

Proficiency Test on pesticide residues in rice kernels

EU Reference Laboratory on Cereals & Feeding stuff

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EUPT-CF14, 2020**

Pesticide Residues in Rice Kernels

Final Report

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PREFACE

Regulation (EU) No 2017/625 [1], defines the general tasks and duties of the European Union Reference Laboratories (EURLs) for Food, Feed and Animal Health including the organisation of comparative tests. These proficiency tests (PTs) are carried out on an annual basis, and aim to improve the quality, accuracy and comparability of the analytical results generated by EU Member States within the framework of the EU multi-annual co-ordinated control and national monitoring programmes. Participation in the proficiency test scheme “European Union Proficiency Tests (EUPTs) for pesticide residues” is mandatory according to Article 28 of Regulation (EC) No 396/2005 on maximum residue levels of pesticides in, or on, food and feed of plant and animal origin [2], as long as the analytical scope of the PT and the laboratory overlap.

The present EUPT was the fourteenth organized within the frame of the EURL activities with cereal or feed matrices as Test Items. The previous PTs were EUPT-C1/SRM2 on wheat, EUPT-C2 on wheat, EUPT-C3/SRM4 on hay, EUPT-C4 on rye, EUPT-C5/SRM6 on rice, EUPT-C6 on barley, EUPT-CF7 on animal feed, EUPT-CF8 on wheat, EUPT-CF9 on maize, EUPT-CF10 on rye flour, EUPT-CF11 on oat flour, EUPT-CF12 on hay flour, and EUPT-CF13 on rye kernels. The PTs in 2007, 2009 and 2011 were jointly organised by the EURL-CF and EURL-SRM using and focusing on both MRM and SRM pesticides. The last EUPT-CF13 on rye as well as the EUPT-CF14 on rice only focused on MRM-pesticides. The test rice Item used for EUPT-CF14 was treated both with formulations in the field and post-harvest in the laboratory.

Participation in EUPT-CF14 was compulsory for all National Reference Laboratories (NRLs) and Official Laboratories (OfLs) within the EU involved in the determination of pesticide residues in cereals for human or animal consumption using multi residue methods for their national programmes. Official laboratories from EFTA countries (Iceland, Norway and Switzerland), as well as official laboratories from EU-candidate states, were invited to take part in this EUPT. Selected laboratories from Third Countries were also allowed to take part in this exercise, but their results, together with the EU-candidate state laboratories, were not used when establishing the Assigned Values for each pesticide.

DG-SANTE will have full access to all data from EUPTs including the lab-code/lab-name key. The same will apply to all NRLs regarding data from laboratories belonging to their own country network. The results of this EUPT may be further presented to the European Commission Standing Committee for Animal Health and the Food Chain.



CONTENTS

PREFACE	7
CONTENTS	9
1. INTRODUCTION	11
1.1 Analytical methods	11
1.2 Selection of Pesticides for the Target Pesticide List.....	11
1.3 Preparation of the Test Item.....	12
1.4 Homogeneity test	12
1.5 Stability tests	13
1.6 Organisational details.....	15
2. EVALUATION OF THE RESULTS	16
2.1 False positives and negatives.....	16
2.2 Estimation of the true concentration (x_{pt})	16
2.3 Uncertainty of the assigned value	16
2.4 Standard deviation of the assigned value (target standard deviation)	16
2.5 Z scores	17
2.6 Category A and B classification and combined z scores (AZ^2)	17
3. RESULTS	18
3.1 Summary of reported results.....	18
3.2 Assigned values and target standard deviations	21
3.3 Assessment of laboratory performance	22
3.4 Trends in numbers of participating laboratories and their performance.....	44
3.5 Summary, conclusions and prospects for the EUPTs on pesticide residues in cereals	45
4. ACKNOWLEDGEMENTS	46
5. REFERENCES	46
APPENDICES	47
Appendix 1 List of Laboratories registered to participate in the EUPT-CF14	47
Appendix 2 Target Pesticide List.....	51
Appendix 3 Homogeneity data	57
Appendix 4 Stability figures.....	59
Appendix 5 Graphical presentation of z scores	61
Annexes	81
Annex 1 General protocol	81
Annex 2 Specific protocol.....	99

EUROPEAN COMMISSION EURL PROFICIENCY TEST ON PESTICIDE RESIDUES IN CEREALS EUPT-CF14, 2019

1. INTRODUCTION

On 28 December 2019 the announcement of the 14th European Commission's Proficiency Test on cereals and feed (EUPC-CF14) was published on the EURL website, together with the Calendar and the Pesticide Target List including all compounds that could potentially be present in the Test Item. The Target Pesticides List included 164 individual compulsory compounds and 38 voluntary requiring the use of multi residue methods (MRMs), along with a minimum required reporting level (MRRL) stipulated for each compound. Links to The General Protocol containing information (**Annex 1**) that is common to all EUPCs, the Specific protocol (**Annex 2**), as well as a list of labs that are obliged to take part in the EUPC-CF14, were provided via the homepage. Laboratories were able to register online from January to 25 May 2020. In total 156 laboratories from EU and EFTA countries agreed to participate in the test as well as 8 laboratories from EU-Candidate States and Third Countries (**Appendix 1**).

The present proficiency test was performed using rice kernels of Indian origin, which had been treated with pesticides in the field, and partly spiked post-harvest at the facilities of the EURL-CF. The Test Item contained 19 compounds that could be evaluated. The rice was grown in India and the field treatment was performed in 2019. The pesticides employed for the field treatment were selected by the EURL-CF and the EUPC quality control group and the application rates and harvest intervals chosen were based on previous experience and data from supervised residue trials. The test material was checked for homogeneity before shipping to participants. Furthermore, the stabilities of the pesticides in the Test Item were checked several times during the period of time allowed for laboratories to complete the PT exercise.

The participating laboratories were provided with 100 g portions of the rice Test Item. The Test Items were shipped to participants on 8 June 2020 and the deadline for submission of results to the Organiser was the 17 August 2019. The deadline for submission of additional information for false negative results was the 24 August 2020. The participants were asked to analyse the Test Item and report the concentrations of any pesticide residues found that were included in the Target Pesticide List (**Appendix 2**). Submission of results was performed online via the DTU Webtool.

1.1 Analytical methods

The QuEChERS method [3] was used by the organiser to test the homogeneity and stability of the Test Items. Determination was performed by GC-MS/MS and LC-MS/MS.

- QuEChERS: Cold water was added to a milled portion of the test item and shaken. Acetonitrile was added immediately and the tube was shaken again. A salt and buffer mixture was then added together with ceramic homogenizers and the sample was shaken vigorously for 1 min. After centrifugation, an aliquot of the supernatant was cleaned by freezing out. After additional centrifugation of the cold extract the supernatant was transferred to a tube containing PSA and MgSO₄. After shaking and centrifugation the extract was ready for analysis by GC-MS/MS and LC-MS/MS.

1.2 Selection of Pesticides for the Target Pesticide List

The pesticides to be included in the target pesticides list were selected by the Organiser and the Quality Control Group taking into account the present and upcoming scope of the EU multi-annual coordinated control programme, the working document and pesticides according to their relevance and risk-potential, as well as pesticides relevant to the specific commodity (rice). The overall capacity and capability of the laboratories within the EU, as assessed from previous PTs and surveys, was also taken into account. The minimum required reporting level (MRRL) for all pesticides in the target list was in general set at 0.01 mg/kg. However, for 15 pesticides the MRRL were set at or below 0.005 mg/kg.

1.3 Preparation of the Test Item

The field spraying was performed in 2019 in India and organised by EURL-SRM and the InDepth Management India Pvt Ltd. Approximately, 21 kg of the harvested rice grain was used for this PT. It was decided to additionally spike in the laboratory with ten pesticides, which were either not included in the field treatments or where residues were too low for the evaluation (**Table 1**). Spiking in the laboratory was performed using formulations or pure standards. Five time one kilogram of the field treated rice was spiked and subsequently mixed with 16 kg of field treated rice and homogenised thoroughly. One hundred gram portions were weighed out into screw-capped polyethylene plastic bottles, sealed, numbered, and stored in a freezer at about -20 °C prior to homogeneity testing and distribution to participants.

1.4 Homogeneity test

Ten bottles of the Test Items were randomly chosen and analyses were performed on duplicate portions taken from each bottle using the analytical methods described in section 1.1. The sequence of analyses and injections were also randomly chosen. Quantification was performed using a 5-point calibration curve constructed from matrix-matched standards.

The statistical evaluation was performed according to the International Harmonized Protocols published by IUPAC, ISO and AOAC [4]. An overview of the statistical analyses of the homogeneity test is shown in **Table 2**. The individual residues data from the homogeneity tests, as well as the results of the statistical analyses, are given in **Appendix 3**.

The homogeneity test is to show that the between-bottle variance is not greater than the within-bottle variance. The acceptance criteria to show that the Test Items were sufficiently homogeneous for the proficiency test was that: $S_s^2 < c$ where S_s is the between-bottle sample standard deviation and $c = F_1 \times \sigma_{all}^2 + F_2 \times s_{an}^2$; F_1 and F_2 being constants with values of 1.83 and 0.93, respectively, from the 11 samples taken, $\sigma_{all}^2 = 0.3 \times \text{FFP RSD (25\%)} \times$ the analytical sampling mean for all pesticides, and s_{an} is the estimate of the analytical standard deviation.

As all pesticides passed the homogeneity test, when the Test Item was stored at -18 °C, the Test Item was considered to be sufficiently homogenous and suitable for the EUPT-CF14.

Table 1. Pesticides used for application in the field and/or spiked in the laboratory.

Pesticides	Application in field	Spike in laboratory	Formulation/standard
Acephate	x		ASATAF
Acetamiprid	x	x	Dhanpreet/analytical standard
Azoxystrobin	x		Amistar
Buprofezin	x		Flotis
Carbendazim	x	x	Ultra/analytical standard
Carbofuran	x		Furadan 3G
Cyproconazole	x	x	Alto 100 SL/analytical standard
Dichlorvos	x	x	DESTA 100 EC/analytical standard
Difenoconazole	x	x	Amistar/analytical standard
Hexaconazole	x		CONTAF
Imidacloprid	x		Confidor®
Isoprothiolane	x		FUJI-ONE
Metalaxyl	x	x	SL 567A/analytical standard
Profenofos	x		Roket Insecticide
Pyriproxyfen	x		Admiral®
Thiamethoxam	x	x	Voliam Flexi/analytical standard
DDE-pp		x	Analytical Standard
Endrin-ketone		x	Analytical Standard
Oxathiapipronil		x	Analytical Standard

1.5 Stability tests

The analytical methods described briefly above (in section 1.1) were also used for the stability tests.

The stability test was performed according to ISO 13528, Annex B [5]. Two different storage temperatures were used; room temperature and -18 °C. Six sub-samples (analytical portions) were analysed on each test day. A pesticide is 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, y_i the mean value of the last stability test and σ the standard deviation used for proficiency assessment (25% of the assigned value):

The dates of testing were as follows:

Day 1: 8 June 2020
Day 2: 13 July 2020
Day 3: 17 August 2020

Table 2. Statistical evaluation of the homogeneity test data (n=22 analyses using a sub-sample of 5 g in each case).
S_s: Between Sampling Standard Deviation

Pesticides	Mean, mg/kg	S _s ²	c	S _s ² < c
Acephate	0.048	0.00000	0.0000	Pass
Acetamiprid	0.067	0.00003	0.0001	Pass
Azoxystrobin	0.286	0.00070	0.0016	Pass
Buprofezin	0.052	0.00000	0.0001	Pass
Carbendazim	0.063	0.00001	0.0001	Pass
Carbofuran	0.062	0.00001	0.0001	Pass
Cyproconazole	0.076	0.00002	0.0001	Pass
DDE-pp	0.037	0.00000	0.0000	Pass
Dichlorvos	0.022	0.00000	0.0000	Pass
Difenoconazole	0.054	0.00001	0.0000	Pass
Endrin-ketone	0.033	0.00000	0.0002	Pass
Hexaconazole	0.094	0.00001	0.0002	Pass
Imidacloprid	0.070	0.00002	0.0001	Pass
Isoprothiolane	0.377	0.00045	0.0025	Pass
Metalaxyl	0.071	0.00001	0.0001	Pass
Oxathiapipronil	0.054	0.00001	0.0000	Pass
Profenofos	0.092	0.00088	0.0014	Pass
Pyriproxyfen	0.160	0.00005	0.0004	Pass
Thiamethoxam	0.052	0.00001	0.0000	Pass

Table 3. Statistical evaluation of the stability test data at 18 °C

Pesticides	Mean, mg/kg	x ₁ - y _i	0.3×σ	x ₁ - y _i ≤ 0.3×σ
Acephate	0.049	0.003	0.004	Pass
Acetamiprid	0.067	0.006	0.006	Pass
Azoxystrobin	0.336	0.001	0.027	Pass
Buprofezin	0.060	0.002	0.005	Pass
Carbendazim	0.066	0.004	0.004	Pass
Carbofuran	0.067	0.001	0.005	Pass
Cyproconazole	0.070	0.005	0.006	Pass
DDE-pp	0.038	0.001	0.003	Pass
Dichlorvos	0.023	0.001	0.001	Pass
Difenoconazole	0.045	0.003	0.004	Pass
Endrin-ketone	0.039	0.003	0.003	Pass
Hexaconazole	0.098	0.003	0.008	Pass
Imidacloprid	0.080	0.004	0.006	Pass
Isoprothiolane	0.353	0.008	0.035	Pass
Metalaxyl	0.073	0.005	0.006	Pass
Oxathiapipronil	0.057	0.004	0.004	Pass
Profenofos	0.105	0.009	0.018	Pass
Pyriproxyfen	0.170	0.005	0.013	Pass
Thiamethoxam	0.061	0.005	0.005	Pass

The results of the stability test for storage temperature -18 °C are given in **Table 3**. All pesticides passed the test at -18 °C. However, acephate, buprofezin, DDE-pp and dichlorvos did not pass the test when stored for 11 weeks at room temperature. But the laboratories were instructed to store the test item at -18 degree and the stability test was consequently accepted. See the individual stability figures for all pesticides in **Appendix 4**.

1.6 Organisational details

1.6.1 Access to documents, registration and confidentiality

In the invitation letter, all NRLs and OfLs were requested to register using the online registration link from January 2020. All documents related to this EUPT (Calendar, Target Pesticides List, Specific Protocol, General Protocol) were uploaded to the EURL website and the CIRCA platform. Laboratories that were intending not to participate were given the opportunity to explain the reasons for their non-participation. Participants from Candidate countries and third countries did also have access to another online registration link. On 9 March, the participants received a link to DTU web tool as well as login credentials and were asked to enter the web tool and to select the scope of pesticides they wanted to be evaluated on. This had to be done before the samples were shipped to the participants (9 June 2020).

1.6.2 Distribution of the Test Item

On 9 June 2020, the Test Item (100 g) was shipped to all participants in insulated polystyrene boxes containing a freezer block. The laboratories were asked to check the state of the sample on receipt and to enter the web tool to report whether they accept/not accept the Test Item.

1.6.3 Submission of results

The participants had to submit their results via a web tool. All participants had access to the result-submission website from a few days after shipment until the result-submission deadline (24 August 2020). Participants were asked not only to report their analytical results, but also to give information regarding accreditation, reporting limits and details regarding the methods they used to analyse the Test Item.

2. EVALUATION OF THE RESULTS

The results were evaluated according to the general and specific protocols (**Annex 1 and 2**). However, the main points are listed below.

2.1 False positives and negatives

2.1.1 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 these 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.

2.1.2 False negatives

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

2.2 Estimation of the true concentration (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 participants' mean (x^*) as described in ISO 13528:2015, taking into account the results reported by EU and EFTA countries laboratories only. In special justifiable cases, the EUPT-Panel may decide to eliminate certain results traceably associated with gross errors 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.

2.3 Uncertainty of the assigned value

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

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

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

2.4 Standard deviation of the assigned value (target standard deviation)

The target standard deviation of the assigned value ($FFP-\sigma_{pt}$) will be calculated using a Fit-For-Purpose approach with a fixed Relative Standard Deviation (FFP-RSD) of 25% as follows:

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

The percentage FFP-RSD is set at 25% based on experience from results of previous EUPTs. 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.

2.5 Z scores

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

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

where x_i is the value reported by the laboratory, x_{pt} is the assigned value, and $FFP-\sigma_{pt}$ is the standard deviation using FFP approach. Z scores was 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.

Z scores will be interpreted in the following way as is set in the ISO 17043:2010 [6]:

$|z| \leq 2$ Acceptable
 $2 < |z| < 3$ Questionable
 $|z| \geq 3$ Unacceptable

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

2.6 Category A and B classification and combined z scores (AZ²)

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% criteria, 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.

For evaluation of the overall performance of laboratories within Category A, the Average of the Squared z Score (AZ²) will be used. The AZ² is calculated as follows:

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

where "n" is the number of each laboratory's z scores that were considered in this formula. For the calculation, any z-score > 5 was set at "5". Based on the AZ² achieved, the laboratories are classified as follows:

$AZ^2 \leq 2$	Good
$2 < AZ^2 < 3$	Satisfactory
$AZ^2 \geq 3$	Unsatisfactory

The AZ² is considered being of lesser importance than the individual z scores.

Laboratories within Category B are 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 is listed as well.

3. RESULTS

3.1 Summary of reported results

In total, 156 EU and EFTA laboratories, from 28 different countries (26 EU member states and UK), agreed to participate in this proficiency test. Additionally, Malta was represented by UK NRL. Six EU participants did not submit results. Additionally, 8 participants from non-EU Countries registered for the PT. The participating laboratories are listed in **Appendix 1**.

An overview of results submitted by laboratories from the EU and EFTA can be seen in **Table 4**. All reported analytical results for the pesticide residues are shown in **Table 10-12** and in **Appendix 5**. However, only results submitted by laboratories from EU and EFTA countries are included in **Table 4, 8-9** and **15** and the z scores histograms are shown in **Appendix 5**.

Table 4. Overview of number of results, number of not analysed (NA), number of not detected (ND = false negatives) and the percentage of laboratories that reported results for the pesticides in the Test Item. Only results submitted by laboratories from the EU and EFTA are included in this table.

Pesticides	No. of reported results	No. of NA	False negatives	% of labs reporting results
Acephate	119	31	4	79
Acetamiprid	128	22	0	85
Azoxystrobin	138	12	0	92
Buprofezin	135	15	1	90
Carbendazim	124	26	0	83
Carbofuran	126	24	3	84
Cyproconazole	135	15	0	90
DDE-pp	127	23	5	85
Dichlorvos	133	17	30	89
Difenoconazole	139	11	1	93
Endrin-ketone	55	95	18	37
Hexaconazole	134	16	3	89
Imidacloprid	127	23	0	85
Isoprothiolane	122	28	2	81
Metalaxyl	131	19	1	87
Oxathiapiprolin	39	111	8	26
Profenofos	132	18	2	88
Pymetrozine	101	49	0	67
Pyriproxyfen	129	21	1	86
Thiamethoxam	125	25	0	83

¹ '% results' have been calculated using the number of laboratories that reported results for each particular compound and the total number of EU laboratories that submitted results (n = 150). False negatives are included in reported results.

Azoxystrobin, buprofezin, cyproconazole, and difenoconazole were the most frequently analysed compounds with ≥ 90 % of the labs submitting results for these compounds. Acephate, acetamiprid, carbendazim, carbofuran, hexaconazole, imidacloprid, isoprothiolane, metalaxyl, profenofos, pyriproxyfen, and thiametoxam were analysed and reported by 79-89% of the participants. Pymetrozine, endrin-ketone, and oxathiapipronil were only analysed and reported by 26-67% of participants. The two pesticides, DDE-pp and dichlorvos, that were present in very low levels, were analysed by 85 and 89% of the labs, respectively.

3.1.1 False positives

Thirteen participants (12 from EU and EFTA) countries reported 15 results for 9 different additional pesticides above the MRRL that had not been used to treat the Test Item (**Table 5**). The pesticides were: ametoctradin, chlordane-cis, cypermethrin, endosulfan alpha, fenpropathrin, formetanate, indoxacarb, isocarbophos, and orthophenylphenol. In all cases the compounds were not detected either by the Organizer, or by the other participating laboratories. The reported results were therefore considered to be false positives. The participant reported that the compounds were also found in the blank test item

Table 5. False positive results at or above 0.01 mg/kg, the concentration detected in mg/kg, the determination technique used, the reporting level and the MRRL in mg/kg.

Lab code	Pesticides	Concentration mg/kg	Determination technique	RL, mg/kg	MRRL, mg/kg
31	Ametoctradin	0.01	LC-MS/MS QQQ	0.01	0.01
135	Chlordane-cis	0.051	GC- (μ) ECD	0.01	0.01
21	Cypermethrin	0.033	GC-MS/MS (QQQ)	0.01	0.01
135	Endosulfan alpha	0.06	GC- (μ) ECD	0.01	0.01
145	Fenpropathrin	0.1671	GC- (μ) ECD	0.01	0.01
87	Formetanate	0.071	LC-MS/MS QQQ	0.01	0.01
91	Formetanate	0.06	LC-MS/MS QQQ	0.01	0.01
150	Formetanate	0.042	LC-MS/MS QQQ	0.01	0.01
151	Formetanate	0.0661	LC - MS/MS	0.01	0.01
154	Formetanate	0.0158	LC-MS	0.01	0.01
114	Indoxacarb	0.013	LC-MS/MS QQQ	0.01	0.01
105	Isocarbophos	0.45	LC-MS/MS QQQ	0.01	0.01
125	Isocarbophos	0.65	LC-MS/MS QQQ	0.01	0.01
148	Orthophenylphenol	0.028	LC-MS/MS QQQ	0.01	0.01

3.1.2 Findings of compounds below 0.01 mg/kg

Apart from the false positive results above and the results for the pesticides listed in **Table 10-12**, five participants reported results for five other pesticides, see **Table 6a**. These results were not evaluated as false positives because the concentrations are below the MRRL at 0.01.

Additionally, 5 laboratories reported low results for chlorpyrifos, also at low level, see **Table 6b**. Chlorpyrifos was used for the field spraying and was also detected by the organiser.

However, all the results reported were below the laboratories own reporting limit, and should therefore not have been reported.

Table 6a. Reported results in mg/kg at or below the MRRL at 0.01 mg/kg

Lab code	Pesticides	Concentration, mg/kg	Determination technique	RL, mg/kg	MRRL, mg/kg
172	Ametoctradin	0.0094	LC-MS/MS QQQ	0.01	0.01
123	Fenpicoxamid	0.005	LC-MS/MS QQQ	0.01	0.01
21	Fenpropimorph	0.004	LC-MS/MS QQQ	0.01	0.01
21	Lindane	0.004	GC-MS/MS QQQ	0.01	0.01
124	Methamidophos	0.002	LC-MS/MS QQQ	0.01	0.01
136	Methamidophos	0.004	LC-MS/MS QQQ	0.01	0.01

Table 6b. Reported results of chlorpyrifos in mg/kg

Lab code	Pesticides	Concentration, mg/kg	Determination technique	RL, mg/kg	MRRL, mg/kg
21	Chlorpyrifos	0.008	GC-MS/MS (QQQ)	0.005	0.005
31	Chlorpyrifos	0.005	GC-MS/MS (QQQ)	0.01	0.005
124	Chlorpyrifos	0.001	LC-MS/MS QQQ	0.005	0.005
139	Chlorpyrifos	0.007	LC-Q-TOF	0.005	0.005
172	Chlorpyrifos	0.005	GC-MS/MS (QQQ)	0.01	0.005

3.1.3 False negatives

Not reported results for pesticides actually present in the Test Item were judged as false negatives. **Table 7** summarizes the number of reported false negatives for each pesticide. Fifty-four participants submitted 87 false negatives results for 19 different pesticides, which represents 2.3% of the total number of results. Around 35% of the participants (54 laboratories) reported false negative results; this is higher than for previous EUPTs on cereals where 20-30% of the labs reported false negative results. False negatives results were reported for all evaluated compounds.

Table 7. False negative results (FN).

Labcode	Acephate	Acetamiprid	Azoxystrobin	Buprofezin	Carbendazim	Carbofuran	Cyproconazole	DDE-pp	Dichlorvos	Difenoconazole	Endrin-ketone	Hexaconazole	Imidacloprid	Isoprothiolane	Metaxyl	Oxathiapiprolin	Profenofos	Pyriproxyfen	Thiamethoxam
9									FN										
12									FN										
18									FN										
20									FN										
21	FN			FN						FN									
25									FN										
31											FN								
33											FN								
36	FN								FN										
41									FN			FN		FN			FN		
42									FN										
44									FN										
52																FN			
55									FN										
60											FN								
62									FN										
64						FN			FN			FN							
70									FN										FN
71									FN										
74									FN										
75									FN										
82											FN					FN			
83											FN					FN			
85											FN								
90									FN										
91											FN								

Labcode	Acephate	Acetamiprid	Azoxystrobin	Buprofezin	Carbendazim	Carbofuran	Cyproconazole	DDE-pp	Dichlorvos	Difenoconazole	Endrin-ketone	Hexaconazole	Imidacloprid	Isoprothiolane	Metalaxyl	Oxathiapiprolin	Profenofos	Pyriproxyfen	Thiamethoxam
93								FN											
94									FN										
100						FN		FN	FN										
103									FN										
105								FN	FN		FN								
106											FN								
107									FN							FN			
108											FN								
112								FN											
114	FN					FN			FN		FN			FN		FN	FN		
116															FN	FN			
120											FN								
130									FN	FN									
135											FN								
137									FN										
139											FN								
142									FN										
143									FN										
145			FN										FN						FN
146									FN										
150									FN		FN								
151									FN										
154									FN										
158	FN							FN	FN		FN	FN				FN			
162											FN								
163									FN										
169											FN					FN			
174							FN												

3.2 Assigned values and target standard deviations

The Assigned Values were calculated as the Algorithm A mean, including the reported results submitted by laboratories from EU and EFTA countries.

All assigned values for the pesticides can be seen in **Table 8**. The assigned values for cypermethrin was less than 3 times the MRRL (equal to 0.03 mg/kg). Consequently, the assigned values for this compound is given for informative purposes only. Deltamethrin and lambda-cyhalothrin were present in very low levels around 0.01 and are not included in the tables below.

The target standard deviation was obtained using a fixed FFP-RSD value of 25 %. In parallel, the Algorithm A standard deviation (Alg A-RSD) was calculated for informative purposes only. The range of Alg A-RSD values was 14-25 % but on average, the Alg A-RSD was 18 %, and thus below the 25 % FFP-RSD used for the calculations.

The uncertainty of the assigned values is calculated according to ISO 13528 [5] as:

$$\mu = 1.25 \frac{s^*}{\sqrt{n}}$$

Where s^* is the robust standard deviation estimate and n is the number of datapoints equal to the number of results used to calculate the assigned value (number of results in **Table 9**)

Table 8. Assigned values and their uncertainty in mg/kg, Fit-For-Purpose Relative Standard Deviation (FFP RSD) and Robust Relative Standard Deviation (Alg A RSD) for the pesticides present in the Test Item.

Pesticides	MRRL, mg/kg	Assigned value, mg/kg	Uncertainty, mg/kg	FFP RSD, %	Alg A RSD, %
Acephate	0.01	0.048	0.001	25	21
Acetamiprid	0.01	0.070	0.001	25	17
Azoxystrobin	0.01	0.308	0.006	25	19
Buprofezin	0.01	0.055	0.001	25	16
Carbendazim	0.01	0.047	0.001	25	24
Carbofuran	0.01	0.057	0.001	25	23
Cyproconazole	0.01	0.069	0.001	25	17
DDE-pp	0.01	0.035	0.001	25	18
Dichlorvos	0.01	0.015	0.000	25	23
Difenoconazole	0.01	0.048	0.001	25	19
Endrin-ketone	0.01	0.036	0.002	25	25
Hexaconazole	0.01	0.090	0.002	25	17
Imidacloprid	0.01	0.069	0.001	25	18
Isoprothiolane	0.01	0.404	0.007	25	16
Metalaxyl	0.01	0.073	0.001	25	18
Oxathiapiprolin	0.01	0.050	0.002	25	18
Profenofos	0.01	0.205	0.005	25	20
Pymetrozine ¹	0.01	0.010	0.000	25	36
Pyriproxyfen	0.01	0.153	0.003	25	17
Thiamethoxam	0.01	0.054	0.001	25	18

¹ The assigned values are less than 3 times the MRRL and consequently shown for informative purposes only.

3.3 Assessment of laboratory performance

3.3.1 Z scores

Z scores have been calculated for all the quantified pesticides using the FFP RSD of 25 %. **Table 9** shows an overview of the acceptable, questionable and unacceptable z scores and Tables 10a/e-12a/e show the individual results and z scores for each laboratory and pesticide together with the assigned values. A graphical representation of the z scores (for EU and EFTA countries) can be seen in **Appendix 4**.

Table 9. Number of acceptable, questionable, unacceptable z scores, and false negatives.

Pesticides	No. of reported results	Assigned values	Acceptable %	Questionable %	Unacceptable ¹ %	False negatives %
Acephate	119	0.048	108	2	9	4
Acetamiprid	128	0.070	120	5	3	0
Azoxystrobin	138	0.308	131	2	5	0
Buprofezin	135	0.055	129	3	3	1
Carbendazim	124	0.047	109	7	8	0
Carbofuran	126	0.057	117	3	6	3
Cyproconazole	135	0.069	128	3	4	0
DDE-pp	127	0.035	114	7	6	5
Dichlorvos	133	0.015	94	6	33	30
Difenoconazole	139	0.048	132	2	5	1
Endrin-ketone	55	0.036	34	2	19	18
Hexaconazole	134	0.090	126	2	6	3
Imidacloprid	127	0.069	117	4	6	0
Isoprothiolane	122	0.404	116	2	4	2
Metalaxyl	131	0.073	124	4	3	1
Oxathiapiprolin	39	0.050	31	0	8	8
Profenofos	132	0.205	122	5	5	2
Pyriproxyfen	129	0.153	121	6	2	1
Thiamethoxam	125	0.054	119	3	3	0

¹ Unacceptable z scores include false negative results.

