Proficiency Test on pesticide residues in oat flour

EU Reference Laboratory on Cereals & Feeding stuff

EUPT-CF11 2017

DTU Food
National Food Institute

EU PROFICIENCY TESTS
EUPT-CF11, 2017

Pesticide Residues in Oat Flour

Final Report

Version 4

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December 2017

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DANAK
PT Reg.nr. 516

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PREFACE

Regulation (EC) No 882/2004 [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 eleventh 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 oats, 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, and EUPT-CF10 on rye flour. 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, whereas the present EUPT-CF11 on oat flour was only focused on MRM-pesticides. The oat Test Item used for EUPT-CF11 was treated with formulations in the field and post-harvest in the laboratory.

Participation in EUPT-CF11 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 multiresidue 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.

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1. INTRODUCTION

On 4 February 2017 the announcement of the 11th European Commission's Proficiency Test on cereals and feed (EUPT-CF11) 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 153 individual compulsory compounds and 9 voluntary requiring the use of multiresidue 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 EUPTs, the Specific protocol (**Annex 2**), as well as a list of labs that are obliged to take part in the EUPT-CF11, were provided via the homepage. Laboratories were able to register on-line from 13 March to 23 April 2017. In total 149 laboratories from EU and EFTA countries agreed to participate in the test as well as 22 laboratories from EU-Candidate States and Third Countries (**Appendix 1**).

The present proficiency test was performed using oat flour of Danish origin, which had been partly treated in the field, and partly spiked post-harvest at the facilities of the EURL-CF. The Test Item contained 18 compounds in total. Danish Centre for Food and Agriculture at Aarhus University grew the oat and performed the field treatments in 2008 (for use in EUPT-C3). The kernels were diluted with organically grown oat kernels, because the amount left over from EUPT-C3 was not enough and the residue levels were relatively high and could be diluted without being too low. The pesticides employed for the treatment were selected by the EURL-CF and the EURL quality control group and the application rates and harvest intervals chosen were based on previous experience and data from supervised residue trials. The harvested oat grains were overspiked with 3 pesticides and spiked with 4 additional compounds post-harvest, and then checked for homogeneity before shipping to participants. Furthermore, the stabilities of the pesticides in the Treated 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 125 g portions of the Treated oat Test Item and 125 g of untreated Blank oat Test Item. Both Test Items were shipped to participants on 8 May 2017 and the deadline for submission of results to the Organiser was the 6 June 2017. The participants were asked to analyse the Treated Test Item as well as the Blank 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 website.

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 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 $MgSO_4$. 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, a pesticide priority list ranking the pesticides according to their relevance and risk-potential, as well as a list of pesticides relevant to the specific commodity (oat). 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 set at 0.01 mg/kg.

1.3 Preparation of the treated Test Item

Before preparing the Test Item, the pesticides and suitable target residue levels for the study were selected. The application rates and harvest intervals for the 14 pesticides used for treatment in the field were chosen based on experience from previous PTs and data from supervised residue trials. The field spraying was performed by the Danish Centre for Food and Agriculture at Aarhus University in 2008 for use in EUPT-C3. Approximately, 70 kg of the harvested oat grain was delivered for preparation of the Test Item. The 12 kg of leftovers were diluted 1:1 with organically grown oat kernels (grown in 2016). The Test material was additionally spiked in the laboratory with four pesticides, which were not included in the field treatments and over spiked with three pesticide where the residue in the oat was too low (**Table 1**). Spiking in the laboratory was performed using formulations or pure standards. One kilogram of the field treated oat was spiked and subsequently mixed with 23 kg of field treated oat and homogenised thoroughly. The resulting 24 kg of mixed oat grain was milled separately per approximately 4 kilograms portions. To ensure that a well-homogenised bulk was obtained, with respect to the spiked residues, the 4 kilogram portions were initially mixed individually by hand. Two 4 kilograms portions were then mixed together by hand and finally all the 8 kg portions were combined. One hundred and ten 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. From 7-18 April the bottles were stored at room temperature to imitate the storage condition that many laboratories has, see details in **Chapter 3.3**. Finally, the Test Item were stored in freezer until it was distributed to the participants.

1.4 Preparation of the 'blank' Test Item

The oat used to prepare the blank Test Item was also produced by the Danish Centre for Food and Agriculture at Aarhus University under similar growing conditions as the treated crop but without any pesticide treatment in the field or spiking in the laboratory. Also these kernels were diluted 1:1 with organically grown oat kernels. One hundred and ten gram portions were weighed out into screw-capped polyethylene plastic bottles, sealed, and stored in a freezer at about -20 °C. From 7-18 April the bottles were stored at room temperature to imitate the storage condition that many laboratories has, see details in **Chapter 3.3**. Finally, the Test Item were stored in freezer until it was distributed to the participants.

1.5 Homogeneity test

Ten bottles of the pesticide treated 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 $c = F_1 \times \sigma_{all}^2 + F_{2\,x}\, s_{an}^2$; F1 and F2 being constants with values of 1.83 and 0.93, respectively, from the 11 samples taken, σ_{all}^2 = 0.3 x FFP RSD (25%) x 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, the treated Test Item was considered to be sufficiently homogenous and suitable for the EUPT-CF11.

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

Pesticides [1]	Application in field	Spike in laboratory	Formulation/standard
Azoxystrobin	x		Amistar
Carbendazim	x		Bavistin DF
Chlordane, trans-		x	Analytical standard
Chlorpyrifos	x		Dursban WG
Cyproconazole	x		Alto
Cyprodinil	x	x	Unix
Endosulfan-alpha		x	Analytical standard
Esfenvalerat [2]	x		Sumi Alpha
Fenbuconazole	x		Indar
Fenpropimorph	x		Corbel
Fludioxonil	x	x	Safir
Flusilazole	x		Lyric
Flutolanil		x	Analytical standard
Lambda-cyhalothrin	x	x	Karate 2,5 WG
Metconazole	x		Juventus
Prothiofos		x	Analytical standard
Pyraclostrobin	x		Comet
Tebuconazole	x		Folicur

[1] Malathion and procymidon were also applied in the field, but too low residue levels were achieved.
[2] Esfenvalerate was used for the field treatment. The participants were asked to analyse for Fenvalerate and Esfenvalerate (Sum of RR/SS and RS/SR isomers). However, this long name is in the rest of the report referred to as fenvalerate only.

