0.0268	0.3
Lab038	0.007	-0.5	0.008	-0.4	0.011	-0.6	0.036	-0.1	0.03	0.0	0.015	0.0	0.011	-0.3	0.025	0.0
Lab039	0.007	-0.5	0.007	-0.9	0.012	-0.3	0.04	0.3	0.031	0.1	0.011	-1.1	0.011	-0.3	0.025	0.0
Lab040	0.011	1.5	0.011	0.9	0.014	0.3	NR	NR	0.029	-0.1	0.019	1.1	0.029	>5	0.026	0.2
Lab041	0.0067	-0.7	0.0072	-0.8	0.014	0.3	0.034	-0.3	0.029	-0.1	0.014	-0.3	0.012	0.0	0.024	-0.2
Lab042	0.007295	-0.4	0.005954	-1.4	0.01608	0.9	NR	NR	0.03467	0.6	0.01367	-0.4	NA	NA	0.03386	1.4
Lab043	0.01	1.0	0.01	0.4	0.011	-0.6	0.039	0.2	0.032	0.3	0.01	-1.3	0.011	-0.3	0.022	-0.5
Lab044	NA	NA	NA	NA	0.011	-0.6	NA	NA	0.034	0.5	0.01	-1.3	0.011	-0.3	0.022	-0.5
Lab045	0.0056	-1.2	0.0051	-1.7	NR	NR	0.029	-0.9	0.024	-0.8	ND	ND	NR	NR	NR	NR
Lab046	0.01	1.0	0.015	2.7	0.015	0.6	0.065	3.0	0.034	0.5	0.014	-0.3	0.019	2.3	0.026	0.2
Lab047	0.01	1.0	0.008	-0.4	0.01	-0.9	0.051	1.5	0.037	0.9	0.014	-0.3	0.016	1.3	0.026	0.2
Lab048	0.0075	-0.3	0.0077	-0.6	0.0133	0.1	0.0368	0.0	0.0307	0.1	0.0143	0.1	0.011	-0.3	0.0262	0.2
Lab049	0.0091	0.6	0.0082	-0.4	0.0114	-0.5	0.034	-0.3	0.0285	-0.2	0.0204	1.4	0.013	0.3	0.0218	-0.5
Lab050	0.0062	-0.9	0.0074	-0.7	0.01	-0.9	0.031	-0.6	0.025	-0.7	0.008	-1.9	0.012	0.0	0.023	-0.3
Lab051	NA	NA	NA	NA	NA	NA	0.037	0.0	0.026	-0.5	ND	ND	NA	NA	0.025	0.0
Lab052	0.0097	0.9	0.0123	1.5	0.0137	0.2	0.0453	0.9	0.0339	0.5	0.0193	1.1	0.0162	1.4	0.026	0.2
Lab053	0.01	1.0	0.009	0.0	0.014	0.3	0.041	0.4	0.033	0.4	0.014	0.0	0.012	0.0	0.025	0.0
Lab054	0.0094	0.7	0.0099	0.4	0.013	0.0	NA	NA	NR	ND*	0.014	-0.3	0.01	-0.7	0.024	-0.2
Lab055	0.0064	-0.8	0.0071	-0.8	0.012	-0.3	0.031	-0.6	0.025	-0.7	0.013	-0.5	0.011	-0.3	0.02	-0.8
Lab056	NA	NA	NA	NA	0.016	0.9	NA	NA	0.021	-1.2	ND	ND	0.011	-0.3	NA	NA
Lab057	0.007	-0.5	0.007	-0.9	0.012	-0.3	0.035	-0.2	0.032	0.3	0.014	-0.3	0.014	0.7	0.022	-0.5

NA: Not analysed; NR: Not reported; ND: Not detected (False negative)


Boscalid
Robust mean: 0.015 mg/kg
CV: 14.9 %
97.9 % Acceptable
0.0 % Questionable
2.1 % Unnacceptable
or L = LC
or G = GC
or B = Both
No Data
B B B L G G B L
5
4
3
2
1
0
-1
-2
-3
-4
-5
lab001
lab022
lab024
lab013
lab021
lab050
lab014
lab055
lab027
lab049
lab012
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lab034
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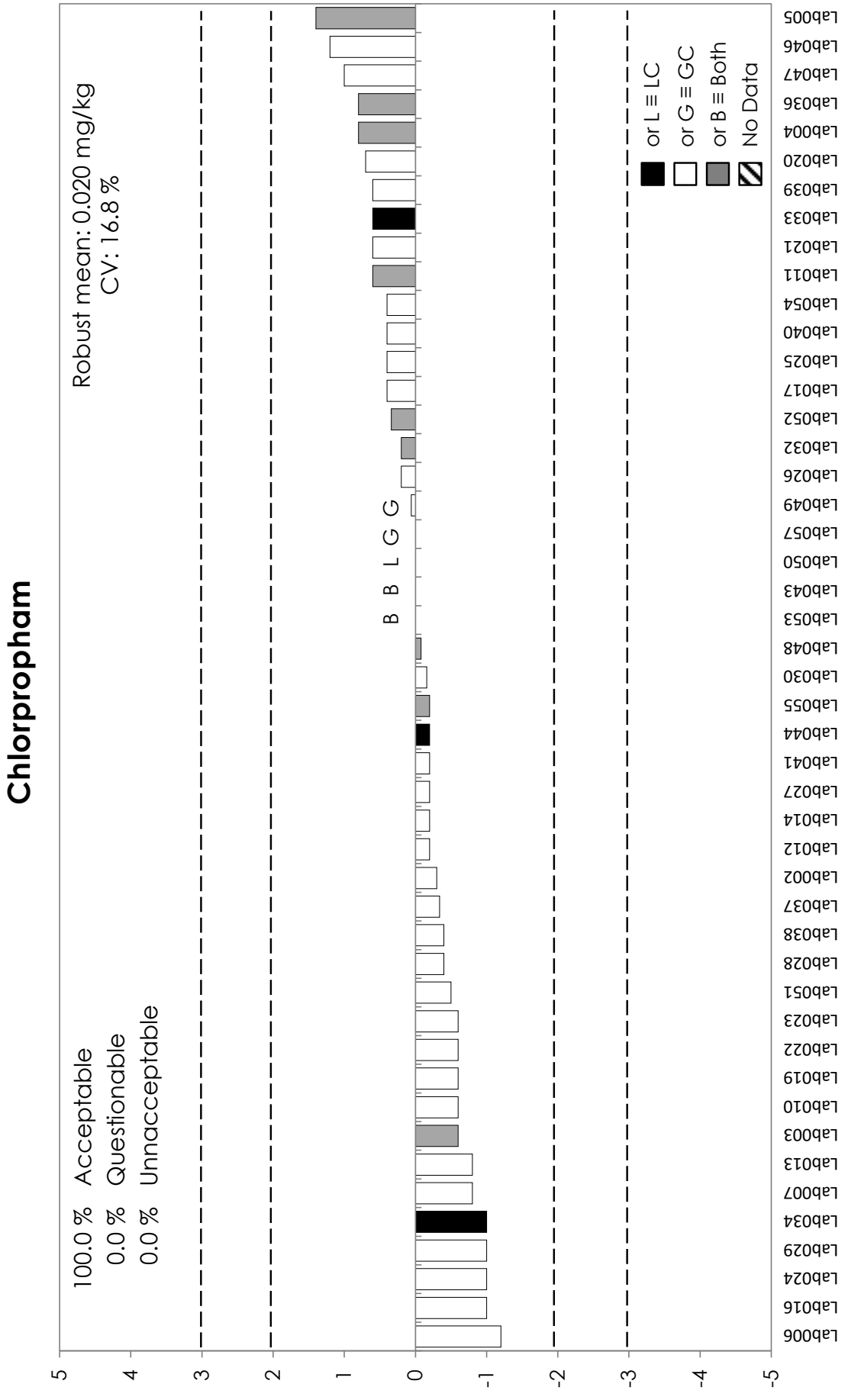
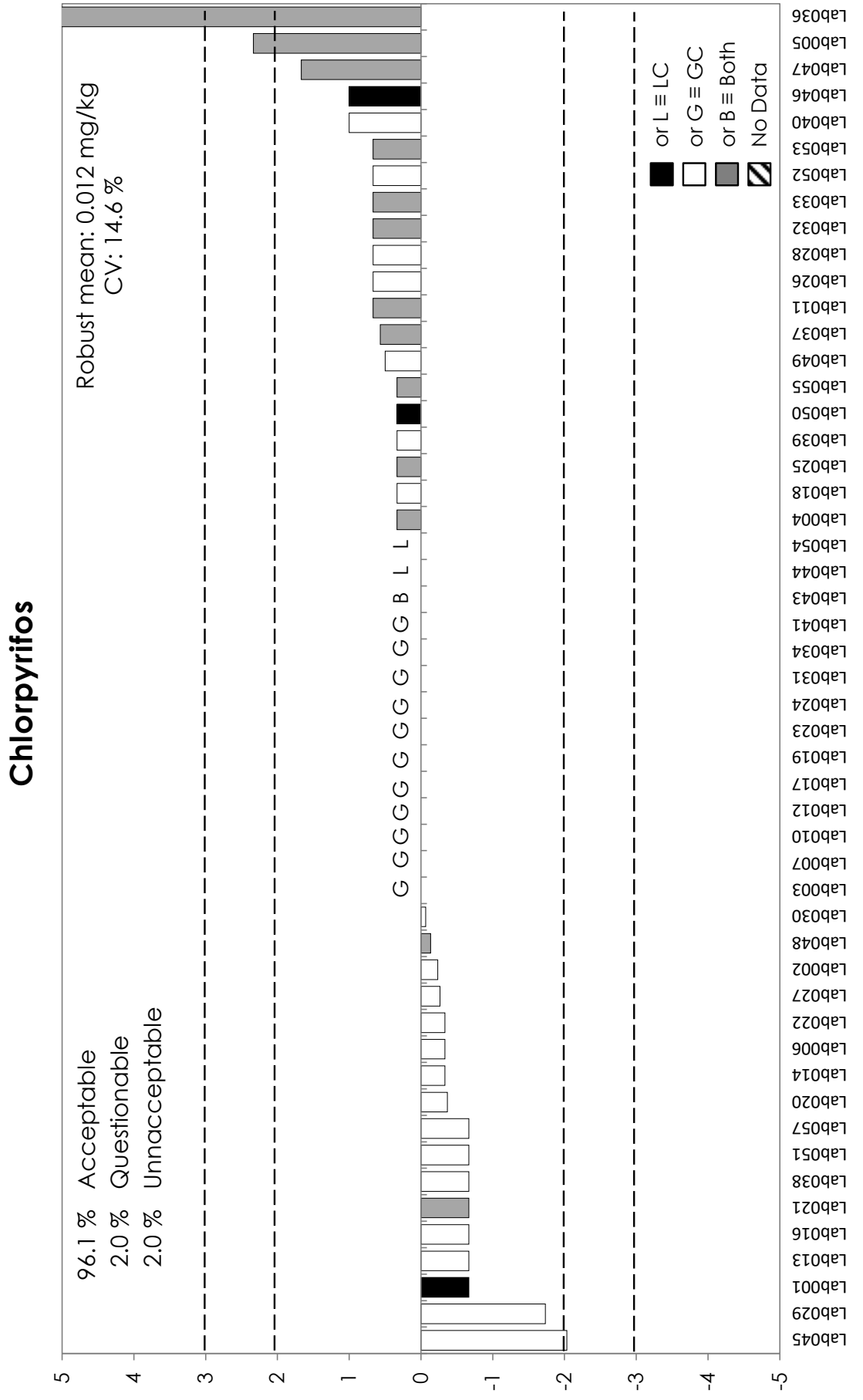
lab036