For azoxystrobin, buprofezin, difenoconazole, isoprothiolane, metalaxyl, and thiamethoxam, acceptable results were obtained by 95-99% of the laboratories. For acephate, acetamiprid, carbofuran, DDE-pp, hexaconazole, imidacloprid, profenofos, and pyriproxyfen, acceptable results were obtained by 90-94% of the laboratories. Only 88% of the laboratories obtained acceptable z scores for carbendazim. Only 79%, 71%, and 62%, of the laboratories obtained acceptable z scores for oxathiapiprolin, dichlorvos, and endrin-ketone, respectively.

Like in previous EUPTs, some laboratories reported very high results for carbendazim. This is probably due to the low solubility of the compound in organic solvents, e.g. solubility in ethyl acetate is only 0.135 mg/ml. It is therefore crucial to check if carbendazim in stock solution is completely dissolved. It is recommended to prepare stock solutions of carbendazim at a concentration not higher than 0.1 mg/ml.

More than five different analytical methods have been used by the laboratories. For the majority of the results, 72%, QuEChERS, Citrate buffered (EN 151662) was used. However, variations in the clean-up procedures were reported by the labs, e.g. some used a freezing out step (20% of the participants), centrifugation (28%), some used d-SPE with PSA/MgSO₄ (24%), some used d-SPE with ODS/ MgSO₄ (3%). Liquid-liquid partition was used by 7% of the participants and some used SPE column. So it was not one specific method.

Other extraction methods have been used; the original QuEChERS version method (J. AOAC 86, 2003) and QuEChERS-Acetate buffered (AOAC Official method 2007.01) were respectively used by 8 and 7% of the participants. The Mini-Luke method and the SweEt method were each used by 3% of the participants. The remaining 6% of the participants used other methods. More than 96% of the reported results derived from a method where water was added before extraction.

For milling, 55% of the labs used a knife mill and 24% of the labs used centrifugal mill. Moreover, 6% used a disk mill, 4% used an horizontal mill, and 2% used a hammer mill. Furthermore, 9% of the labs did not specify the type of mill used.

GC instruments were used for 38% of the results, mainly GC-MS/MS and GC-MSD (30% and 5%), 1% used GC-TOF or GC-Orbitrap instruments, and the rest used GC with specific detectors, ECD and NPD. LC instruments were used for 62% of the reported results, mainly LC-MS/MS (58%) but 3% used high resolution instrument like LC-Orbitrap or LC-Q-TOF. No results were analysed using specific detectors such as LC-Fluorescence, LC-UV or LC-DAD.

Table 10a. Results for acephate, acetamiprid, axoxystrobin, buprofezin, carbendazim, carbofuran, cyproconazole, and dichlorvos in mg/kg, and the corresponding z scores, MRRs and the assigned values.

Laboratory code	Acephate	Z-scores (FFP RSD (25%))	Acetamiprid	Z-scores (FFP RSD (25%))	Azoxystrobin	Z-scores (FFP RSD (25%))	Buprofezin	Z-scores (FFP RSD (25%))	Carbendazim	Z-scores (FFP RSD (25%))	Carbofuran	Z-scores (FFP RSD (25%))	Cyproconazole	Z-scores (FFP RSD (25%))	Dichlorvos	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01		0.01		0.005		0.01		0.005	
Assigned value	0.048		0.070		0.308		0.055		0.047		0.057		0.069		0.015	
1	0.0488	0.0	0.0701	0.0	0.291	-0.2	0.051	-0.3	0.055	0.7	0.0425	-1.0	0.0804	0.7	0.0138	-0.3
2	0.055	0.6	0.07	0.0	0.304	-0.1	0.075	1.5	0.046	-0.1	0.037	-1.4	0.074	0.3	0.018	0.9
3																
4			0.058	-0.7	0.275	-0.4	0.063	0.6					0.064	-0.3		
5	0.0305	-1.5	0.0309	-2.2	0.132	-2.3	0.0241	-2.2	0.02	-2.3	0.0309	-1.8	0.0259	-2.5	0.0094	-1.5
6																
9	0.042	-0.5	0.072	0.1	0.304	-0.1	0.069	1.0	0.042	-0.4	0.066	0.6	0.074	0.3	FN	-3.5
11	0.05	0.1	0.069	-0.1	0.321	0.2	0.058	0.2	0.046	-0.1	0.062	0.3	0.075	0.4	0.017	0.6
12											0.0315	-1.8			FN	-3.5
14	0.036	-1.0	0.071	0.0	0.205	-1.3	0.05	-0.3	0.086	3.3	0.048	-0.7	0.059	-0.6	0.01	-1.3
15	0.048	0.0	0.071	0.0	0.346	0.5	0.043	-0.9	0.044	-0.3	0.061	0.3	0.076	0.4	0.013	-0.5
17	0.0587	0.9	0.083	0.7	0.445	1.8	0.0637	0.7	0.0487	0.2	0.072	1.0	0.0823	0.8	0.0167	0.5
18					0.36	0.7	0.057	0.2			0.055	-0.2	0.084	0.9	FN	-3.5
19	0.048	0.0	0.076	0.3	0.36	0.7	0.058	0.2	0.056	0.8	0.065	0.5	0.064	-0.3	0.019	1.1
20	0.182	>5	0.07	0.0	0.304	-0.1	0.06	0.4	0.069	1.9	0.054	-0.2	0.079	0.6	FN	-3.5
21	FN	-3.2	0.173	>5	0.75	>5	FN	-3.3	0.088	3.5	0.162	>5	0.186	>5	0.089	20.0
22	0.0538	0.5	0.0783	0.4	0.422	1.5	0.0634	0.6	0.0527	0.5	0.0617	0.3	0.0891	1.2	0.0176	0.7
23	0.041	-0.6	0.07	0.0	0.277	-0.4	0.055	0.0	0.036	-0.9	0.069	0.8	0.06	-0.5	0.01	-1.3
24			0.06	-0.6			0.049	-0.4								
25	0.062	1.1	0.123	3.0	0.373	0.8			0.096	4.2	0.07	0.9	0.072	0.2	FN	-3.5
26	0.0637	1.3	0.076	0.3	0.424	1.5	0.0705	1.2	0.0472	0.0	0.0698	0.9	0.064	-0.3	0.0184	1.0
27					0.344	0.5	0.0544	0.0					0.0748	0.3	0.0117	-0.8
28	0.064	1.3	0.081	0.6	0.573	3.4	0.074	1.4	0.054	0.6	0.066	0.6	0.191	>5		
29	0.0351	-1.1	0.0469	-1.3	0.219	-1.2	0.0334	-1.6	0.0291	-1.5	0.0411	-1.1	0.0479	-1.2	0.0104	-1.2
30	0.053	0.4	0.069	-0.1	0.269	-0.5	0.059	0.3	0.049	0.2	0.06	0.2	0.071	0.1	0.018	0.9
31	0.044	-0.4	0.07	0.0	0.33	0.3	0.06	0.4	0.045	-0.2	0.065	0.5	0.071	0.1	0.012	-0.8
33	0.047	-0.1	0.065	-0.3	0.31	0.0	0.052	-0.2	0.097	4.3	0.061	0.3	0.064	-0.3	0.016	0.3
34	0.062	1.1	0.074	0.2	0.333	0.3	0.056	0.1	0.049	0.2	0.064	0.5	0.074	0.3	0.017	0.6
35	0.055	0.6	0.1	1.7	0.33	0.3	0.065	0.8	0.056	0.8			0.097	1.6		
36	FN	-3.2	0.0758	0.3	0.32	0.2	0.048	-0.5	0.0545	0.6	0.0608	0.2	0.0668	-0.1	FN	-3.5
37	0.0432	-0.4	0.0425	-1.6							0.0305	-1.9			0.0046	-2.8
38	0.0466	-0.1	0.081	0.6	0.324	0.2	0.0604	0.4	0.0614	1.2	0.0715	1.0	0.0645	-0.3	0.0152	0.1
39																
40					0.349	0.5	0.062	0.5					0.076	0.4	0.0132	-0.4
41					0.422	1.5	0.078	1.7			0.085	1.9	0.089	1.2	FN	-3.5

Table 10b. Results for acephate, acetamiprid, axoxystrobin, buprofezin, carbendazim, carbofuran, cyproconazole, and dichlorvos in mg/kg, and the corresponding z scores, MRRLs and the assigned values.

Laboratory code	Acephate	Z-scores (FFP RSD (25%))	Acetamiprid	Z-scores (FFP RSD (25%))	Azoxystrobin	Z-scores (FFP RSD (25%))	Buprofezin	Z-scores (FFP RSD (25%))	Carbendazim	Z-scores (FFP RSD (25%))	Carbofuran	Z-scores (FFP RSD (25%))	Cyproconazole	Z-scores (FFP RSD (25%))	Dichlorvos	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01		0.01		0.005		0.01		0.005	
Assigned value	0.048		0.070		0.308		0.055		0.047		0.057		0.069		0.015	
42	0.056	0.6	0.084	0.8	0.242	-0.9	0.058	0.2	0.056	0.8	0.077	1.4	0.064	-0.3	FN	-3.5
44	0.0584	0.8	0.0727	0.1	0.355	0.6	0.0512	-0.3	0.0572	0.9	0.0663	0.6	0.0736	0.3	FN	-3.5
45			0.065	-0.3	0.275	-0.4	0.059	0.3	0.051	0.3	0.062	0.3				
46					0.33	0.3	0.054	-0.1					0.073	0.2	0.011	-1.0
47					0.272	-0.5	0.048	-0.5	0.0475	0.0			0.068	-0.1	0.014	-0.2
48	0.047	-0.1	0.05	-1.2	0.25	-0.8	0.037	-1.3	0.036	-0.9	0.049	-0.6	0.056	-0.7	0.016	0.3
49	0.0536	0.4	0.0813	0.6	0.338	0.4	0.0652	0.8	0.0462	-0.1	0.0608	0.2	0.0803	0.7	0.0198	1.3
50	0.021	-2.3	0.099	1.6	0.379	0.9	0.057	0.2	0.057	0.9	0.046	-0.8	0.078	0.5	0.025	2.7
51	0.0476	-0.1	0.0677	-0.2	0.332	0.3			0.0567	0.8			0.0773	0.5		
52	0.04	-0.7	0.055	-0.9	0.332	0.3	0.039	-1.1	0.043	-0.3	0.049	-0.6	0.06	-0.5	0.026	3.0
53	0.048	0.0	0.068	-0.1	0.278	-0.4	0.046	-0.6	0.049	0.2	0.063	0.4	0.052	-1.0	0.014	-0.2
54					0.355	0.6	0.0565	0.1					0.0769	0.5	0.0127	-0.6
55	0.044	-0.4	0.073	0.1	0.28	-0.4	0.056	0.1	0.049	0.2	0.051	-0.4	0.07	0.1	FN	-3.5
57	0.044	-0.4	0.067	-0.2	0.338	0.4	0.055	0.0	0.044	-0.3	0.061	0.3	0.075	0.4	0.015	0.0
58	0.045	-0.3	0.06	-0.6	0.275	-0.4	0.039	-1.1	0.039	-0.7	0.055	-0.2	0.068	-0.1	0.016	0.3
59	0.035	-1.1	0.065	-0.3	0.297	-0.1	0.04	-1.1	0.032	-1.3	0.045	-0.9	0.043	-1.5	0.026	3.0
60	0.044	-0.4	0.074	0.2	0.289	-0.3	0.053	-0.1	0.043	-0.3	0.057	0.0	0.07	0.1	0.013	-0.5
61	0.045	-0.3	0.069	-0.1	0.316	0.1	0.058	0.2	0.027	-1.7	0.05	-0.5	0.069	0.0	0.013	-0.5
62	0.085	3.0	0.094	1.3	0.292	-0.2	0.049	-0.4	0.079	2.7	0.067	0.7	0.064	-0.3	FN	-3.5
63	0.054	0.5	0.07	0.0	0.305	0.0	0.06	0.4	0.052	0.4	0.083	1.8	0.074	0.3	0.013	-0.5
64	0.057	0.7	0.059	-0.7	0.604	3.8	0.054	-0.1	0.037	-0.8	FN	-3.7	0.021	-2.8	FN	-3.5
65	0.0404	-0.7	0.0696	0.0	0.33	0.3	0.0435	-0.8	0.0388	-0.7	0.0555	-0.1	0.0864	1.0	0.0267	3.2
66					0.316	0.1	0.0559	0.1					0.0699	0.1	0.0121	-0.7
67	0.0457	-0.2	0.0758	0.3	0.305	0.0	0.0558	0.1	0.0482	0.1	0.0554	-0.1	0.0639	-0.3	0.0148	0.0
68					0.298	-0.1	0.051	-0.3					0.0695	0.0	0.0136	-0.3
69	0.0475	-0.1	0.0652	-0.3	0.334	0.3	0.0547	0.0	0.0447	-0.2	0.0584	0.1	0.0717	0.2	0.0157	0.2
70	0.04	-0.7	0.091	1.2	0.389	1.0	0.055	0.0	0.051	0.3	0.057	0.0	0.047	-1.3	FN	-3.5
71	0.0411	-0.6	0.0694	-0.1	0.323	0.2	0.0571	0.2	0.0382	-0.7	0.0451	-0.9	0.0701	0.1	FN	-3.5
72	0.0477	-0.1	0.0748	0.2	0.3584	0.6	0.0593	0.3	0.0529	0.5	0.0492	-0.6	0.0799	0.6	0.014	-0.2
73			0.029	-2.4	0.24	-0.9	0.028	-2.0	0.072	2.1	0.026	-2.2	0.039	-1.7	0.02	1.4
74			0.074	0.2	0.364	0.7	0.053	-0.1	0.043	-0.3	0.039	-1.3			FN	-3.5
75	0.049	0.1	0.075	0.3	0.34	0.4	0.069	1.0	0.048	0.1	0.077	1.4	0.098	1.7	FN	-3.5
76	0.055	0.6	0.07	0.0	0.4	1.2	0.08	1.9	0.05	0.3	0.035	-1.6	0.07	0.1	0.015	0.0
77	0.05	0.1	0.062	-0.5	0.313	0.1	0.0565	0.1	0.0407	-0.5	0.0555	-0.1	0.0646	-0.3	0.017	0.6
78	0.039	-0.8	0.0631	-0.4	0.332	0.3	0.0422	-0.9	0.0646	1.5	0.0486	-0.6	0.0582	-0.6	0.0115	-0.9

Table 10c. Results for acephate, acetamiprid, axoxystrobin, buprofezin, carbendazim, carbofuran, cyproconazole, and dichlorvos in mg/kg, and the corresponding z scores, MRRLs and the assigned values.

Laboratory code	Acephate	Acetamiprid		Azoxystrobin		Buprofezin		Carbendazim		Carbofuran		Cyproconazole		Dichlorvos		
	MRRL	Z-scores (FFP RSD (25%))	0.01	Z-scores (FFP RSD (25%))	0.01	Z-scores (FFP RSD (25%))	0.01	Z-scores (FFP RSD (25%))	0.01	Z-scores (FFP RSD (25%))	0.005	Z-scores (FFP RSD (25%))	0.01	Z-scores (FFP RSD (25%))	0.005	
Assigned value	0.048		0.070		0.308		0.055		0.047		0.057		0.069		0.015	
79	0.037	-0.9	0.075	0.3	0.303	-0.1	0.06	0.4	0.062	1.3	0.06	0.2	0.074	0.3	0.013	-0.5
80	0.04	-0.7	0.066	-0.3	0.422	1.5	0.059	0.3	0.03	-1.4	0.079	1.5	0.062	-0.4	0.015	0.0
82	0.064	1.3	0.073	0.1	0.38	0.9	0.069	1.0	0.06	1.1	0.074	1.2	0.081	0.7	0.016	0.3
83	0.0571	0.7	0.0805	0.6	0.285	-0.3	0.0692	1.1	0.0504	0.3	0.0695	0.8	0.0597	-0.5	0.0138	-0.3
85	0.045	-0.3	0.075	0.3	0.396	1.1	0.052	-0.2	0.06	1.1	0.046	-0.8	0.076	0.4	0.017	0.6
86	0.0608	1.0	0.0702	0.0	0.358	0.6	0.055	0.0	0.045	-0.2	0.076	1.3	0.0766	0.4	0.012	-0.8
87	0.152	>5	0.069	-0.1	0.307	0.0	0.051	-0.3	0.045	-0.2	0.069	0.8	0.07	0.1	0.014	-0.2
88	0.049	0.1	0.072	0.1	0.335	0.3	0.059	0.3	0.046	-0.1	0.067	0.7	0.076	0.4	0.017	0.6
89	0.0429	-0.4	0.0693	-0.1	0.314	0.1	0.0461	-0.6	0.0374	-0.8	0.0574	0.0	0.0673	-0.1	0.0138	-0.3
90	0.042	-0.5	0.069	-0.1	0.242	-0.9	0.051	-0.3	0.162	>5	0.037	-1.4	0.051	-1.0	FN	-3.5
91	0.054	0.5	0.075	0.3	0.336	0.4	0.068	1.0	0.048	0.1	0.06	0.2	0.076	0.4	0.017	0.6
92	0.055	0.6	0.096	1.4	0.369	0.8	0.053	-0.1	0.046	-0.1	0.071	1.0	0.089	1.2	0.014	-0.2
93							0.051	-0.3							0.017	0.6
94	0.05	0.1	0.097	1.5	0.312	0.0	0.061	0.5	0.04	-0.6	0.038	-1.3	0.069	0.0	FN	-3.5
95	0.043	-0.4	0.052	-1.0	0.25	-0.8	0.054	-0.1	0.038	-0.8	0.066	0.6	0.068	-0.1	0.015	0.0
96			0.185	>5	0.996	>5	0.183	>5	0.124	>5	0.168	>5	0.22	>5	0.0313	4.4
97	0.048	0.0	0.063	-0.4	0.294	-0.2	0.052	-0.2	0.049	0.2	0.04	-1.2	0.063	-0.3	0.018	0.9
98	0.048	0.0	0.059	-0.7	0.34	0.4	0.056	0.1	0.046	-0.1	0.059	0.1	0.052	-1.0	0.005	-2.7
99	0.048	0.0	0.06	-0.6	0.276	-0.4	0.048	-0.5	0.041	-0.5	0.055	-0.2	0.06	-0.5	0.014	-0.2
100	0.0465	-0.2	0.0715	0.1	0.336	0.4	0.0742	1.4	0.0459	-0.1	FN	-3.7	0.0901	1.2	FN	-3.5
101	0.056	0.6	0.068	-0.1	0.324	0.2	0.051	-0.3	0.049	0.2	0.052	-0.4	0.062	-0.4	0.009	-1.6
102																
103	0.034	-1.2	0.074	0.2	0.246	-0.8	0.04	-1.1	0.055	0.7	0.045	-0.9	0.066	-0.2	FN	-3.5
104	0.0388	-0.8	0.0659	-0.3	0.282	-0.3	0.054	-0.1	0.0504	0.3	0.0598	0.2	0.0605	-0.5	0.0121	-0.7
105	0.065	1.4	0.074	0.2	0.103	-2.7	0.05	-0.3	0.053	0.5	0.063	0.4	0.072	0.2	FN	-3.5
106	0.058	0.8	0.0624	-0.5	0.266	-0.5	0.0425	-0.9	0.0529	0.5	0.0609	0.2	0.0529	-0.9	0.016	0.3
107	0.03	-1.5	0.055	-0.9	0.31	0.0	0.052	-0.2	0.045	-0.2	0.055	-0.2	0.061	-0.5	FN	-3.5
108	0.0505	0.2	0.09	1.1	0.347	0.5	0.071	1.2	0.054	0.6	0.073	1.1	0.078	0.5	0.019	1.1
109					0.256	-0.7					0.068	0.7	0.064	-0.3		
110	0.07	1.8	0.069	-0.08	0.23	-1.02	0.053	-0.12	0.037	-0.85	0.05	-0.51	0.07	0.06	0.023	2.21
111	0.0392	-0.8	0.0677	-0.2	0.227	-1.1	0.0511	-0.3	0.0485	0.1	0.0548	-0.2	0.0682	0.0	0.016	0.3
112	0.0382	-0.8	0.0666	-0.2	0.334	0.3	0.0578	0.2	0.0446	-0.2	0.0524	-0.3	0.0708	0.1	0.0062	-2.3
113	0.0478	0.0	0.0673	-0.2	0.292	-0.2	0.0554	0.1	0.0458	-0.1	0.0487	-0.6	0.0693	0.0	0.0135	-0.4
114	FN	-3.2	0.096	1.4	0.257	-0.7	0.088	2.4	0.115	>5	FN	-3.7	0.091	1.3	FN	-3.5

Table 10d. Results for acephate, acetamiprid, axoxystrobin, buprofezin, carbendazim, carbofuran, cyproconazole, and dichlorvos in mg/kg, and the corresponding z scores, MRRs and the assigned values.

Laboratory code	Acephate	Z-scores (FFP RSD (25%))	Acetamiprid	Z-scores (FFP RSD (25%))	Azoxystrobin	Z-scores (FFP RSD (25%))	Buprofezin	Z-scores (FFP RSD (25%))	Carbendazim	Z-scores (FFP RSD (25%))	Carbofuran	Z-scores (FFP RSD (25%))	Cyproconazole	Z-scores (FFP RSD (25%))	Dichlorvos	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01		0.01		0.005		0.01		0.005	
Assigned value	0.048		0.070		0.308		0.055		0.047		0.057		0.069		0.015	
115	0.048	0.0	0.057	-0.8	0.27	-0.5	0.06	0.4	0.04	-0.6	0.06	0.2	0.062	-0.4	0.012	-0.8
116	0.0523	0.3	0.0721	0.1	0.282	-0.3	0.05	-0.3	0.0476	0.1	0.0634	0.4	0.0555	-0.8	0.0154	0.2
117	0.0511	0.2	0.0681	-0.1	0.305	0.0	0.0534	-0.1	0.045	-0.2	0.0609	0.2	0.0703	0.1	0.0147	0.0
118	0.049	0.1	0.074	0.2	0.26	-0.6	0.049	-0.4	0.049	0.2	0.057	0.0	0.067	-0.1	0.013	-0.5
119					0.357	0.6	0.0539	-0.1					0.069	0.0	0.0117	-0.8
120	0.067	1.5	0.075	0.3	0.21	-1.3	0.053	-0.1	0.05	0.3	0.064	0.5	0.065	-0.2	0.015	0.0
121																
122																
123	0.045	-0.3	0.064	-0.4	0.322	0.2	0.056	0.1	0.037	-0.8	0.047	-0.7	0.065	-0.2	0.017	0.6
124	0.04	-0.7	0.08	0.5	0.34	0.4	0.06	0.4	0.05	0.3	0.05	-0.5	0.06	-0.5	0.02	1.4
125	0.071	1.9	0.092	1.2	0.389	1.0	0.08	1.9	0.041	-0.5	0.059	0.1	0.094	1.5	0.014	-0.2
126	0.049	0.1	0.082	0.7	0.353	0.6	0.058	0.2	0.076	2.5	0.034	-1.6	0.084	0.9	0.019	1.1
127	0.043	-0.4	0.068	-0.1	0.305	0.0	0.052	-0.2	0.039	-0.7	0.048	-0.7	0.063	-0.3	0.012	-0.8
128																
129	0.039	-0.8	0.0811	0.6	0.281	-0.4	0.0573	0.2	0.0438	-0.3	0.0642	0.5	0.0708	0.1	0.0107	-1.1
130	0.046	-0.2	0.104	1.9	0.63	4.2	0.088	2.4	0.065	1.5	0.085	1.9	0.09	1.2	FN	-3.5
131	0.05	0.1	0.074	0.2	0.308	0.0	0.059	0.3	0.05	0.3	0.061	0.3	0.068	-0.1	0.017	0.6
132	0.051	0.2	0.051	-1.1	0.385	1.0	0.045	-0.7	0.042	-0.4	0.08	1.6	0.061	-0.5	0.005	-2.7
133	0.045	-0.3	0.061	-0.5	0.297	-0.1	0.062	0.5	0.047	0.0	0.055	-0.2	0.067	-0.1	0.014	-0.2
134	0.039	-0.8	0.046	-1.4	0.245	-0.8	0.045	-0.7	0.024	-2.0	0.048	-0.7	0.061	-0.5	0.014	-0.2
135																
136	0.08	2.6	0.065	-0.3	0.225	-1.1	0.047	-0.6	0.05	0.3	0.06	0.2	0.046	-1.3	0.015	0.0
137	0.047	-0.1	0.08	0.5	0.253	-0.7	0.053	-0.1	0.033	-1.2	0.042	-1.1	0.034	-2.0	FN	-3.5
138																
139	0.047	-0.1	0.083	0.7	0.335	0.3	0.092	2.7	0.052	0.4	0.066	0.6	0.076	0.4	0.02	1.4
140																
141																
142	0.05	0.1	0.05	-1.2	0.242	-0.9			0.0325	-1.2			0.059	-0.6	FN	-3.5
143					0.22	-1.1	0.047	-0.6							FN	-3.5
144	0.049	0.1	0.065	-0.3	0.253	-0.7	0.047	-0.6	0.034	-1.1	0.036	-1.5	0.072	0.2	0.021	1.7
145					FN	-3.9										
146			0.0814	0.6	0.254	-0.7	0.0564	0.1	0.0329	-1.2	0.0785	1.5	0.0605	-0.5	FN	-3.5
147	0.047	-0.1	0.056	-0.8	0.253	-0.7	0.034	-1.5	0.042	-0.4			0.055	-0.8		
148	0.069	1.7	0.079	0.5	0.336	0.4	0.068	1.0	0.047	0.0	0.066	0.6	0.158	>5	0.018	0.9
149	0.034	-1.2	0.056	-0.8	0.2	-1.4	0.046	-0.6	0.036	-0.9	0.052	-0.4	0.059	-0.5	0.016	0.3

Table 10e. Results for acephate, acetamidrid, axoxystrobin, buprofezin, carbendazim, carbofuran, cyproconazole, and dichlorvos in mg/kg, and the corresponding z scores, MRRLs and the assigned values.

Laboratory code	Acephate	Z-scores (FFP RSD (25%))	Acetamidrid	Z-scores (FFP RSD (25%))	Azoxystrobin	Z-scores (FFP RSD (25%))	Buprofezin	Z-scores (FFP RSD (25%))	Carbendazim	Z-scores (FFP RSD (25%))	Carbofuran	Z-scores (FFP RSD (25%))	Cyproconazole	Z-scores (FFP RSD (25%))	Dichlorvos	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01		0.01		0.005		0.01		0.005	
Assigned value	0.048		0.070		0.308		0.055		0.047		0.057		0.069		0.015	
150	0.036	-1.0	0.059	-0.6	0.288	-0.2	0.043	-0.8	0.042	-0.3	0.051	-0.4	0.054	-0.8	FN	-3.5
151	0.0995	4.3	0.0947	1.4	0.3834	1.0			0.3823	>5	0.0657	0.6	0.0987	1.8	FN	-3.5
152	0.177	>5	0.074	0.2	0.308	0.0	0.052	-0.2	0.02	-2.3	0.053	-0.3	0.04	-1.7	0.01	-1.3
153															0.016	0.3
154			0.0484	-1.2	0.185	-1.6	0.04	-1.1	0.0324	-1.2	0.044	-0.9	0.0514	-1.0	FN	-3.5
155	0.044	-0.4	0.074	0.2	0.28	-0.3	0.055	0.1	0.039	-0.6	0.057	0.0	0.067	-0.1	0.013	-0.5
156	0.063	1.2	0.13	3.4	0.69	>5	0.1	3.4			0.1	3.0	0.12	3.0		
157	0.049	0.1	0.098	1.6	0.424	1.6	0.052	-0.2	0.075	2.6	0.081	1.7	0.073	0.3	0.014	-0.2
158	FN	-3.2	0.0254	-2.5	0.333	0.4	0.0506	-0.3			0.053	-0.3	0.0678	0.0	FN	-3.5
159	0.031	-1.4	0.039	-1.8	0.171	-1.8	0.033	-1.6	0.023	-2.0	0.036	-1.5	0.036	-1.9		
160																
161	0.033	-1.3	0.056	-0.8	0.26	-0.6	0.048	-0.5	0.033	-1.1	0.048	-0.6	0.056	-0.7	0.011	-1.0
162	0.0492	0.1	0.0637	-0.4	0.328	0.3	0.0595	0.4	0.0424	-0.3	0.0551	-0.1	0.0665	-0.1	0.0098	-1.4
163	0.037	-0.9	0.074	0.2	0.27	-0.5	0.049	-0.4	0.046	0.0	0.056	-0.1	0.072	0.2	FN	-3.5
164			0.068	-0.1	0.33	0.3	0.049	-0.4	0.048	0.2	0.095	2.7	0.071	0.2	0.021	1.7
165	0.05	0.1	0.074	0.2	0.294	-0.1	0.054	0.0	0.048	0.2	0.062	0.3	0.07	0.1	0.014	-0.2
166	0.029	-1.6	0.053	-1.0	0.332	0.4	0.06	0.4	0.022	-2.1	0.066	0.6	0.07	0.1	0.015	0.0
167	0.0473	-0.1	0.0504	-1.1	0.214	-1.2	0.0439	-0.8	0.0258	-1.7	0.0344	-1.6	0.0706	0.1	0.0221	2.0
169	0.029	-1.6	0.03	-2.3	0.33	0.3	0.051	-0.2	0.023	-2.0	0.007	-3.5	0.069	0.0	0.016	0.3
170	0.05	0.1	0.08	0.6	0.221	-1.1	0.048	-0.5	0.034	-1.0	0.052	-0.4	0.051	-1.0	0.013	-0.5
171	0.051	0.2	0.083	0.7	0.34	0.5	0.053	-0.1	0.064	1.6	0.05	-0.5	0.069	0.0	0.014	-0.2
172	0.045	-0.3	0.084	0.8	0.28	-0.3	0.06	0.4	0.05	0.4	0.058	0.1	0.064	-0.3	0.0086	-1.7
173																
174			0.09	1.1	0.42	1.5	0.1	3.4	0.1	4.7	0.07	0.9	FN	-3.4	0.01	-1.3
175	0.046	-0.2	0.07	0.0	0.31	0.1	0.054	0.0	0.052	0.5	0.057	0.0	0.084	0.9	0.011	-1.0

Table 11a. Results for difenoconazole, hexaconazole, imidacloprid, isoprothiolane, metalaxyl, profenofos, pyriproxyfen, and thiametoxam in mg/kg, and the corresponding z scores, MRRLs and the assigned values.