1.6 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 $| x1 - yi | \leq 0.3 \times \sigma$, where x1 is the mean value of the first stability test, yi the mean value of the last stability test and σ the standard deviation used for proficiency assessment (25% of the assigned value):

The dates of testing were as follows:

Day 1: 9 May 2017
Day 2: 23 May 2017
Day 3: 06 June 2017

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

Pesticide	Mean, mg/kg	S_s^2	c	$S_s^2 < c$
Azoxystrobin	0.102	0.00000	0.0001	Pass
Carbendazim	0.262	0.00013	0.0009	Pass
Chlordane, trans	0.035	0.00000	0.0000	Pass
Chlorpyrifos	0.536	0.00036	0.0033	Pass
Cyproconazole	0.266	0.00008	0.0008	Pass
Cyprodinil	0.076	0.00001	0.0001	Pass
Endosulfan , alpha	0.054	0.00000	0.0000	Pass
Fenbuconazole	0.291	0.00000	0.0010	Pass
Fenpropimorph	0.087	0.00001	0.0001	Pass
Fenvalerate	0.049	0.00000	0.0001	Pass
Fludioxonil	0.088	0	0.0001	Pass
Flusilazole	0.440	0.00029	0.0028	Pass
Flutolanil	0.057	0.00000	0.0000	Pass
Cyhalothrin, lambda	0.044	0.00000	0.0000	Pass
Metconazole	0.303	0	0.0012	Pass
Prothiofos	0.043	0.00000	0.0000	Pass
Pyraclostrobin	0.353	0.00023	0.0017	Pass
Tebuconazole	0.782	0.00072	0.0069	Pass

Table 3. Statistical evaluation of the stability test data

Pesticide	Mean, mg/kg	$\lvert x1 - yi \rvert$	0.3×σ	$\lvert x1 - yi \rvert \leq 0.3\times\sigma$
Azoxystrobin	0.098	0.006	0.007	Pass
Carbendazim	0.315	0.018	0.021	Pass
Chlordane, trans-	0.036	0.000	0.004	Pass
Chlorpyriphos	0.580	0.036	0.041	Pass
Cyhalothin, lambda	0.068	0.003	0.004	Pass
Cyproconazole	0.239	0.011	0.019	Pass
Cyprodinil	0.067	0.001	0.006	Pass
Endosulfan, alpha	0.053	0.002	0.004	Pass
Fenbuconazole	0.275	0.019	0.021	Pass
Fenpropimorph	0.068	0.002	0.005	Pass
Fenvalerate	0.062	0.004	0.004	Pass
Fludioxonil	0.076	0.002	0.006	Pass
Flusilazole	0.395	0.017	0.030	Pass
Flutolanil	0.050	0.003	0.004	Pass
Metconazole	0.300	0.006	0.020	Pass
Prothiofos	0.042	0.000	0.003	Pass
Pyraclostrobin	0.443	0.020	0.030	Pass
Tebuconazole	0.668	0.041	0.053	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 and at room temperature. See the individual stability figures for all pesticides in Appendix 4.

1.7 Organisational details

1.7.1 Access to documents, registration and confidentiality

In the invitation letter, all NRLs and OfLs were requested to register using the online registration link from 13 March to 23 April 2017. 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 third countries also had access to another online registration link after contacting the EURL. After registration, the participants were provided with username, password, laboratory code and link for the online result submission website. This ensured confidentiality throughout the entire duration of the PT.

1.7.2 Distribution of the Test Item

On 8 May 2017, one bottle of treated Test Item (125 g) and one bottle of blank Test Item (125 g) were 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 website (see above) and report whether they accept/not accept the Test Items. Test Items for Third Countries were shipped one week earlier due the often very time-consuming customs procedures at the borders.

1.7.3 Submission of results

An online submission tool was developed that allowed participants to submit their results via the internet. All participants had access to the result-submission website (http://pesticide.food.dtu.dk) from a few days after shipment until the result-submission deadline (6 June 2017). 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 Items.

2. EVALUATION OF THE RESULTS

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 participant's 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 (see "Omission or Exclusion of results" below) or to use only the results of a subgroup consisting of laboratories that have repeatedly demonstrated good performance for the specific compound in the past.

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-σ_{pt}) will be calculated using a Fit-For-Purpose approach with a fixed Relative Standard Deviation (FFP-RSD) of 25% as follows:

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

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

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:

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

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

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

Z scores will be interpreted in the following way as is set in the ISO 17043:2010 [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 the RL < MRRL. The EUPT-Panel will decide whether, or not, these values should appear in the z-score histograms.

2.6 Category A and B classification and combined z scores (AZ^2)

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

For evaluation of the overall performance of laboratories within Category A, the Average of the Squared z Score (AZ^2) 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 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 AZ^2 achieved, the laboratories are classified as follows:

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

The AZ^2 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, 149 EU and EFTA laboratories, from 30 different countries (27 EU member states), agreed to participate in this proficiency test. Additionally, Malta was represented by UK NRL. Four EU/EFTA participants did not submit results among these was one NRL. Furthermore, 22 Third Countries registered for the PT. Two of these laboratories did not submit results. 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 8-12(a/b)** and in **Appendix 5**. The methods used are presented in **Appendix 6** but in a separate electronic file. However, only results submitted by laboratories from EU and EFTA countries are included in **Table 4-7, 16** and in the z scores histograms 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 treated Test Item. Only results submitted by laboratories from the EU and EFTA are included in this table.

Pesticide	No. of reported results	No. of NA [1]	No. of false negatives	% of labs reporting results [2]
Azoxystrobin	129	16	3	89
Carbendazim	110	35	2	76
Chlordane, trans	101	44	9	70
Chlorpyrifos	135	10	6	93
Cyproconazole	129	16	9	89
Cyprodinil	132	13	4	91
Endosulfan, alpha-	132	13	16	91
Fenbuconazole	124	21	1	86
Fenpropimorph	119	26	9	82
Fenvalerate	125	20	10	86
Fludioxonil	129	16	8	89
Flusilazole	128	17	9	88
Flutolanil	100	45	1	69
Lambda-cyhalothrin	132	13	11	91
Metconazole	105	40	1	72
Prothiofos	94	51	12	65
Pyraclostrobin	116	29	2	80
Tebuconazole	132	13	1	91

[1] NA = not analysed

[2] '% 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 = 145). False negatives are included in reported results.

Chlorpyrifos was the most frequently analysed compound with 93 % of the labs submitting results for this compound. Cyprodinil, endosulfan (alpha-), lambda-cyhalothrin and tebuconazole were analysed by 91% of the participating laboratories. Azoxystrobin, carbendazim, cyproconazole, fenbuconazole, fenpropimorph, fenvalerate, fludioxonil, flusilazole, metconazole and pyraclostrobin were analysed and reported by 75-89% of the participants. Less than 70% analysed for flutolanil and prothiofos and the voluntary compound trans-chlordane. The Test Item contained low levels of malathion and 19 laboratories reported results between 0.003-0.015 with a median of 0.0062. These levels were too low to evaluate.

3.1.1 False positives

Nineteen participants from EU and EFTA countries reported 19 results for 9 different additional pesticides above the MRRL that had not been used to treat the Test Item (**Table 5**). The pesticides were: acrinathrin, chlordane, cis-, fenthion, paclobutrazol, pendimethalin, pirimiphos-methyl, pyridaben, tebufenozide and triticonazole. 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.