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




Chlorpyrifos
Robust mean: 0.012 mg/kg
CV: 14.6 %
96.1 % Acceptable
2.0 % Questionable
2.0 % Unacceptable
G G G G G G G G G B L L
or L = LC
or G = GC
or B = Both
No Data
5
4
3
2
1
0
-1
-2
-3
-4
-5
lab045
lab029
lab001
lab013
lab016
lab021
lab038
lab051
lab057
lab020
lab014
lab006
lab022
lab027
lab002
lab048
lab030
lab003
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lab036

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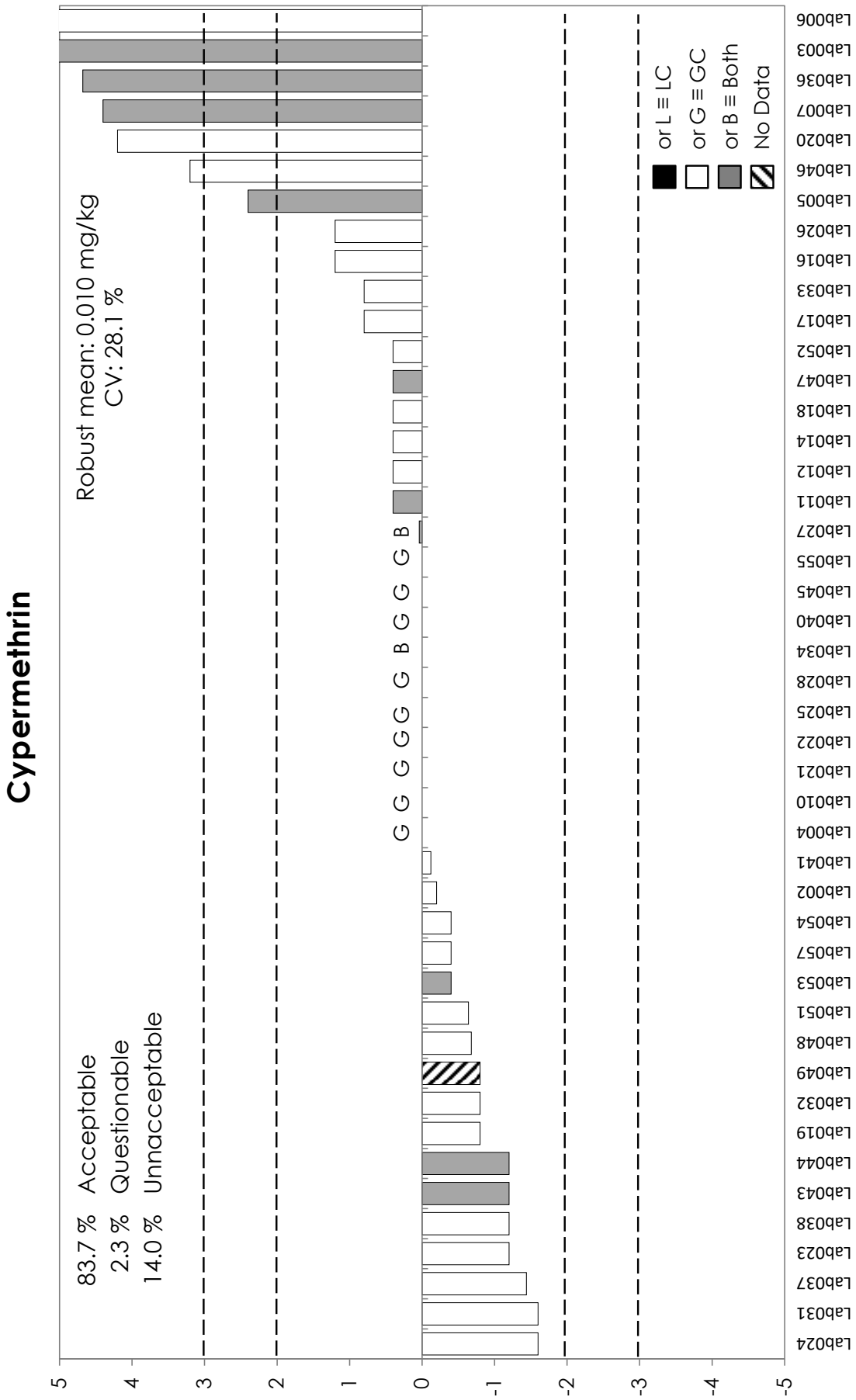
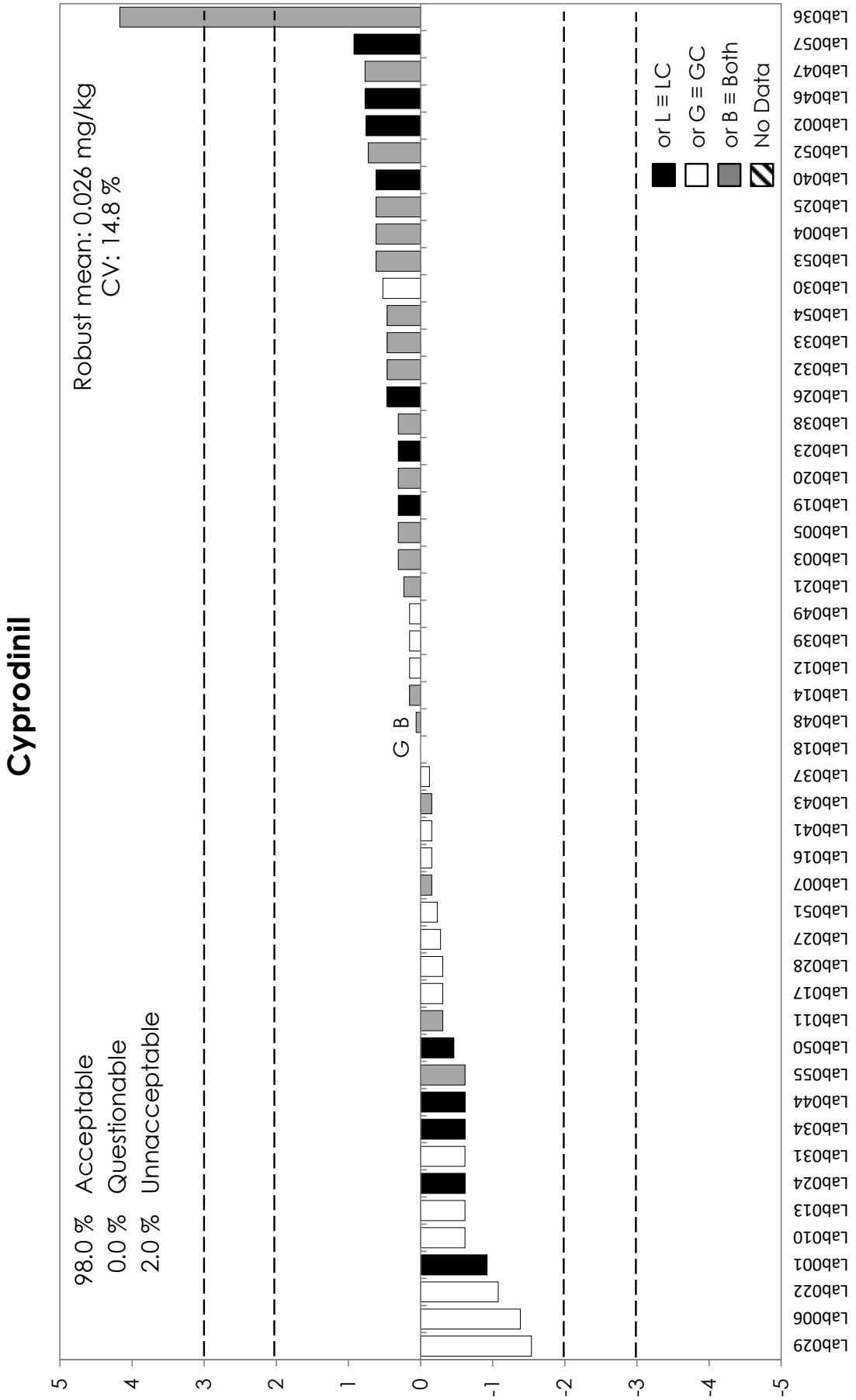
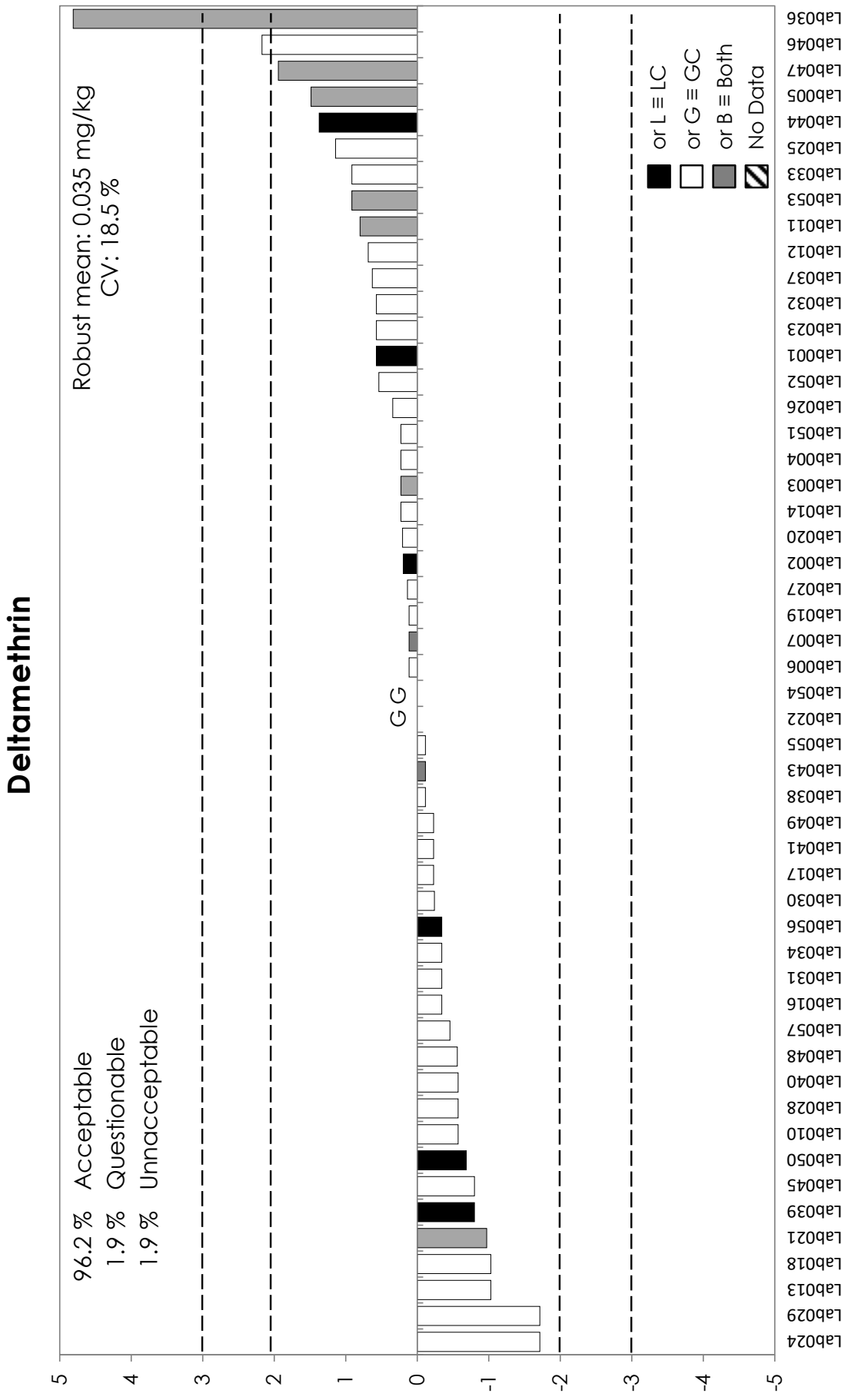
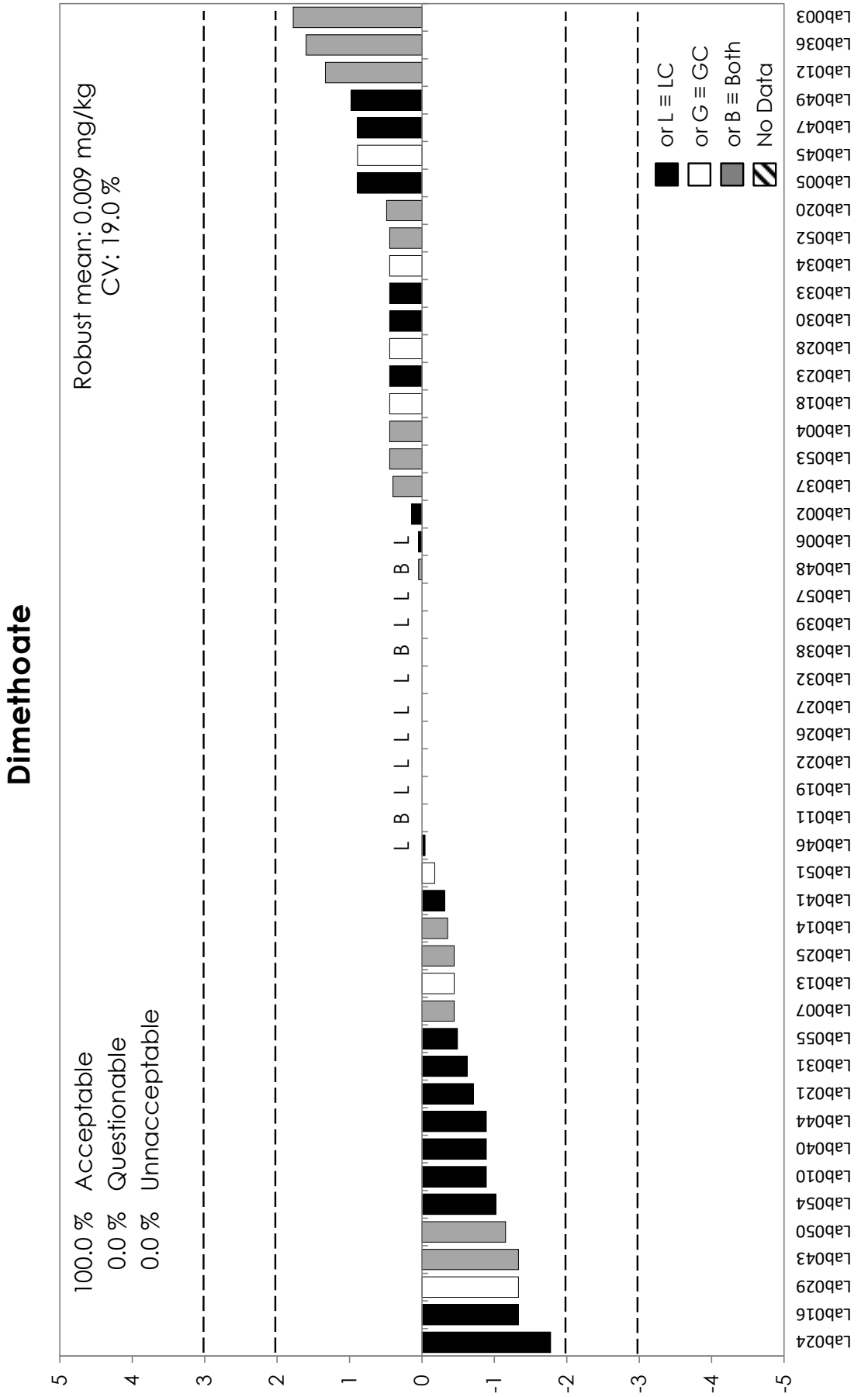