Laboratory code	Difenoconazole	Hexaconazole		Imidacloprid		Isoprothiolane		Metaxyl	Profenofos		Pyriproxyfen		Thiametoxam			
MRRL	0.01	Z-scores (FFP RSD (25%))	0.01	Z-scores (FFP RSD (25%))	0.01	Z-scores (FFP RSD (25%))	0.01	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	0.01	Z-scores (FFP RSD (25%))	0.01	Z-scores (FFP RSD (25%))	0.01		
Assigned value	0.048		0.090		0.069		0.404		0.073		0.205		0.153		0.054	
1	0.057	0.7	0.0859	-0.2	0.0636	-0.3	0.406	0.0	0.063	-0.6	0.194	-0.2	0.132	-0.6	0.057	0.2
2	0.051	0.2	0.087	-0.1	0.068	0.0	0.437	0.3	0.087	0.8	0.235	0.6	0.208	1.4	0.056	0.2
3																
4	0.058	0.8	0.094	0.2	0.067	-0.1			0.07	-0.2	0.227	0.4	0.152	0.0	0.06	0.5
5	0.0346	-1.1	0.0662	-1.1	0.0319	-2.1	0.187	-2.1	0.035	-2.1	0.107	-1.9	0.0758	-2.0	0.0262	-2.0
6																
9	0.045	-0.3	0.071	-0.9	0.111	2.5			0.072	-0.1			0.16	0.2	0.06	0.5
11	0.051	0.2	0.083	-0.3	0.068	0.0	0.437	0.3	0.077	0.2	0.21	0.1	0.155	0.0	0.05	-0.3
12											0.203	0.0				
14	0.033	-1.3	0.084	-0.3	0.049	-1.2	0.279	-1.2	0.041	-1.8	0.157	-0.9	0.156	0.1	0.085	2.3
15	0.049	0.0	0.074	-0.7	0.07	0.1	0.373	-0.3	0.068	-0.3	0.191	-0.3	0.157	0.1	0.053	-0.1
17	0.0557	0.6	0.103	0.6	0.0787	0.6	0.476	0.7	0.0933	1.1	0.227	0.4	0.18	0.7	0.0567	0.2
18	0.056	0.6	0.098	0.3			0.45	0.5	0.083	0.5	0.21	0.1	0.15	-0.1		
19	0.056	0.6	0.1	0.4	0.066	-0.2	0.42	0.2	0.086	0.7	0.22	0.3	0.19	1.0	0.053	-0.1
20	0.053	0.4	0.092	0.1	0.056	-0.7			0.082	0.5	0.305	1.9	0.161	0.2	0.048	-0.4
21	FN	-3.2	0.225	>5	0.27	>5			0.18	>5			0.266	2.9	0.18	>5
22	0.0467	-0.1	0.103	0.6	0.064	-0.3	0.555	1.5	0.0899	0.9	0.248	0.8	0.1915	1.0	0.0555	0.1
23	0.039	-0.8	0.08	-0.5	0.077	0.5	0.362	-0.4	0.067	-0.3	0.231	0.5	0.182	0.7	0.045	-0.6
24	0.037	-0.9														
25	0.051	0.2	0.276	>5	0.126	3.3	0.289	-1.1	0.073	0.0	0.183	-0.4	0.167	0.4	0.091	2.8
26	0.0538	0.4	0.119	1.3	0.075	0.4	0.569	1.6	0.0839	0.6	0.253	0.9	0.196	1.1	0.0587	0.4
27	0.0466	-0.1	0.106	0.7			0.397	-0.1								
28	0.063	1.2	0.107	0.7	0.064	-0.3			0.102	1.6	0.298	1.8	0.186	0.9	0.066	0.9
29	0.0326	-1.3	0.0593	-1.4	0.042	-1.6	0.297	-1.1	0.0569	-0.9	0.13	-1.5	0.116	-1.0	0.0334	-1.5
30	0.045	-0.3	0.093	0.1	0.077	0.5	0.419	0.1	0.072	-0.1	0.241	0.7	0.163	0.3	0.081	2.0
31	0.049	0.0	0.098	0.3	0.067	-0.1	0.45	0.5	0.083	0.5	0.2	-0.1	0.15	-0.1	0.048	-0.4
33	0.046	-0.2	0.084	-0.3	0.06	-0.5	0.397	-0.1	0.073	0.0	0.193	-0.2	0.163	0.3	0.048	-0.4
34	0.052	0.3	0.097	0.3	0.06	-0.5	0.424	0.2	0.08	0.4	0.25	0.9	0.169	0.4	0.044	-0.7
35	0.068	1.6	0.12	1.3	0.091	1.3			0.11	2.0	0.31	2.0	0.18	0.7	0.066	0.9
36	0.0536	0.4	0.0962	0.3	0.067	-0.1	0.369	-0.3	0.0534	-1.1	0.189	-0.3	0.126	-0.7	0.0717	1.3
37					0.0696	0.0					0.196	-0.2			0.0585	0.4
38	0.0586	0.8	0.0945	0.2	0.0761	0.4	0.577	1.7	0.108	1.9	0.213	0.1	0.162	0.2	0.0691	1.1
39																
40	0.0544	0.5	0.101	0.5			0.402	0.0								
41	0.065	1.4	FN	-3.6			FN	-3.9	0.105	1.7	FN	-3.8	0.173	0.5		

Table 11b. Results for difenoconazole, hexaconazole, imidacloprid, isoprothiolane, metalaxyl, profenofos, pyriproxyfen, and thiametoxam in mg/kg, and the corresponding z scores, MRRs and the assigned values.

Laboratory code	Difenoconazole	Hexaconazole		Imidacloprid		Isoprothiolane		Metalaxyl		Profenofos		Pyriproxyfen		Thiametoxam		
MRR	0.01	0.01		0.01		0.01		0.01		0.01		0.01		0.01		
Assigned value	0.048	0.090		0.069		0.404		0.073		0.205		0.153		0.054		
		Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	
42	0.036	-1.0	0.085	-0.2	0.081	0.7	0.487	0.8	0.064	-0.5	0.199	-0.1	0.135	-0.5	0.06	0.5
44	0.0517	0.3	0.109	0.8	0.0604	-0.5	0.331	-0.7	0.0778	0.3	0.246	0.8	0.162	0.2	0.0662	0.9
45	0.041	-0.6			0.077	0.5			0.065	-0.4	0.26	1.1	0.174	0.5	0.057	0.2
46	0.049	0.0	0.091	0.0			0.38	-0.2								
47	0.043	-0.4	0.085	-0.2			0.384	-0.2	0.077	0.2	0.186	-0.4	0.133	-0.5		
48	0.04	-0.7	0.071	-0.9	0.05	-1.1	0.38	-0.2	0.054	-1.0	0.15	-1.1	0.1	-1.4	0.041	-0.9
49	0.0536	0.4	0.0991	0.4	0.0728	0.2	0.428	0.2	0.0892	0.9	0.231	0.5	0.162	0.2	0.0568	0.2
50	0.048	0.0	0.105	0.7	0.086	1.0	0.486	0.8	0.093	1.1	0.22	0.3	0.187	0.9	0.057	0.2
51	0.06	1.0	0.0865	-0.2	0.0619	-0.4	0.432	0.3	0.0738	0.0	0.221	0.3	0.14	-0.3	0.0507	-0.2
52	0.039	-0.8	0.074	-0.7	0.052	-1.0	0.374	-0.3	0.059	-0.8	0.178	-0.5	0.119	-0.9	0.048	-0.4
53	0.037	-0.9	0.07	-0.9	0.07	0.1	0.234	-1.7	0.045	-1.5	0.165	-0.8	0.129	-0.6	0.057	0.2
54	0.051	0.2	0.0972	0.3			0.381	-0.2								
55	0.053	0.4	0.088	-0.1	0.061	-0.5	0.43	0.3	0.08	0.4	0.21	0.1	0.13	-0.6	0.052	-0.1
57	0.047	-0.1	0.098	0.3	0.074	0.3	0.523	1.2	0.077	0.2	0.266	1.2	0.15	-0.1	0.07	1.2
58	0.047	-0.1	0.088	-0.1	0.062	-0.4	0.39	-0.1	0.069	-0.2	0.232	0.5	0.141	-0.3	0.047	-0.5
59	0.049	0.0	0.112	1.0	0.069	0.0	0.397	-0.1	0.067	-0.3	0.238	0.6	0.158	0.1	0.042	-0.9
60	0.053	0.4	0.088	-0.1	0.071	0.1	0.433	0.3	0.063	-0.6	0.194	-0.2	0.114	-1.0	0.068	1.1
61	0.047	-0.1	0.088	-0.1	0.073	0.2	0.429	0.2	0.077	0.2	0.209	0.1	0.149	-0.1	0.058	0.3
62	0.037	-0.9	0.107	0.7	0.103	2.0	0.438	0.3	0.078	0.3	0.221	0.3	0.152	0.0	0.078	1.8
63	0.041	-0.6	0.094	0.2	0.073	0.2	0.369	-0.3	0.075	0.1	0.21	0.1	0.173	0.5	0.058	0.3
64	0.064	1.3	FN	-3.6	0.061	-0.5	0.52	1.1	0.086	0.7	0.084	-2.4	0.254	2.6	0.038	-1.2
65	0.0511	0.2	0.101	0.5	0.0681	0.0	0.382	-0.2	0.0718	-0.1	0.205	0.0	0.166	0.3	0.041	-0.9
66	0.0493	0.1	0.0971	0.3			0.357	-0.5								
67	0.0446	-0.3	0.0769	-0.6	0.0738	0.3	0.387	-0.2	0.076	0.2	0.197	-0.2	0.137	-0.4	0.0562	0.2
68	0.0524	0.3	0.0958	0.2			0.36	-0.4								
69	0.0564	0.7	0.094	0.2	0.0758	0.4	0.452	0.5	0.0827	0.5	0.247	0.8	0.177	0.6	0.0533	0.0
70	0.076	2.3	0.085	-0.2	0.083	0.8	0.468	0.6	0.089	0.9	0.151	-1.1	FN	-3.7	0.045	-0.6
71	0.0477	-0.1	0.094	0.2	0.0686	0.0	0.458	0.5	0.0715	-0.1	0.201	-0.1	0.149	-0.1	0.0482	-0.4
72	0.0611	1.0	0.0994	0.4	0.0933	1.4	0.443	0.4	0.0853	0.7	0.228	0.4	0.1779	0.6	0.058	0.3
73	0.036	-1.0	0.033	-2.5	0.05	-1.1	0.3	-1.0	0.049	-1.3	0.22	0.3	0.039	-3.0	0.032	-1.6
74	0.108	4.9			0.062	-0.4			0.065	-0.4			0.12	-0.9	0.06	0.5
75	0.057	0.7	0.11	0.9	0.076	0.4	0.6	1.9	0.086	0.7	0.21	0.1	0.19	1.0	0.051	-0.2
76	0.065	1.4	0.12	1.3	0.07	0.1	0.4	0.0	0.07	-0.2	0.25	0.9	0.25	2.5	0.045	-0.6
77	0.0506	0.2	0.0945	0.2	0.0702	0.1	0.439	0.3	0.077	0.2	0.0304	-3.4	0.2	1.2	0.0514	-0.2
78	0.0401	-0.7	0.0677	-1.0	0.0601	-0.5	0.356	-0.5	0.067	-0.3	0.173	-0.6	0.14	-0.3	0.0437	-0.7

Table 11c. Results for difenoconazole, hexaconazole, imidacloprid, isoprothiolane, metalaxyl, profenfos, pyriproxyfen, and thiametoxam in mg/kg, and the corresponding z scores, MRRLs and the assigned values.

Laboratory code	Difenoconazole	Hexaconazole		Imidacloprid		Isoprothiolane		Metalaxyl		Profenfos		Pyriproxyfen		Thiametoxam		
MRRL	0.01	0.01		0.01		0.01		0.01		0.01		0.01		0.01		
Assigned value	0.048	0.090		0.069		0.404		0.073		0.205		0.153		0.054		
		Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	Z-scores (FFP RSD (25%))	
79	0.051	0.2	0.084	-0.3	0.071	0.1	0.454	0.5	0.071	-0.1	0.196	-0.2	0.137	-0.4	0.056	0.2
80	0.058	0.8	0.098	0.3	0.066	-0.2	0.471	0.7	0.075	0.1	0.199	-0.1	0.145	-0.2	0.053	-0.1
82	0.057	0.7	0.109	0.8	0.073	0.2	0.338	-0.7	0.084	0.6	0.283	1.5	0.163	0.3	0.064	0.8
83	0.0466	-0.1	0.0982	0.4	0.0665	-0.1	0.444	0.4	0.0817	0.5	0.354	2.9	0.159	0.1	0.0484	-0.4
85	0.052	0.3	0.085	-0.2	0.075	0.4	0.483	0.8	0.077	0.2	0.16	-0.9	0.15	-0.1	0.059	0.4
86	0.046	-0.2	0.0916	0.1	0.125	3.3	0.455	0.5	0.0862	0.7	0.24	0.7	0.161	0.2	0.0546	0.1
87	0.043	-0.4	0.082	-0.4	0.067	-0.1	0.431	0.3	0.073	0.0	0.169	-0.7	0.16	0.2	0.053	-0.1
88	0.052	0.3	0.096	0.3	0.072	0.2	0.448	0.4	0.081	0.4	0.206	0.0	0.161	0.2	0.056	0.2
89	0.047	-0.1	0.0951	0.2	0.0692	0.0	0.403	0.0	0.0749	0.1	0.199	-0.1	0.107	-1.2	0.0534	0.0
90	0.042	-0.5	0.062	-1.2	0.06	-0.5	0.323	-0.8	0.035	-2.1	0.148	-1.1	0.138	-0.4	0.049	-0.4
91	0.055	0.5	0.096	0.3	0.065	-0.2	0.049	-3.5	0.087	0.8	0.23	0.5	0.23	2.0	0.052	-0.1
92	0.048	0.0	0.1	0.4	0.073	0.2	0.504	1.0	0.067	-0.3	0.268	1.2	0.173	0.5	0.059	0.4
93							0.309	-0.9	0.061	-0.7	0.203	0.0	0.145	-0.2		
94	0.055	0.5	0.089	-0.1	0.07	0.1	0.399	-0.1	0.083	0.5	0.238	0.6	0.15	-0.1	0.064	0.8
95	0.037	-0.9	0.069	-0.9	0.047	-1.3			0.074	0.0	0.17	-0.7	0.15	-0.1	0.04	-1.0
96	0.171	>5			0.0973	1.7					0.544	>5			0.152	>5
97	0.043	-0.4	0.072	-0.8	0.061	-0.5	0.384	-0.2	0.059	-0.8	0.182	-0.5	0.137	-0.4	0.046	-0.6
98	0.038	-0.9	0.07	-0.9	0.057	-0.7			0.06	-0.7	0.195	-0.2	0.148	-0.1	0.052	-0.1
99	0.04	-0.7	0.078	-0.5	0.06	-0.5	0.368	-0.4	0.067	-0.3	0.162	-0.8	0.156	0.1	0.046	-0.6
100	0.035	-1.1	0.109	0.8	0.072	0.2	0.352	-0.5	0.0762	0.2	0.38	3.4	0.187	0.9	0.058	0.3
101	0.047	-0.1	0.095	0.2	0.066	-0.2	0.428	0.2	0.074	0.0	0.19	-0.3	0.143	-0.3	0.051	-0.2
102																
103	0.053	0.4	0.081	-0.4	0.172	>5	0.264	-1.4	0.051	-1.2	0.186	-0.4	0.135	-0.5	0.031	-1.7
104	0.0408	-0.6	0.0909	0.0	0.0636	-0.3	0.426	0.2	0.0587	-0.8	0.181	-0.5	0.147	-0.2	0.0464	-0.5
105	0.041	-0.6	0.091	0.0	0.078	0.5	0.345	-0.6	0.063	-0.6	0.143	-1.2	0.113	-1.1	0.061	0.5
106	0.0355	-1.1	0.0776	-0.6	0.0625	-0.4	0.332	-0.7	0.0667	-0.4	0.154	-1.0	0.117	-0.9	0.0491	-0.3
107	0.04	-0.7	0.062	-1.2	0.061	-0.5	0.43	0.3	0.071	-0.1	0.16	-0.9	0.12	-0.9	0.055	0.1
108	0.065	1.4	0.102	0.5	0.082	0.8	0.493	0.9	0.0925	1.1	0.239	0.7	0.188	0.9	0.07	1.2
109	0.047	-0.1	0.086	-0.2							0.158	-0.9				
110	0.06	1.0	0.11	0.9	0.065	-0.2	0.44	0.4	0.07	-0.2	0.22	0.3	0.12	-0.9	0.05	-0.3
111	0.0452	-0.3	0.0996	0.4	0.0694	0.0	0.535	1.3	0.0712	-0.1	0.244	0.8	0.156	0.1	0.0516	-0.2
112	0.0476	-0.1	0.0932	0.1	0.0608	-0.5	0.429	0.2	0.0748	0.1	0.199	-0.1	0.159	0.1	0.047	-0.5
113	0.0479	0.0	0.0817	-0.4	0.0718	0.2	0.387	-0.2	0.0737	0.0	0.182	-0.5	0.138	-0.4	0.0504	-0.2
114	0.065	1.4	0.089	-0.1	0.164	>5	FN	-3.9	0.077	0.2	FN	-3.8	0.296	3.7	0.142	>5

Table 11d Results for difenoconazole, hexaconazole, imidacloprid, isoprothiolane, metalaxyl, profenofos, pyriproxyfen, and thiametoxam in mg/kg, and the corresponding z scores, MRRRLs and the assigned values.

Laboratory code	Difenoconazole	Z-scores (FFP RSD (25%))	Hexaconazole	Z-scores (FFP RSD (25%))	Imidacloprid	Z-scores (FFP RSD (25%))	Isoprothiolane	Z-scores (FFP RSD (25%))	Metalaxyl	Z-scores (FFP RSD (25%))	Profenofos	Z-scores (FFP RSD (25%))	Pyriproxyfen	Z-scores (FFP RSD (25%))	Thiametoxam	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01		0.01		0.01		0.01		0.01	
Assigned value	0.048		0.090		0.069		0.404		0.073		0.205		0.153		0.054	
115	0.042	-0.5	0.082	-0.4	0.062	-0.4	0.398	-0.1	0.064	-0.5	0.177	-0.6	0.129	-0.6	0.047	-0.5
116	0.0405	-0.7	0.075	-0.7	0.0711	0.1	0.389	-0.2	FN	-3.5	0.194	-0.2	0.139	-0.4	0.0543	0.0
117	0.0467	-0.1	0.0902	0.0	0.0731	0.3	0.393	-0.1	0.0727	0.0	0.196	-0.2	0.154	0.0	0.0499	-0.3
118	0.046	-0.2	0.088	-0.1	0.065	-0.2	0.38	-0.2	0.069	-0.2	0.17	-0.7	0.14	-0.3	0.067	1.0
119	0.047	-0.1	0.0915	0.1			0.388	-0.2								
120	0.038	-0.9	0.11	0.9	0.059	-0.6	0.54	1.3	0.076	0.2	0.209	0.1	0.154	0.0	0.056	0.2
121																
122																
123	0.046	-0.2	0.073	-0.8	0.06	-0.5	0.41	0.1	0.074	0.0	0.211	0.1	0.156	0.1	0.043	-0.8
124	0.05	0.1	0.09	0.0	0.07	0.1	0.41	0.1	0.08	0.4	0.21	0.1	0.15	-0.1	0.05	-0.3
125	0.059	0.9	0.105	0.7	0.081	0.7	0.498	0.9	0.088	0.8	0.258	1.0	0.151	-0.1	0.061	0.5
126	0.056	0.6	0.084	-0.3	0.079	0.6	0.434	0.3	0.076	0.2	0.23	0.5	0.167	0.4	0.058	0.3
127	0.041	-0.6	0.103	0.6	0.06	-0.5	0.408	0.0	0.07	-0.2	0.124	-1.6	0.154	0.0	0.051	-0.2
128																
129	0.0407	-0.6	0.0719	-0.8	0.0762	0.4	0.428	0.2	0.0822	0.5	0.231	0.5	0.159	0.1	0.0559	0.2
130	FN	-3.2	0.145	2.4	0.09	1.2			0.1	1.5	0.25	0.9	0.225	1.9	0.11	4.2
131	0.056	0.6	0.087	-0.1	0.112	2.5	0.426	0.2	0.075	0.1	0.221	0.3	0.147	-0.2	0.084	2.3
132	0.038	-0.9	0.077	-0.6	0.07	0.1	0.32	-0.8	0.049	-1.3	0.202	-0.1	0.14	-0.3	0.05	-0.3
133	0.053	0.4	0.087	-0.1	0.064	-0.3	0.392	-0.1	0.071	-0.1	0.198	-0.1	0.157	0.1	0.048	-0.4
134	0.046	-0.2	0.082	-0.4	0.046	-1.3	0.334	-0.7	0.065	-0.4	0.19	-0.3	0.13	-0.6	0.044	-0.7
135																
136	0.032	-1.4	0.053	-1.6	0.073	0.2	0.267	-1.4	0.053	-1.1	0.13	-1.5	0.115	-1.0	0.056	0.2
137	0.05	0.1	0.092	0.1	0.066	-0.2	0.408	0.0	0.04	-1.8	0.189	-0.3	0.151	-0.1	0.052	-0.1
138																
139	0.075	2.2	0.147	2.5	0.082	0.8	0.463	0.6	0.085	0.6	0.228	0.4	0.174	0.5	0.061	0.5
140																
141																
142	0.044	-0.4	0.0775	-0.6	0.06	-0.5			0.06	-0.7	0.165	-0.8	0.122	-0.8		
143	0.038	-0.9							0.029	-2.4	0.135	-1.4				
144	0.041	-0.6	0.074	-0.7	0.055	-0.8	0.352	-0.5	0.076	0.2	0.122	-1.6	0.104	-1.3	0.047	-0.5
145					FN	-3.4					0.1997	-0.1			FN	-3.3
146	0.054	0.5	0.103	0.6	0.0686	0.0	0.595	1.9	0.057	-0.9	0.225	0.4	0.152	0.0	0.0526	-0.1
147	0.039	-0.8	0.08	-0.5	0.056	-0.7			0.066	-0.4	0.16	-0.9	0.09	-1.7	0.042	-0.9
148	0.102	4.4	0.122	1.4	0.074	0.3	0.434	0.3	0.094	1.1	0.24	0.7	0.234	2.1	0.057	0.2
149	0.038	-0.9	0.067	-1.0	0.06	-0.5	0.35	-0.5	0.054	-1.0	0.15	-1.1	0.14	-0.3	0.044	-0.7

Table 11e Results for difenoconazole, hexaconazole, imidacloprid, isoprothiolane, metalaxyl, profenofos, pyriproxyfen, and thiametoxam in mg/kg, and the corresponding z scores, MRRs and the assigned values.

Laboratory code	Difenoconazole	Z-scores (FFP RSD (25%))	Hexaconazole	Z-scores (FFP RSD (25%))	Imidacloprid	Z-scores (FFP RSD (25%))	Isoprothiolane	Z-scores (FFP RSD (25%))	Metalaxyl	Z-scores (FFP RSD (25%))	Profenofos	Z-scores (FFP RSD (25%))	Pyriproxyfen	Z-scores (FFP RSD (25%))	Thiametoxam	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01		0.01		0.01		0.01		0.01	
Assigned value	0.048		0.090		0.069		0.404		0.073		0.205		0.153		0.054	
150	0.042	-0.5	0.071	-0.9	0.06	-0.5	0.369	-0.3	0.067	-0.3	0.189	-0.3	0.125	-0.7	0.052	-0.1
151	0.0716	1.9	0.1043	0.6	0.0778	0.5			0.0946	1.2	0.2151	0.2			0.0663	0.9
152	0.038	-0.9	0.077	-0.6	0.051	-1.0	0.344	-0.6	0.072	-0.1	0.174	-0.6	0.155	0.0	0.056	0.2
153									0.069	-0.2	0.184	-0.4	0.147	-0.2		
154	0.0325	-1.3	0.0653	-1.1	0.0512	-1.0	0.316	-0.9	0.0547	-1.0	0.313	2.1	0.106	-1.2	0.043	-0.8
155	0.044	-0.4	0.085	-0.2	0.06	-0.5	0.423	0.2	0.073	0.0	0.192	-0.3	0.166	0.3	0.048	-0.4
156	0.09	3.4	0.19	4.4	0.14	4.1			0.15	4.2	0.34	2.6	0.18	0.7		
157	0.048	0.0	0.072	-0.8	0.102	1.9	0.728	3.2	0.072	-0.1	0.278	1.4	0.245	2.4	0.069	1.1
158	0.044	-0.4	FN	-3.6	0.03	-2.3	0.381	-0.2	0.07	-0.2	0.173	-0.6	0.147	-0.2	0.0264	-2.0
159	0.024	-2.0	0.049	-1.8	0.035	-2.0	0.147	-2.5	0.021	-2.9	0.098	-2.1	0.083	-1.8	0.029	-1.8
160																
161	0.038	-0.9	0.08	-0.5	0.055	-0.8	0.36	-0.4	0.057	-0.9	0.21	0.1	0.13	-0.6	0.048	-0.4
162	0.0468	-0.1	0.129	1.7	0.0742	0.3	0.445	0.4	0.0755	0.1	0.276	1.4	0.168	0.4	0.0453	-0.6
163	0.05	0.1	0.078	-0.5	0.068	0.0	0.345	-0.6	0.071	-0.1	0.17	-0.7	0.153	0.0	0.059	0.4
164	0.058	0.8	0.096	0.3	0.062	-0.4	0.385	-0.2	0.071	-0.1	0.226	0.4	0.16	0.2	0.034	-1.5
165	0.05	0.1	0.096	0.3	0.07	0.1	0.422	0.2	0.076	0.2	0.198	-0.1	0.16	0.2	0.056	0.2
166	0.048	0.0	0.091	0.0	0.07	0.1			0.074	0.0	0.211	0.1	0.149	-0.1	0.054	0.0
167	0.0463	-0.2	0.0787	-0.5	0.0565	-0.7	0.3179	-0.9	0.0764	0.2	0.1777	-0.5	0.1393	-0.4	0.075	1.6
169	0.043	-0.4	0.086	-0.2	0.037	-1.8	0.222	-1.8	0.077	0.2	0.297	1.8	0.161	0.2	0.034	-1.5
170	0.042	-0.5	0.089	-0.1	0.087	1.1	0.366	-0.4	0.05	-1.3	0.216	0.2	0.148	-0.1	0.057	0.2
171	0.051	0.2	0.13	1.8	0.084	0.9	0.42	0.2	0.064	-0.5	0.21	0.1	0.2	1.2	0.056	0.2
172	0.051	0.2	0.1	0.4	0.091	1.3	0.38	-0.2	0.11	2.0	0.21	0.1	0.16	0.2	0.055	0.1
173																
174	0.07	1.8	0.26	>5	0.07	0.1	0.14	-2.6	0.09	0.9			0.21	1.5	0.08	2.0
175	0.053	0.4	0.088	-0.1	0.064	-0.3	0.4	0.0	0.087	0.8	0.232	0.5	0.172	0.5	0.049	-0.4

Table 12a Results for voluntary compound DDE-pp, endrin-ketone and oxathiaprolin, in mg/kg, and the corresponding z scores, MRRLs and the assigned values. The results pymetrozin is only shown for informative purposes because the residue level was too low to evaluate.

Laboratory code	DDE-pp	Z-scores (FFP RSD (25%))	Endrin-ketone	Z-scores (FFP RSD (25%))	Oxathiaprolin	Z-scores (FFP RSD (25%))	Pymetrozin	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01	
Assigned value	0.035		0.036		0.050		0.010	
1	0.042	0.8	0.0282	-0.9				
2	0.033	-0.3	0.054	2.0	0.051	0.1	0.008	-0.7
3	0.034	-0.1	0.029	-0.8				
4								
5	0.0174	-2.0						
6	0.037	0.2	0.028	-0.9				
9	0.033	-0.3						
11	0.037	0.2	0.038	0.2	0.046	-0.3		
12	0.0378	0.3	0.0356	-0.1				
14	0.031	-0.5			0.037	-1.0		
15	0.028	-0.8						
17	0.032	-0.4			0.0627	1.0		
18	0.043	0.9						
19	0.033	-0.3	0.034	-0.2				
20	0.022	-1.5						
21	0.043	0.9	0.026	-1.1			0.012	0.9
22	0.0376	0.3	0.0369	0.1				
23	0.042	0.8			0.047	-0.2		
24	0.034	-0.1						
25								
26	0.0412	0.7	0.0402	0.4				
27								
28	0.038	0.3	0.063	3.0			0.01	0.1
29	0.024	-1.3	0.0185	-2.0				
30	0.035	0.0	0.041	0.5	0.044	-0.5		
31	0.039	0.4	FN	-3.5	0.062	1.0	0.009	-0.3
33	0.036	0.1	FN	-3.5	0.039	-0.9		
34	0.037	0.2			0.048	-0.2		
35								
36	0.0396	0.5						
37	0.0411	0.7	0.0381	0.2				
38	0.0403	0.6			0.0576	0.6		
39								
40	0.0338	-0.2						
41	0.029	-0.7						

Table 12b Results for voluntary compound DDE-pp, endrin-ketone and oxathiaprolin, in mg/kg, and the corresponding z scores, MRRs and the assigned values. The results pymetrozin is only shown for informative purposes because the residue level was too low to evaluate.