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	Pesticide	Concentration mg/kg	Determination technique	RL, mg/kg	MRRL, mg/kg
10	Paclobutrazol	0.08	LC-MS/MS QQQ	0.01	0.01
13	Chlordane, cis-	0.063	GC-MS/MS (QQQ)	0.01	0.01
27	Chlordane, cis-	0.528	GC-MS/MS (QQQ)	0.01	0.01
39	Fenthion	0.016	GC-MSD	0.01	0.01
47	Chlordane, cis-	0.061	GC-MS/MS (QQQ)	0.01	0.01
48	Paclobutrazol	0.0283	LC-MS/MS QQQ	0.01	0.01
67	Chlordane, cis-	0.034	GC-MS/MS (QQQ)	0.01	0.01
83	Chlordane, cis-	0.041	GC-Ion Trap	0.01	0.01
112	Paclobutrazol	0.222	GC-MS/MS (QQQ)	0.01	0.01
130	Paclobutrazol	0.56	LC-MS/MS QQQ	0.005	0.01
135	Pendimethalin	0.0251	LC-MS/MS QQQ	0.01	0.01
138	Acrinathrin	0.036	GC-MS/MS (QQQ)	0.01	0.01
138	Tebufenozide	0.045	LC-MS/MS QQQ	0.01	0.01
138	Chlordane, cis-	0.45	[1)]	0.01	0.01
149	Pyridaben	0.065	GC-MS/MS (QQQ)	0.01	0.01
155	Tebufenozide	0.033	LC-MS/MS QQQ	0.02	0.01
170	Chlordane, cis-	0.05	GC-MS/MS (QQQ)	0.01	0.01
174	Fenthion	0.021	GC-MSD	0.02	0.01
179	Pirimiphos-methyl	0.028	GC-MSD	0.01	0.01

[1)] No information received.

3.1.2 False negatives

Not reported results for pesticides actually present in the treated Test Item were judged as false negatives. **Table 6** summarizes the number of reported false negatives for each pesticide. Fifty one laboratories submitted 119 false negatives results for the 18 different pesticides (all pesticide included in the PT), which represents more than 5% of the total number of results. Around 35% of the participants (51 laboratories) reported false negative results. This is the highest frequency ever seen in EUPTs on cereals. In previous PTs typically 20-30% of the labs reported false negative results.

The reason for the high number of false negatives is assumed to be the high fatty acid level that appears in the cereal flour when stored at room temperature, see details in **Chapter 3.3**. However, it should be noted the one NRL that reported 7 false negatives informed before deadline that their GC instrument broke down.

Table 6. False negative results (FN) for EU and EFTA country as well as for Candidate and Third Countries.

Lab code	Azoxystrobin	Carbendazim	Chlordane, trans	Chlorpyrifos	Cyproconazole	Cyprodinil	Endosulfan, alpha-	Fenbuconazole	Fenpropimorph	Fenvalerate	Fludioxonil	Flusilazole	Flutolanil	Lambda-cyhalothrin	Metconazole	Prothiofos	Pyraclostrobin	Tebuconazole
8					FN													
12							FN											
13			FN															
14											FN							
15				FN		FN	FN			FN		FN		FN		FN		
22					FN													
26															FN			
27							FN											
33			FN				FN									FN		
35							FN											
47			FN															
48									FN							FN		
50						FN	FN			FN				FN		FN		
51							FN							FN				
61									FN			FN						
67			FN															
68									FN			FN	FN		FN			
74																FN		
77			FN															
78				FN			FN									FN		
80									FN									
83			FN															
84							FN											
86												FN						
97					FN							FN						
98	FN			FN					FN	FN	FN			FN				
110			FN		FN												FN	
112	FN	FN							FN	FN				FN				
114							FN											
126					FN													
129						FN			FN	FN								
130			FN	FN			FN		FN	FN				FN		FN		
131	FN			FN			FN			FN	FN			FN		FN		
132											FN							
134																FN		
135							FN			FN						FN		
137																FN		

Lab code	Azoxystrobin	Carbendazim	Chlordane, trans	Chlorpyrifos	Cyproconazole	Cyprodinil	Endosulfan, alpha-	Fenbuconazole	Fenpropimorph	Fenvalerate	Fludioxonil	Flusilazole	Flutolanil	Lambda-cyhalothrin	Metconazole	Prothiofos	Pyraclostrobin	Tebuconazole
138						FN			FN									
140					FN		FN	FN		FN	FN	FN		FN				FN
148	FN																	
152																	FN	
155									FN									
162					FN						FN							
163					FN													
166												FN	FN					
167												FN						
169					FN							FN						
170			FN								FN			FN				
173														FN				
174		FN					FN				FN							
179				FN			FN			FN		FN		FN		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. However, because of earlier experience with significantly biased results from laboratories not adding water to the sample before extraction (or using a mixture of water and extraction solvent) these results were not included in the calculation of the Algorithm A mean. Also results from laboratories that did not provide information about their extraction method were excluded from the calculations.

All Assigned Values for the pesticides can be seen in **Table 7**. 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 14-23 % but on average the Alg A-RSD was 16 %, and thus below the 25 % FFP-RSD used for the calculations.

The uncertainty of the assigned values is calculated according 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 8**)

Table 7. 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, %
Azoxystrobin	0.01	0.097	0.002	25	15
Carbendazim	0.01	0.274	0.009	25	29
Chlordane, trans	0.01	0.050	0.001	25	20
Chlorpyrifos	0.01	0.555	0.009	25	15
Cyproconazole	0.01	0.251	0.004	25	14
Cyprodinil	0.01	0.075	0.001	25	18
Endosulfan, alpha-	0.01	0.055	0.002	25	24
Fenbuconazole	0.01	0.275	0.005	25	16
Fenpropimorph	0.01	0.065	0.001	25	18
Fenvalerate	0.01	0.057	0.001	25	21
Fludioxonil	0.01	0.082	0.002	25	18
Flusilazole	0.01	0.405	0.007	25	16
Flutolanil	0.01	0.053	0.001	25	17
Lambda-cyhalothrin	0.01	0.048	0.001	25	20
Metconazole	0.01	0.268	0.006	25	17
Prothiofos	0.01	0.044	0.001	25	19
Pyraclostrobin	0.01	0.406	0.009	25	18
Tebuconazole	0.01	0.706	0.010	25	13

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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 8** shows an overview of the acceptable, questionable and unacceptable z scores and **Tables 8a/b-13a/b** 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 8. Number of acceptable, questionable, unacceptable z scores and false negatives.