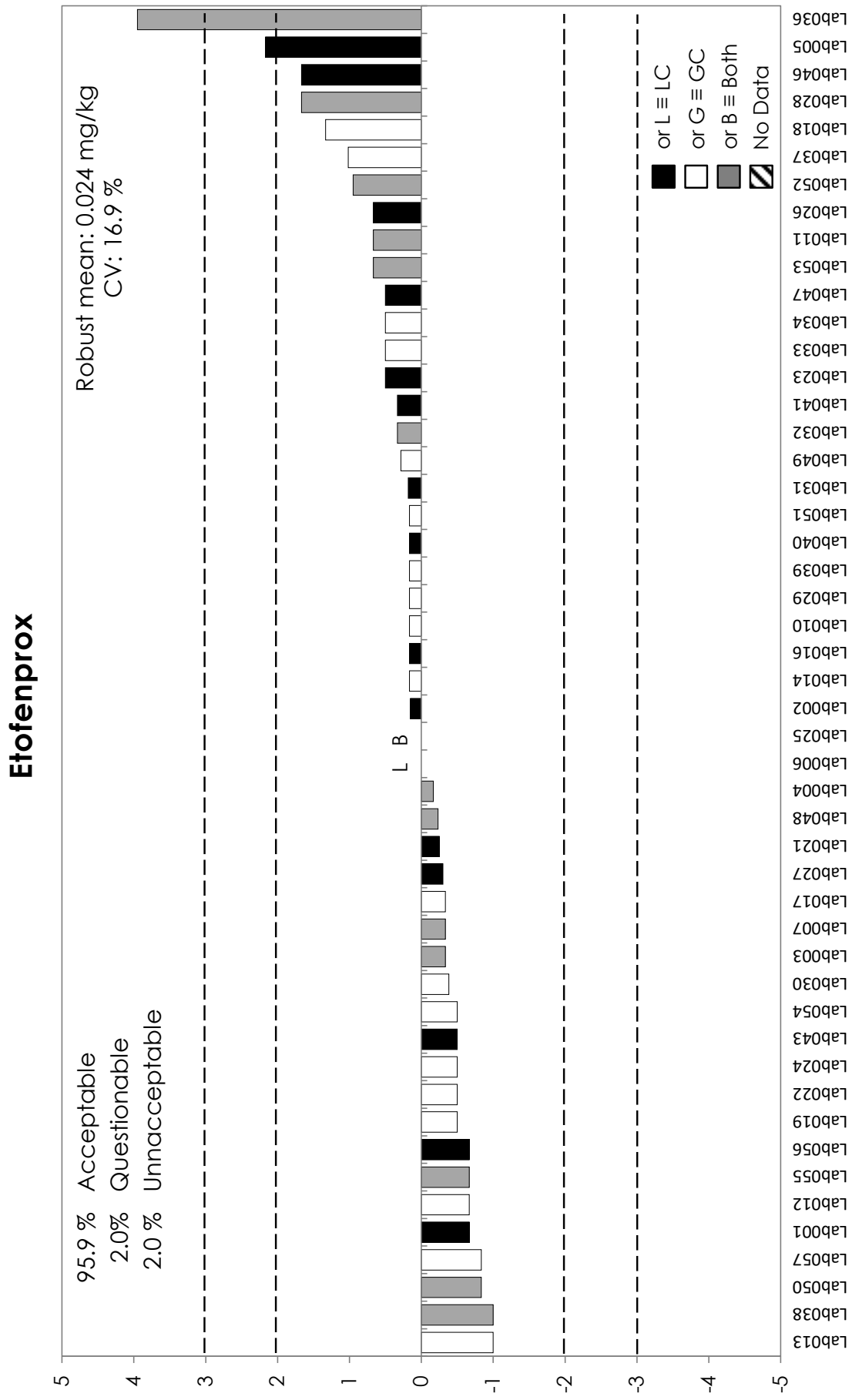

Cyprodinil
Robust mean: 0.026 mg/kg
CV: 14.8 %
98.0 % Acceptable
0.0 % Questionable
2.0 % Unnacceptable
or L = LC
or G = GC
or B = Both
No Data
G B
5
4
3
2
1
0
-1
-2
-3
-4
-5
lab029
lab006
lab022
lab001
lab010
lab013
lab024
lab031
lab034
lab044
lab055
lab050
lab011
lab017
lab028
lab027
lab051
lab007
lab016
lab041
lab043
lab037
lab018
lab048
lab014
lab012
lab039
lab049
lab021
lab003
lab005
lab019
lab020
lab023
lab038
lab026
lab032
lab033
lab054
lab030
lab053
lab004
lab025
lab040
lab052
lab002
lab046
lab047
lab057
lab036

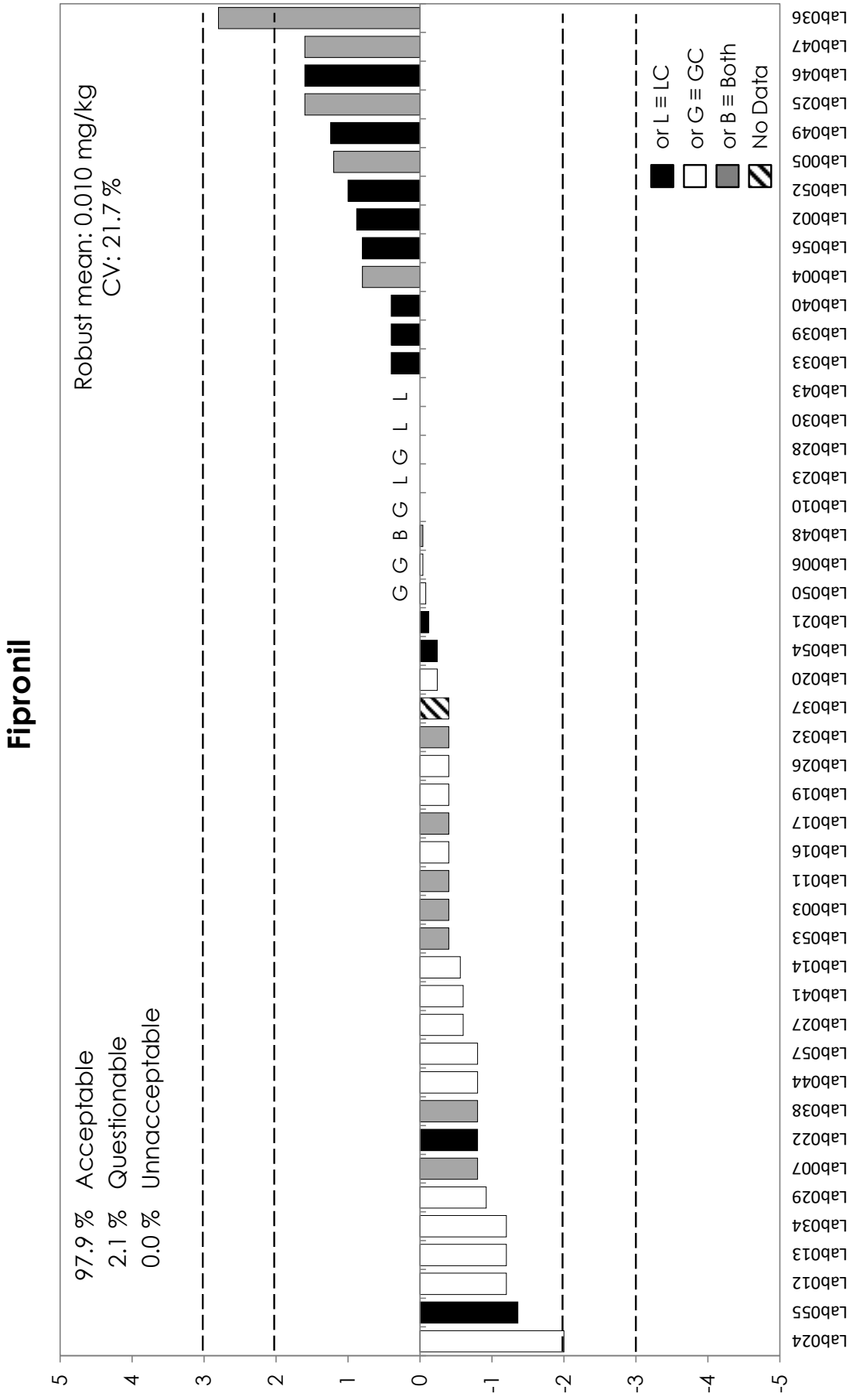
APPENDIX 4. Graphical representation of z scores for FFP RSD (25 %).