Laboratory code	DDE-pp	Z-scores (FFP RSD (25%))	Endrin-ketone	Z-scores (FFP RSD (25%))	Oxathiaprolin	Z-scores (FFP RSD (25%))	Pymetrozin	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01	
Assigned value	0.035		0.036		0.050		0.010	
42	0.047	1.3						
44	0.0323	-0.3			0.052	0.2	0.0033	-2.7
45								
46	0.032	-0.4						
47	0.029	-0.7						
48								
49	0.0338	-0.2	0.0323	-0.4				
50								
51								
52	0.037	0.2	0.043	0.8	FN	-3.2		
53								
54								
55	0.038	0.3						
57	0.035	0.0						
58	0.033	-0.3	0.029	-0.8				
59	0.037	0.2						
60	0.037	0.2	FN	-3.5				
61								
62	0.014	-2.4					0.01	0.1
63	0.029	-0.7	0.024	-1.3				
64	0.046	1.2	0.035	-0.1			0.014	1.7
65								
66								
67	0.04	0.5						
68								
69	0.033	-0.3			0.0546	0.4	0.0077	-0.8
70								
71	0.028	-0.8	0.0291	-0.8				
72	0.034	-0.1						
73	0.035	0.0	0.017	-2.1				
74	0.033	-0.3	0.043	0.8				
75	0.049	1.6	0.1	>5				
76	0.04	0.5						
77								
78	0.0316	-0.4						

Table 12c Results for voluntary compound DDE-pp, endrin-ketone and oxathiaprolin, in mg/kg, and the corresponding z scores, MRRs and the assigned values. The results pymetrozin is only shown for informative purposes because the residue level was too low to evaluate.

Laboratory code	DDE-pp	Z-scores (FFP RSD (25%))	Endrin-ketone	Z-scores (FFP RSD (25%))	Oxathiaprolin	Z-scores (FFP RSD (25%))	Pymetrozin	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01	
Assigned value	0.035		0.036		0.050		0.010	
79	0.033	-0.3						
80	0.041	0.6	0.036	0.0	0.047	-0.2		
82	0.06	2.8	FN	-3.5	FN	-3.2		
83	0.035	0.0	FN	-3.5	FN	-3.2		
85	0.057	2.5	FN	-3.5	0.048	-0.2		
86	0.0257	-1.1						
87	0.032	-0.4	0.054	2.0	0.045	-0.4		
88	0.033	-0.3	0.033	-0.4	0.053	0.2		
89	0.0337	-0.2						
90	0.042	0.8						
91	0.039	0.4	FN	-3.5			0.007	-1.1
92	0.035	0.0						
93	FN	-3.5						
94	0.036	0.1						
95								
96	0.148	>5						
97	0.028	-0.8						
98	0.034	-0.1	0.03	-0.7				
99	0.034	-0.1	0.03	-0.7	0.04	-0.8		
100	FN	-3.5						
101	0.04	0.5	0.032	-0.5	0.055	0.4		
102	0.039	0.4						
103	0.032	-0.4					0.006	-1.5
104	0.037	0.2	0.037	0.1	0.0381	-0.9		
105	FN	-3.5	FN	-3.5				
106	0.0326	-0.3	FN	-3.5				
107	0.054	2.1			FN	-3.2		
108	0.045	1.1	FN	-3.5	0.052	0.2		
109	0.031	-0.5						
110								
111	0.0513	1.8						
112	FN	-3.5						
113	0.0321	-0.4						
114	0.035	0.0	FN	-3.5	FN	-3.2	0.016	2.6

Table 12d Results for voluntary compound DDE-pp, endrin-ketone and oxathiaprolin, in mg/kg, and the corresponding z scores, MRRs and the assigned values. The results pymetrozin is only shown for informative purposes because the residue level was too low to evaluate.

Laboratory code	DDE-pp	Z-scores (FFP RSD (25%))	Endrin-ketone	Z-scores (FFP RSD (25%))	Oxathiaprolin	Z-scores (FFP RSD (25%))	Pymetrozin	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01	
Assigned value	0.035		0.036		0.050		0.010	
115	0.041	0.6						
116	0.0345	-0.1	0.032	-0.5	FN	-3.2		
117	0.0328	-0.3						
118	0.03	-0.6						
119	0.0317	-0.4						
120	0.036	0.1	FN	-3.5	0.047	-0.2	0.007	-1.1
121	0.0581	2.6						
122								
123	0.038	0.3	0.037	0.1	0.062	1.0		
124	0.03	-0.6						
125	0.046	1.2			0.062	1.0	0.017	3.0
126								
127	0.04	0.5						
128								
129	0.0303	-0.6						
130	0.0355	0.0						
131	0.037	0.2			0.038	-1.0	0.01	0.1
132	0.028	-0.8	0.025	-1.2				
133	0.037	0.2			0.047	-0.2	0.008	-0.7
134	0.028	-0.8						
135	0.03	-0.6	FN	-3.5				
136	0.027	-0.9					0.005	-1.9
137	0.024	-1.3						
138	0.043	0.9	0.039	0.3				
139	0.032	-0.4	FN	-3.5	0.055	0.4		
140								
141	0.019	-1.8						
142								
143	0.034	-0.1						
144	0.023	-1.4						
145								
146	0.0335	-0.2						
147	0.015	-2.3						
148	0.034	-0.1	0.032	-0.5				
149	0.03	-0.6						

Table 12d Results for voluntary compound DDE-pp, endrin-ketone and oxathiaprolin, in mg/kg, and the corresponding z scores, MRRLs and the assigned values. The results pymetrozin is only shown for informative purposes because the residue level was too low to evaluate.

Laboratory code	DDE-pp	Z-scores (FFP RSD (25%))	Endrin-ketone	Z-scores (FFP RSD (25%))	Oxathiaprolin	Z-scores (FFP RSD (25%))	Pymetrozin	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01	
Assigned value	0.035		0.036		0.050		0.010	
150	0.025	-1.2	FN	-3.5	0.04	-0.8		
151	0.044	1.0						
152	0.031	-0.5						
153								
154	0.0267	-1.0						
155	0.037	0.2						
156	0.026	-1.1					0.016	2.6
157	0.041	0.6	0.05	1.5	0.05	0.0		
158	FN	-3.5	FN	-3.5	FN	-3.2		
159								
160								
161	0.033	-0.3	0.04	0.4				
162	0.0486	1.5	FN	-3.5	0.0479	-0.2		
163	0.031	-0.5					0.008	-0.7
164	0.036	0.1	0.041	0.5				
165	0.033	-0.3			0.064	1.1	0.01	0.1
166								
167	0.0308	-0.5	0.0262	-1.1				
169	0.035	0.0	FN	-3.5	FN	-3.2		
170	0.029	-0.7						
171	0.04	0.5						
172	0.057	2.5			0.06	0.8	0.009	-0.3
173								
174	0.04	0.5						
175	0.038	0.3	0.048	1.3	0.047	-0.2		

3.3.2 Sum of Weighted Z scores (AZ²) – Category A

To be classified into Category A, the laboratories had to submit quantitative results for at least 90 % of the compulsory pesticides present in the Test Item (≥ 14 pesticide residues, exclusive of any false negatives results), analyse for more than 90% of the compulsory pesticides on the target list and also report no false positive results. For the 88 EU and EFTA laboratories in Category A (59%), the results were additionally evaluated by calculating the Average of the Squared -Score (AZ²). Of the 88 participants, 80 participants (91%) obtained AZ² values at or below 2 (good), 6 participants (6.5%) obtained AZ² values between 2-3 (satisfactory) and 2 participants (2.2%) obtained AZ² values ≥ 3 (unsatisfactory). An additional four laboratories from Third Countries were evaluated and classified into Category A. The AZ² scores achieved by the labs can be seen in **Table 13**.

Table 13 Sum of Weighted z scores (AZ²) for laboratories in Category A, the number of pesticides detected and quantified by the laboratories, the number of false negatives reported and the classification as good, satisfactory and unsatisfactory. The table includes data for both EU and non-EU participants.

Lab code	No. of detected mandatory pesticides	No. of detected voluntary pesticides	AZ ²	False negative	Classification	NRL
1	16	2	0.2	0	Good	
2	16	3	0.5	0	Good	
5	16	1	3.9	0	Unsatisfactory	NRL-CF
11	16	3	0.1	0	Good	
14	16	2	1.9	0	Good	
15	16	1	0.1	0	Good	NRL-CF
17	16	2	0.6	0	Good	
19	16	2	0.3	0	Good	
22	16	2	0.7	0	Good	
23	16	2	0.4	0	Good	NRL-CE
26	16	2	0.9	0	Good	
29	16	2	1.7	0	Good	NRL-CF
30	16	3	0.4	0	Good	NRL-CF
33	16	3	1.2	1	Good	
34	16	2	0.3	0	Good	
36	14	1	1.7	2	Good	
38	16	2	0.8	0	Good	
42	15	1	1.2	1	Good	NRL-CF
44	15	2	1.1	1	Good	
48	16	0	0.8	0	Good	
49	16	2	0.3	0	Good	
50	16	0	1.4	0	Good	NRL-CF
52	16	3	1.0	1	Good	
53	16	0	0.6	0	Good	
55	15	1	0.9	1	Good	
57	16	1	0.3	0	Good	NRL-FE
58	16	2	0.2	0	Good	NRL-CF
59	16	1	1.1	0	Good	

Lab code	No. of detected mandatory pesticides	No. of detected voluntary pesticides	AZ ²	False negative	Classification	NRL
60	16	2	0.2	1	Good	
61	16	0	0.2	0	Good	
62	15	1	2.5	1	Satisfactory	NRL-FE
63	16	2	0.3	0	Good	NRL-FE
65	16	0	0.9	0	Good	
67	16	1	0.1	0	Good	NRL-CF
69	16	2	0.2	0	Good	
70	14	0	2.5	2	Satisfactory	
71	15	2	0.9	1	Good	
72	16	1	0.4	0	Good	
75	15	2	1.6	1	Good	
76	16	1	1.2	0	Good	
77	16	0	0.9	0	Good	
78	16	1	0.5	0	Good	NRL-CF
79	16	1	0.3	0	Good	NRL-FE
80	16	3	0.5	0	Good	
82	16	3	0.7	2	Good	NRL-CE
83	16	3	0.8	2	Good	NRL-CF
85	16	3	0.4	1	Good	
86	16	1	1.0	0	Good	
88	16	3	0.1	0	Good	
92	16	1	0.6	0	Good	
94	15	1	1.2	1	Good	
97	16	1	0.3	0	Good	
98	15	2	0.7	0	Good	
99	16	3	0.2	0	Good	
100	14	1	2.8	3	Satisfactory	
101	16	3	0.2	0	Good	
103	15	1	3.0	1	Unsatisfactory	
104	16	3	0.2	0	Good	
106	16	2	0.5	1	Good	
107	15	2	1.2	2	Good	NRL-CF
108	16	3	0.8	1	Good	NRL-CF
111	16	1	0.3	0	Good	NRL-CE
113	16	1	0.1	0	Good	0
115	16	1	0.2	0	Good	NRL-CE
116	15	3	0.9	2	Good	NRL-CE
117	16	1	0.0	0	Good	NRL-CE

Lab code	No. of detected mandatory pesticides	No. of detected voluntary pesticides	AZ ²	False negative	Classification	NRL
118	16	1	0.2	0	Good	NRL-CE
120	16	3	0.5	1	Good	
123	16	3	0.2	0	Good	
127	16	1	0.3	0	Good	
129	16	1	0.3	0	Good	NRL-CF
131	16	2	0.8	0	Good	
132	16	2	1.0	0	Good	
133	16	2	0.1	0	Good	NRL-CF
134	16	1	0.7	0	Good	
136	16	1	1.3	0	Good	
139	16	3	1.5	1	Good	
144	16	1	0.8	0	Good	NRL-CF
149	16	1	0.7	0	Good	NRL-CF
152	16	1	2.3	0	Satisfactory	NRL-FE
155	16	1	0.1	0	Good	
157	16	3	2.3	0	Satisfactory	NRL-CF
161	16	2	0.6	0	Good	
162	16	3	0.5	1	Good	NRL-CF
163	15	1	0.9	1	Good	
164	15	2	0.9	0	Good	NRL-CF
165	16	2	0.0	0	Good	
167	16	2	1.1	0	Good	
169	16	3	2.3	2	Satisfactory	
170	17	1	0.5	0	Good	
172	17	2	0.6	0	Good	
175	17	3	0.2	0	Good	

The laboratories that did not fulfil the requirements described above, were classified in Category B. The number of reported quantitative results, analysed compounds from the Target List and acceptable z scores as well as information on false negative and positive results are shown in **Table 14**. Seventeen participants fulfilled the criteria of detecting 90% of the compulsory pesticides in the Test Item but did not fulfil the criteria of analysing for 90% of the compulsory pesticides on the Target List. The reverse was the case for four participants. Forty-four participants (27%) analysed and detected less than 70% of the pesticides present in the Test Item.

Table 14 Number of pesticides detected and quantified and the percentage. Number of compulsory compounds analysed from the Target List, number of voluntary pesticides detected and quantified, number of acceptable z scores, false negative and positive results, and NRL status for the laboratories in Category B.

Lab code	No. of compulsory pesticides detected	Compulsory pesticides detected in test item, %	Analysed of compulsory pesticides on Target List, %	No. Of voluntary pesticides detected	No. of acceptable z score	No. of false negative	No. of false positive	NRL
3	0	0	3	2	0	0	0	
4	11	69	62	0	11	0	0	
6	0	0	5	2	0	0	0	
9	13	88	60	1	12	1	0	
12	2	19	25	2	2	1	0	
18	10	69	54	1	10	1	0	
20	14	94	80	1	12	1	0	
21	11	88	76	2	0	3	1	
24	3	19	17	1	3	0	0	
25	14	94	69	0	9	1	0	
27	7	44	43	0	7	0	0	
28	14	88	81	2	12	0	0	
31 ¹	16	100	99	3	16	1	1	
35	13	81	54	0	11	0	0	
37	7	44	31	2	6	0	0	NRL-CF
39	0	0	99	0	0	0	0	
40	7	44	45	1	7	0	0	
41	7	69	78	1	7	4	0	
45	11	69	52	0	11	0	0	
46	7	44	45	1	7	0	0	
47	11	69	67	1	11	0	0	
51	13	81	74	0	13	0	0	
54	7	44	45	0	7	0	0	
64	13	100	77	2	9	3	0	
66	7	44	43	0	7	0	0	
68	7	44	44	0	7	0	0	
73	15	94	85	2	10	0	0	NRL-CF
74	10	69	58	2	9	1	0	
87 ¹	16	100	100	3	15	0	1	
89	16	100	88	1	16	0	0	
90	15	100	86	1	13	1	0	
91 ¹	16	100	95	2	14	1	1	
93	6	38	46	1	6	1	0	
95	15	94	62	0	15	0	0	
96	11	69	59	1	1	0	0	NRL-FE
102	0	0	4	1	0	0	0	
105 ¹	15	100	91	2	14	3	1	
109	6	38	37	1	6	0	0	
110	16	100	69	0	15	0	0	

Lab code	No. of compulsory pesticides detected	Compulsory pesticides detected in test item, %	Analysed of compulsory pesticides on Target List, %	No. Of voluntary pesticides detected	No. of acceptable z score	No. of false negative	No. of false positive	NRL
112	16	100	88	1	15	1	0	
114 ¹	11	100	100	3	6	7	1	
119	7	44	43	1	7	0	0	
121	0	0	4	1	0	0	0	
122	0	0	97	0	0	0	0	
124	16	100	69	1	16	0	0	
125 ¹	16	100	100	2	16	0	1	
126	16	100	66	0	15	0	0	
128	0	0	97	0	0	0	0	
130	13	94	85	1	9	2	0	
135	0	0	3	2	0	1	2	NRL-FE
137	15	100	89	1	14	1	0	
138	0	0	5	2	0	0	0	
140	0	0	39	0	0	0	0	
141	0	0	4	1	0	0	0	
142	11	75	60	0	11	1	0	
143	5	38	54	1	4	1	0	
145	1	25	18	0	1	3	1	
146	14	94	74	1	14	1	0	NRL-CE
147	13	81	83	1	13	0	0	
148 ¹	16	100	91	2	13	0	1	
150 ¹	15	100	99	3	15	2	1	
151	12	81	58	1	10	1	1	
153	4	25	30	0	4	0	0	
154 ¹	14	94	99	1	13	1	1	
156	12	75	71	1	2	0	0	
158	12	94	99	3	9	6	0	NRL-CF
159	15	94	71	0	12	0	0	
160	0	0	100	0	0	0	0	
166	15	94	83	0	14	0	0	
171	17	106	89	1	16	0	0	
173	0	0	52	0	0	0	0	
174	14	94	63	1	8	1	0	

¹ Laboratories that reported false positive results and consequently were moved from Category A to Category B

3.4 Trends in numbers of participating laboratories and their performance

The number of EU and EFTA laboratories participating in the EUPTs on cereals has increased steadily over the years but has settled at around 150. In EUPT-C3 in 2009, 102 labs participated and in the latest EUPT-CF14, 156 labs participated. The numbers from EUPT-CF8 and forward can be seen in **Table 15**. The number of pesticides included in the Target Pesticide List has also increased during this 14-years period, from 43 to 164 compulsory compounds and 38 voluntary compounds. Thus, the demands put on the participating laboratories has increased every year. Many laboratories have a limited scope and are therefore not able to cover all pesticides in the PT. In this EUPT, 18% of the laboratories were not able to analyse and detect more than 70% of pesticides present in the Test Item. This is a small trend to the better. Last EUPT the number was 25%.

Table 15. Overall trends in participation of laboratories, pesticides in the target list and test item, and performance of laboratories in the 7 latest EUPTs cereals (excluding EUPT-CF7 on feed and EUPT-CF12 on hay).

PT and types of test item	EUPT-CF8 Wheat flour	EUPT-CF9 Maize flour	EUPT-CF10 Rye flour	EUPT-CF11 Oat flour	EUPT-CF12 Hay flour	EUPT-CF13 Oat kernels	EUPT-CF14 Rice kernels
Participants submitting results (EU + EFTA)	142	143	160	149	111	149	156
MRM pesticides in the Target Pesticide List	111	117	134/7	153/9	155/23	160/32	164/38
MRM pesticides in the test material	17	18	16	18	8	18	19
No. of results for MRM pesticides	1893	2012	2012	2172	808	2007	2298
Average of 'reported results', %	78	78	79	83	74	75	80
Range of 'reported results', %	49-93	61-94	58-90	65-93	40-91	44-94	26-93
Acceptable z scores, %	90	89	95	89	93	93	91
Questionable z scores, %	6	2	2	3	3	3.1	3
Unacceptable z scores, %	4	3	2	8	3	3.4	6
False negatives, %	3	2	2	4	1	2.3	3.4
Number of false positives	4	9	0	19	7	3	14
Category A, % of participating laboratories	50	57	53	45	51	57	57
Good SWZ/AZ², %	85	96	93	92	92	91	91
Satisfactory SWZ/AZ², %	10	1	5	1.5	3.4	5.7	6.7
Unsatisfactory SWZ/AZ², %	4	3	2	6.2	5.1	3.4	2.2
Alg A RSD%	20	19	17	17	20	18	19

The overall analytical performance (accuracy of measurement) if looking at the percentage of acceptable, questionable, unacceptable z scores has not changed significantly during the last 6 EUPTs, which has also been the case for the analytical scope. The average % of reported results has in the last four cereal EUPT-CF been between 75-83%. This was because a lot of participants analysed for less than 50% of the pesticide residues present in the test Item. The false negative results have increased to 3.4% due to a low content of dichlorvos that many were not able to detect and quantify. The number of false positive results reported also increased, which indicate that rice matrix is not so easy to analyse.

The percentage of Category A laboratories has varied slightly over the years and 57% of the participants were evaluated as Category A in this EUPT. For Category A an improvement in AZ² was seen in EUPT-CF9 where 96% of the results were Good, and in EUPT-CF14, still, 91% of the laboratories were evaluated as Good. However, it is difficult to assess any improvement/deterioration in laboratory performance between the Proficiency Tests, because the number of pesticides in the Test item and the number of laboratories participating in the PTs have both significantly increased.

3.5 Summary, conclusions and prospects for the EUPTs on pesticide residues in cereals

The EUPT-CF14 Test Item consisted of rice kernels containing incurred and spiked pesticides. The rice crop had been sprayed in the field with commercially available pesticide formulations and additionally spiked post-harvest in the laboratory. The final Test Item contained the following pesticides: acephate, acetamiprid, azoxystrobin, buprofezin, carbendazim, carbofuran, cyproconazole, DDE-pp, dichlorvos, difenoconazole, endrin-ketone, hexaconazole, imidacloprid, isoprothiolane, metalaxyl, oxathiapiprolin, profenofos, pyriproxyfen, and thiamethoxam. One hundred fifty-two EU and EFTA laboratories, from 28 different countries (26 EU member states and UK), agreed to participate in this proficiency test. Six more laboratories registered, but did not submit any results. All NRLs, participated in the PT. Malta was represented in the PT by the NRL for the UK. An additional 8 laboratories from EU candidate states and Third Countries registered for the PT and all submitted results. The Target Pesticide List distributed to the laboratories prior to the test contained 164 individual compulsory and 38 voluntary compounds..

The number of false positives and false negatives has varied between the EUPTs. Fourteen false positive results were reported and the number of false negatives represented 3.4% of the total number of results. This is in the high end compared to previous EUPTs indicating that rice is a difficult commodity to analyse. The average Alg A-RSD was at 19%, well below the FFP-RSD of 25% with a range from 16-25% for the individual compounds.

For azoxystrobin, buprofezin, difenoconazole, isoprothiolane, metalaxyl, and thiamethoxam, acceptable results were obtained by 95-99% of the laboratories. For acephate, acetamiprid, carbofuran, DDE-pp, hexaconazole, imidacloprid, profenofos, and pyriproxyfen, acceptable results were obtained by 90-94% of the laboratories. Only 88% of the laboratories obtained acceptable z scores for carbendazim. Only 79%, 71%, and 62%, of the laboratories obtained acceptable scores for oxathiapiprolin, dichlorvos, and endrin-ketone, respectively.

The EUPT-CF15 will have rape seed cake as test item, which will be shipped to the laboratories in February/March 2021. The selection of pesticides will continue to be focused on pesticides included in the scope of the EU multi-annual coordinated control programme, as well as additional pesticides of relevance to feed and/or cereal production in Europe and in other parts of the world from where significant quantities of feed and cereals are imported.

4. ACKNOWLEDGEMENTS

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5. REFERENCES

[1] Regulation (EU) No 2017/625 of the European Parliament and of the Council on official controls and other official activities performed to ensure the application of food and feed law, rules on animal health and welfare, plant health and plant protection products. Published at OJ of the EU L 95/1 of 07.04.2017

[2] 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.

[3] CEN EN 15662 - Foods of plant origin - Determination of pesticide residues using GC-MS and/or LC-MS/MS following acetonitrile extraction/partitioning and clean-up by dispersive SPE - QuEChERS-method

[4] Thompson M., Ellison S. L. R. and Wood R., The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories. Pure & Appl. Chem., Vol.78, No. 1, pp. 145-196, 2006.

[5] ISO 13528:2015 – Statistical methods for use in proficiency testing by interlaboratory comparison

[6] ISO 17043:2010 – Conformity assessment -- General requirements for proficiency testing-

APPENDICES

Appendix 1 List of laboratories registered to participate in the EUPT-CF14

Participating labs from EU and EFTA member states

Country	Institution	City	NRL-CF	Report data
Austria	AGES Innsbruck, Pesticide and Food Analytics/PLMA	Innsbruck	NRL-CF	Yes
Belgium	Sciensano	Brussels	NRL-CF	Yes
Austria	AGES Innsbruck, Pesticide and Food Analytics/PLMA	Innsbruck	NRL-CF	Yes
Belgium	Sciensano	Brussels	NRL-CF	Yes
Belgium	Primoris Belgium	Gent - Zwijnaarde		Yes
Belgium	FLVV - Tervuren	Tervuren		Yes
Bulgaria	Primoris - Bulgaria, Plovdiv	Plovdiv		Yes
Bulgaria	CLCTC - Sofia Pesticide Lab	Sofia		Yes
Croatia	Bioinstitut Ltd.	Cakovec		Yes
Croatia	Nastavni Zavod za javno zdravstvo Primorsko-goranske zupanije	Kotar County, Rijeka		Yes
Croatia	Sample Control - Pesticide Lab	Lucko		Yes
Croatia	INSPECTO d.o.o. Laboratorij (Osijek)	Osijek		Yes
Croatia	Croatian National Institute of Public Health-HZJZ	Zagreb		Yes
Croatia	Croatiakontrola - Pesticide Lab	Zagreb		Yes
Croatia	Center for Food Control - PBF, Zagreb	Zagreb		Yes
Croatia	Croatian veterinary institute	Zagreb	NRL-FE	Yes
Croatia	Dr. Andrija Štampar - Pesticide Lab	Zagreb	NRL-CE	Yes
Cyprus	Pesticides Residues Laboratory of S.G.L	Nicosia	NRL-CE	Yes
Cyprus	Animal Feeds and Feed Additives - Pesticide Lab	Nicosia	NRL-FE	Yes
Czech Republic	Pesticide Lab (Brno)	Brno	NRL-FE	Yes
Czech Republic	Czech Agriculture and Food Inspection Authority (CAFIA)	Praha	NRL-CE	Yes
Czech Republic	Metrological and Testing laboratory, University of Chemistry and Technology	Praha		Yes
Denmark	Laboratoriet Ringsted - Pesticide Lab	Ringsted	NRL-FE	Yes
Estonia	Agricultural Research Center - Estonia, Saku	Saku	NRL-CF	Yes
Finland	Finnish Customs Laboratory	Espoo	NRL-CE	Yes
Finland	Finnish Food Authority	Helsinki	NRL-FE	Yes
France	GIRPA-POLLENIZ - Pesticide Lab	Beaucouzé		Yes
France	CERECO (GARONS)	GARONS		Yes
France	CAPINOV	Landerneau		Yes
France	INOVALYS - Le Mans	Le Mans		Yes
France	SCL Paris	Massy Cedex	NRL-CF	Yes
France	SCL Montpellier	Montpellier		Yes
France	Phytocontrol (Nimes) - Pesticide Lab	Nimes		Yes
France	CAMP Méditerranée (Perpignan)	PERPIGNAN		Yes
Germany	Federal Office of Consumer Protection and Food Safety, NRL for Pesticide Residues	Berlin	NRL-CF	Yes
Germany	LUA Sachsen - Pesticide Lab	Dresden		Yes
Germany	Bavarian Health and Food Safety Authority	Erlangen		Yes
Germany	Bundeswehr - Pesticide Lab (Garching-Hochbrück)	Garching-Hochbrück		Yes
Germany	LLG - Pesticide Lab	Halle/Saale		Yes
Germany	LAV Sachsen-Anhalt Fachbereich 3	Halle/Saale		Yes

Germany	Eurofins Dr. Specht Laboratorien GmbH	Hamburg		Yes
Germany	GALAB Laboratories GmbH	Hamburg		Yes
Germany	Thüringer Landesanstalt für Landwirtschaft, Jena	Jena		Yes
Germany	LTZ Augustenberg	Karlsruhe		Yes
Germany	LUFÄ Kiel - Pesticide Lab	Kiel		Yes
Germany	Chemisches und Veterinäruntersuchungsamt Rhein Ruhr Wupper	Krefeld		Yes
Germany	Landeslabor Schleswig-Holstein	Neumünster		Yes
Germany	Staatliche Betriebsgesellschaft für Umwelt und Landwirtschaft - FB42	Nossen		Yes
Germany	Niedersächsisches Landesamt für Verbraucherschutz und Lebensmittelsicherheit, LVI Oldenburg	Oldenburg		Yes
Germany	Landeslabor Berlin-Brandenburg, Potsdam	Potsdam		Yes
Germany	Landesamt für Landwirtschaft, Lebensmittelsicherheit und Fischerei	Rostock		Yes
Germany	LUA Saarbrücken	Saarbrücken		No
Germany	LUFÄ Speyer	Speyer		Yes
Germany	LUA Rheinland-Pfalz, Institut für LM-Chemie Speyer	Speyer		Yes
Germany	LAVES Futtermittelinstitut	Stade		Yes
Greece	General Chemical State Laboratory	Athens	NRL-CF	Yes
Greece	Pesticide Residues Laboratory of the Benaki Phytopathological Institute	Kifissia	NRL-CF	Yes
Greece	Regional Center of Plant Prot. - Thessaloniki	Thessaloniki		Yes
Hungary	Food Chain Safety Centre Non-profit Ltd.	Hódmezovásárhely		Yes
Hungary	FCSCN Ltd., Pesticide Residue Analytical Laboratory	Miskolc		Yes
Hungary	Food Chain Safety Centre Non-profit Ltd	Szolnok		Yes
Hungary	National Food Chain Safety Office Pesticide Analytical National Reference Laboratory	Velence	NRL-CF	Yes
Iceland	Matís - Iceland, Reykjavík	Reykjavík	NRL-CF	Yes
Ireland	Pesticide Residue Lab	Co. Kildare	NRL-CF	Yes
Italy	APPA-Puglia Polo Alimenti Bari - Pesticide Lab	Bari		Yes
Italy	Laboratorio di Prevenzione (Bergamo)	Bergamo		Yes
Italy	APPA Bolzano	Bolzano		Yes
Italy	Istituto Zooprofilattico Sperimentale Lombardia Emilia Romagna	Brescia		Yes
Italy	Water & Life Lab srl	Entratico		Yes
Italy	ARPA-ER - Pesticide Lab	Ferrara		Yes
Italy	Laboratorio di Sanità Pubblica Firenze	Firenze		Yes
Italy	IZS PB - Pesticide Lab	Foggia		Yes
Italy	IZS PLV (sez. Genova) - Pesticide Lab	Genova	NRL-CF	Yes
Italy	ARPAL Sez. di La Spezia	La Spezia		Yes
Italy	ARPA Lazio (sez. Latina) - Pesticide Lab	Latina		Yes
Italy	IZSve - Pesticide Lab	Legnano (Padova)		Yes
Italy	ARPAM - Pesticide Lab	Macerata		No
Italy	ATS Milano - Laboratorio di Prevenzione	Milano		Yes
Italy	IZS Sicilia - Pesticide Lab	Palermo		Yes
Italy	IZSUM - Italy, Perugia	Perugia		Yes
Italy	IZS LT - Italy, Rome	Roma		Yes
Italy	ISS - Pesticide Lab	Roma		No
Italy	ARPA VDA - Pesticide Lab	Saint Christophe		Yes
Italy	IZS LT (sezione Firenze)	San Martino alla Palma Scandicci (FI)		Yes