Pesticide	No. of reported results	Assigned values	Acceptable z scores	Questionable z scores	Unacceptable z scores [1]	False negatives[1]
Azoxystrobin	129	0.097	119	5	5	3
Carbendazim	110	0.274	89	11	10	2
Chlordane, trans	101	0.050	85	2	14	9
Chlorpyrifos	135	0.555	128	0	7	6
Cyproconazole	129	0.251	113	4	12	9
Cyprodinil	132	0.075	126	0	6	4
Endosulfan, alpha-	132	0.055	108	4	20	16
Fenbuconazole	124	0.275	118	2	4	1
Fenpropimorph	119	0.065	101	6	12	9
Fenvalerate	125	0.057	104	4	17	10
Fludioxonil	129	0.082	113	5	11	8
Flusilazole	128	0.405	112	4	12	9
Flutolanil	100	0.053	90	4	6	1
Lambda-cyhalothrin	132	0.048	118	1	13	11
Metconazole	105	0.268	99	3	3	1
Prothiofos	94	0.044	77	3	14	12
Pyraclostrobin	116	0.406	108	4	4	2
Tebuconazole	132	0.706	122	6	4	1

[1] Unacceptable z scores include those for false negative results.

For azoxystrobin, chlorpyrifos, cyprodinil, fenbuconazole, flutolanil, metconazole, pyraclostrobin and tebuconazole acceptable results were obtained by 90-95% of the laboratories. For carbendazim, chlordane-trans, cyproconazole, endosulfan, alpha-, fenpropimorph, fenvalerate, fludioxonil, flusilazole, lambda-cyhalothrin, and prothiofos acceptable results were obtained by 82-89% of the laboratories. As seen in previous EUPTs on cereals some laboratories reported very high results for carbendazim. This is probably due to the low solubility of the compound in organic solvent, e.g. for ethyl acetate it is only 0.135 mg/ml. It is therefore crucial to check if the carbendazim stock solution is completely dissolved. It is recommended to prepare stock solutions of carbendazim at a concentration no higher than 0.1 mg/ml.

Several different analytical methods have been used by the laboratories. QuEChERS, Citrate buffered (EN 151662) was used for 60% of the reported results. However, variations in the clean-up procedures were reported by the labs, e.g. some used a freezing out step, some used PSA, others PSA/C18 or PSA/ODS or PSA/GCB or $CaCl_2$. So it is not one specific method. Two other QuEChERS methods were used, the Original Version (J. AOAC 86, (2003) 412) and the Acetate buffered (AOAC Official Method 2007.01). These were used for 5% and 6% of the results, respectively. The SweEt method (NMKL 195, 2013) was used for 4%, Mini Luke 5%, the methanol extraction method (Klein, Alder, J. AOAC 86 (2003) 1015) 1% and the German S-19 method for 1% of the results. Finally 9% of the results were analysed by other methods (e.g. in house method) and for 13% of the results no information on the reference method was given by the laboratories.

More than 92% of the reported results derived from a method where water was added before extraction and for 4% of the results no information was given. Likewise, no information was given concerning the use of an ISTD for 8% of the results, while 52% of the results were produced with the use of an ISTD and 40% without an ISTD. Around 75% of the results were based on a mechanical agitation. Finally, no information on the type of instruments used was reported for 4% of the reported results. GC instruments was used for 48% of the results, mainly GC-MS/MS and GC-MSD (32% and 6%), 1 % used GC-TOF instruments, 3% GC-iontrap and the rest (6%) used GC with specific detectors, mainly ECD but also NPD and FPD. LC instruments was used for 48% of the reported results, mainly LC-MS/MS (45%) but 4% used high resolution instrument like LC-Orbitrap or LC-Q-TOF. Only 3 results were analysed using specific detectors such as LC-Fluorescence, LC-UV or LC-DAD.

The results from this PT was not as good as results from previous EUPT-CFs. There have not been reported as many false negatives and false positives before. The number of acceptable results were also lower than in previous EUPTs for cereals. This is probably due to oat being a quite difficult matrix. Oat is the cereal type that contains the highest amount of fat and furthermore, the bottles containing the Test Item, was deliberate stored at room temperature for 11 days, to imitate the storage condition that many laboratories use for dry samples like cereal kernels and flour. However, flour matrix will rapidly change at room temperature where free fatty acids are formed in relative high amount. These free fatty acids can interfere with the co-eluting pesticides especially when analysed on GC resulting in distorted peaks and shifting retention times. However, the free fatty acids can be removed by e.g. PSA if these used in adequate amount. Figure 1 shows the GC chromatogrammes (injection of 1µl) of the sample extracts from the QuEChERS method clean-up with the standard amount of PSA (25mg/ml extract) and with 6 times higher amount of PSA (150mg/ml extract), respectively. The chromatogrammes clearly show that the standard amount of PSA was not enough to remove the fatty acid peak, but the six times amount effectively removed the fatty acid.