Dimethoate
Robust mean: 0.009 mg/kg
CV: 19.0 %
100.0 % Acceptable
0.0 % Questionable
0.0 % Unnacceptable
or L = LC
or G = GC
or B = Both
No Data
L B L L L L L B L L B L L
5
4
3
2
1
0
-1
-2
-3
-4
-5
lab024
lab016
lab029
lab043
lab050
lab054
lab010
lab040
lab044
lab021
lab031
lab055
lab007
lab013
lab025
lab014
lab041
lab051
lab046
lab011
lab019
lab022
lab026
lab027
lab032
lab038
lab039
lab057
lab048
lab006
lab002
lab037
lab053
lab004
lab018
lab023
lab028
lab030
lab033
lab034
lab052
lab020
lab005
lab045
lab047
lab049
lab012
lab036
lab003

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




Fipronil
Robust mean: 0.010 mg/kg
CV: 21.7 %
97.9 % Acceptable
2.1 % Questionable
0.0 % Unnacceptable
or L ≡ LC
or G ≡ GC
or B ≡ Both
No Data
G G B G L G L L L
5
4
3
2
1
0
-1
-2
-3
-4
-5
lab024
lab055
lab012
lab013
lab034
lab029
lab007
lab022
lab038
lab044
lab057
lab027
lab041
lab014
lab053
lab003
lab011
lab016
lab017
lab019
lab026
lab032
lab037
lab020
lab054
lab021
lab050
lab006
lab048
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lab047
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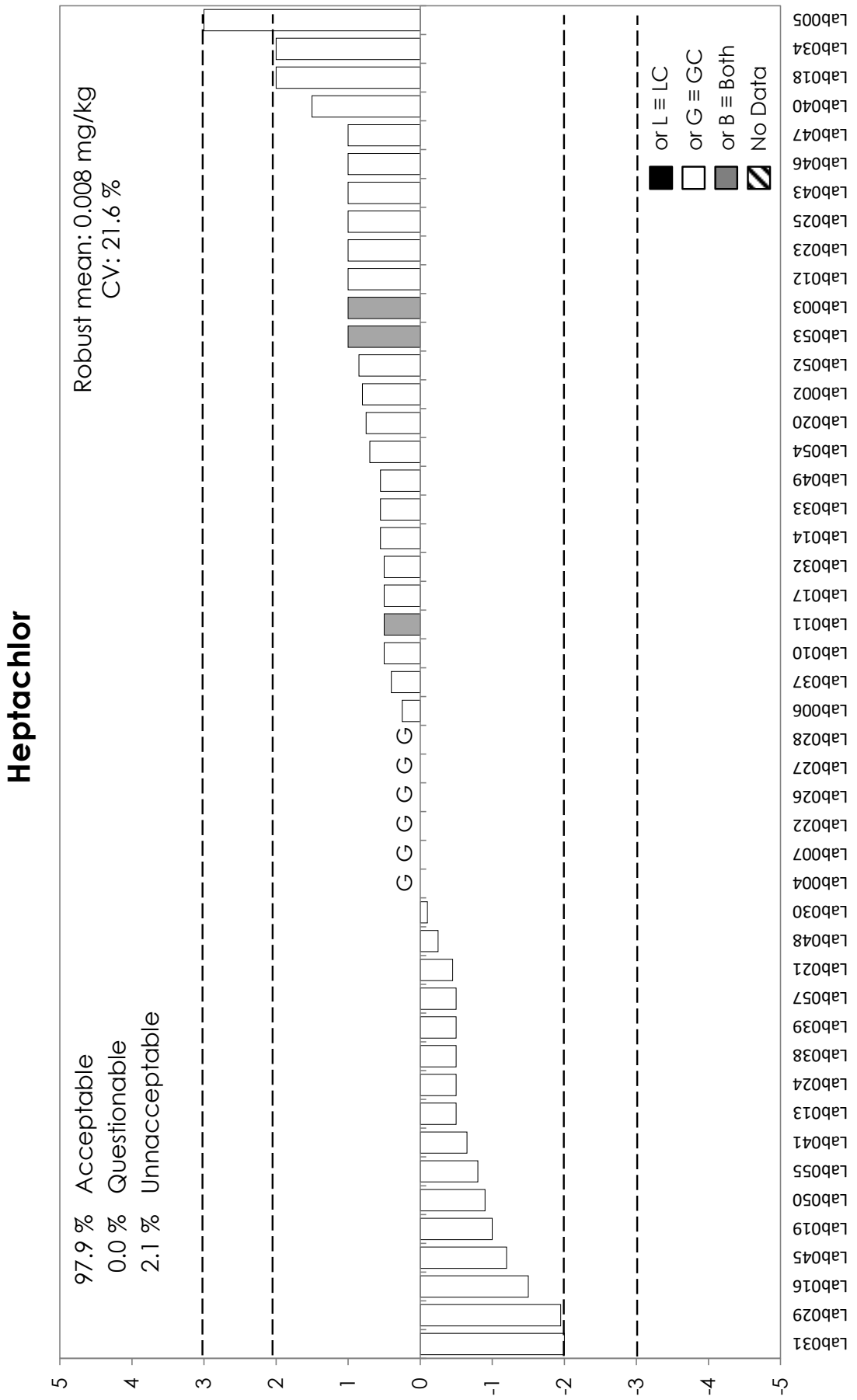
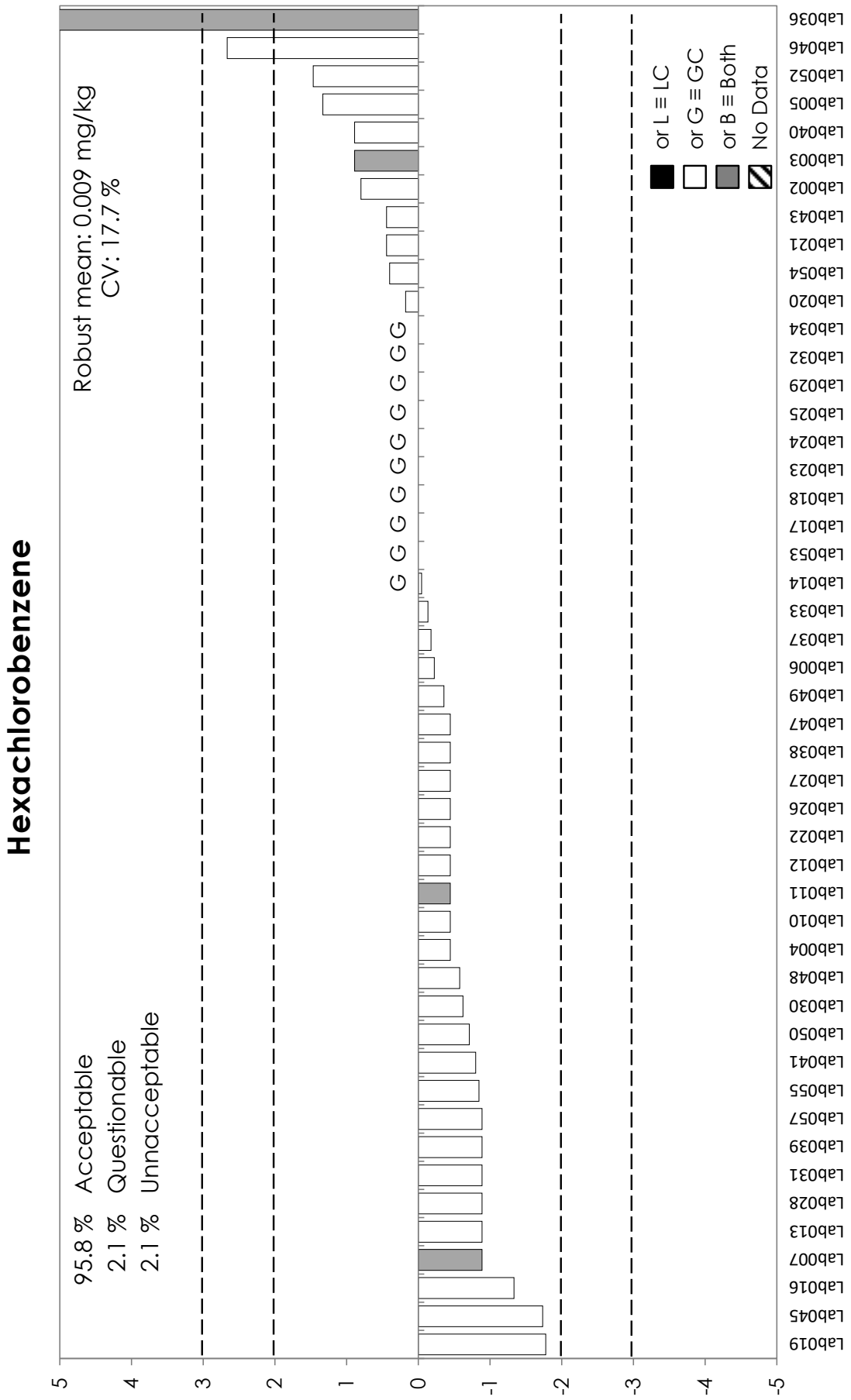
APPENDIX 4. Graphical representation of z scores for FFP RSD (25 %).