Italy	IZS Sardegna	Sassari		Yes
Italy	IZSAM	Teramo		Yes
Italy	APPA-SL Trento	Trento		Yes
Italy	ARPA FVG	Udine		Yes
Italy	ARPA Veneto	Verona		Yes
Latvia	Institute of Food Safety, Animal Health and Environment "BIOR"	Riga	NRL-CF	Yes
Lithuania	NFVRAI	Vilnius	NRL-CF	Yes
Luxembourg	LNS Food lab	Dudelange	NRL-CE	Yes
Netherlands	Groen Agro Control - Netherlands	Delfgauw		Yes
Netherlands	Eurofins Lab Zeeuws-Vlaanderen	Graauw		Yes
Netherlands	Dr. A. Verwey B.V.	Rotterdam		Yes
Netherlands	NofaLab B.V.	Schiedam		No
Netherlands	WFSR - Wageningen Food Safety Research	Wageningen	NRL-CF	Yes
Norway	NIBIO - Department of Pesticide Chemistry	Ås	NRL-CF	Yes
Poland	WIW ZHW (Bialystok) - Pesticide Lab	Bialystok		Yes
Poland	Laboratory of Food & Feed Safety in Bialystok	Bialystok		Yes
Poland	WIW ZHW (Gdansk) - Pesticide Lab	Gdansk		Yes
Poland	UO-Technologia (Grojec) - Pesticide Lab	Grojec		Yes
Poland	WIW ZHW (Katowice) - Pesticide Lab	Katowice		Yes
Poland	WIW ZHW (Opole) - Pesticide Lab	Opole		Yes
Poland	VSES Opole - Pesticide Lab	Opole		Yes
Poland	WIW ZHW Poznan - Pesticide Lab	Poznan		Yes
Poland	IPP-NRI - Pesticide Lab (Poznan)	Poznan	NRL-FE	Yes
Poland	InHort (Skierniewice) - Pesticide Lab	Skierniewice		Yes
Poland	IPP-NRI - Pesticide Lab (Sosnicowice)	Sosnicowice		Yes
Poland	WIW ZHW (Szczecin) - Pesticide Lab	Szczecin		Yes
Poland	VSES Warszawa - Pesticide Lab	Warszaw	NRL-CE	Yes
Poland	WIW ZHW (Wroclaw) - Pesticide Lab	Wroclaw		Yes
Portugal	Laboratorio Regional de Veterinaria e Seguranca Alimentar	Funchal - Madeira Island		Yes
Portugal	INIAV-UEISTSA - Pesticide Lab.	Oeiras	NRL-CE	Yes
Portugal	Labiagro – Laboratório Químico	Oeiras - Lisboa		Yes
Romania	LSVSA Bistrita-Nasaud	Bistrita		Yes
Romania	Institute for Hygiene and Veterinary Public Health	Bucharest	NRL-CF	Yes
Romania	Laboratory for Pesticides Residues in Plants and Vegetable Products	Bucharest	NRL-CF	Yes
Romania	DSVSA Bucuresti - Pesticide Lab	Bucharest		Yes
Romania	Pesticide Lab (Cluj Napoca)	Cluj Napoca		Yes
Romania	DSVSA DOLJ	Craiova		Yes
Romania	Regional Laboratory for Pesticide Residues Control in Plant and Plant Products Mures	Tirgu Mures		Yes
Slovakia	Veterinary and Food Institute in Bratislava	Bratislava	NRL-CF	Yes
Slovakia	Pesticide Lab of PHA SR - Bratislava	Bratislava		Yes
Slovenia	NLZOH-MB-location Ljubljana	Ljubljana		Yes
Slovenia	Kmetijski inštitut Slovenije Centralni laboratorij	Ljubljana	NRL-FE	Yes
Slovenia	Pesticide Lab - Maribor	Maribor	NRL-CE	Yes
Spain	Laboratorio de Residuos, Inst. Tecnol. de Canarias	Agüimes, Gran Canaria		Yes
Spain	Laboratorio Analítico Bioclínico - Spain, Almeria	Almeria		Yes

Spain	Analytica Alimentaria GmbH - Almeria, Spain	Almería		Yes
Spain	EURL-FV University of Almeria	Almeria		Yes
Spain	Laboratorio del SOIVRE - Almeria	Almería		No
Spain	Laboratorio Salud Pública de Badajoz	Badajoz		Yes
Spain	Laboratori Agència Salut Pública Barcelona	Barcelona		Yes
Spain	Lab. Agrario Regional - Junta de Castilla y Leon	Burgos		Yes
Spain	Labs and technological services AGQ	Burguillos		Yes
Spain	LAC - Generalitat de Catalunya	Cabrils		Yes
Spain	Laboratorio Agroalimentario de Extremadura	Cáceres		Yes
Spain	Laboratorio Regional de la CCAA de La Rioja	Logroño		Yes
Spain	EUROFINS ECOSUR, S.A.	Lorquí		Yes
Spain	Laboratorio de Salud Pública de Galicia, Lugo	Lugo		Yes
Spain	Madrid Salud	Madrid		Yes
Spain	Laboratorio Arbitral Agroalimentario	Madrid	NRL-CF	Yes
Spain	National Centre for Food (Majadahonda)	Majadahonda	NRL-CF	Yes
Spain	LABORATORIO KUDAM, S.L.	Pilar de la Horadada (Alicante)		Yes
Spain	National Center for Technology and Food Safety	San Adrián (Navarra)		Yes
Spain	LABORATORIO QUIMICO MICROBIOLOGICO, S.L.	SAN GINES (MURCIA)		Yes
Spain	Soivre Tenerife - Pesticide Lab	Santa Cruz de Tenerife		No
Spain	LARAGA - Pesticide Lab (Toledo)	Toledo		Yes
Spain	Ainia (Valencia)	Valencia		Yes
Spain	Laboratorio Agroalimentario - Spain, Valencia	Valencia		Yes
Spain	Nasertic - Spain, Villava	Villava		Yes
Spain	Laboratorio Agroambiental de Zaragoza	Zaragoza		Yes
Sweden	Eurofins Food and Feed Testing Sweden	Lidköping		Yes
United Kingdom	Concept Life Sciences - United Kingdom, Cambridge	Bar Hill		Yes
United Kingdom	AFBI	Belfast		Yes
United Kingdom	Fera Science Ltd	York	NRL-CF	Yes

Participating labs from EU candidate states and other non EU countries

Country	Institution	City	Report data
Bosnia and Herzegovina	Federal Institute for Agriculture - B-H, Sarajevo	Sarajevo	Yes
Ghana	Ghana Standards Authority, Accra	Accra	Yes
Peru	Bureau Veritas - Lab Lima	LIMA - CALLAO	Yes
Serbia	SP Laboratorija - Pesticide Lab	BECEJ	Yes
Serbia	Inst. of Public Health of Belgrade - Pesticide Lab	Belgrade	Yes
Singapore	SFA NCFS	Singapore	Yes
Thailand	Central Laboratory - Pesticide Lab (Bangkok)	Bangkok	Yes
Uruguay	UdelaR - Faculty of Chemistry (Montevideo)	Montevideo	Yes

Appendix 2

Target Pesticide List

Pesticides	MRRL (mg/kg)
Compulsory Compounds (will be considered in Category A/B classification)	
2-phenylphenol	0.01
Acephate	0.01
Acetamiprid	0.01
Acrinathrin	0.01
Aldrin	0.005
Ametoctradin	0.01
Azinphos-methyl	0.01
Azoxystrobin	0.01
Bifenthrin	0.01
Biphenyl	0.01
Bitertanol (sum of isomers)	0.01
Bixafen	0.01
Boscalid	0.01
Bromuconazole (sum of isomers)	0.01
Buprofezin	0.01
Cadusafos	0.005
Carbaryl	0.01
Carbendazim	0.01
Carbofuran	0.005
Carbofuran-3-hydroxy	0.01
Carboxin	0.01
Chlorantraniliprole	0.01
Chlorfenapyr	0.01
Chlorfenvinphos	0.01
Chlorpropham	0.01
Chlorpyrifos	0.005
Chlorpyrifos-methyl	0.01
Clothianidin	0.01
Cyazofamid	0.01
Cyfluthrin (sum of isomers)	0.01
Cymoxanil	0.01
Cypermethrin (sum of isomers)	0.01
Cyproconazole	0.01
Cyprodinil	0.01
Deltamethrin, cis-	0.01
Demeton-S-methylsulfone	0.005
Diazinon	0.005
Dichlorvos	0.005
Dieldrin	0.005
Difenoconazole	0.01
Diflubenzuron	0.01

Pesticides	MRRL (mg/kg)
Dimethoate	0.003
Dimethomorph (sum of isomers)	0.01
Diniconazole (sum of isomers)	0.01
Endosulfan-alpha	0.01
Endosulfan-beta	0.01
Endosulfan-sulfate	0.01
Epoxiconazole	0.01
Ethion	0.01
Ethirimol	0.01
Ethoprophos	0.005
Etoxazole	0.01
Famoxadone	0.01
Fenbuconazole	0.01
Fenhexamid	0.01
Fenitrothion	0.01
Fenpropathrin	0.01
Fenpropidin	0.01
Fenpropimorph (sum of isomers)	0.01
Fenpyrazamine	0.01
Fenpyroximate	0.01
Fenthion	0.01
Fenthion-oxon	0.01
Fenthion-oxon-sulfone	0.01
Fenthion-oxon-sulfoxide	0.01
Fenthion-sulfone	0.01
Fenthion-sulfoxide	0.01
Fenvalerate (sum of isomers)	0.01
Fipronil	0.004
Fipronil-sulfone	0.004
Flonicamid	0.01
Flubendiamide	0.01
Fludioxonil	0.01
Flufenoxuron	0.01
Fluopicolide	0.01
Fluopyram	0.01
Fluquinconazole	0.01
Flusilazole	0.01
Flutolanil	0.01
Flutriafol	0.01
Fluxapyroxad	0.01
Formetanate	0.01
Hexaconazole	0.01
Imazalil	0.01
Imidacloprid	0.01
Indoxacarb	0.01
Iprodione	0.01

Pesticides	MRRL (mg/kg)
Isocarbophos	0.01
Isoprothiolane	0.01
Isoproturon	0.01
Kresoxim-methyl	0.01
Lambda-cyhalothrin	0.01
Lindane	0.01
Linuron	0.01
Malaoxon	0.01
Malathion	0.01
Mandipropamid	0.01
Metalaxyl	0.01
Metconazole (sum of isomers)	0.01
Methacrifos	0.01
Methamidophos	0.01
Methomyl	0.01
Metolachlor	0.01
Metrafenone	0.01
Metribuzin	0.01
Omethoate	0.003
Oxydemeton-methyl	0.005
Paclobutrazol	0.01
Parathion	0.01
Penconazole	0.01
Pencycuron	0.01
Pendimethalin	0.01
Permethrin (sum of isomers)	0.01
Phosphamidon	0.01
Pirimicarb	0.01
Pirimicarb-desmethyl	0.01
Pirimiphos-methyl	0.01
Prochloraz (parent compound only)	0.01
Procymidone	0.01
Profenofos	0.01
Propamocarb (only parent compound)	0.01
Propiconazole (sum of isomers)	0.01
Proquinazid	0.01
Prosulfocarb	0.01
Prothioconazole-desthio	0.01
Prothiofos	0.01
Pymetrozine	0.01
Pyraclostrobin	0.01
Pyridaben	0.01
Pyrimethanil	0.01
Pyriproxyfen	0.01
Quinoxifen	0.01
Spirodiclofen	0.01

Pesticides	MRRL (mg/kg)
Spiromesifen	0.01
Spirotetramat	0.01
SpirotetramatmetaboliteBYI08330-enol	0.01
SpirotetramatmetaboliteBYI08330enol-glucoside	0.01
SpirotetramatmetaboliteBYI08330-ketohydroxy	0.01
SpirotetramatmetaboliteBYI08330-monohydroxy	0.01
Spiroxamine	0.01
Tau-Fluvalinate	0.01
Tebuconazole	0.01
Tebufenozide	0.01
Teflubenzuron	0.01
Tefluthrin	0.01
Terbuthylazine	0.01
Tetraconazole	0.01
Tetradifon	0.01
Tetramethrin	0.01
Thiabendazole	0.01
Thiacloprid	0.01
Thiamethoxam	0.01
Thiodicarb	0.01
Thiophanate-methyl	0.01
Tolclofos-methyl	0.01
Triadimefon	0.01
Triadimenol	0.01
Triazophos	0.005
Tricyclazole	0.01
Trifloxystrobin	0.01
Trifluralin	0.01
Triticonazole	0.01
Vinclozolin (parent compound only)	0.01
Zoxamide	0.01
Voluntary Compounds	
<i>(will not be considered in Category A/B classification)</i>	
Benalaxyl (sum)	0.01
Benzovindiflupyr	0.01
Chlordane-cis	0.01
Chlordane-cis	0.01
Chlordane-oxy	0.01
Chlorfluazuron	0.01
Clomazone	0.01
Cyflufenamid	0.01
DDD-pp	0.01
DDE-pp	0.01
DDT-op	0.01
DDT-pp	0.01

Pesticides	MRRL (mg/kg)
Dinotefuran	0.01
Endrin	0.01
Endrin, ketone-	0.01
Fenobucarb	0.01
Fenpicoxamid	0.01
Fluensulfone	0.01
HCH-alpha	0.01
HCH-beta	0.01
Heptachlor	0.01
Heptachlorepoxyd-cis	0.01
Heptachlorepoxyd-trans	0.01
Isopyrazam	0.01
Novaluron	0.01
Oxathiapiprolin	0.01
Penflufen	0.01
Pentachloro-aniline	0.01
Penthiopyrad	0.01
Pyrethrins	0.01
Pyridalil	0.01
Pyriofenone	0.01
Quinalphos	0.01
Quintozene	0.01
Spinetoram	0.01
Sulfoxaflor	0.01
Tolfenpyrad	0.01
Tri-allate	0.01

Appendix 3 Homogeneity data

Sample no.	Acephate, mg/kg		Acetamiprid, mg/kg		Azoxystrobin, mg/kg	
	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
9	0.039	0.042	0.065	0.075	0.278	0.299
19	0.048	0.045	0.093	0.076	0.335	0.322
31	0.041	0.039	0.067	0.066	0.321	0.259
51	0.041	0.042	0.059	0.062	0.292	0.270
88	0.041	0.044	0.081	0.060	0.310	0.333
103	0.046	0.039	0.068	0.066	0.313	0.279
121	0.042	0.042	0.065	0.063	0.279	0.276
140	0.042	0.043	0.065	0.068	0.000	0.000
167	0.039	0.039	0.066	0.066	0.296	0.296
173	0.039	0.034	0.058	0.053	0.232	0.212
190	0.041	0.039	0.067	0.062	0.237	0.278

Sample no.	Buprofezin, mg/kg		Carbendazim, mg/kg		Carbofuran, mg/kg	
	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
9	0.058	0.054	0.063	0.069	0.061	0.066
19	0.059	0.051	0.065	0.064	0.067	0.062
31	0.050	0.053	0.065	0.060	0.062	0.059
51	0.047	0.050	0.057	0.059	0.059	0.059
88	0.051	0.051	0.062	0.062	0.058	0.060
103	0.052	0.047	0.068	0.067	0.069	0.065
121	0.054	0.056	0.063	0.064	0.060	0.061
140	0.048	0.055	0.064	0.064	0.063	0.063
167	0.052	0.047	0.065	0.064	0.061	0.065
173	0.053	0.042	0.059	0.056	0.056	0.056
190	0.050	0.054	0.063	0.060	0.062	0.061

Sample no.	DDE-pp, mg/kg		Dichlorvos, mg/kg		Difenoconazole, mg/kg	
	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
9	0.036	0.038	0.022	0.023	0.052	0.049
19	0.039	0.040	0.024	0.022	0.059	0.051
31	0.040	0.037	0.023	0.021	0.048	0.048
51	0.036	0.034	0.022	0.021	0.047	0.047
88	0.039	0.040	0.021	0.021	0.047	0.048
103	0.040	0.037	0.023	0.022	0.049	0.041
121	0.037	0.036	0.020	0.022	0.053	0.051
140	0.000	0.000	0.021	0.020	0.054	0.053
167	0.039	0.041	0.022	0.022	0.052	0.044
173	0.034	0.032	0.021	0.019	0.049	0.046
190	0.035	0.036	0.024	0.022	0.054	0.051

Sample no.	Endrin-ketone, mg/kg		Hexaconazole, mg/kg		Imidacloprid, mg/kg	
	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
9	0.026	0.058	0.094	0.104	0.070	0.085
19	0.052	0.033	0.101	0.093	0.088	0.078
31	0.052	0.023	0.095	0.085	0.073	0.066
51	0.031	0.048	0.085	0.087	0.064	0.062
88	0.022	0.058	0.084	0.090	0.087	0.064
103	0.028	0.024	0.104	0.091	0.077	0.066
121	0.039	0.025	0.101	0.085	0.073	0.066
140	0.000	0.000	0.100	0.100	0.067	0.068
167	0.024	0.029	0.098	0.089	0.070	0.068
173	0.022	0.018	0.091	0.085	0.061	0.053
190	0.024	0.027	0.101	0.095	0.071	0.068

Sample no.	imidacloprid mg/kg		Isoprothiolane, mg/kg		Metalaxyl, mg/kg	
	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
9	0.070	0.085	0.360	0.398	0.070	0.080
19	0.088	0.078	0.419	0.396	0.077	0.069
31	0.073	0.066	0.420	0.359	0.072	0.068
51	0.064	0.062	0.368	0.355	0.066	0.064
88	0.087	0.064	0.392	0.425	0.070	0.067
103	0.077	0.066	0.403	0.361	0.077	0.071
121	0.073	0.066	0.379	0.361	0.072	0.071
140	0.067	0.068	0.000	0.000	0.073	0.072
167	0.070	0.068	0.392	0.407	0.070	0.073
173	0.061	0.053	0.335	0.314	0.064	0.066
190	0.071	0.068	0.343	0.361	0.071	0.070

Sample no.	Oxathiapronil mg/kg		Profenofos, mg/kg		Pyriproxyfen, mg/kg		Thiamethoxam, mg/kg	
	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
9	0.053	0.058	0.111	0.131	0.160	0.161	0.053	0.059
19	0.060	0.053	0.175	0.132	0.181	0.158	0.062	0.052
31	0.052	0.054	0.166	0.046	0.149	0.143	0.053	0.051
51	0.052	0.053	0.090	0.089	0.164	0.160	0.050	0.049
88	0.051	0.048	0.102	0.179	0.181	0.157	0.055	0.048
103	0.062	0.059	0.077	0.052	0.152	0.164	0.058	0.055
121	0.052	0.052	0.097	0.082	0.160	0.147	0.052	0.051
140	0.054	0.059	0.000	0.000	0.143	0.167	0.053	0.052
167	0.055	0.054	0.064	0.062	0.173	0.171	0.054	0.053
173	0.052	0.047	0.044	0.030	0.144	0.142	0.045	0.043
190	0.058	0.051	0.034	0.069	0.173	0.164	0.053	0.053

Appendix 4 Stability figures

The stability test was performed according to ISO 13528, Annex B [5]. Two different storage temperatures were used; room temperature and -18 °C.

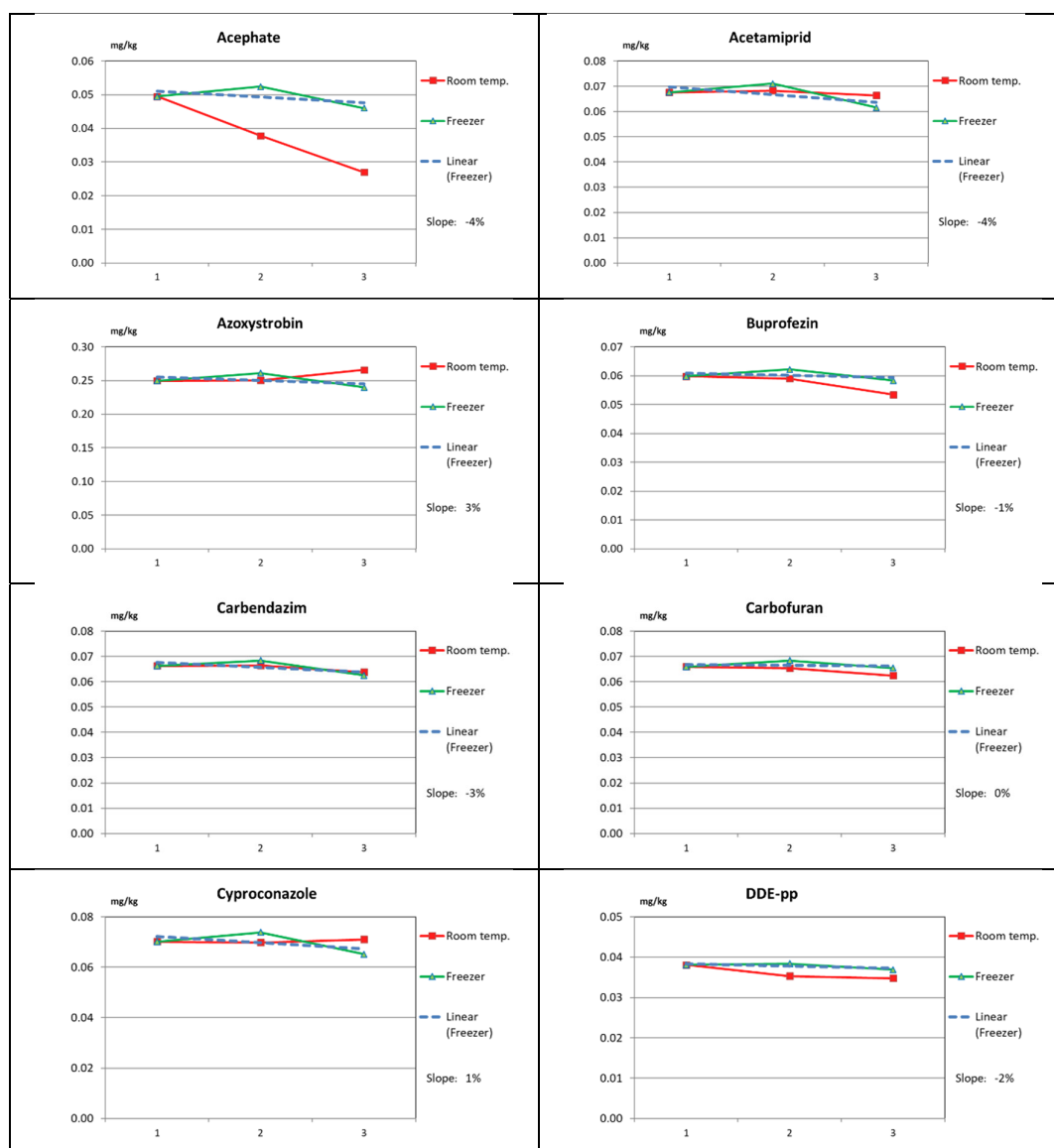
The dates of testing were as follows:

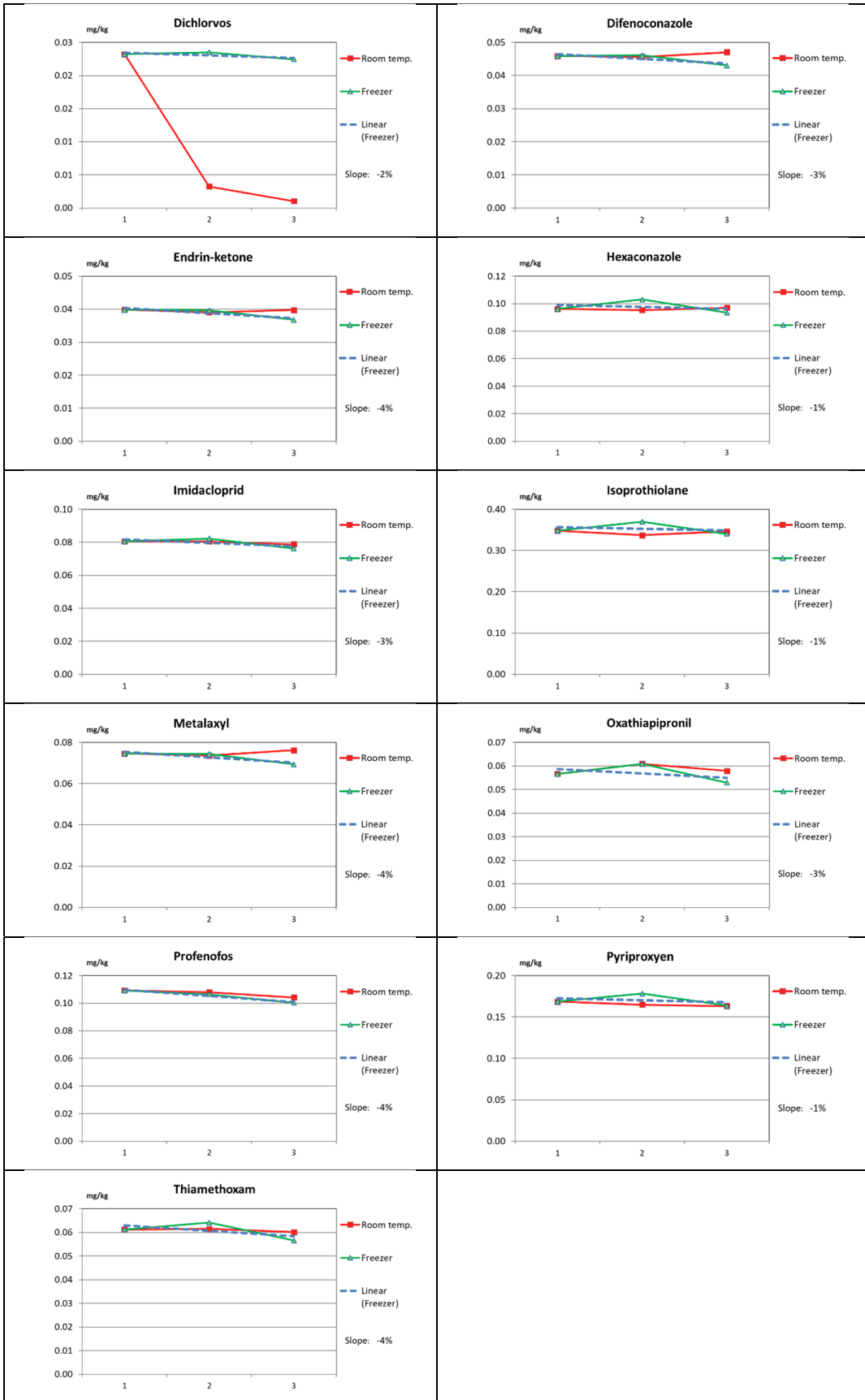
Day 1: 8 June 2020

Day 2: 13 July 2020

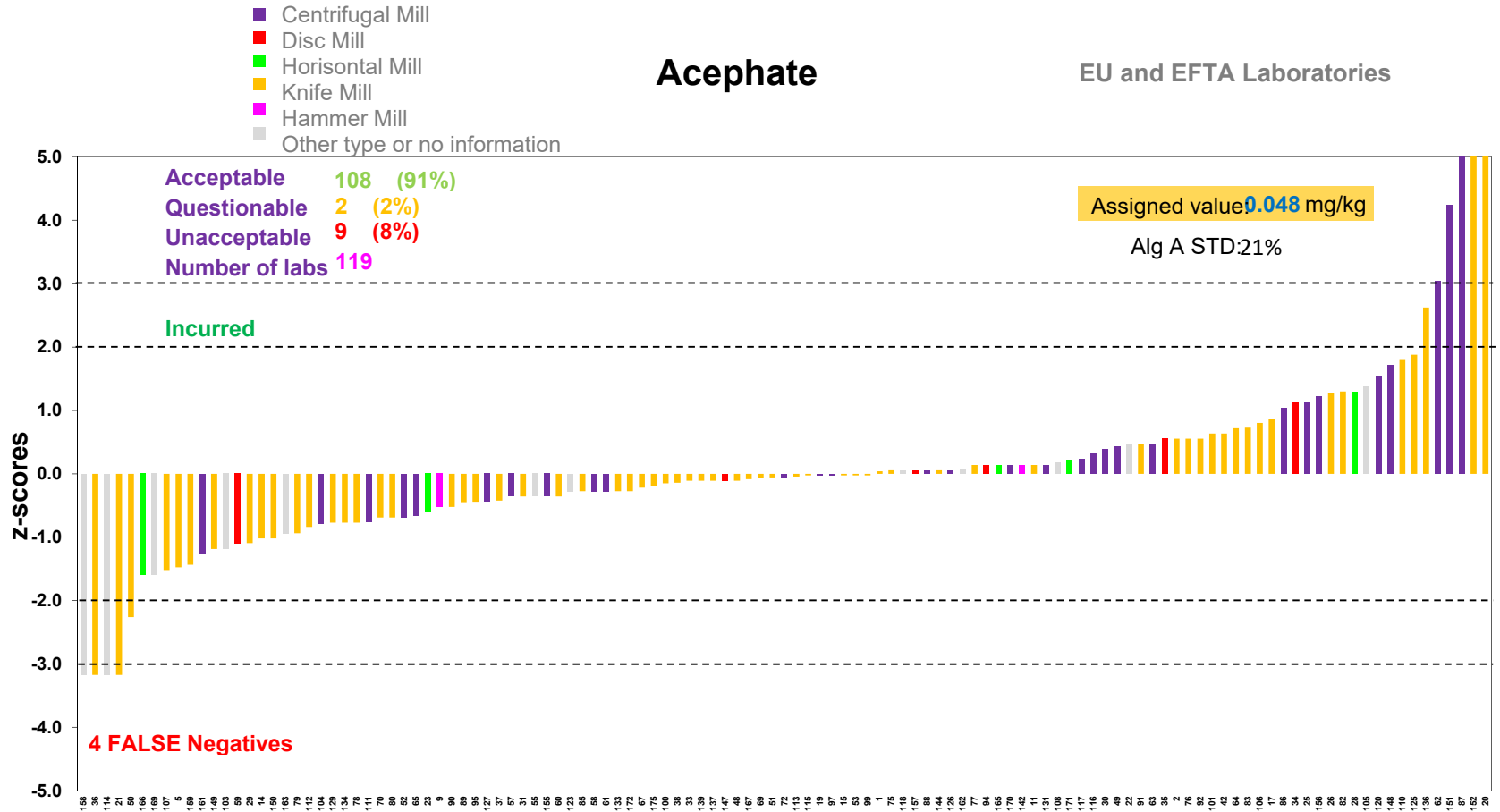
Day 3: 17 August 2020

All pesticides passed the test at -18 °C, see **1.6 Stability test**. However, acephate, buprofezin, DDE-pp and dichlorvos did not pass the test when stored for 11 weeks at room temperature.