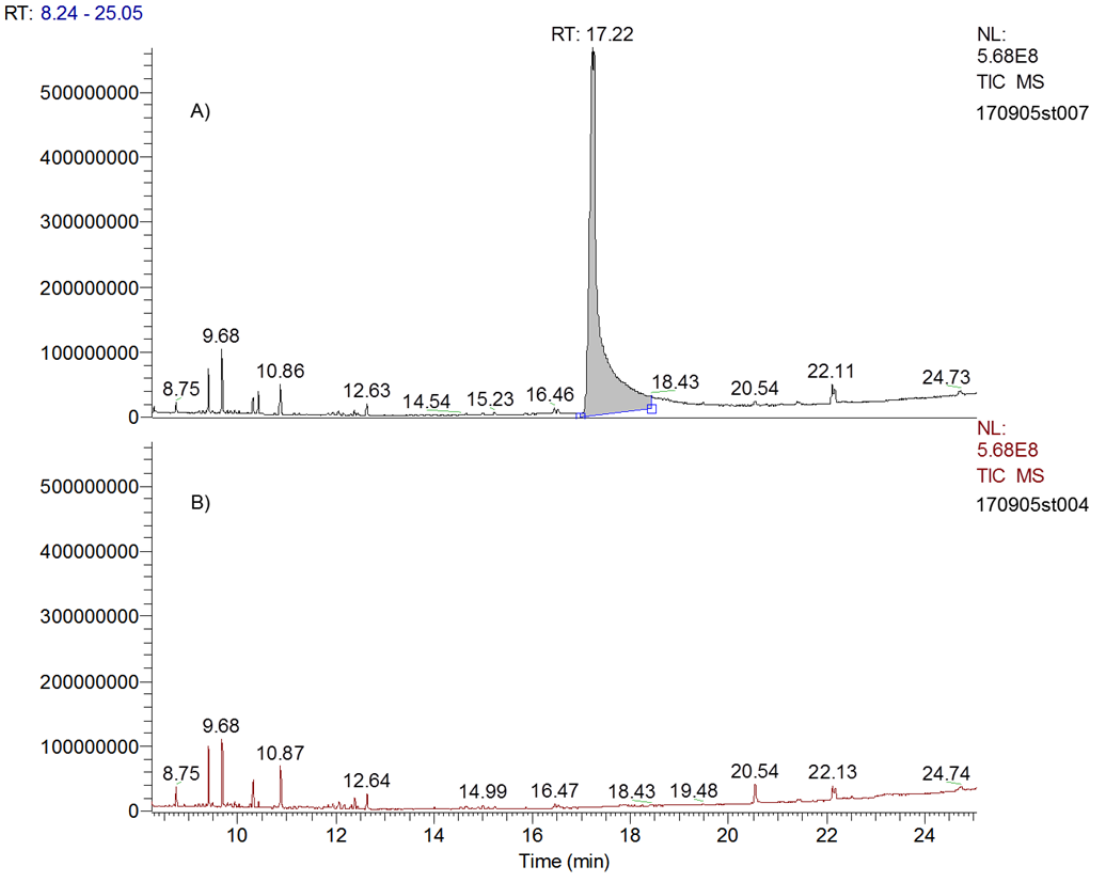


Figure 1. GC chromatogrammes (injection of 1µl) of sample extracts from the QuEChERS method cleaned-up with A) standard amount of PSA (25mg/ml extract) and B) 6 times higher amount of PSA (150mg/ml extract)respectively.

Table 9a. Results for azoxystrobin, carbendazim, chlorpyrifos, cyproconazole, cyprodinil, endosulfan, alpha-, fenbuconazole, fenpropimorph and fenvalerate in mg/kg, and the corresponding z scores, MRRLs and the assigned values.

Laboratory code	Azoxystrobin	Z score (FFP RSD (25%)	Carbendazim	Z score (FFP RSD (25%)	Chlorpyrifos	Z score (FFP RSD (25%)	Cyproconazole	Z score (FFP RSD (25%)	Cyprodinil	Z score (FFP RSD (25%)	Endosulfan, alpha-	Z score (FFP RSD (25%)	Fenbuconazole	Z score (FFP RSD (25%)	Fenpropimorph	Z score (FFP RSD (25%)	Fenvalerate	Z scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.097**		**0.274**		**0.555**		**0.251**		**0.075**		**0.055**		**0.275**		**0.065**		**0.057**	
1			0.135	-2.0	0.393	-1.2											0.0267	-2.1
2	0.095	-0.1	0.177	-1.4	0.498	-0.4	0.258	0.1	0.06	-0.8			0.257	-0.3	0.074	0.5	0.056	0.0
3	0.107	0.4	0.782	>5	0.954	2.9	0.202	-0.8	0.097	1.2			0.299	0.4	0.071	0.4	0.066	0.7
5	0.114	0.7			0.703	1.1	0.323	1.2	0.108	1.8	0.056	0.1	0.332	0.8	0.122	3.5	0.057	0.0
6	0.09	-0.3	0.384	1.6	0.71	1.1	0.22	-0.5	0.066	-0.5	0.069	1.0	0.246	-0.4	0.126	3.7	0.053	-0.3
7																		
8	0.0924	-0.2	0.247	-0.4	0.576	0.2	FN	-3.8	0.087	0.7	0.052	-0.2	0.3	0.4	0.071	0.4	0.0427	-1.0
9	0.085	-0.5	0.279	0.1	0.533	-0.2	0.251	0.0	0.073	0.0	0.0562	0.1	0.251	-0.3	0.06	-0.3	0.0542	-0.2
10	0.102	0.2	0.128	-2.1	0.596	0.3	0.225	-0.4	0.072	-0.1	0.08	1.8	0.29	0.2	0.057	-0.5	0.116	4.2
11	0.096	0.0	0.244	-0.4	0.598	0.3	0.244	-0.1	0.080	0.3	0.041	-1.0	0.277	0.0	0.065	0.0	0.057	0.0
12	0.084	-0.5	0.252	-0.3	0.639	0.6	0.279	0.5	0.093	1.0	FN	-3.3	0.317	0.6	0.064	-0.1	0.069	0.9
13	0.1	0.1	0.284	0.1	0.489	-0.5	0.285	0.5	0.070	-0.2			0.285	0.1	0.064	0.0	0.052	-0.3
14	0.109	0.5	0.316	0.6	0.547	-0.1	0.282	0.5	0.098	1.3	0.0688	1.0	0.356	1.2	0.081	1.0	0.0567	0.0
15	0.104	0.3	0.259	-0.2	FN	-3.9	0.283	0.5	FN	-3.5	FN	-3.3	0.254	-0.3	0.051	-0.9	FN	-3.3
16	0.103	0.3			0.625	0.5	0.266	0.2	0.081	0.4	0.063	0.6	0.309	0.5			0.057	0.0
17																		
18	0.112	0.6	0.444	2.5	0.442	-0.8	0.239	-0.2	0.079	0.2			0.377	1.5	0.075	0.6	0.072	1.1
19	0.099	0.1	0.284	0.1	0.518	-0.3	0.245	-0.1	0.073	-0.1	0.064	0.6	0.251	-0.3	0.069	0.2	0.053	-0.3
20	0.118	0.9	0.298	0.4	0.536	-0.1	0.239	-0.2	0.075	0.0	0.0645	0.7	0.289	0.2	0.058	-0.4	0.0527	-0.3
21	0.071	-1.1	0.205	-1.0	0.147	-2.9	0.123	-2.0	0.048	-1.4			0.147	-1.9	0.045	-1.2	0.071	1.0
22	0.1	0.1	0.291	0.2	0.679	0.9	FN	-3.8	0.099	1.3	0.072	1.2	0.305	0.4	0.084	1.2	0.07	0.9
23	0.111	0.6	0.199	-1.1	0.565	0.1	0.281	0.5	0.083	0.5	0.0628	0.6	0.324	0.7	0.076	0.7	0.0587	0.1
24	0.146	2.0	0.383	1.6	0.724	1.2	0.35	1.6	0.094	1.0	0.0671	0.9	0.333	0.8	0.076	0.7	0.0458	-0.8
25					0.595	0.3					0.0652	0.7					0.0579	0.1
26	0.103	0.3			0.599	0.3	0.272	0.3	0.071	-0.2	0.0493	-0.4	0.271	-0.1	0.070	0.3	0.0485	-0.6
27	0.113	0.7	0.282	0.1	0.635	0.6	0.344	1.5	0.065	-0.5	FN	-3.3	0.323	0.7	0.053	-0.7	0.052	-0.3
28											0.0674	0.9						
29	0.0935	-0.1	0.358	1.2	0.507	-0.3	0.278	0.4	0.065	-0.5	0.0606	0.4	0.277	0.0	0.069	0.2	0.0541	-0.2
31	0.0842	-0.5	0.17	-1.5	0.616	0.4	0.228	-0.4	0.069	-0.3	0.059	0.3	0.373	1.4	0.086	1.3	0.1	3.1
32	0.111	0.6	0.343	1.0	0.621	0.5	0.289	0.6	0.076	0.1	0.0609	0.4	0.288	0.2	0.066	0.1	0.0621	0.4
33	0.11	0.6	0.205	-1.0	0.453	-0.7	0.286	0.6	0.065	-0.5	FN	-3.3	0.27	-0.1	0.056	-0.6	0.049	-0.5
34	0.092	-0.2	0.298	0.4	0.568	0.1	0.29	0.6	0.058	-0.9	0.041	-1.0	0.24	-0.5	0.063	-0.1	0.073	1.1
35	0.1	0.1	0.25	-0.3	0.48	-0.5	0.244	-0.1	0.072	-0.1	FN	-3.3	0.257	-0.3	0.054	-0.7	0.051	-0.4
36					0.569	0.1	0.179	-1.1					0.193	-1.2			0.03	-1.9
37	0.094	-0.1	0.259	-0.2	0.473	-0.6	0.276	0.4	0.079	0.2			0.284	0.1	0.058	-0.4	0.047	-0.7
38	0.085	-0.5	0.249	-0.4	0.592	0.3	0.284	0.5	0.061	-0.7	0.039	-1.2	0.317	0.6	0.055	-0.6	0.061	0.3
39	0.085	-0.5	0.261	-0.2	0.581	0.2	0.233	-0.3	0.069	-0.3	0.06	0.4	0.273	0.0	0.064	-0.1	0.06	0.2
40	0.093	-0.2	0.259	-0.2	0.587	0.2	0.223	-0.4	0.082	0.4	0.058	0.2	0.323	0.7	0.079	0.8	0.065	0.6
41	0.08	-0.7	0.349	1.1	0.437	-0.8	0.151	-1.6	0.061	-0.7	0.0335	-1.6	0.231	-0.6	0.046	-1.2	0.0478	-0.6
42	0.11	0.6	0.309	0.5	0.568	0.1	0.257	0.1	0.079	0.2	0.074	1.4	0.324	0.7	0.061	-0.3	0.065	0.6
43	0.0902	-0.3	0.281	0.1	0.498	-0.4	0.261	0.2	0.077	0.1	0.0611	0.4	0.26	-0.2	0.066	0.1	0.0547	-0.1