Hexachlorobenzene
Robust mean: 0.009 mg/kg
CV: 17.7 %
95.8 % Acceptable
2.1 % Questionable
2.1 % Unnacceptable
or L = LC
or G = GC
or B = Both
No Data
G G G G G G G G G G G
5
4
3
2
1
0
-1
-2
-3
-4
-5
lab019
lab045
lab016
lab007
lab013
lab028
lab031
lab039
lab057
lab055
lab041
lab050
lab030
lab048
lab004
lab010
lab011
lab012
lab022
lab026
lab027
lab038
lab047
lab049
lab006
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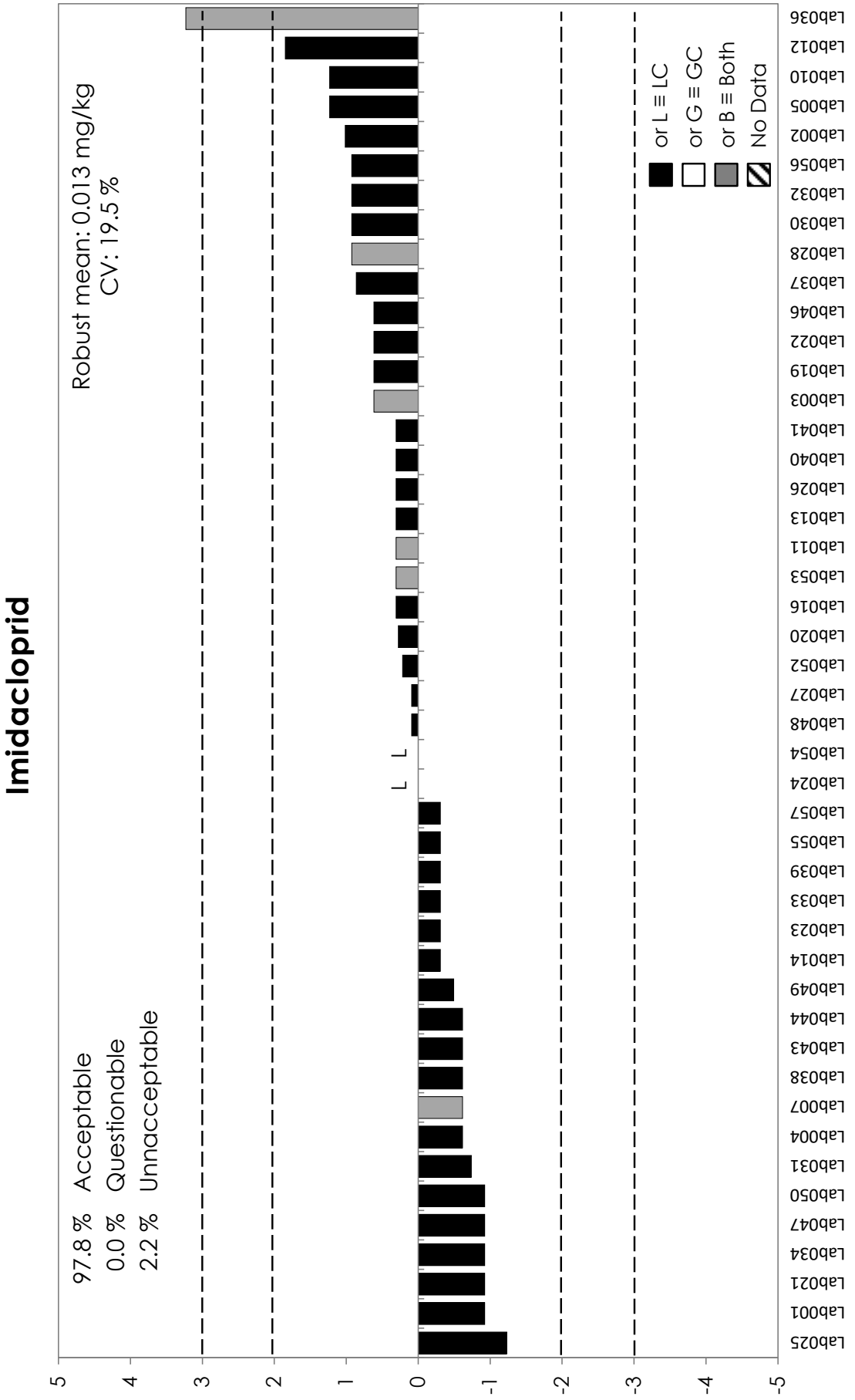

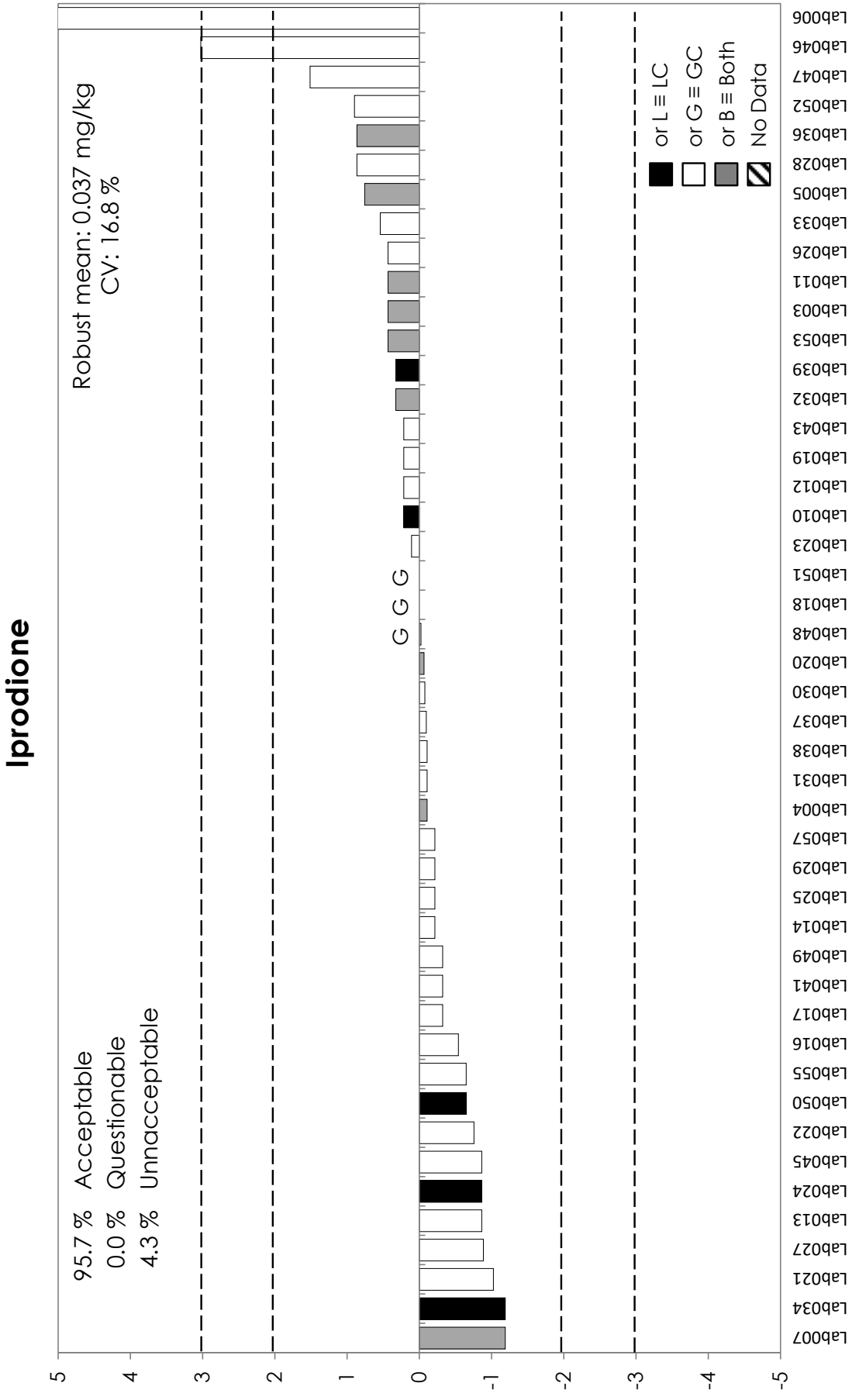
Iprodione
Robust mean: 0.037 mg/kg
CV: 16.8 %
95.7 % Acceptable
0.0 % Questionable
4.3 % Unacceptable
or L ≡ LC
or G ≡ GC
or B ≡ Both
No Data
G G G
5
4
3
2
1
0
-1
-2
-3
-4
-5
lab007
lab034
lab021
lab027
lab013
lab024
lab045
lab022
lab050
lab055
lab016
lab017
lab041
lab049
lab014
lab025
lab029
lab057
lab004
lab031
lab038
lab037
lab030
lab020
lab048
lab018
lab051
lab023
lab010
lab012
lab019
lab043
lab032
lab039
lab053
lab003
lab011
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lab005
lab028
lab036
lab052
lab047
lab046
lab006

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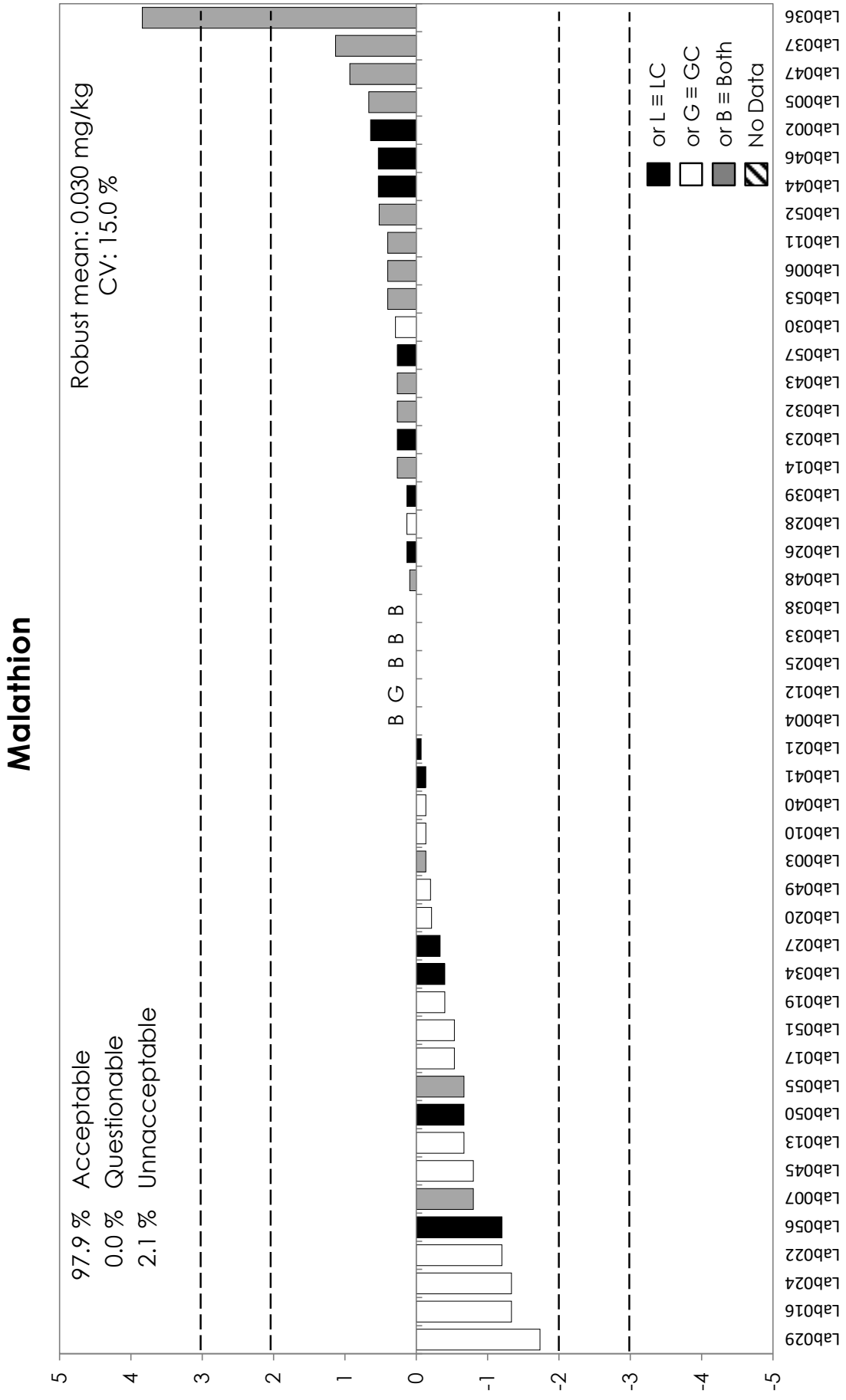