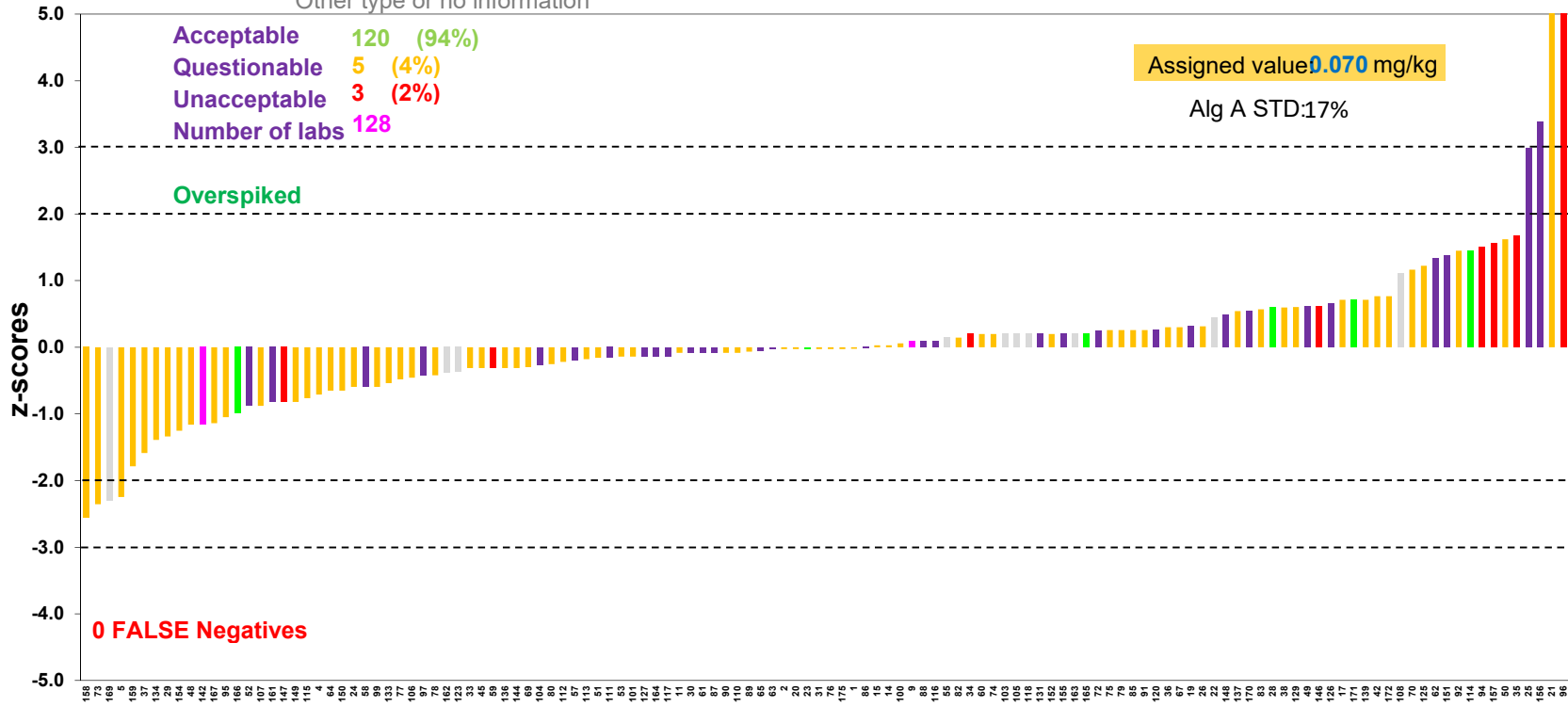
Appendix 5 Graphical presentation of z-scores



Acetamidrid

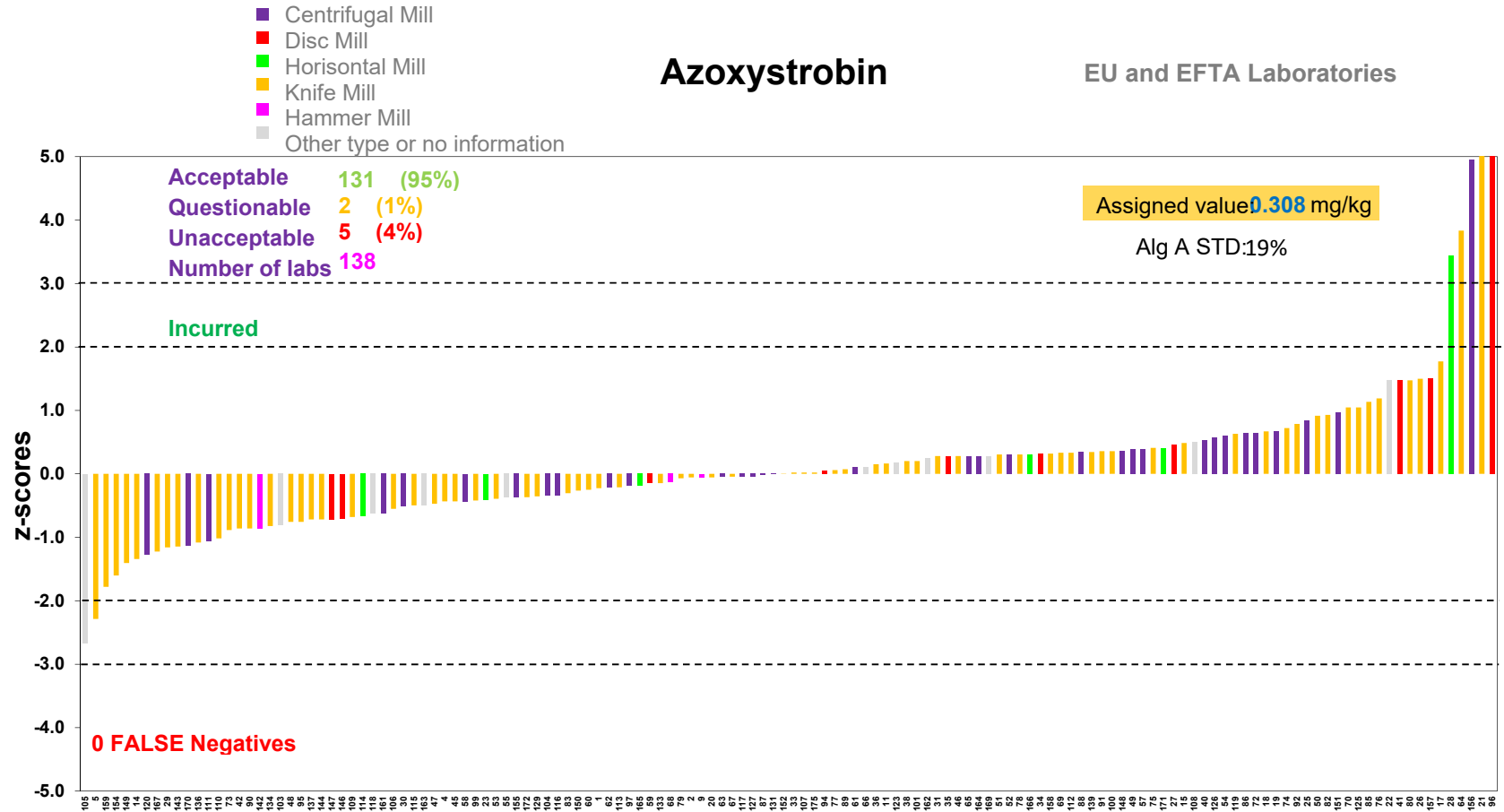
EU and EFTA Laboratories

- Centrifugal Mill
- Disc Mill
- Horizontal Mill
- Knife Mill
- Hammer Mill
- Other type or no information