Table 9b. Results for fludioxonil, flusilazole , flutolanil, lambda-cyhalothrin, metconazole , prothiofos, pyraclostrobin, tebuconazole and chlordane, trans in mg/kg, and the corresponding z scores, MRRLs and the assigned values.

Laboratory code	Fludioxonil	Z score (FFP RSD (25%)	Flusilazole	Z score (FFP RSD (25%)	Flutolanil	Z score (FFP RSD (25%)	Lambda-cyhalothrin	Z score (FFP RSD (25%)	Metconazole	Z score (FFP RSD (25%)	Prothiofos	Z score (FFP RSD (25%)	Pyraclostrobin	Z score (FFP RSD (25%)	Tebuconazole	Z score (FFP RSD (25%)	Chlordane, trans	Z score (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.082**		**0.405**		**0.053**		**0.048**		**0.268**		**0.044**		**0.406**		**0.706**		**0.050**	
1			0.107	-2.9									0.109	-2.9	0.166	-3.1		
2	0.093	0.5	0.48	0.7			0.057	0.8	0.219	-0.7			0.367	-0.4	0.655	-0.3	0.053	0.3
3	0.09	0.4	0.457	0.5	0.059	0.5	0.064	1.4	0.299	0.5	0.057	1.2	0.46	0.5	0.755	0.3	0.065	1.2
5	0.1	0.9	0.531	1.2	0.062	0.7	0.039	-0.7	0.341	1.1			0.611	2.0	0.837	0.7	0.062	1.0
6	0.11	1.4	0.361	-0.4	0.034	-1.4	0.038	-0.8	0.257	-0.2	0.032	-1.1	0.375	-0.3	0.593	-0.6		
7																		
8	0.092	0.5	0.406	0.0	0.096	3.3	0.044	-0.3	0.262	-0.1	0.044	0.1	0.547	1.4	0.833	0.7	0.036	-1.0
9	0.041	-2.0	0.368	-0.4	0.049	-0.3	0.041	-0.5	0.221	-0.7	0.039	-0.4	0.414	0.1	0.695	-0.1	0.058	0.7
10	0.092	0.5	0.38	-0.2	0.061	0.6	0.083	2.9	0.25	-0.3	0.033	-1.0	0.41	0.0	0.62	-0.5	0.092	3.4
11	0.080	-0.1	0.388	-0.2	0.052	0.0	0.05	0.2	0.264	-0.1	0.051	0.6	0.388	-0.2	0.679	-0.2	0.05	0.0
12	0.059	-1.1	0.474	0.7	0.053	0.0	0.043	-0.4	0.294	0.4	0.042	-0.2	0.38	-0.3	0.797	0.5	0.047	-0.2
13	0.090	0.4	0.411	0.1	0.053	0.1	0.050	0.2	0.316	0.7			0.421	0.1	0.787	0.5	FN	-3.2
14	FN	-3.5	0.459	0.5			0.045	-0.2					0.468	0.6	0.784	0.4	0.063	1.1
15	0.117	1.7	FN	-3.9	0.066	1.0	FN	-3.2	0.318	0.7	FN	-3.1	0.432	0.3	0.766	0.3		
16	0.103	1.0	0.413	0.1	0.056	0.3	0.065	1.5	0.29	0.3			0.45	0.4	0.727	0.1	0.046	-0.3
17																		
18	0.065	-0.8	0.364	-0.4	0.08	2.1	0.049	0.1	0.255	-0.2			0.534	1.3	0.468	-1.3		
19	0.087	0.2	0.368	-0.4	0.046	-0.5	0.043	-0.4	0.259	-0.1	0.043	-0.1	0.416	0.1	0.691	-0.1	0.043	-0.5
20	0.090	0.4	0.043	-3.6	0.060	0.6	0.046	-0.1	0.246	-0.3	0.040	-0.3	0.424	0.2	0.627	-0.4	0.048	-0.1
21	0.053	-1.4	0.107	-2.9	0.028	-1.9	0.018	-2.5	0.163	-1.6			0.234	-1.7	0.423	-1.6	0.026	-1.9
22	0.084	0.1	0.444	0.4	0.054	0.1	0.041	-0.6	0.289	0.3	0.044	0.0	0.585	1.8	0.76	0.3	0.071	1.7
23	0.089	0.3	0.183	-2.2	0.053	0.1	0.053	0.4	0.14	-1.9	0.045	0.1	0.452	0.5	0.808	0.6	0.059	0.8
24	0.070	-0.5	0.559	1.5	0.058	0.5	0.049	0.1	0.403	2.0	0.057	1.2	0.643	2.3	0.959	1.4	0.058	0.7
25																	0.061	1.0
26	0.087	0.3	0.426	0.2			0.055	0.6	FN	-3.9					0.715	0.1	0.043	-0.5
27	0.09	0.4	0.403	0.0	0.055	0.2	0.05	0.2	0.317	0.7	0.035	-0.8	0.49	0.8	0.753	0.3	0.528	>5
28																	0.063	1.1
29	0.086	0.2	0.416	0.105	0.044	-0.6	0.044	-0.3	0.301	0.5	0.055	1.0	0.426	0.2	0.736	0.2	0.049	0.0
31	0.076	-0.3	0.453	0.5	0.095	3.2	0.224	>5			0.056	1.1	0.418	0.1	0.724	0.1	0.012	-3.0
32	0.081	0.0	0.429	0.2	0.053	0.1	0.054	0.5	0.301	0.5	0.046	0.2	0.452	0.5	0.731	0.1	0.050	0.1
33	0.101	0.9	0.47	0.6	0.057	0.3	0.043	-0.4	0.29	0.3	FN	-3.1	0.475	0.7	0.76	0.3	FN	-3.2
34	0.082	0.0	0.313	-0.9	0.048	-0.3	0.06	1.0	0.224	-0.7			0.301	-1.0	0.508	-1.1		
35	0.084	0.1	0.385	-0.2	0.050	-0.1	0.036	-1.0	0.275	0.1	0.045	0.1	0.41	0.0	0.649	-0.3		
36	0.064	-0.9					0.029	-1.6					0.267	-1.4	0.605	-0.6		
37	0.043	-1.9	0.424	0.2	0.044	-0.7	0.067	1.6	0.263	-0.1			0.437	0.3	0.568	-0.8	0.044	-0.5
38	0.07	-0.6	0.434	0.3	0.05	-0.2	0.053	0.4	0.278	0.1	0.047	0.3	0.399	-0.1	0.664	-0.2	0.047	-0.2
39	0.075	-0.3	0.414	0.1	0.047	-0.4	0.045	-0.2	0.249	-0.3	0.045	0.1	0.422	0.2	0.655	-0.3		
40	0.066	-0.8	0.459	0.5	0.056	0.3	0.055	0.6	0.25	-0.3	0.051	0.6	0.472	0.6	0.738	0.2	0.05	0.0
41	0.063	-0.9	0.316	-0.9	0.046	-0.5	0.031	-1.4	0.211	-0.9	0.034	-0.9	0.302	-1.0	0.619	-0.5	0.038	-0.9
42	0.090	0.4	0.427	0.2	0.053	0.1	0.060	1.0	0.34	1.1	0.079	3.2	0.465	0.6	0.74	0.2	0.050	0.1
43	0.082	0.0	0.249	-1.5	0.044	-0.6	0.042	-0.4	0.291	0.3	0.051	0.6	0.38	-0.3	0.659	-0.3	0.051	0.1