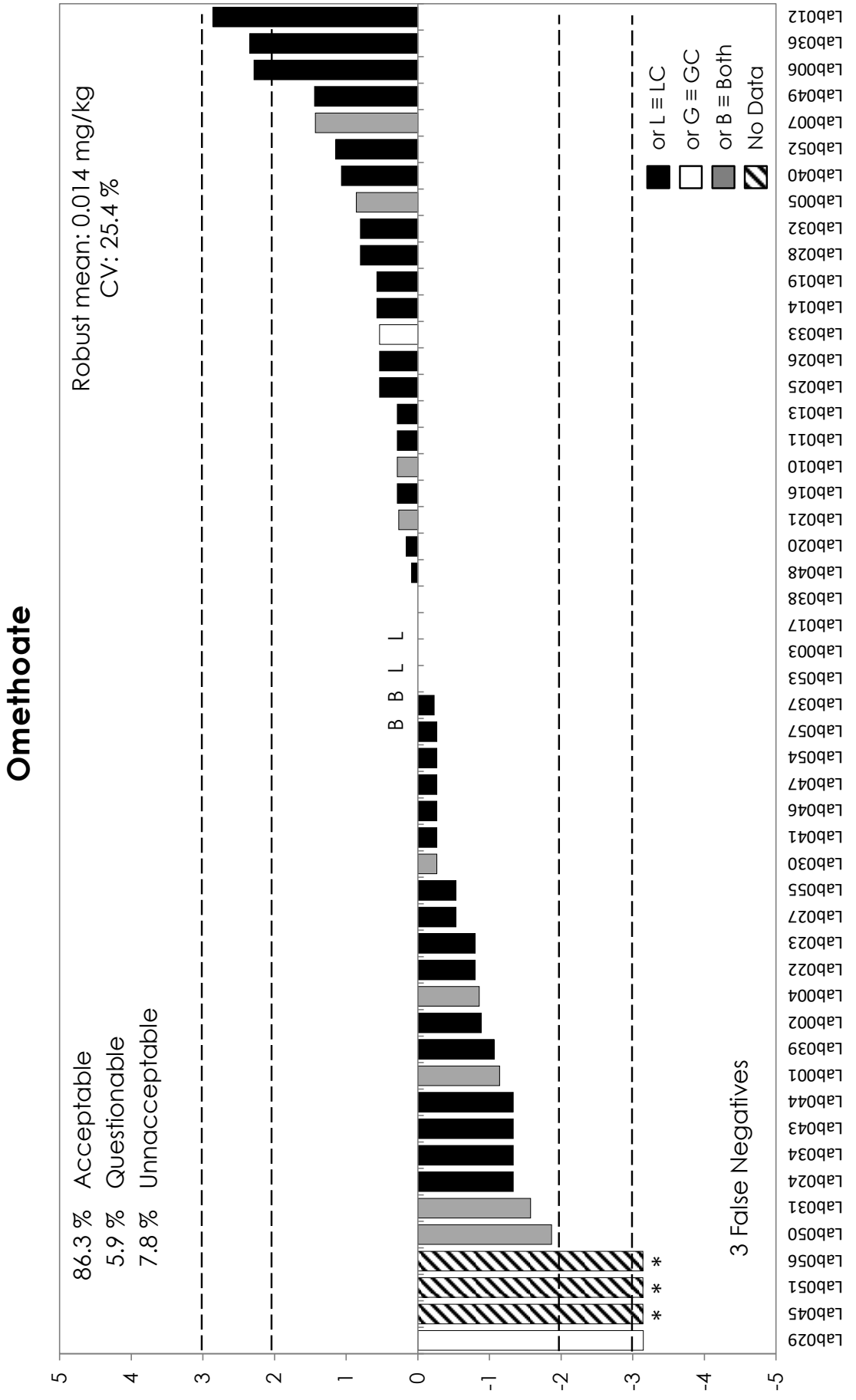
Omethoate
Robust mean: 0.014 mg/kg
CV: 25.4 %
86.3 % Acceptable
5.9 % Questionable
7.8 % Unnacceptable
or L ≡ LC
or G ≡ GC
or B ≡ Both
No Data
3 False Negatives
B B L L
5
4
3
2
1
0
-1
-2
-3
-4
-5
lab029
lab045
lab051
lab056
lab050
lab031
lab024
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lab043
lab044
lab001
lab039
lab002
lab004
lab022
lab023
lab027
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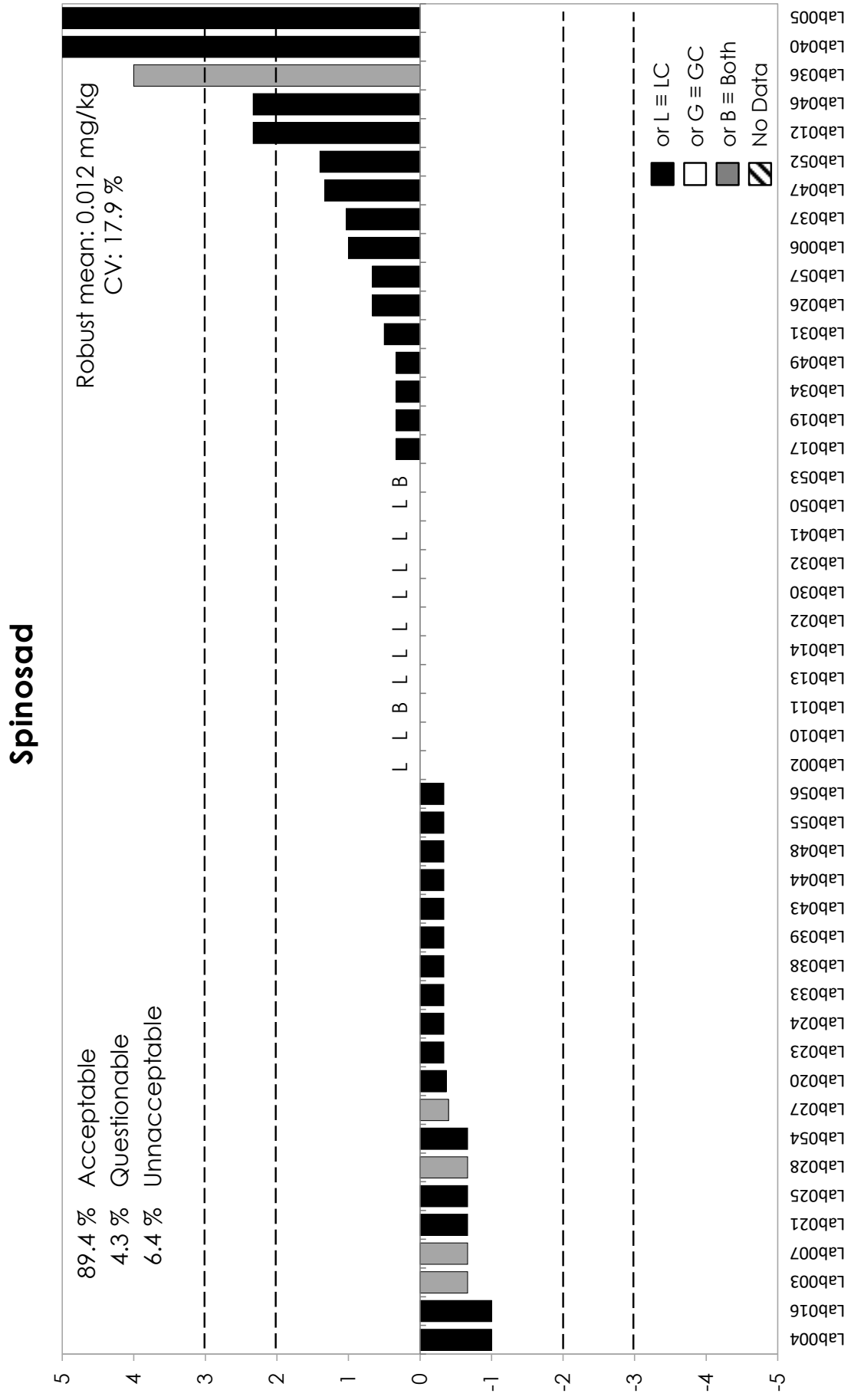

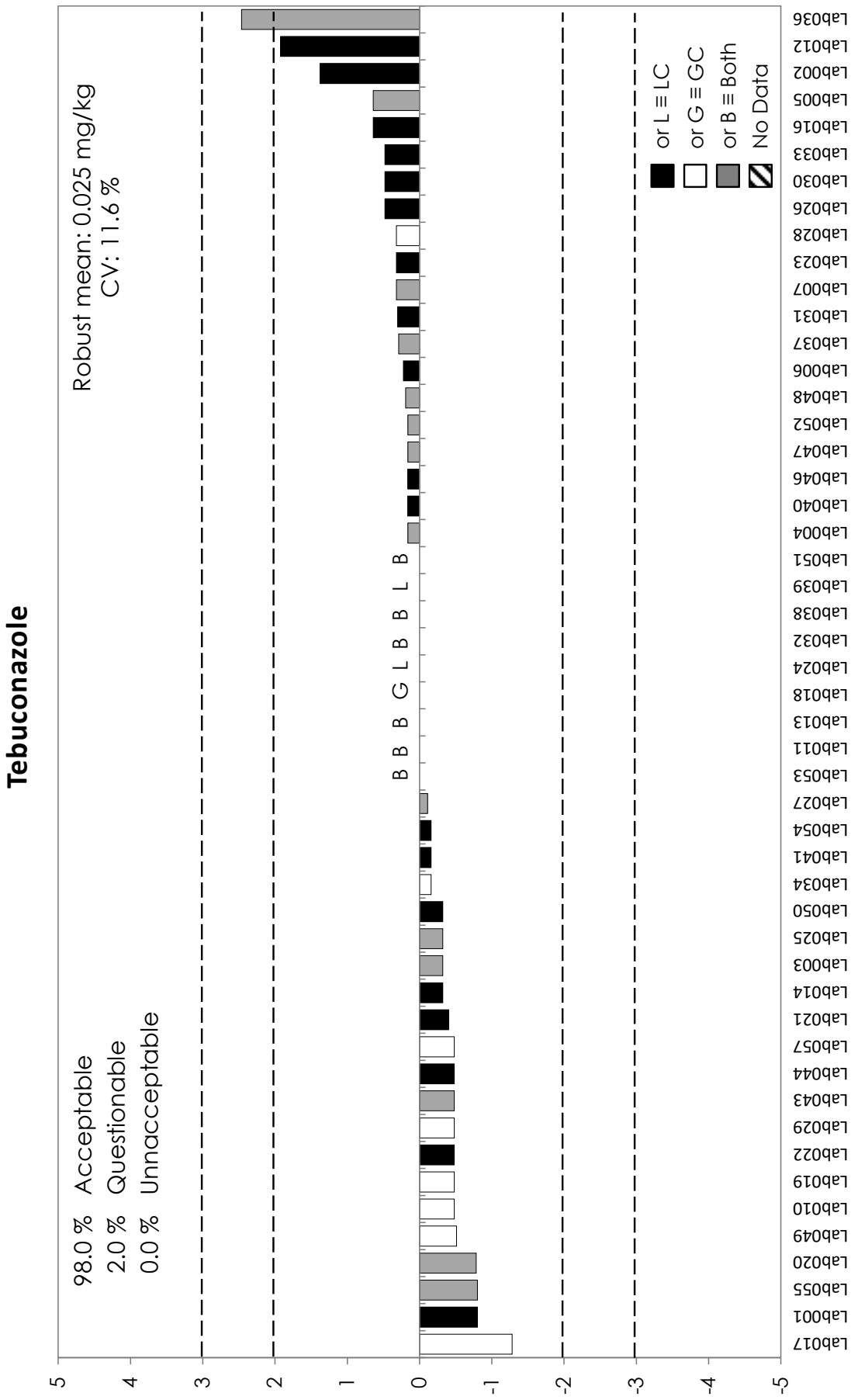
Tebuconazole
Robust mean: 0.025 mg/kg
CV: 11.6 %
98.0 % Acceptable
2.0 % Questionable
0.0 % Unnacceptable
or L ≡ LC
or G ≡ GC
or B ≡ Both
No Data
B B B G L B B L B
5
4
3
2
1
0
-1
-2
-3
-4
-5
lab017
lab001
lab055
lab020
lab049
lab010
lab019
lab022
lab029
lab043
lab044
lab057
lab021
lab014
lab003
lab025
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lab011
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6th Edition
Revised: February 2016

GENERAL PROTOCOL
for EU Proficiency Tests for 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[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-Organizers and Scientific Committee

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

An **Organising Team** 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, production of Test Item and Blank Material, the undertaking of homogeneity and

[5] DG-SANTÉ = 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 (EC) No 882/2004 of the European Parliament and of the Council on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules. Published at OJ of the EU L191 of 28.05.2004
[8] European Commission Proficiency Tests for Pesticide Residues in Fruits and Vegetables, Trends in Analytical Chemistry, 2010, 29 (1), 70 – 83.

stability tests, packing and shipment of the Test Item and Blank Material, handling and evaluation of the results and method information submitted by the participants and the drafting of the preliminary and final reports.