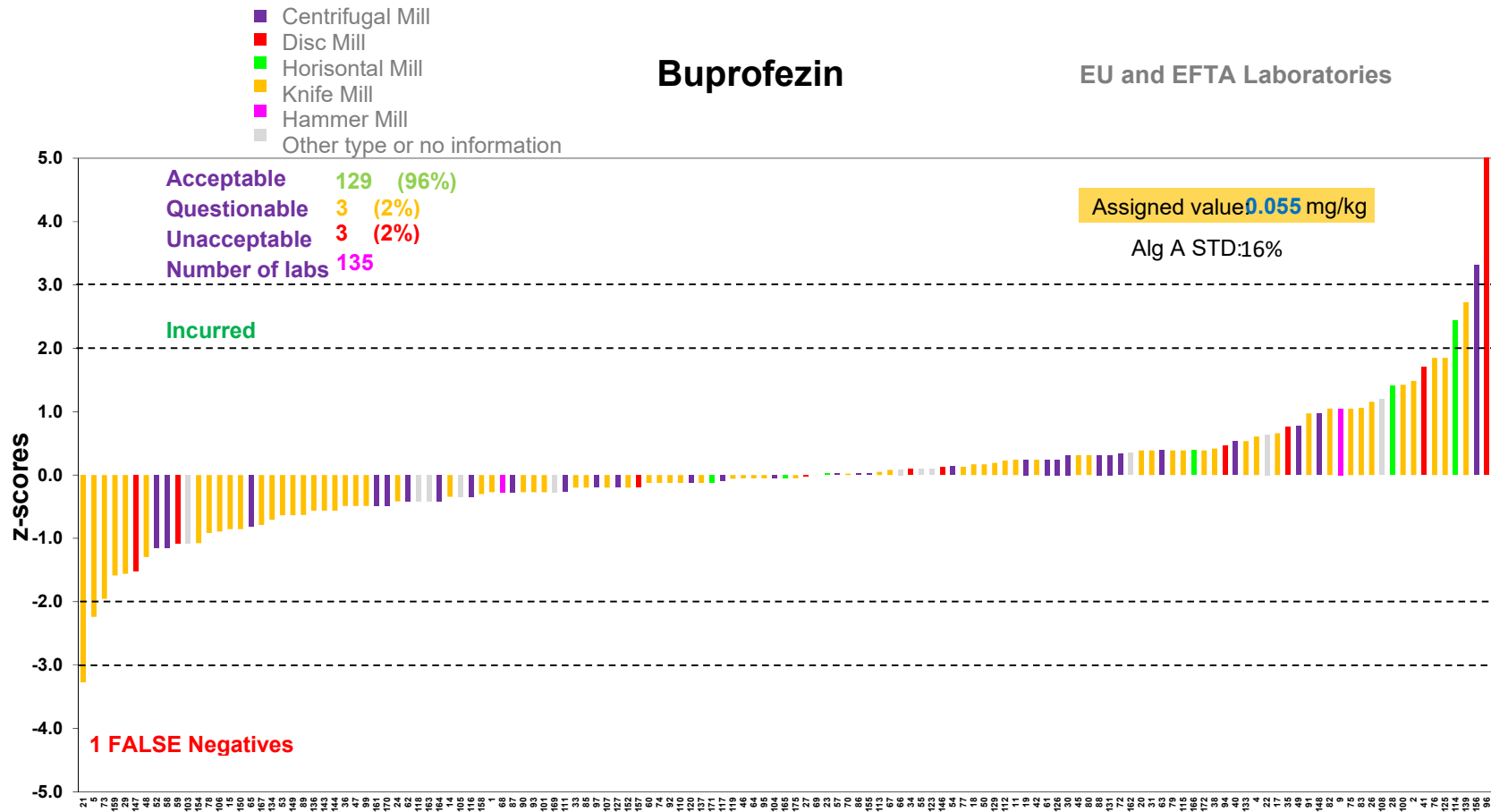
Azoxystrobin

EU and EFTA Laboratories



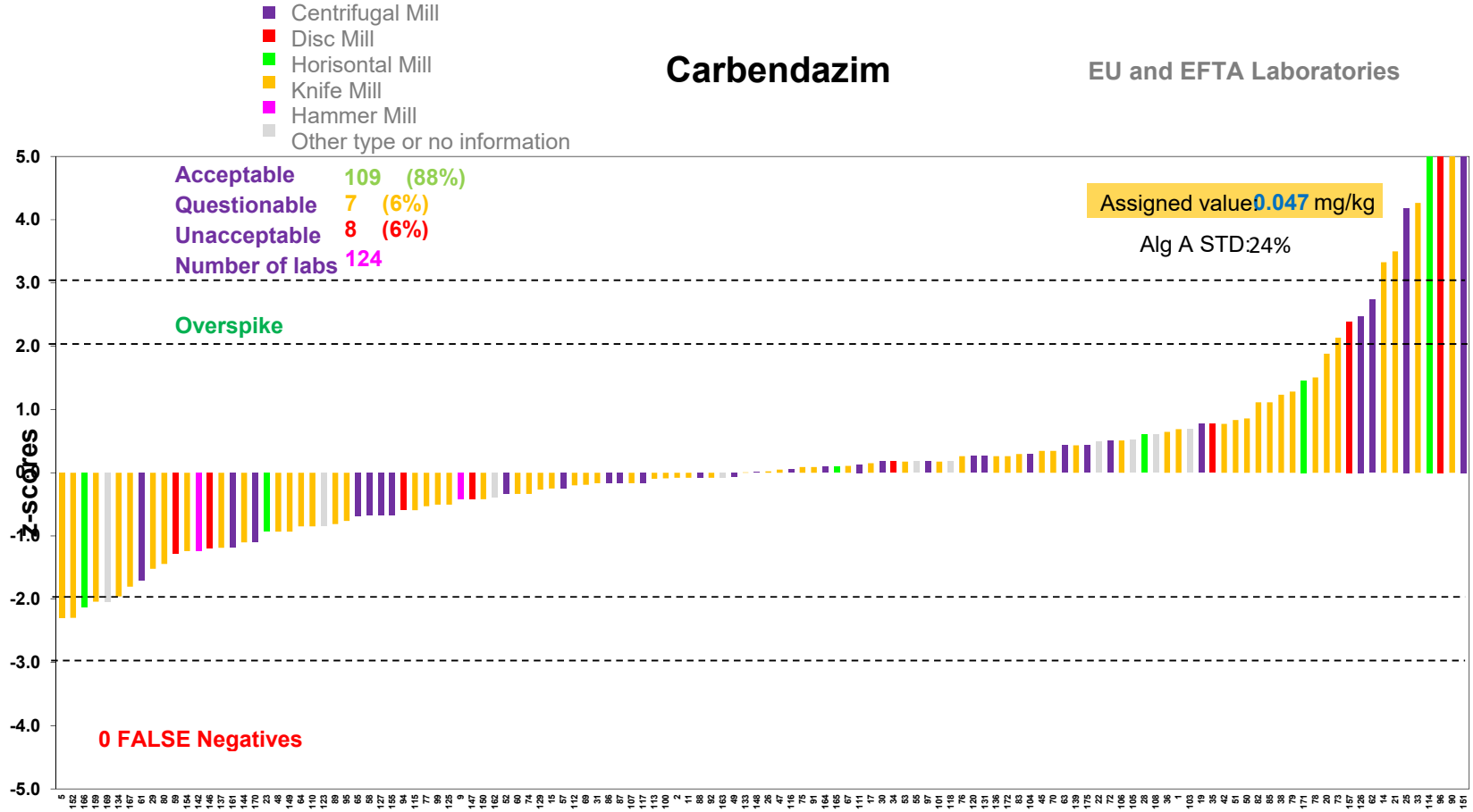
Buprofezin

EU and EFTA Laboratories



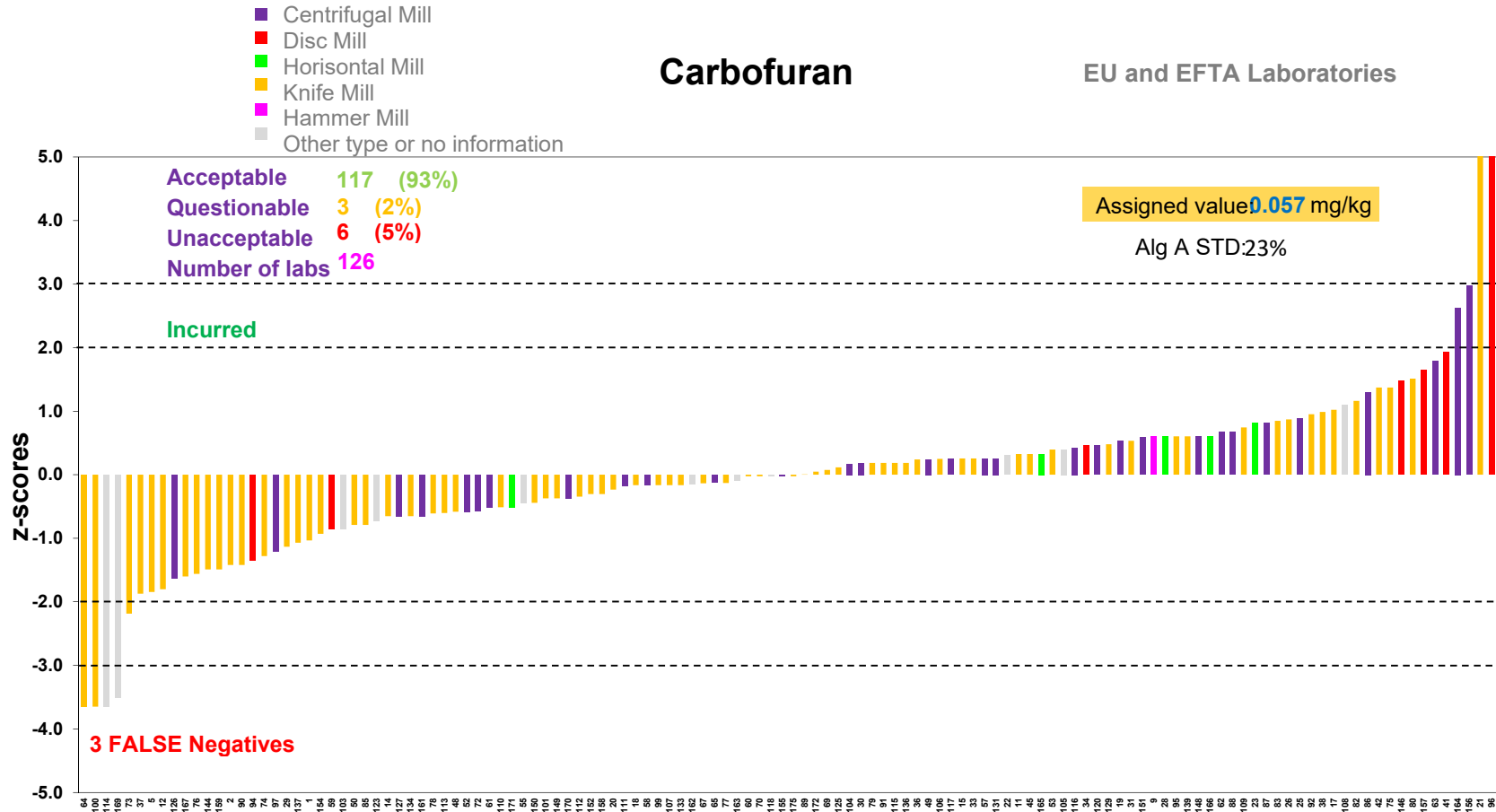
Carbendazim

EU and EFTA Laboratories



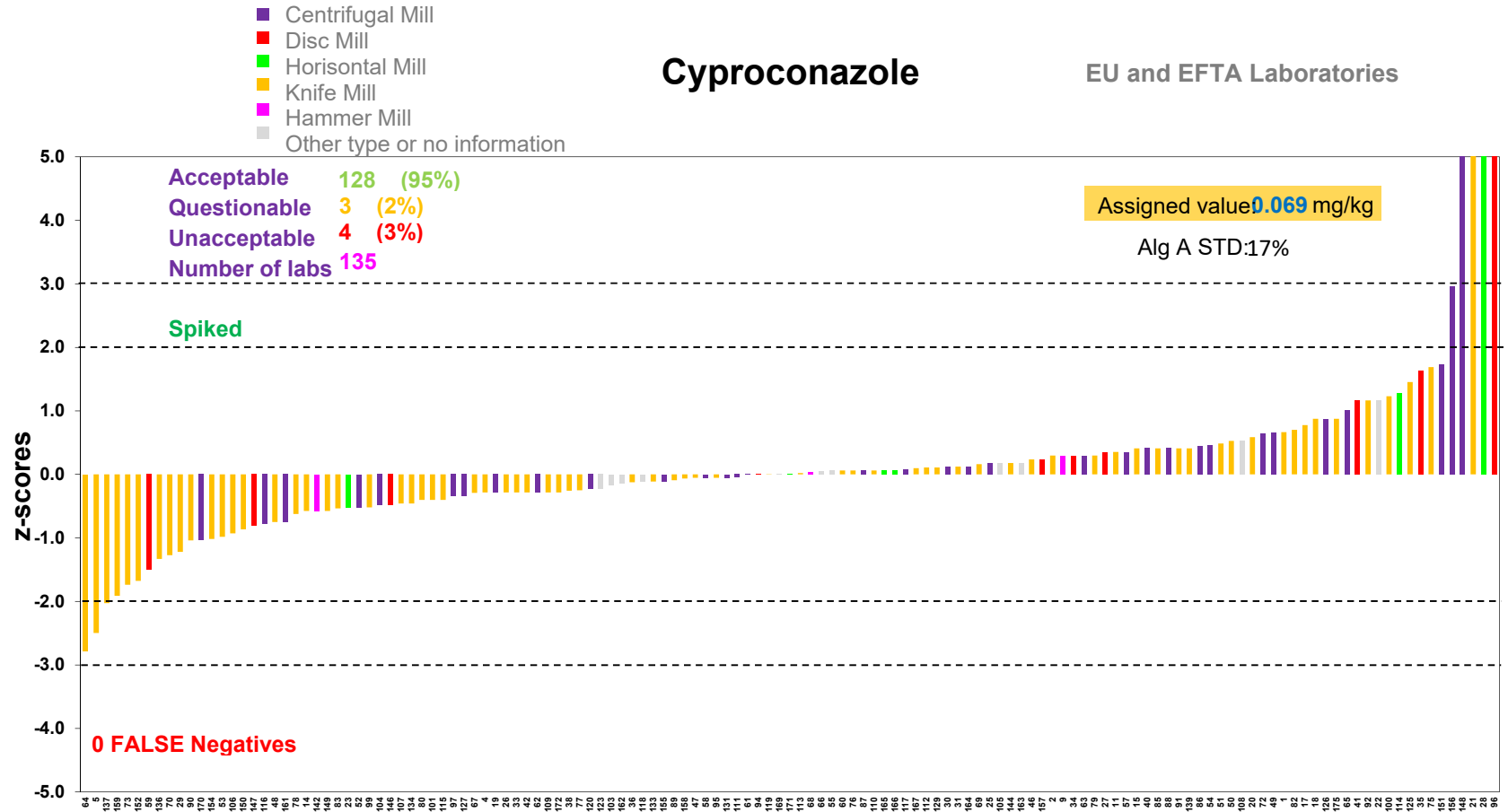
Carbofuran

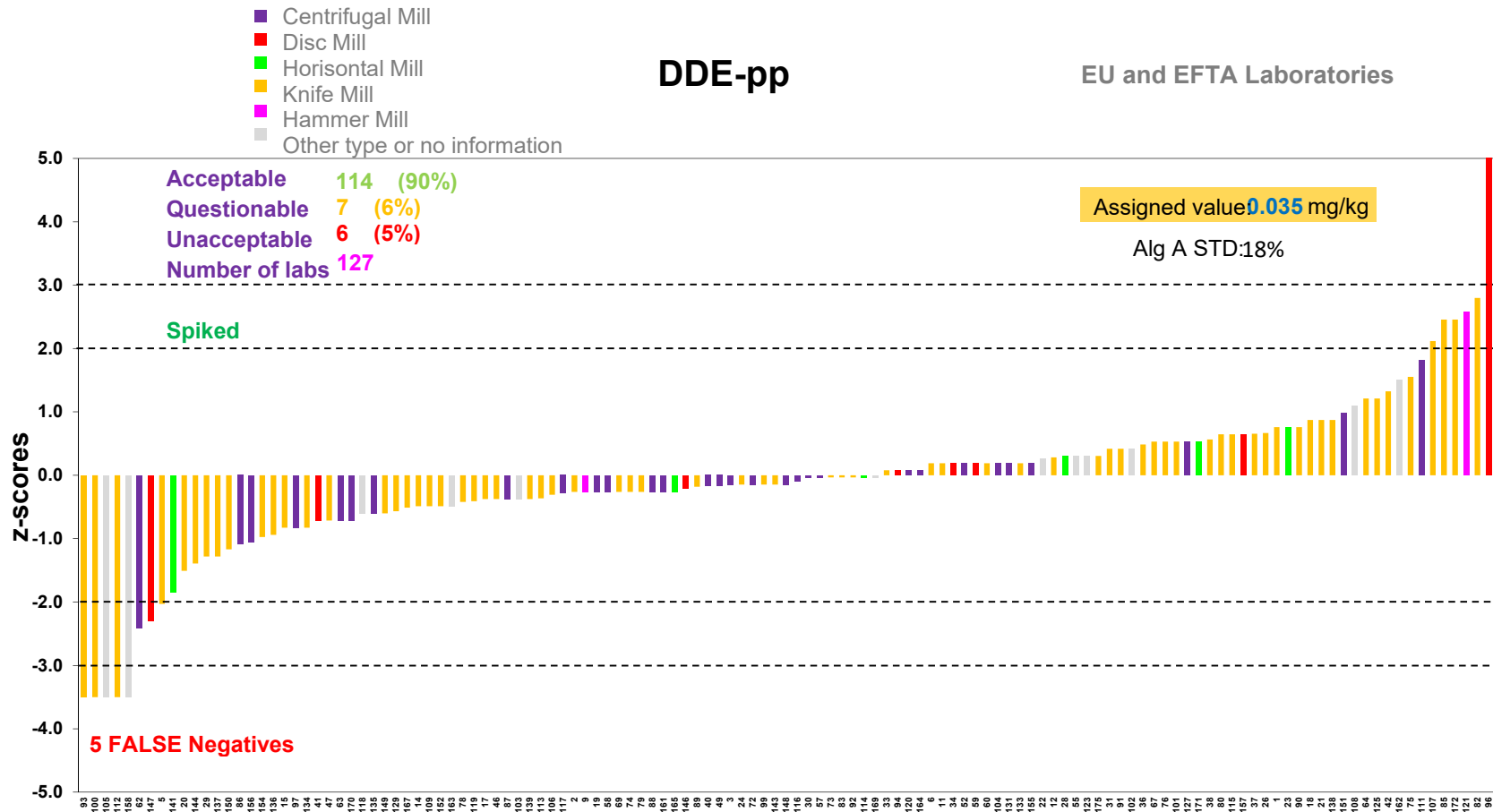
EU and EFTA Laboratories



Cyproconazole

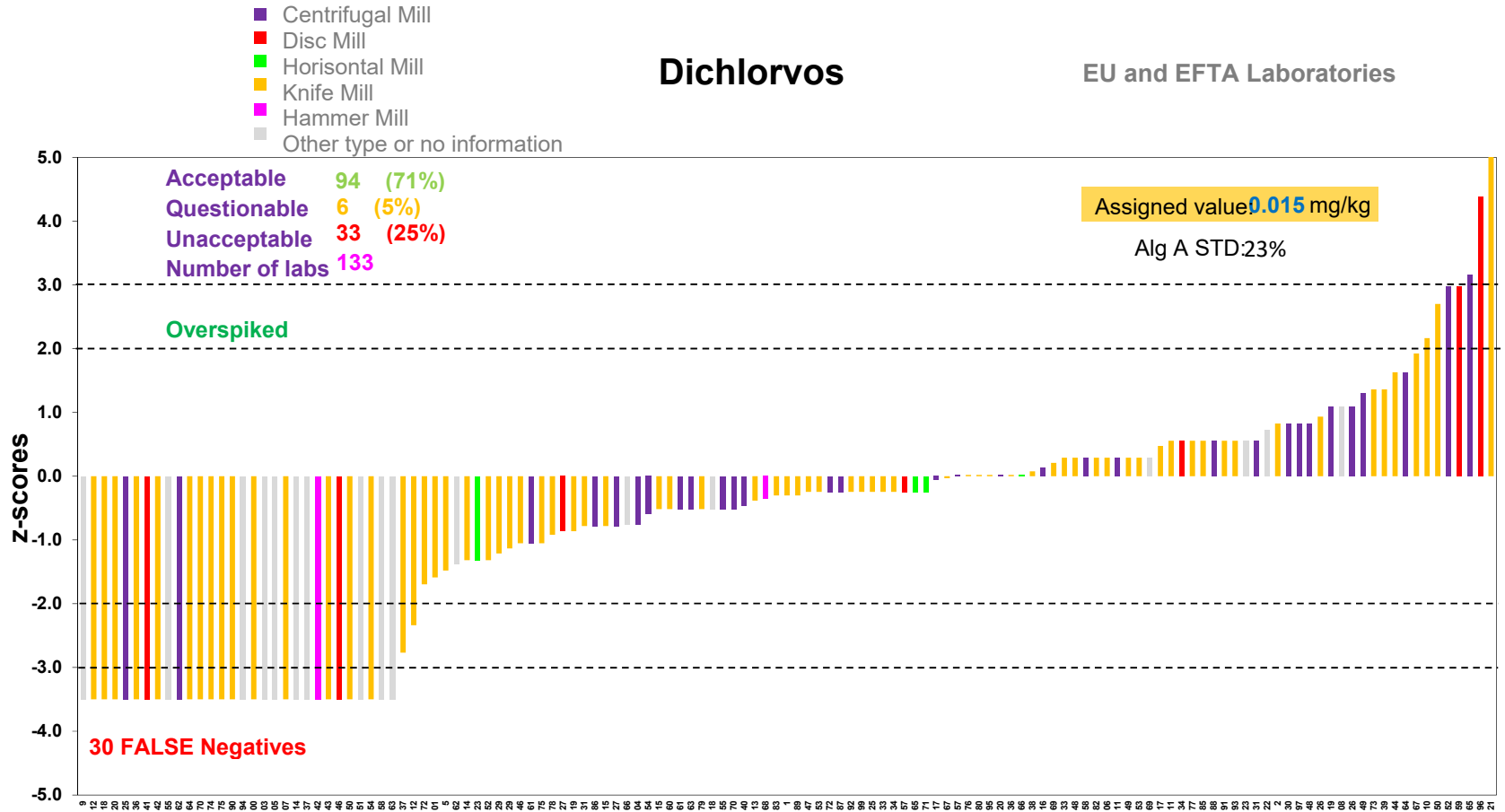
EU and EFTA Laboratories





Dichlorvos

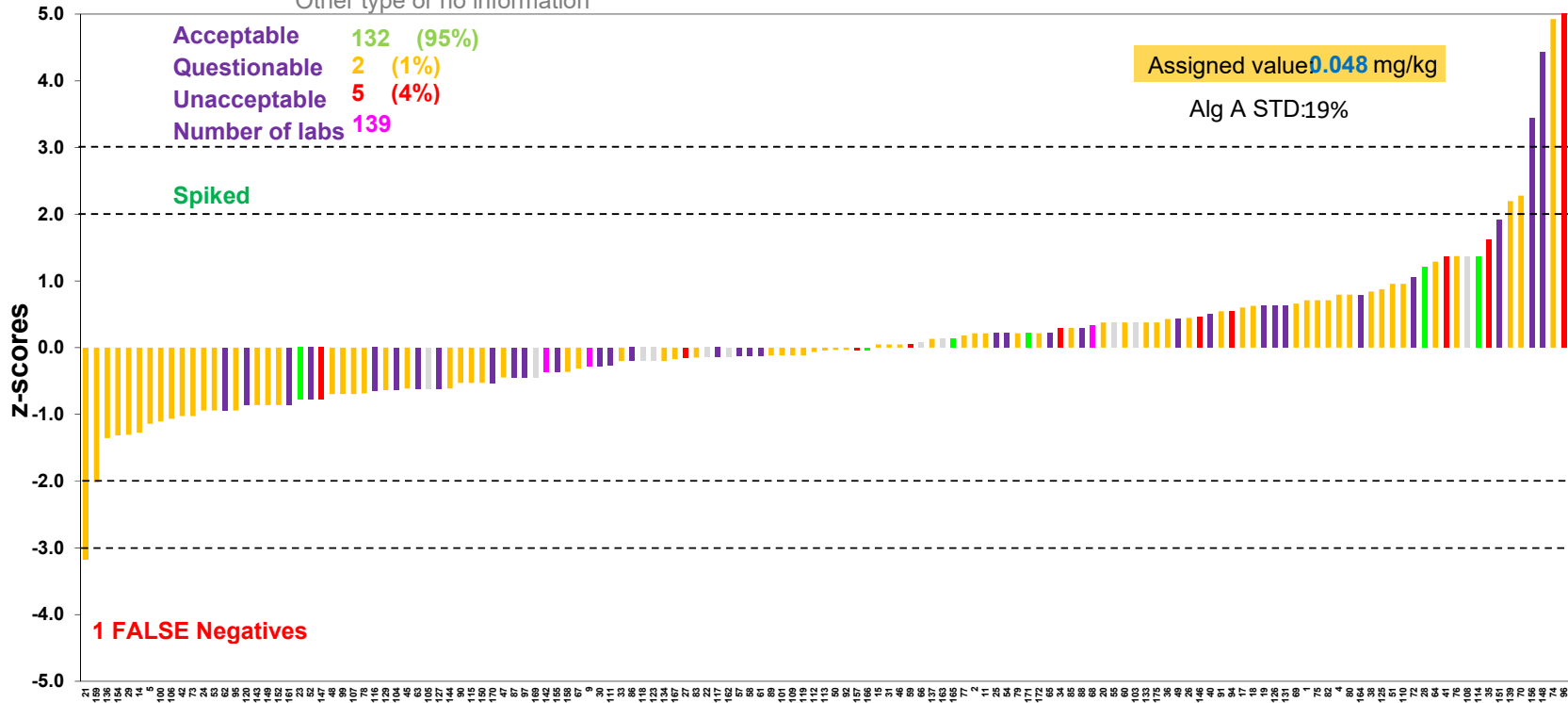
EU and EFTA Laboratories



Difenoconazole

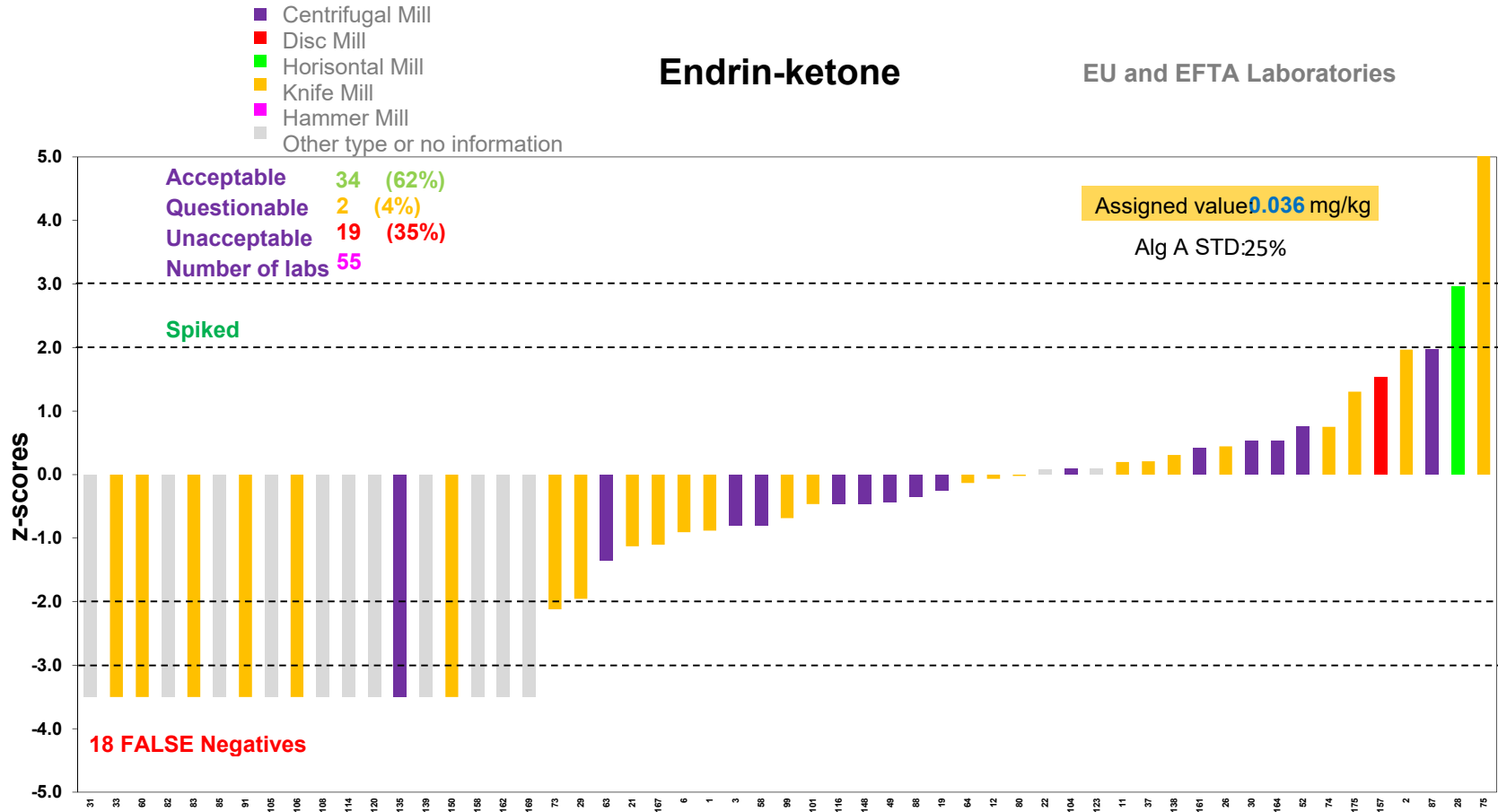
EU and EFTA Laboratories

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- Disc Mill
- Horizontal Mill
- Knife Mill
- Hammer Mill
- Other type or no information



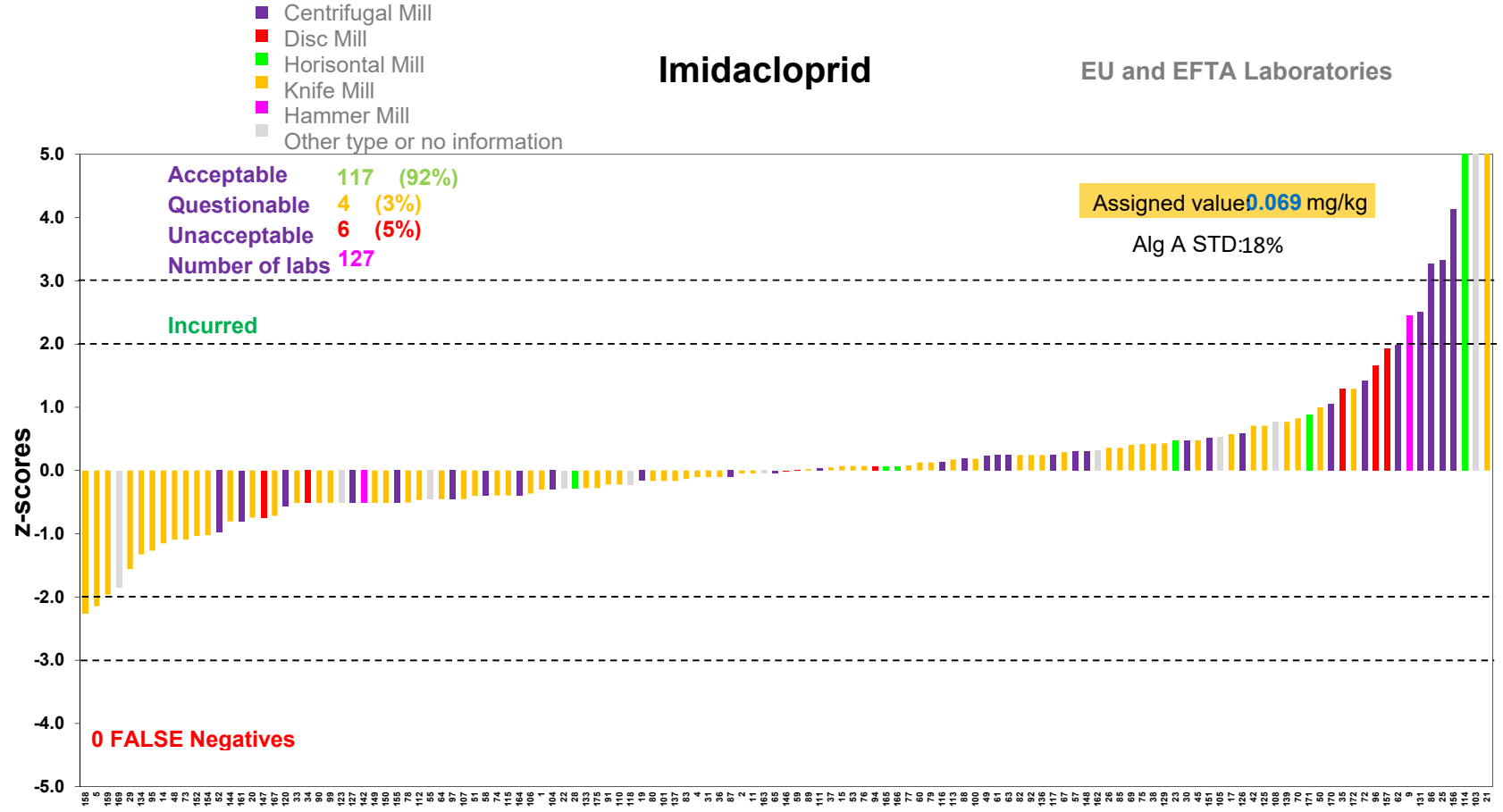
Endrin-ketone

EU and EFTA Laboratories



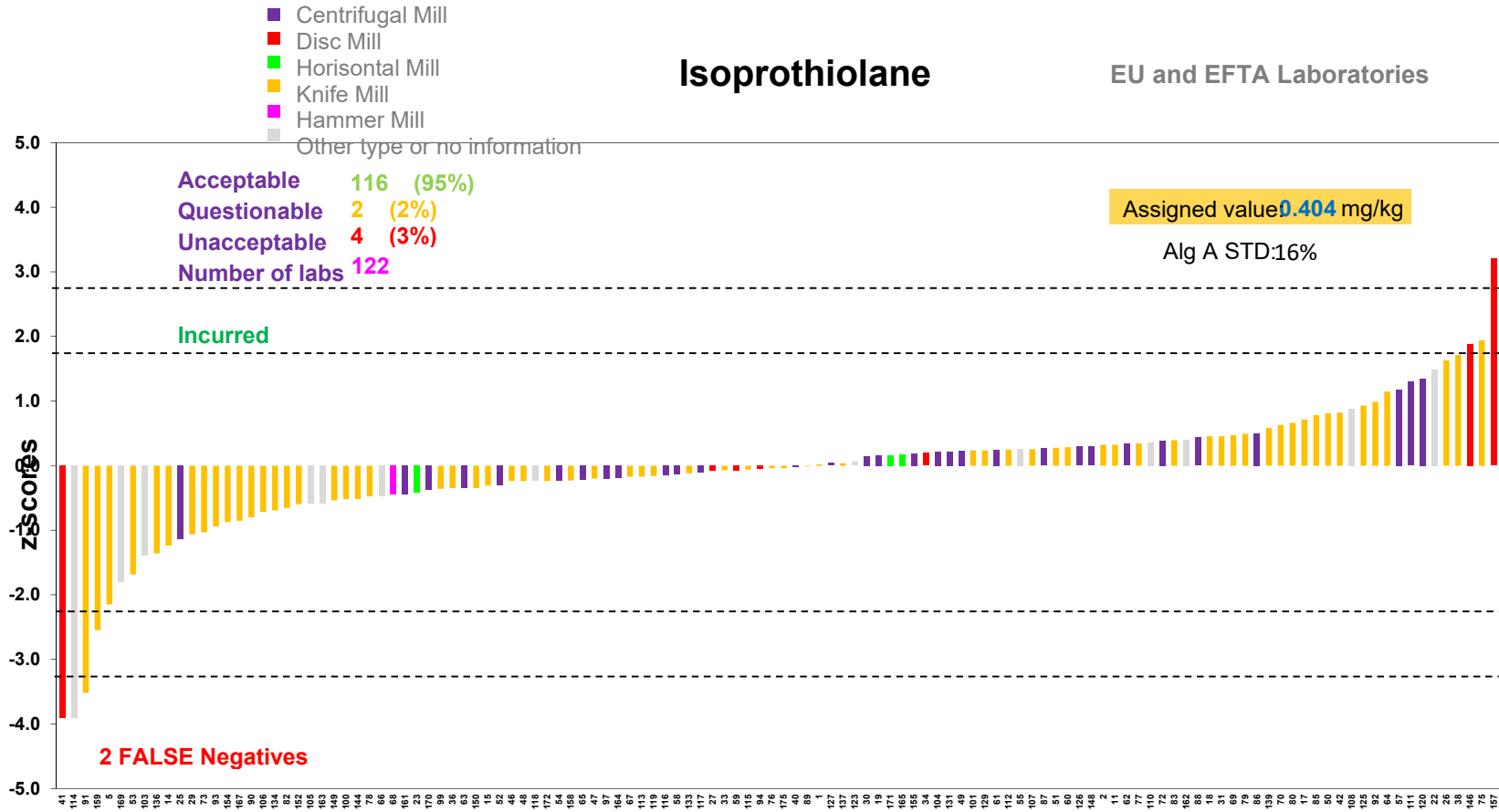
Imidacloprid

EU and EFTA Laboratories



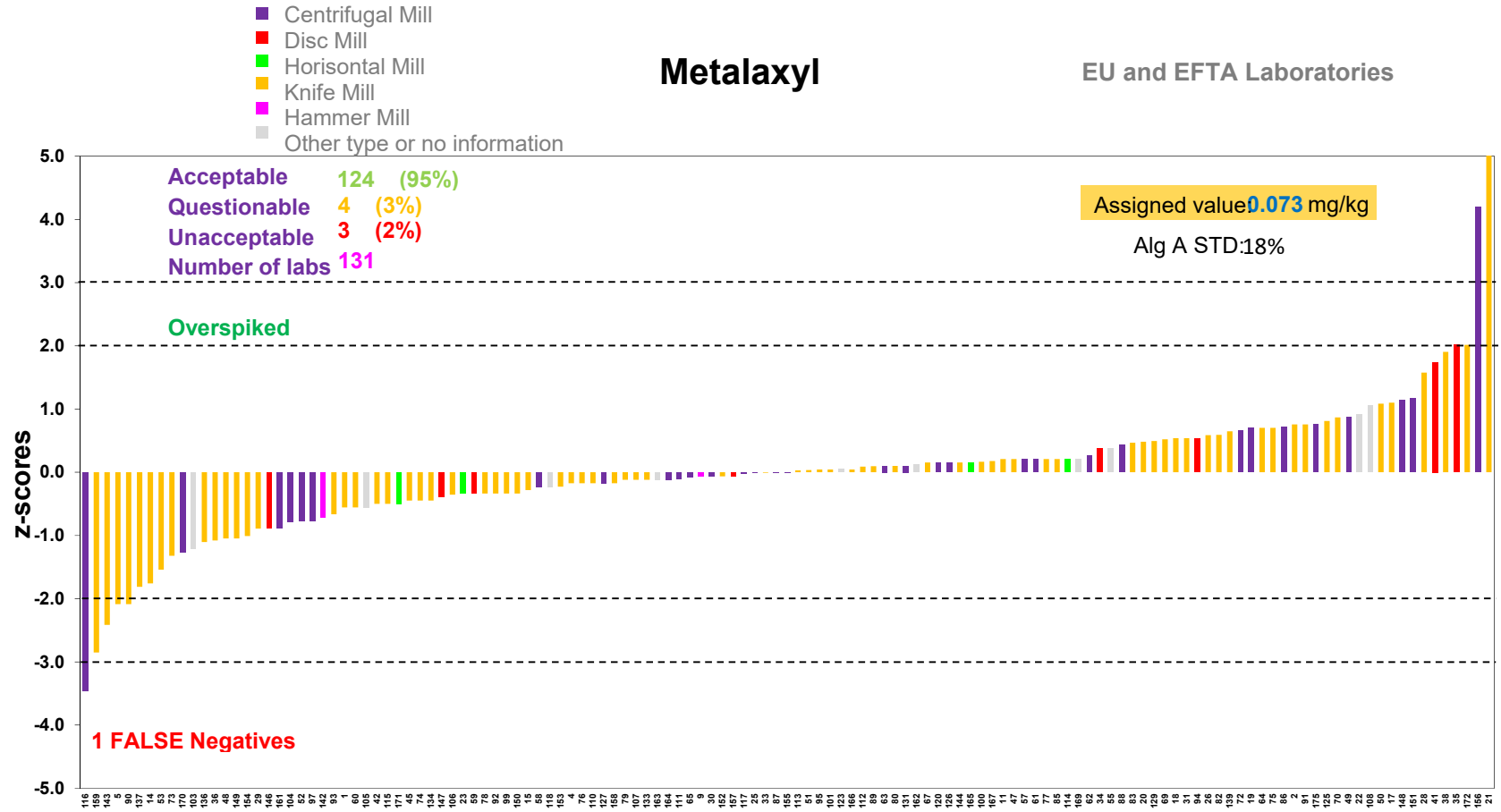
Isoprothiolane

EU and EFTA Laboratories



Metalaxyl

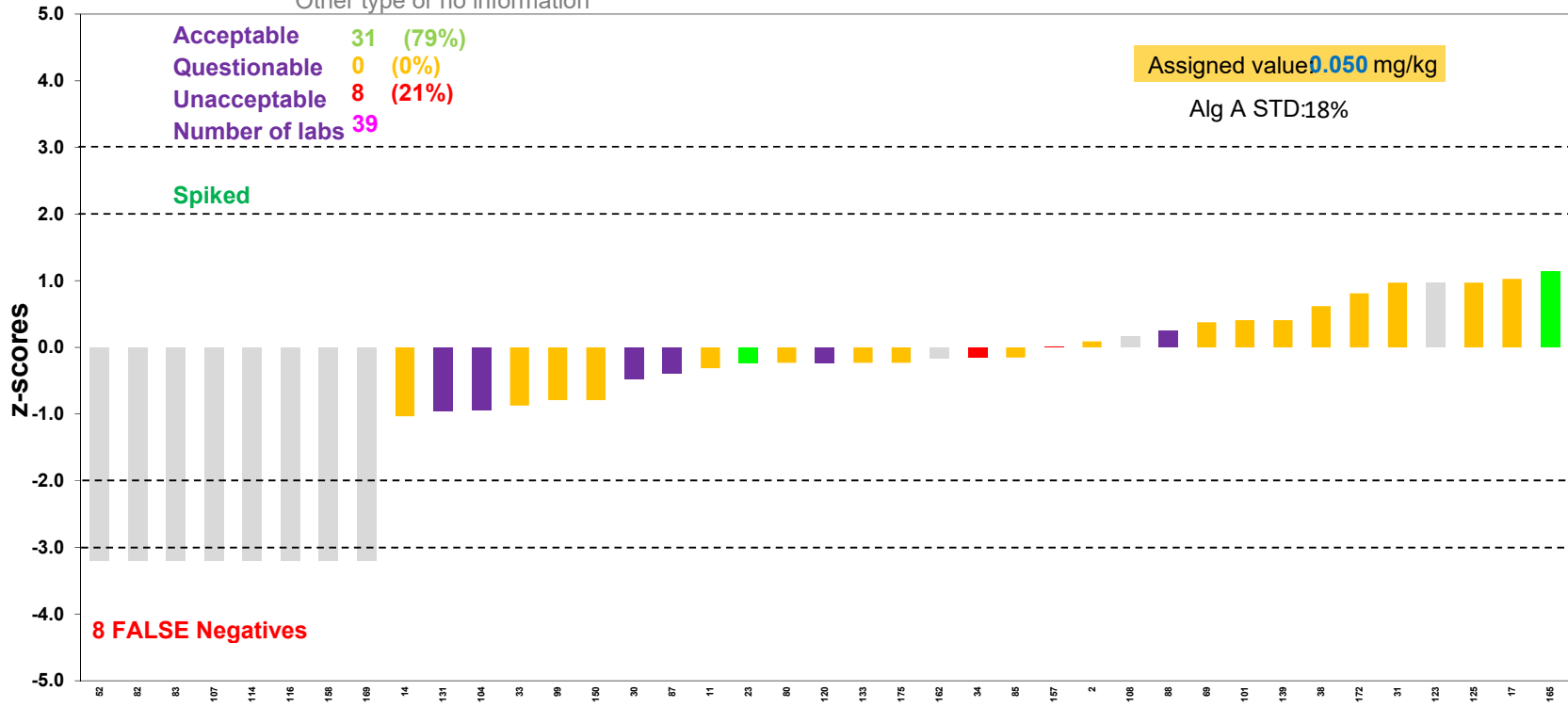
EU and EFTA Laboratories



Oxathiapiprolin

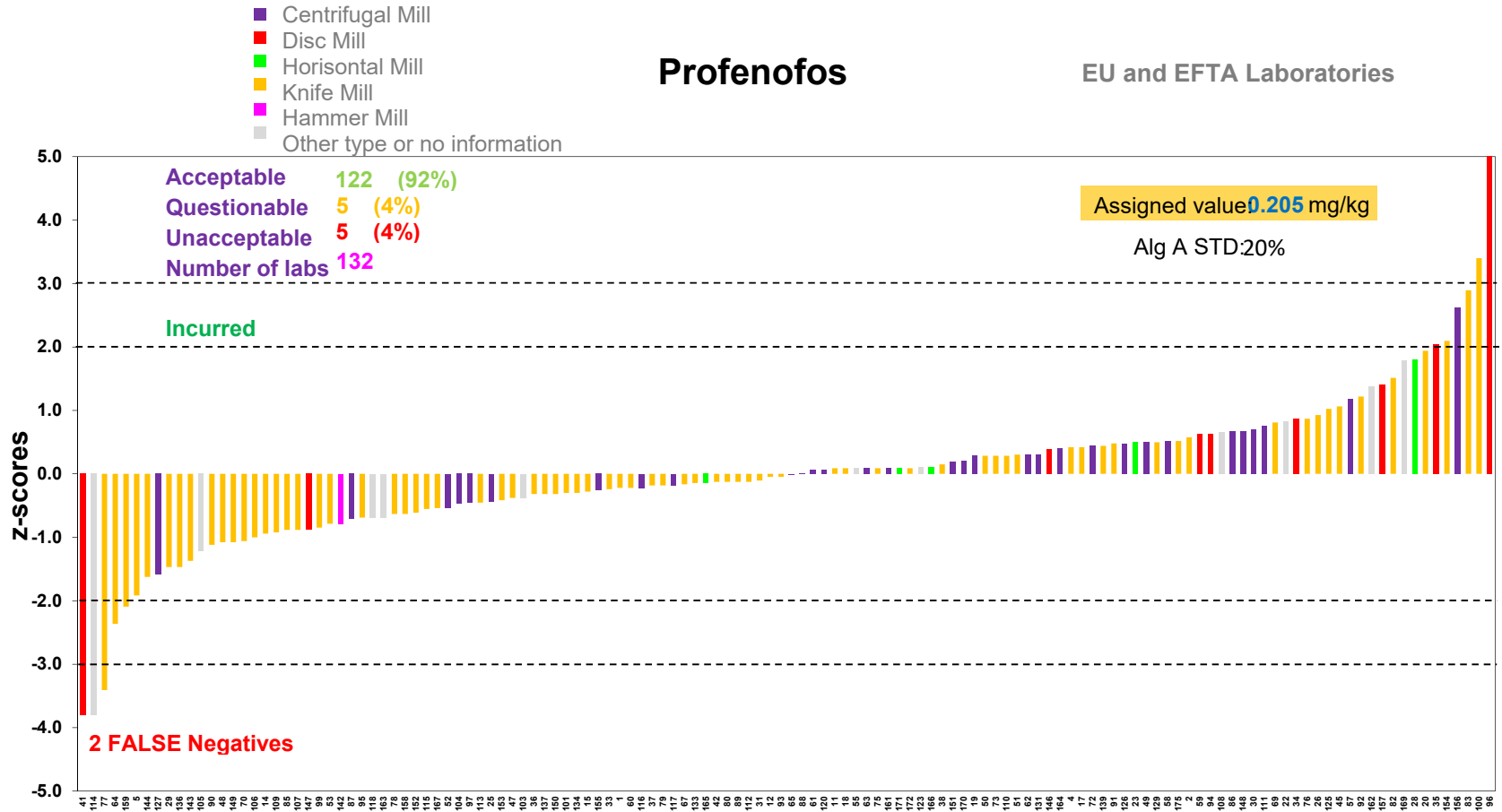
EU and EFTA Laboratories

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- Horizontal Mill
- Knife Mill
- Hammer Mill
- Other type or no information