Table 10a. Results for azoxystrobin, carbendazim, chlorpyrifos, cyproconazole, cyprodinil, endosulfan, alpha-, fenbuconazole, fenpropimorph and fenvalerate in mg/kg, and the corresponding z scores, MRRLs and the assigned values.

Laboratory code	Azoxystrobin	Z score (FFP RSD (25%)	Carbendazim	Z score (FFP RSD (25%)	Chlorpyrifos	Z score (FFP RSD (25%)	Cyproconazole	Z score (FFP RSD (25%)	Cyprodinil	Z score (FFP RSD (25%)	Endosulfan, alpha-	Z score (FFP RSD (25%)	Fenbuconazole	Z score (FFP RSD (25%)	Fenpropimorph	Z score (FFP RSD (25%)	Fenvalerate	Z scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.097**		**0.274**		**0.555**		**0.251**		**0.075**		**0.055**		**0.275**		**0.065**		**0.057**	
44											0.102	3.4						
45	0.096	0.0	0.353	1.2	0.604	0.4	0.272	0.3	0.082	0.4			0.271	-0.1	0.064	-0.1	0.034	-1.6
46	0.106	0.4			0.618	0.5	0.243	-0.1	0.072	-0.1	0.051	-0.3	0.262	-0.2	0.062	-0.2	0.053	-0.3
47	0.115	0.8	0.303	0.4	0.585	0.2	0.156	-1.5	0.066	-0.5	0.035	-1.5	0.291	0.2	0.058	-0.4	0.05	-0.5
48	0.0851	-0.5	0.458	2.7	0.507	-0.3	0.265	0.2	0.071	-0.2	0.0583	0.2	0.268	-0.1	FN	-3.4	0.0498	-0.5
49	0.095	-0.1	0.271	0.0			0.207	-0.7	0.073	-0.1			0.291	0.2	0.062	-0.2		
50	0.091	-0.2			0.574	0.1	0.361	1.8	FN	-3.5	FN	-3.3	0.271	-0.1	0.035	-1.9	FN	-3.3
51	0.102	0.2	0.324	0.7	0.539	-0.1	0.173	-1.2	0.061	-0.7	FN	-3.3						
52																		
53	0.097	0.0	0.284	0.1	0.483	-0.5	0.245	-0.1	0.068	-0.4	0.065	0.7	0.296	0.3	0.055	-0.6	0.069	0.9
54																		
55	0.0914	-0.2	0.165	-1.6	0.589	0.2	0.233	-0.3	0.067	-0.4	0.0501	-0.4	0.27	-0.1	0.065	0.0	0.0576	0.1
56					0.588	0.2					0.0688	1.0					0.0632	0.5
57																		
58	0.102	0.2	0.306	0.5	0.686	0.9	0.306	0.9	0.092	1.0	0.069	1.0	0.317	0.6	0.062	-0.2	0.0703	1.0
59	0.0529	-1.8	0.105	-2.5	0.34	-1.5	0.155	-1.5	0.044	-1.6	0.377	>5	0.176	-1.4	0.035	-1.8	0.042	-1.0
60	0.1	0.1	0.221	-0.8	0.586	0.2	0.265	0.2	0.086	0.6	0.0524	-0.2	0.311	0.5	0.073	0.5	0.0381	-1.3
61	0.0848	-0.5	0.303	0.4	0.59	0.3	0.225	-0.4	0.079	0.3	0.0421	-0.9	0.217	-0.8	FN	-3.4	0.0432	-1.0
62	0.0972	0.0	0.251	-0.3	0.632	0.6	0.274	0.4	0.077	0.2	0.058	0.2	0.243	-0.5	0.062	-0.2	0.0769	1.4
63	0.0523	-1.8	0.0974	-2.6	0.272	-2.0	0.215	-0.6	0.046	-1.5	0.0555	0.0	0.281	0.1	0.052	-0.8	0.142	>5
64	0.0652	-1.3	0.234	-0.6	0.532	-0.2	0.234	-0.3	0.061	-0.7	0.0619	0.5	0.262	-0.2	0.046	-1.2	0.0558	-0.1
66	0.099	0.1	0.287	0.2	0.609	0.4	0.265	0.2	0.091	0.9			0.282	0.1	0.082	1.0	0.035	-1.5
67	0.111	0.6	0.218	-0.8	0.47	-0.6	0.382	2.1	0.087	0.7	0.06	0.4	0.321	0.7	0.052	-0.8	0.044	-0.9
68	0.065	-1.3	0.06	-3.1	0.525	-0.2	0.15	-1.6	0.062	-0.7	0.027	-2.0	0.15	-1.8	FN	-3.4	0.057	0.0
70					0.472	-0.6					0.04	-1.1					0.054	-0.2
71	0.108	0.5	0.288	0.2	0.55	0.0	0.274	0.4	0.09	0.8	0.057	0.1	0.308	0.5	0.07	0.3	0.047	-0.7
72	0.0862	-0.4	0.248	-0.4	0.442	-0.8	0.193	-0.9	0.069	-0.3	0.0528	-0.2	0.21	-0.9	0.049	-0.9	0.0422	-1.0
73	0.108	0.5	0.243	-0.5	0.587	0.2	0.231	-0.3	0.074	0.0	0.056	0.1	0.309	0.5	0.047	-1.1	0.059	0.2
74	0.0945	-0.1	0.61	4.9	0.705	1.1	0.24	-0.2	0.042	-1.7	0.0695	1.0	0.281	0.1	0.073	0.5	0.033	-1.7
75					0.495	-0.4					0.0504	-0.3					0.0732	1.2
76	0.102	0.2	0.273	0.0	0.574	0.1	0.269	0.3	0.078	0.2	0.0632	0.6	0.288	0.2	0.056	-0.5	0.0622	0.4
77									0.09	0.8	0.064	0.6						
78	0.207	4.6			FN	-3.9			0.096	1.1	FN	-3.3						
79											0.04	-1.1						
80	0.082	-0.6	0.316	0.6	0.405	-1.1	0.24	-0.2	0.099	1.3	0.078	1.7	0.272	0.0	FN	-3.4	0.07	0.9
81	0.107	0.4			0.788	1.7	0.236	-0.2			0.494	>5					0.0647	0.6
82	0.088	-0.4	0.147	-1.9	0.31	-1.8	0.158	-1.5	0.046	-1.5	0.035	-1.5	0.176	-1.4			0.026	-2.2
83	0.09	-0.3	0.171	-1.5	0.461	-0.7	0.27	0.3	0.046	-1.5	0.041	-1.0	0.237	-0.6			0.032	-1.7
84	0.0962	0.0	0.332	0.8	0.497	-0.4	0.257	0.1	0.070	-0.2	FN	-3.3	0.255	-0.3	0.051	-0.9		
85	0.129	1.3	0.279	0.1	0.558	0.0	0.337	1.4	0.068	-0.4	0.052	-0.2	0.348	1.1	0.072	0.4	0.064	0.5
86	0.106	0.4			0.556	0.0	0.295	0.7	0.067	-0.4	0.052	-0.2	0.286	0.2	0.062	-0.2	0.052	-0.3