To complement the internal expertise of the EURLs, a group of external consultants that form 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, the affiliation of each member is shown on the EURL-Website. The members of the EUPT-SC will also be 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

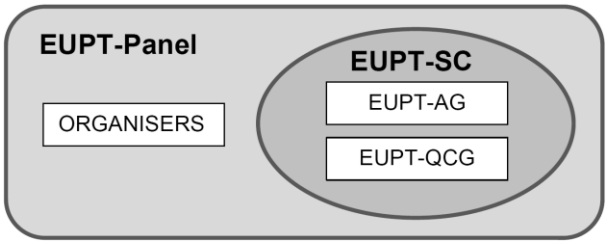
b) An **Advisory Group** (EUPT-AG).

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 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 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 consult with the EURLs in their decision making. Upcoming EUPTs are also planned during these meetings.

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



The decisions of the EUPT-Panel will be documented.

This present EUPT General Protocol was jointly drafted by the EUPT-SC and the EURLs and was approved by DG-SANTE.

[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 1. Protocols and Target list of pesticides to be sought.

EUPT Participants

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

- Art. 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. 33 of Reg. 882/2004/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 and whether the contact information and commodity-scopes are correct.

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 without prejudice of any legal action taken against them for not participating. This also applies to any participating laboratories that then fail to report results.

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 882/2004, 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

[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. 882/2004/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.

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 relevant documents in their latest version are linked.

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, along with the Minimum Required Reporting Levels (MRRLs) valid for the specific EUPT. The MRRLs are typically based upon the lowest MRLs found either in Regulation 396/2005/EC or Commission Directive 2006/125/EC (Baby Food Directive).

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

Specific Protocol

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

Homogeneity of the Test Item

The Test Item will be tested for homogeneity typically before distribution to participants. The homogeneity tests 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 the International Harmonized Protocols published by ISO and IUPAC. The acceptance criterion for the Test Items to be sufficiently homogeneous for the Proficiency Test is that s_{sam}^2 is less than c with s_{sam} being the between-bottle sampling standard deviation and $c = F_1 \times \sigma_{all}^2 + F_2 \times s_{an}^2$. F_1 and F_2 are constants,

with values of 1.88 and 1.01, respectively, if 10 samples are used. $\sigma_{all}^2 = 0.3 \times$ FFP-RSD[12] (FFP-RSD=0.25 × the mean of the homogeneity test), and s_{an} is the estimate of the analytical standard deviation.

The results of all homogeneity tests are presented to the EUPT-SC. In special cases where the above homogeneity test criteria are not met, the EUPT-SC considering all relevant aspects (e.g. the homogeneity results of other pesticides spiked at the same time, the overall distribution the participants' results, 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.

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-SC will finally decide whether analytes for which the stability test was not undertaken will be included in the final report, considering all relevant aspects such as the distribution of the participant's results (CV*).

A pesticide is considered to be adequately stable if $| y_i - y | \leq 0.3 \times \sigma_{pt}$, where y_i the mean value of the last period of the stability test, y is the mean value of the first period of the stability test and σ_{pt} 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 analytical difficulties faced during the test, 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.

[12] FFP-RSD = fit for purpose relative standard deviation, see also p. 11.

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.

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 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 considering all relevant aspects including the shipment time of the samples to each 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.

The Test Item is intentionally treated with pesticides whereas the Blank Material is analysed to ensure that it does not contain any of the pesticides in the Target Pesticides List, at or above, the specified MRRLs. Both the Test Item and Blank Material have to be analysed by the participating laboratories and any pesticide detected in them must be reported.

Correction of results for recovery

According to the Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed[13], it is common practice that pesticide analysis results are not corrected for recovery, but may be corrected if the average recovery is significantly different from 100 % (typically if outside the 70 – 120 % range, but also exhibiting good precision). Other approaches for recovery correction explicitly allowed in the SANTE document are the use of stable isotope labelled analogues of the target analytes used as Internal Standards (ISTDs), the 'procedural calibration' approach as well as the approach of 'standard addition' with additions of analyte(s) being made to analytical portions. Where reported residue data have been automatically adjusted for recovery by the method, or have subsequently been adjusted using a recovery factor, this must be indicated on the specific field of the 'Result Submission Form'. Results

[13] Document N° SANTE/11945/2015; Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed

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 (in percentage) must also be reported. No recovery data are required where correction for recovery is automatic by using the 'standard addition approach, or isotopically-labelled internal standards (in both cases with spiking into the Test Item at the beginning of the extraction procedures). In these cases, the laboratories should report the actual approach that was followed.

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 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 Organiser reserves the right not to accept the analytical results reported by the participants concerned.

Results evaluation

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

- ***False Positives 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 four 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 robust estimate of the participant's mean (x*) as described in ISO 13528:2015[14]. 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 compound 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), major deviations from the analytical procedure, inappropriate storage or transport conditions (in case of susceptible compounds), and the use of inappropriate procedures that demonstrably lead to significantly biased results (e.g. due to degradation or incomplete extraction). 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").

Uncertainty of the assigned value

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

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

[14] 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).

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) of 25% as follows:

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

The percentage FFP-RSD is set at 25% based on experience from results of previous EUPTs[15]. The EUPT-Panel reserves the right to also employ other approaches 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*) is calculated according to ISO 13528:2015; Chapter 7.7 (Consensus value from participant results) following Algorithm A in Annex C.

- ***z scores***

This parameter is calculated using the following formula:

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

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 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 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[16]:

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

[15] 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.

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

For results considered as false negatives, z scores will be calculated using the MRRL or RL (the laboratory's Reporting Limit) if the RL < MRRL. The EUPT-Panel will decide whether, or not, these values should appear in the z score histograms.

- ***Category A and B classification***

The EUPT-Panel will decide if and how to classify the laboratories into two categories - A or B. Currently, laboratories that are able to analyse at least 90 % of the compulsory pesticides in the target pesticides list, have correctly detected and quantified a sufficiently high percentage of the pesticides present in the Test Item (at least 90 %) and reported no false positives will have demonstrated 'sufficient scope' and can therefore be 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	

- ***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)[17,18] (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 classified 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 the individual z scores. The EUPT-Panel retains the right not to calculate AZ^2 if it is considered as not being useful or if the number of results reported by any participant is considered to be too low.

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

Laboratories within Category B will be ranked according to the total number of pesticides that 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 report may be published up to 10 months after the deadline for results submission.

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

[18] 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.

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 Category A or B.

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. The online version of the final 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. Even results within $|z| \leq 2.0$ may have to be checked if there are indications of a significant positive or negative bias.

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 traceability 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.

ANNEX 1. Protocols and Target list of pesticides to be sought.

NRLs will be considered as **underperforming in relation to scope** if in two EUPTs of the last four EUPTs falling within their responsibility area if 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 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[19]:

Phase 1:

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

Phase 2:

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

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

Disclaimer

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

[19] Article 32 of the Regulation 882/2004

EUPT-FV-BF01 SPECIFIC PROTOCOL

European Union Proficiency Test for Pesticide Residues in Baby Food (2016)

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 peach baby food spiked with pesticides. The baby food was supplied by a Spanish baby food company, and it was spiked with pesticide analytical standards.

The test item was homogenised and sub-sampled into polyethylene bottles that had previously been coded. Ten of those bottles containing the test item have been chosen randomly, and analysed to check for homogeneity.

The test item is 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).

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 and return 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 200 euros for EU national reference laboratories and EU official laboratories and 270 euros for the rest of laboratories.
4. The sample will be delivered to the participant laboratories on November 7th 2016. At the same time, they will receive by e-mail an Excel file where they will be able to report the results.
5. The deadline for submitting the results of this proficiency test is 28th November 2016.
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 peach based baby food (puree).

Shipment of Test Item

The test item will be packed in polystyrene boxes surrounded by dry ice and packed into cardboard boxes.

The shipment of the test item will be carried out over a one-week period from the 7th November 2016. 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 (-18°C or less) prior to analysis thus avoiding any possible deterioration/spoilage. The test item should be mixed thoroughly before taking the analytical portion(s).

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

Test Item Receipt

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 11th November 2016. 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 11th November, they must inform the Organiser **immediately** by e-mail (cferrer@ual.es)

Submission of results:

Once the laboratory has analysed the test item and is ready to submit their data, they must enter their results in the Excel file provided by the Organisers and send it to the following e-mail address: cferrer@ual.es.

All analyte concentrations must be expressed in mg/kg together with the associated recovery expressed as a percentage.

The number of significant figures should be based on the guidelines provided in SANTE/11945/2015. 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) or <LOQ. 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 or Additional Information

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.

ANNEX 1. Protocols and Target list of pesticides to be sought.

Calendar

ACTIVITY	DATE
Publishing the Target Pesticide List, Calendar and Matrix on the Web page.	4th August 2016
Opening Registration period	5th August 2016
Deadline for receiving Application Form from laboratories.	3th October 2016
Sample distribution.	7th November 2016
Deadline for receiving results	28th November 2016
Preliminary Report: only results, no statistical treatment.	5th December 2016
Preliminary Report with statistical treatment.	19th December 2016
Final Report	February 2017

Cost of test item shipment.

The sample delivery will be **200 € for EU National Reference Laboratories and EU Official Laboratories and 270 € 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

ACCOUNT NUMBER: ES0730580130172731005000

SWIFT: CCRIES2A

REFERENCE GIVEN: Lab Code

Contact information

The official organising group details are as follows:

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Stewart Reynolds, Fera Science Ltd., York, United Kingdom

Antonio Valverde, University of Almería, Spain

ANNEX 1. Protocols and Target list of pesticides to be sought.

TARGET PESTICIDE LIST FOR THE EUPT-FV-BF01

Pesticide	MRRL (mg/Kg)
Acephate	0.010
Acetamiprid	0.010
Acrinathrin	0.010
Aldicarb	0.010
Aldicarb Sulfone	0.010
Aldicarb Sulfoxide	0.010
Aldrin	**0.003**
Azinphos-methyl	0.010
Azoxystrobin	0.010
Benfuracarb	0.002*
Bifenthrin	0.010
Biphenyl	0.010
Bitertanol	0.010
Boscalid	0.010
Bromopropylate	0.010
Bromuconazole	0.010
Bupirimate	0.010
Buprofezin	0.010
Cadusafos	**0.006**
Carbaryl	0.010
Carbendazim and benomyl (sum of benomyl and carbendazim expressed as carbendazim)	0.010
Carbofuran	0.002*
Carbofuran-3-hydroxy	0.002*
Carbosulfan	0.002*
Chlorantraniliprole	0.010
Chlorfenapyr	0.010
Chlorfenvinphos	0.010
Chlorobenzilate	0.010
Chlorothalonil	0.010
Chlorpropham	0.010
Chlorpyrifos	0.010
Chlorpyrifos-methyl	0.010
Clofentezine	0.010
Clothianidin	0.010
Cyfluthrin (cyfluthrin incl. other mixtures of constituent isomers (sum of isomers))	0.010
Cymoxanil	0.010
Cypermethrin (cypermethrin incl. other mixtures of constituent isomers (sum of isomers))	0.010
Cyproconazole	0.010
Cyprodinil	0.010
Deltamethrin (cis-deltamethrin)	0.010
Demeton-S-methyl	**0.006**
Demeton-S-methyl sulfone	**0.006**
Demeton-S-methyl sulfoxide (Oxydemeton-methyl)	**0.006**
Diazinon	0.010
Dichlofluanid	0.010
Dichlorvos	0.010
Dicloran	0.010
Dicofol (sum of p, p´ and o,p´ isomers)	0.010
Dieldrin	**0.003**
Diethofencarb	0.010
Difenoconazole	0.010
Diflubenzuron	0.010
Dimethoate	0.003
Dimethomorph	0.010
Dimethylaminosulfotoluidide (DMST)	0.010
Diniconazole	0.010
Diphenylamine	0.010
Disulfoton	**0.003**
Disulfoton sulfone	**0.003**
Disulfoton sulfoxide	**0.003**
Endosulfan alpha	0.010
Endosulfan beta	0.010