Profenofos

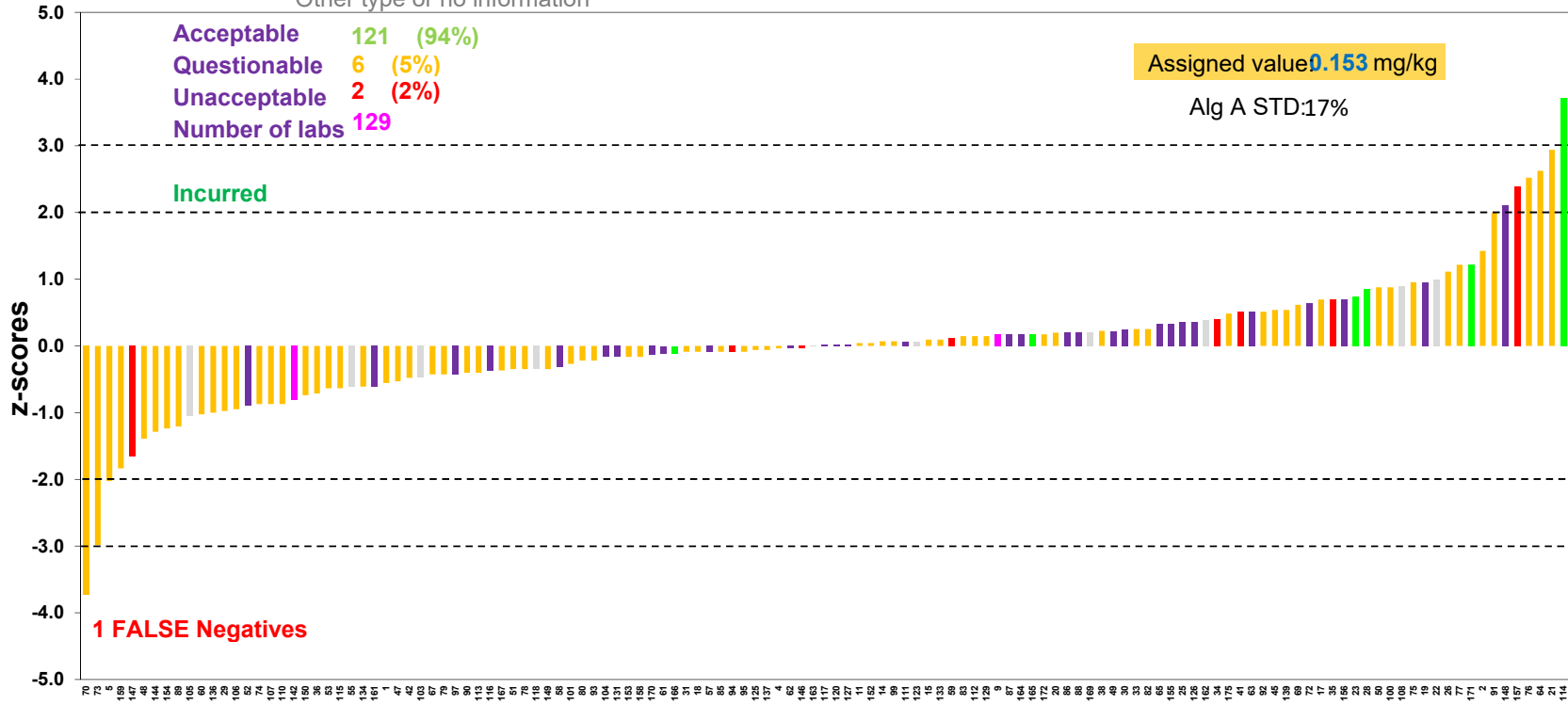
EU and EFTA Laboratories



Pyriproxyfen

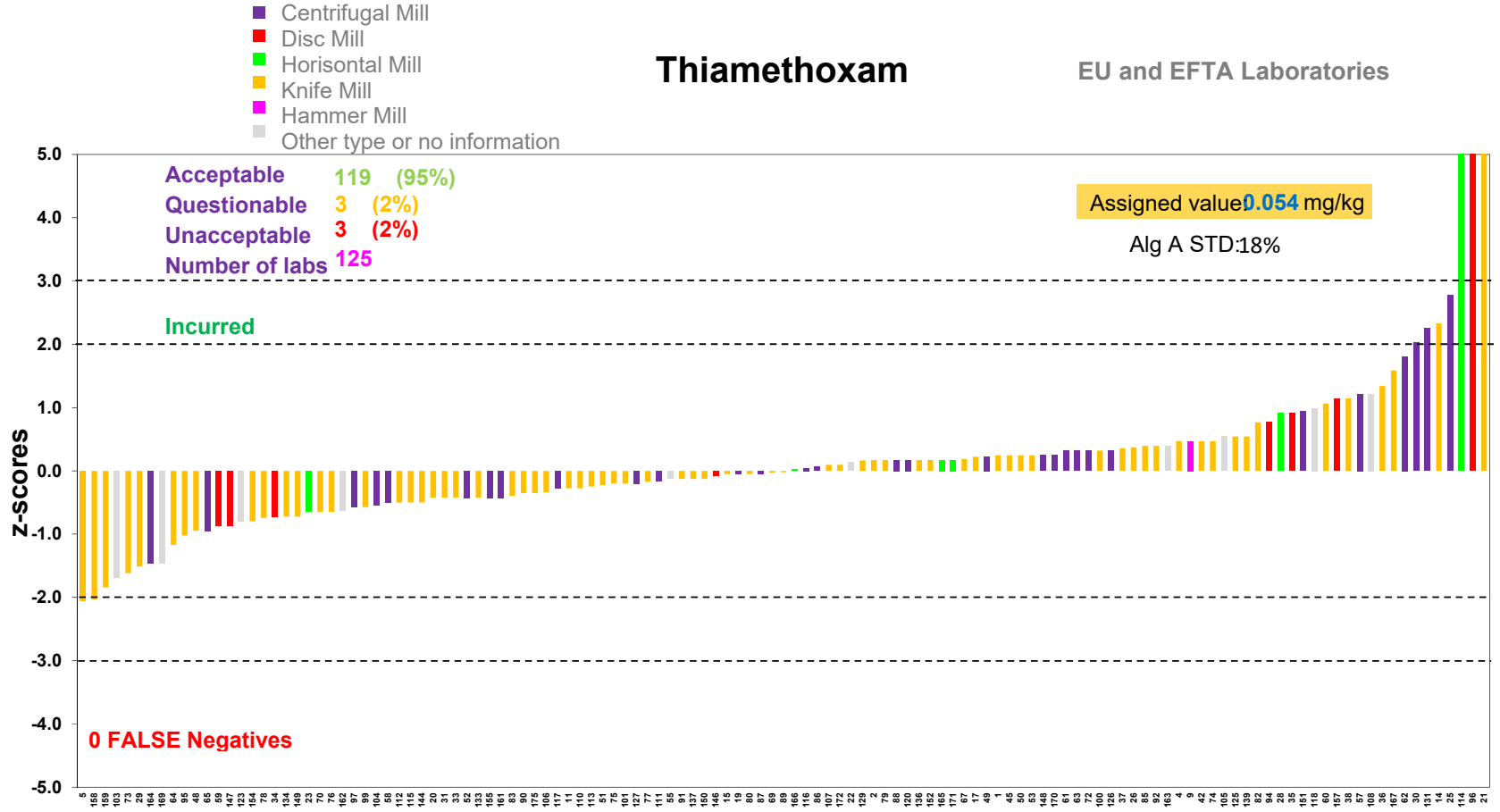
EU and EFTA Laboratories

- Centrifugal Mill
- Disc Mill
- Horizontal Mill
- Knife Mill
- Hammer Mill
- Other type or no information



Thiamethoxam

EU and EFTA Laboratories





GENERAL PROTOCOL

for EU Proficiency Tests on Pesticide Residues

in Food and Feed

Introduction

This protocol contains general procedures valid for all European Union Proficiency Tests (EUPTs) organised on behalf of the European Commission, DG-SANTE¹ 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 Network² of National Reference Laboratories (NRLs) and Official Laboratories (OfLs) of the EU Member States. OfLs from EFTA countries and EU-Candidate countries are also welcome to participate in the EUPTs. OfLs from Third countries may be permitted to participate on a case-by-case basis.

The following four EURLs for pesticide residues were appointed by DG-SANTE based on regulation 882/2004/EC that was repealed by regulation 625/2017/EC³:

- 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 programme⁴. Participating laboratories will be provided with an assessment of their analytical performance that

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

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

³ Regulation (EU) 2017/625 of the European Parliament and of the Council on official controls and other official activities performed to ensure the application of food and feed law, rules on animal health and welfare, plant health and plant protection products.. Published at OJ of the EU L95 of 07.04.2017

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



they can use to demonstrate their analytical performance and compare themselves with other participating laboratories.

EUPT-Organisers and Scientific Committee

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

An **Organising Team** (in the following named Organisers) is appointed by the EURL(s) in charge. This team is responsible for all administrative and technical matters concerning the organisation of the PT, e.g. the PT-announcement, the production of the PT-material (Test Item), the undertaking of homogeneity and stability tests, the packing and shipment of the PT-materials, the handling and evaluation of the results and method information submitted by the participants, the drafting of the preliminary and final reports as well as generation and distribution of EUPT-participation certificates.

To complement the internal expertise of the EURLs, a group of external consultants forming the **EUPT-Scientific Committee** (EUPT-SC)⁵ has been established and approved by DG-SANTE. The EUPT-SC consists of expert scientists with many years of experience in PTs and/or pesticide residue analysis. The actual composition of the EUPT-SC and the affiliation of each of its members is shown on the EURL-Website. The members of the EUPT-SC are also listed in the Specific Protocol and the Final Report of each EUPT.

The EUPT-SC is made up of the following two subgroups:

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

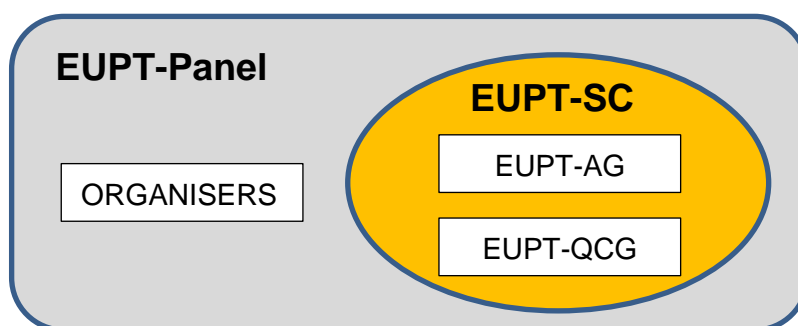
The EUPT-SC's role is to help the Organisers make decisions regarding the EUPT design: the selection of the commodity, the selection of pesticides to be included in the Target Pesticide List (see below), the establishment of the Minimum Required Reporting Levels (MRRLs), the statistical treatment and evaluation of the participants' results (in anonymous form), and the drafting and updating of documents, such as the General and Specific PT Protocols and the Final EUPT-Reports.

⁵ Link to the List of current members of the EUPT Scientific Committee:
<http://www.eurl-pesticides.eu/library/docs/allcrl/EUPT-SC.pdf>

The EUPT-QCG has the additional function of supervising the quality of EUPTs and of assisting the EURLs in confidential aspects such as the choice of the pesticides to be present in the Test Item and the approximate concentrations at which they should be present.

The EUPT-SC typically meets once a year, after the EUPTs of all four pesticide EURLs have been conducted, to discuss the evaluation of the EUPT-results and to assist the EURLs in their decision making. Upcoming EUPTs are also planned during these meetings.

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



The decisions of the EUPT-Panel will be documented.

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

EUPT Participants

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

- Art 38 (b) of Reg. 625/2017/EC and Art. 28 of Reg. 396/2005/EC⁶ (for all OfLs analysing for pesticide residues within the framework of official controls⁷ of food or feed)
- Art. 101 (1)(a) of Reg. 625/2017/EC (for all NRLs)

⁶ 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.

⁷ Official controls in the sense of Reg. 625/2017/EC. This includes labs involved in controls within the framework of national and/or EU-controlled programmes as well as labs involved in import controls according to Regulation 669/2009/EC.

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 obliged laboratories with their actual commodity-scopes and contact information.

OfLs are furthermore urged to keep their own profiles within the EURL-DataPool up-to-date, especially their commodity and pesticide scopes and their contact information.

Labs that are obliged to participate in a given EUPT, and that are not able to participate, must provide the reasons for their non-participation. This also applies to any participating laboratories that fail to report results.

OfLs not paying the EUPT sample delivery fee will be initially warned that their participation in subsequent EUPTs could be denied. In case of a repetitive non-payment, the EUPT organisers will inform the corresponding NRL to take action.

Confidentiality and Communication

The proprietor of all EUPT data is DG-SANTE and as such has access to all information.

For each EUPT, the laboratories are given a unique code (lab code), initially only known to themselves and the Organisers. In the final EUPT-Report, the names of participating laboratories will not be linked to their laboratory codes. It should be noted, however, that the Organisers, at the request by DG-SANTE, may present the EUPT-results on a country-by-country basis. It may therefore be possible that a link between codes and laboratories could be made, especially for those countries where only one laboratory has participated. Furthermore, the EURLs reserve the right to share EUPT results and codes amongst themselves: for example, for the purpose of evaluating overall lab or country performance as requested by DG-SANTE.

As laid down in Regulation 625/2017/EC, NRLs are responsible for evaluating and improving their own OfL-Network. On request from the NRLs, the EURLs will provide them with the PT-codes of the participating OfLs belonging to their OfL-Network. This will allow NRLs to follow the participation and performance of the laboratories within their network.

Communication between participating laboratories during the test, on matters concerning a PT exercise, is not permitted from the start of the PT exercise until the distribution of the preliminary report.

For each EUPT the organising EURL prepares a specific EUPT-Website where all PT-relevant documents in their latest version are linked. In case of important modifications on any of these documents, the participating laboratories will be informed via e-mail. In any case, as soon as the PT-period starts the participants are encouraged to visit the particular EUPT-Website, to make sure that they are using the latest versions of all PT-relevant documents.

The official language used in all EUPTs is English.

Announcement / Invitation Letter

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

Target Pesticide List

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

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

Specific Protocol

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



Homogeneity of the Test Item

The Test Item will be tested for homogeneity typically before distribution to participants. The homogeneity tests usually involve the analysis of two replicate analytical portions, taken from at least ten randomly chosen units of treated Test Item. Both, sample preparation and measurements should be conducted in random order.

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

Stability of the analytes contained in the Test Item

The Test Items will also be tested for stability - according to ISO 13528, Annex B. The time delay between the first and the last stability test must exceed the period of the EUPT-exercise. Typically the first analysis is carried out shortly before the shipment of the Test Items and the last one shortly after the deadline for submission of results. To better recognise trends and gain additional certainty one or more additional tests may be conducted by the Organisers. At least 6 sub-samples (analytical portions) should be analysed on each test day (e.g. 2 analytical portions withdrawn from three randomly chosen containers OR 6 portions withdrawn from a single container). In principle all pesticides contained in the Test Item should be checked for stability. However, in individual cases, where sufficient knowledge exists that the stability of a certain analyte is very unlikely to be significantly affected during storage (e.g. based on experience from past stability tests or knowledge of its physicochemical properties), the Organisers, after consultation with the EUPT-QCG, may decide to omit a specific stability test. The EUPT-Panel will finally decide whether



analytes for which the stability test was not undertaken will be included in the Final EUPT-Report, considering all relevant aspects such as the distribution of the participant's results (CV*).

A pesticide is considered to be adequately stable if $|y_i - y| \leq 0.3 \times \sigma_{pt}$, with y_i being the mean value of the results of the last phase of the stability test, y being the mean value of the results of the first phase of the stability test and σ_{pt} being the standard deviation used for proficiency assessment (typically 25 % of the assigned value).

The results of all stability tests are presented to the EUPT-SC. In special cases where the above stability test criteria are not met, the EUPT-SC considering all relevant aspects (e.g. the past experience with the stability of the compound, the overall distribution the participants' results, the measurement variability, analytical difficulties faced during the test and knowledge about the analytical behaviour of the pesticide question) may decide to overrule the test. The reasons of this overruling will be transparently explained in the Final EUPT-Report.

The Organisers may also decide to conduct additional stability tests at different storage conditions than those recommended to the participants e.g. at ambient temperature.

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

Methodologies to be used by the participants

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



General procedures for reporting results

Participating laboratories are responsible for reporting their own quantitative results to the Organiser within the stipulated deadline. Any pesticide that was targeted by a participating laboratory should be reported as “analysed”. Each laboratory will be able to report only one result for each analyte detected in the Test Item. The concentrations of the pesticides detected should be expressed in ‘mg/kg’ unless indicated otherwise in the specific protocol. Laboratories should not report results below their reporting limits.

Correction of results for recovery

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

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

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

Methodology information

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

Results evaluation

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

– **False Positive results**

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

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

– **False Negative results**

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

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

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

In order to minimise the influence of out-lying results on the statistical evaluation, the assigned value x_{pt} (= consensus concentration) will typically be estimated using the robust estimate of the

participant's mean (\bar{x}^*) as described in ISO 13528:2015⁸, taking into account the results reported by EU and EFTA countries laboratories only. In special justifiable cases, the EUPT-Panel may decide to eliminate certain results traceably associated with gross errors (see "Omission or Exclusion of results" below) or to use only the results of a subgroup consisting of laboratories that have repeatedly demonstrated good performance for the specific or similar compounds in the past.

– ***Omission or Exclusion of results***

Before estimating the assigned value, results associated with obvious mistakes have to be examined to decide whether they should be removed from the population. Such gross errors may include incorrect recording (e.g. due to transcription errors by the participant, decimal point faults or transposed digits, incorrect unit), calculation errors (e.g. missing factors), analysis of a wrong sample/extract (e.g. a spiked blank), use of wrong concentrations of standard solutions, incorrect data processing (e.g. integration of wrong peak), inappropriate storage or transport conditions (in case of susceptible compounds), and the use of inappropriate analytical steps or procedures that demonstrably lead to significantly biased results (e.g. employing inappropriate internal standards or analytical steps or conditions leading to considerable losses, due to degradations, adsorptions, incomplete extractions, partitioning etc.). Where the Organisers (e.g. after the publication of the preliminary report) receive information of such gross errors, having a significant impact on a generated result, the affected results will be examined on a case-by-case basis to decide whether, or not, they should be excluded from the population used for robust statistics. Results may also be omitted e.g. if an inappropriate method has been used even if they are not outliers. All decisions to omit/exclude results will be discussed with the EUPT-SC and the reasoning for the omission of each result clearly stated in the Final EUPT-Report. However, z scores will be calculated for all results irrespective of the fact that they were omitted from the calculation of the assigned value.

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

Results reported by laboratories from non EU member states are typically excluded from the population that is used to derive the assigned value (see also "Estimation of the assigned value").

⁸ 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).

– ***Uncertainty of the assigned value***

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

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

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

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 ($FFP-\sigma_{pt}$) will be calculated using a Fit-For-Purpose approach with a fixed Relative Standard Deviation (FFP-RSD).

Based on experience from previous EUPTs⁹, a percentage FFP-RSD of 25 % is currently used for all analyte-matrix combination, with the target standard deviation being calculated as follows:

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

The EUPT-Panel reserves the right to also employ other FFP-RSDs or other approaches for setting the assigned value on a case-by-case basis, considering analytical difficulties and experience gained from previous proficiency tests.

For informative purposes the robust relative standard deviation (CV^*) of the participants results is calculated according to ISO 13528:2015; Chapter 7.7 following Algorithm A in Annex C (so called “consensus approach”).

⁹ 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.

– **z scores**

This parameter is calculated using the following formula:

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

where x_i is the value reported by the laboratory, x_{pt} is the assigned value, and $FFP - \sigma_{pt}$ is the standard deviation using the FFP approach. Z scores will be rounded to one decimal place. For the calculation of combined z scores (see below) the original z scores will be used and the combined z-scores will be rounded to one decimal place after calculation.

Any z scores > 5 will be typically reported as '> 5' and a value of '5' will be used to calculate combined z scores (see below).

Z scores will be interpreted in the following way, as is set in the ISO 17043:2010¹⁰:

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

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

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

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

¹⁰ ISO/IEC 17043:2010. Conformity assessment – General requirements for proficiency testing

– **Category classification**

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

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

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

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

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

For evaluation of the overall performance of laboratories within Category A, the Average of the Squared z score (AZ^2)^{11,12} (see below) will be used. The AZ^2 is calculated as follows:

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

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

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

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

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

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

¹¹ Formerly named “Sum of squared z scores (SZ^2)”

¹² 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.



Publication of results

The EURLs will publish a preliminary report, containing tentative assigned values and z score values for all pesticides present in the Test Item, within 2 months of the deadline for result submission.

The Final EUPT-Report will be published after the EUPT-Panel has discussed the results. Taking into account that the EUPT-Panel meets normally only once a year (typically in late summer or autumn) to discuss the results of all EUPTs organised by the EURLs earlier in the year, the Final EUPT-Report may be published up to 10 months after the deadline for results submission. Results submitted by non-EU/EFTA laboratories might not always be used in the tables or figures in the Final EUPT-Report.

Certificates of participation

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

Feedback

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

Correction of errors

Should errors be discovered in any of the documents issued prior to the EUPT (Calendar, Target Pesticides List, Specific Protocol, General Protocol) the corrected documents will be uploaded onto the website and in the case of substantial errors the participants will be informed. **Before starting the exercise, participants should make sure to download the latest version of these documents.**

If substantial errors are discovered in the Preliminary EUPT-Report the Organisers will distribute a new corrected version, where it will be stated that the previous version is no longer valid.

Where substantial errors are discovered in the Final EUPT-Report the EUPT-Panel will decide whether a corrigendum will be issued and how this should look like. The online version of the Final EUPT report will be replaced by the new one and all affected labs will be contacted.

Where errors are discovered in EUPT-Certificates the relevant laboratories will be sent new corrected ones. Where necessary the laboratories will be asked to return the old ones.

Follow-up activities

Laboratories are expected to undertake follow-up activities to trace back the sources of erroneous or strongly deviating results (typically those with $|z| > 2.0$) - including all false positives. In exceptional cases, follow-up activities may even be indicated for results within $|z| \leq 2.0$ (e.g. where two errors with opposed tendency cancel each other leading to acceptable results).

Upon request, the laboratory's corresponding NRL and EURL are to be informed of the outcome of any investigative activities for false positives, false negatives and for results with $|z| \geq 3.0$. Concerning z scores between 2.0 and 3.0 the communication of the outcome of follow-up activities is optional but highly encouraged where the source of deviation could be identified and could be of interest to other labs.

According to instructions from DG-SANTE, the "Protocol for management of underperformance in comparative testing and/or lack of collaboration of National Reference Laboratories (NRLs) with EU Reference Laboratories (EURLs) activities" is to be followed.

NRLs will be considered as **underperforming in relation to scope** if in at least two of the last four EUPTs falling within their responsibility area they: a) haven't participated, or b) targeted less than 90% of the compulsory pesticides in the target lists (80% for SRM-compounds), or c) detected less than 90% of the compulsory compounds present in the test items (80% for SRM-compounds). Additionally, NRLs that obtained AZ^2 higher than 3 (AAZ higher than 1.3 for SRM-compounds) in two consecutive EUPTs of the last four EUPTs, will be considered as **underperforming in accuracy**. A two-step protocol established by DG-SANTE will be applied as soon as underperformance of an NRL is detected¹³:

Phase 1:

- Identifying the origin of the bad results (failure in EUPTs).

¹³ Article 101 of Regulation (EC) 625/2017

- 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.

SPECIFIC PROTOCOL

for the EU Proficiency Test for Pesticide Residues in Cereals/Feeding stuff using Multi Residue Methods, EUPT-CF14 (2020)

(last updated: 19 May 2020)

Introduction

This protocol is complementary to the [General Protocol for EU Proficiency Tests for Pesticide Residues in Food and Feed](#) (9th Edition). The current proficiency test covers pesticides that are determined by Multi Residue Methods. This EUPT is to be performed by all National Reference Laboratories for Cereals and/or Feeding stuffs (NRL-CFs) as well as by all official EU laboratories (OfLs) responsible for official pesticide residue controls on cereals and/or feeding stuff, as far as their scope overlaps with that of the EUPT-CF14.

Test Item (Test Material)

This proficiency test concerns the analysis of pesticide residues rice kernels. The rice was grown in India and pesticides were applied in the field.

The Organiser, will check the Test Items for sufficient homogeneity and for stability at conditions reproducing sample shipment and storage during the duration of the test, according to ISO 13528, Annex B. All these tests will be conducted by the organiser, the EURL-CF which is (ISO 17025 accredited).

Analytical Parameters

The Test Item contains several pesticides from the **Target Pesticides List**.

Laboratories must report their results as stated in the Target Pesticides List.

Amount of Test Item

The participants will receive:

- approximately 100 g of rice kernel Test Item with incurred and spiked pesticides and

Blank material will not be distributed to the participants.

Shipment of Test Items

The Test Items are planned to be shipped on 8 June 2020.

Test Items will be shipped frozen and packed in thermo-boxes together with a freezer block. The organiser will aim to ensure that all participating laboratories will receive their shipments on the same day. Prior to shipment a reminder will be sent to the participating laboratories by e-mail.

Laboratories must make their own arrangements for the receipt of the package. They should inform the Organiser of any public holidays in their country/city during the week of the shipment, and must make the necessary arrangements to receive the shipment, even if the laboratory is closed.

Instructions on Test Item Handling

Once received, the Test Items should be stored deep-frozen (at -18°C or below) before analysis to avoid any possible deterioration/spoilage and to minimize pesticide losses. The test Item should be milled before analysis. After milling mix the flour thoroughly, before taking the analytical portion(s).

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

The homogeneity test is conducted using 5 g of milled Test Item in all cases. As sub-sampling variability increases with decreasing analytical portion size, sufficient homogeneity can only be guaranteed where participants employ sample portions that are equal to or larger than the ones stated above.

EUPT Webtool and Deadlines

To select pesticide scope and report results and method information, the participants should log in to the **EUPT Webtool** using the username and password send by email. Please, save the credentials, as it will be valid for the EUPTs next year.

Selection/deselection of scope: The analytical scope must be selected prior to the shipment of the samples. This is done via the **EUPT Webtool**. The scope selection subpage will be open from 9 March to 5 June 2020. As default all pesticides are preselected.

Results and method submission: The **EUPT Webtool** will be accessible from 9 June 2020 for sample receipt acknowledgement and submission results and method information.

The deadline for submission is 17 August 2020 at 24.00 CEST.

IMPORTANT: After the final submission it will NOT be possible to edit the results. Participants will receive an email confirming the submission of their results. Attached to the email will be an excel file with their submitted data.

Test Item Receipt and Acceptance: Once the laboratory has received the Test Items it must report to the organiser, via the **EUPT Webtool**, the date of receipt, and its acceptance. If the laboratory does not

respond by 12 June 2020, the Organiser will assume that the Test Items have been received and accepted.

If participants have not received the Test Items by **the 12 June 2020 at noon**, they must inform the Organiser immediately by e-mail to eurl-cf@food.dtu.dk.

Reporting Quantitative Results:

Results should not be reported where a pesticide

- a) was not detected,
- b) was detected below the RL (Reporting Limit) of the laboratory, or

Results reported as <RL will be considered as „Not Detected“.

Significant Figures:

Residue levels <0.010 mg/kg;

- to be expressed by two significant figures (e.g. 0.0058 mg/kg).

Residue levels \geq 0.010 mg/kg;

- to be expressed by three significant figures, e.g. 0.156, 1.64, 10.3 mg/kg.

Reporting Analytical method: The laboratory must to report details of the analytical methods they used. If not it will not be possible to submit results.

Reporting of supplementary information in case of false negative results

In case of false negative results the affected laboratories will be asked to provide details on the methodology used after the deadline for result submission. This can also be done by accessing EUPT Webtool. Deadline for this is 24 August 2020.

Follow-up actions

In accordance with Art. 32 1b of Regulation (EC) No 882/2004, underperformance of any NRL-CF in comparative testing will be followed by EURL-CF.

Documents

All documents related to EUPT-CF14 can be found on EUPT-CF14 website.

Calendar

Activity	Dates
Announcement Calendar Target Pesticide List	December 2019
EUPT-Registration Website open	January 2020
Deadline for registration	25 May 2020
Specific Protocol published	19 May 2020
Website for selecting pesticide scope open	9 March 2020
Website for selecting pesticide scope closed	9 June 2020
Distribution of Test items	8 June 2020
Deadline for receipt and acceptance of Test Materials	within 24 hr on receipt
Deadline for Result Submission	15 August 2020 at 24.00 CET
Deadline for submission of additional method information for false negative results	24 August 2020 at 24.00 CET
Preliminary Report (only compilation of results) published	17 October 2020
Final Report published	December 2020

Participation Fees

For participating laboratories from the EU, EU-candidate states and EFTA states the participation fee will be:

- 200 €

The participation fees for laboratories from third countries will be:

- 350 €

For further information, visit www.eurl-pesticides.eu.

Delays in Payment

The participants will receive an invoice from DTU. The terms of payment are 30 days net. After this deadline reminders will be sent. From the second reminder onwards an administration fee of DKK 100.00 excluding VAT (ca. 13 €) will be charged per reminder.

Any questions concerning invoices must be directed to Tom Schmidt Christensen, tomsc@adm.dtu.dk at the financial department of DTU.

Contact information:

DTU Food
National Food Institute



Mette Erecius Poulsen

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