Pesticide	MRRL (mg/Kg)
Endosulfan sulfate	0.010
Endrin	**0.003**
EPN	0.010
Epoxiconazole	0.010
Ethion	0.010
Ethirimol	0.010
Ethoprophos	**0.008**
Etofenprox	0.010
Famoxadone	0.010
Fenamidone	0.010
Fenamiphos	0.010
Fenamiphos sulfone	0.010
Fenamiphos sulfoxide	0.010
Fenarimol	0.010
Fenazaquin	0.010
Fenbuconazole	0.010
Fenhexamid	0.010
Fenitrothion	0.010
Fenoxycarb	0.010
Fenpropathrin	0.010
Fenpropidin	0.010
Fenpropimorph	0.010
Fenpyroximate	0.010
Fensulfothion	**0.003**
Fensulfothion sulfone	**0.003**
Fenthion	0.010
Fenthion oxon	0.010
Fenthion oxon sulfone	0.010
Fenthion oxon sulfoxide	0.010
Fenthion sulfone	0.010
Fenthion sulfoxide	0.010
Fenvalerate (any ratio of constituent isomers (RR, SS, RS & SR) including esfenvalerate)	0.010
Fipronil	**0.004**
Fipronil-desulfinyl	**0.004**
Fludioxonil	0.010
Flufenoxuron	0.010
Fluopicolide	0.010
Fluopyram	0.010
Fluquinconazole	0.010
Flusilazole	0.010
Flutolanil	0.010
Flutriafol	0.010
Fosthiazate	0.010
Heptachlor	**0.003**
Heptachlor-Trans epoxide	**0.003**
Hexachlorobenzene	**0.003**
Hexaconazole	0.010
Hexythiazox	0.010
Imazalil	0.010
Imidacloprid	0.010
Indoxacarb (sum of indoxacarb and its R enantiomer)	0.010
Iprodione	0.010
Iprovalicarb	0.010
Isocarbophos	0.010
Isofenphos-methyl	0.010
Isoprothiolane	0.010
Kresoxim-methyl	0.010
Lambda-Cyhalothrin	0.010
Linuron	0.010
Lufenuron	0.010
Malaoxon	0.010
Malathion	0.010
Mandipropamid	0.010
Mepanipyrim	0.010
Metaflumizone (sum of E- and Z- isomers)	0.010

ANNEX 1. Protocols and Target list of pesticides to be sought.

Pesticide	MRRL (mg/Kg)
Metalaxyl and metalaxyl-M	0.010
Metconazole (sum of isomers)	0.010
Methamidophos	0.010
Methidathion	0.010
Methiocarb	0.010
Methiocarb sulfone	0.010
Methiocarb sulfoxide	0.010
Methomyl	0.010
Methoxyfenozide	0.010
Monocrotophos	0.010
Myclobutanyl	0.010
Nitrofen	**0.003**
Omethoate	**0.003**
Orthophenylphenol	0.010
Oxadixyl	0.010
Oxamyl	0.010
Paclobutrazole	0.010
Paraoxon-methyl	0.010
Parathion-ethyl	0.010
Parathion-methyl	0.010
Penconazole	0.010
Pencycuron	0.010
Pendimethalin	0.010
Permethrin (sum of isomers)	0.010
Phenthoate	0.010
Phosalone	0.010
Phosmet	0.010
Phosmet oxon	0.010
Phoxim	0.010
Pirimicarb	0.010
Pirimicarb-desmethyl	0.010
Pirimiphos-methyl	0.010
Prochloraz (only parent compound)	0.010
Procymidone	0.010
Profenofos	0.010
Propamocarb	0.010
Propargite	0.010
Propiconazole	0.010
Propyzamide	0.010
Prothioconazole (Prothioconazole-desthio)	0.010
Prothiofos	0.010
Pyraclostrobin	0.010
Pyridaben	0.010
Pyrimethanil	0.010
Pyriproxyfen	0.010
Quinoxyfen	0.010
Spinosad (sum of spinosyn A and spinosyn D, expr. as spinosad)	0.010
Spirodiclofen	0.010
Spiromesifen	0.010
Spiroxamine	0.010
Tau-Fluvalinate	0.010
Tebuconazole	0.010
Tebufenozide	0.010
Tebufenpyrad	0.010
Teflubenzuron	0.010
Tefluthrin	0.010
Terbufos	**0.003**
Terbufos_Sulfone	**0.003**
Terbufos_Sulfoxide	**0.003**
Terbuthylazine	0.010
Tetraconazole	0.010
Tetradifon	0.010
Thiabendazole	0.010
Thiacloprid	0.010
Thiamethoxam	0.010

Pesticide	MRRL (mg/Kg)
Thiodicarb	0.010
Thiophanate-methyl	0.010
Tolclofos-methyl	0.010
Tolylfluanid	0.010
Triadimefon	0.010
Triadimenol	0.010
Triazophos	0.010
Trichlorfon	0.010
Trifloxystrobin	0.010
Triflumuron	0.010
Trifluralin	0.010
Triticonazole	0.010
Vinclozolin (only parent compound)	0.010
Zoxamide	0.010

Pesticides included in Commission Directive 2006/125/EC.

This list is based on Commission implementing Regulation (EU) 2015/595 of 15 April 2015 and Commission Directive 2006/125/EC.
The MRRLs are based on Commission Directive 2006/125/EC, except those indicated with an asterix, which are set considering Regulation (EC) No. 396/2005

ANNEX 2. List of laboratories that agreed to participate in EUPT-FV-BF01.

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
Austria	Austrian Agency For Health And Food Safety (AGES Gmbh). Department Pesticide and Food Analytics	Innsbruck	Yes
Belgium	Primoris Belgium	Zwijnaarde	Yes
Cyprus	Pesicides Residues Laboratory of the State General Laboratory of Cyprus.	Nicosia	Yes
Czech Republic	University of Chemistry and Technology Prague, Metrological and Testing Laboratory	Prague 6	Yes
Egypt	Central Lab of Residue Analysis of Pesticides and Heavy Metals in Foods	Giza	Yes
Finland	Finnish Customs Laboratory	Espoo	Yes
France	CERECO SUD	Garons	Yes
France	INOVALYS Le Mans	Le Mans	Yes
France	Laboratoire du SCL de Montpellier	Montpellier	Yes
France	Service Commun des Laboratoires - Ile de France - Massy	Massy Cedex	Yes
Germany	Bavarian health and food safety authority	Erlangen	Yes
Germany	Chemisches und Veterinäruntersuchungsamt Münsterland Emscher Lippe	48147 Münster	Yes
Germany	Chemisches und Veterinäruntersuchungsamt Stuttgart	Fellbach	Yes
Germany	Eurofins Dr. Specht Laboratorien GmbH	Hamburg	Yes
Germany	GALAB Laboratories GmbH	Hamburg	Yes
Germany	Landesamt für Verbraucherschutz Sachsen-Anhalt	Halle/Saale	Yes
Germany	Landeslabor Berlin-Brandenburg (LLBB)	Frankfurt (Oder)	Yes
Germany	Niedersächsisches Landesamt für Verbraucherschutz und Lebensmittelsicherheit Lebensmittel- und Veterinärinstitut Oldenburg	Oldenburg	Yes
Greece	BENAKI Pesticide Residue Laboratory	Kifissia	Yes
Hungary	National Food Chain Safety Office, Directorate of Plant Protection, Soil Conservation and Agri-environment Pesticide Residue Analytical Laboratory, Miskolc	Miskolc	Yes
Hungary	WESSLING Hungary Ltd. Food Testing Laboratory	Budapest	Yes
Indonesia	ANGLER BIOCHEMLAB, PT	Surabaya	No
Ireland	The Pesticide Control Laboratory	Celbridge, Co. Kildare	Yes
Italy	ARPALAZIO - Sezione Provinciale Di Latina	Latina	Yes
Italy	Laboratorio Di Prevenzione - Ats Milano Cittá' Metropolitana	Milano	Yes

ANNEX 2. List of laboratories that agreed to participate in EUPT-FV-BF01.

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
Italy	APPA - Settore Laboratorio - Provincia Autonoma di Trento	Trento	Yes
Italy	ARPA PUGLIA - Polo Di Specializzazione Alimenti Bari	Bari	Yes
Italy	ARPA Veneto - Laboratorio di Verona	Verona	Yes
Italy	ARPAE - Laboratorio Tematico Fitofarmaci Sezione Provinciale Di Ferrara	Ferrara	Yes
Italy	Istituto Zooprofilattico dell'Abruzzo e el Molise "G.Caporale" Teramo Italy	Teramo	Yes
Italy	WATER & LIFE LAB SRL	Entratico	Yes
Kenya	SGS Kenya Limited	Mombasa	Yes
Latvia	Institute of Food Safety, Animal Health and Environment "BIOR"	Riga	Yes
Norway	NIBIO - Norwegian Institute of Bioeconomy Research. Division of Biotechnology and Plant Health Department of Pesticides and Natural Products Chemistry	Aas	Yes
Poland	Research Institute of Horticulture. Food Safety Laboratory	Skierniewice	Yes
Poland	Voivodship Sanitary-Epidemiological Station in Warsaw Pesticide Residues Laboratory	Warszawa	Yes
Portugal	Laboratório Regional de Veterinária e Segurança Alimentar - Madeira	Funchal	Yes
Serbia	Center for Food analysis, Belgrade	Belgrade	Yes
Slovenia	NLZOH (National Laboratory for Health, Environment and Food)	Maribor	Yes
Spain	CNTA (National Centre for Technology and Food Safety)	San Adrian (Navarra)	Yes
Spain	Eurofins SICA AgriQ	Vicar - Almeria	Yes
Spain	Laboratori Agroalimentari	Cabrils	Yes
Spain	Laboratorio Agroalimentario Generalitat Valenciana	Burjassot. Valencia	Yes
Spain	Laboratorio Agroambiental de Zaragoza	Zaragoza	Yes
Spain	Laboratorio Arbitral Agroalimentario	Madrid	Yes
Spain	Laboratorio De Salud Publica (Madrid Salud)	Madrid	Yes
Spain	Laboratorio del Servicio de Inspección SOIVRE de la Dirección provincial de Comercio de Almería	Almería	Yes
Spain	Laboratorio Quimico Microbiologico, S.A.	Pol. Industrial Oeste, Parcela 21/1 30169 San Ginés Murcia	Yes
Spain	Laboratorios Ecosur,S.A	Lorqui - Murcia	Yes
Sweden	Eurofins Food & Feed testing Sweden AB	Lidköping	Yes

ANNEX 2. List of laboratories that agreed to participate in EUPT-FV-BF01.

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
Sweden	Swedish National Food Agency	Uppsala	Yes
Switzerland	Amt für Verbraucherschutz Aargau (Cantonal Office of Consumer Protection Aargau)	Aarau	Yes
Taiwan (R.O.C.)	SGS Taiwan Ltd. Food Laboratory-Taipei	New Taipei Industrial Park, Wu Ku District, New Taipei City	Yes
The Nerherlands	NVWA- Netherlands Food and Consumer Product Safety Authority NRL for Pesticide Residues in Food and Feed	Wageningen	Yes
The Nerherlands	Groen Agro Control	Delfgauw	Yes
The Nerherlands	Eurofins lab Zeeuws-Vlaanderen B.V. (LZV)	Graauw	Yes
United Kingdom	Fera Science Ltd	York	Yes