Table 10b. Results for fludioxonil, flusilazole, flutolanil, lambda-cyhalothrin, metconazole , prothiofos, pyraclostrobin, tebuconazole and chlordane, trans in mg/kg, and the corresponding z scores, MRRLs and the assigned values.

Laboratory code	Fludioxonil	Z score (FFP RSD (25%)	Flusilazole	Z score (FFP RSD (25%)	Flutolanil	Z score (FFP RSD (25%)	Lambda-cyhalothrin	Z score (FFP RSD (25%)	Metconazole	Z score (FFP RSD (25%)	Prothiofos	Z score (FFP RSD (25%)	Pyraclostrobin	Z score (FFP RSD (25%)	Tebuconazole	Z score (FFP RSD (25%)	Chlordane, trans	Z score (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.082**		**0.405**		**0.053**		**0.048**		**0.268**		**0.044**		**0.406**		**0.706**		**0.050**	
44																	0.084	2.8
45	0.084	0.1	0.373	-0.3	0.062	0.7	0.052	0.4	0.309	0.6			0.541	1.3	0.626	-0.5	0.064	1.2
46	0.084	0.1	0.435	0.3			0.050	0.3							0.63	-0.4		
47	0.086	0.2	0.418	0.1	0.056	0.3	0.051	0.3	0.258	-0.2	0.039	-0.5	0.446	0.4	0.766	0.3	FN	-3.2
48	0.087	0.2	0.365	-0.4	0.049	-0.3	0.046	-0.1	0.258	-0.2	FN	-3.1	0.384	-0.2	0.659	-0.3	0.043	-0.5
49	0.087	0.2	0.423	0.2					0.287	0.3			0.458	0.5	0.823	0.7		
50	0.037	-2.2	0.367	-0.4	0.055	0.2	FN	-3.2			FN	-3.1			0.555	-0.9		
51	0.083	0.0	0.391	-0.1			FN	-3.2					0.404	0.0	0.739	0.2		
52																		
53	0.086	0.2	0.454	0.5	0.068	1.2	0.06	1.0	0.294	0.4	0.053	0.8	0.404	0.0	0.768	0.4		
54																	0.054	0.3
55	0.073	-0.4	0.335	-0.7	0.050	-0.2	0.045	-0.2	0.267	0.0	0.043	-0.1	0.281	-1.2	0.663	-0.2	0.048	-0.1
56							0.053	0.5									0.065	1.3
57																		
58	0.096	0.7	0.409	0.0	0.052	0.0	0.056	0.7	0.318	0.7	0.049	0.5	0.491	0.8	0.732	0.1	0.058	0.7
59	0.056	-1.3	0.25	-1.5	0.030	-1.7	0.027	-1.7	0.153	-1.7	0.024	-1.8	0.311	-0.9	0.434	-1.5	0.034	-1.3
60	0.074	-0.4	0.304	-1.0	0.047	-0.4	0.049	0.2	0.181	-1.3	0.045	0.2	0.454	0.5	0.822	0.7		
61	0.068	-0.7	FN	-3.9	0.041	-0.8	0.033	-1.2	0.256	-0.2	0.041	-0.3	0.368	-0.4	0.741	0.2	0.043	-0.5
62	0.041	-2.0	0.42	0.1	0.05	-0.2	0.056	0.7	0.287	0.3	0.041	-0.2	0.471	0.6	0.729	0.1	0.045	-0.3
63	0.074	-0.4	0.182	-2.2	0.017	-2.6	0.044	-0.3	0.213	-0.8	0.041	-0.2	0.267	-1.4	0.629	-0.4	0.042	-0.6
64	0.073	-0.4	0.367	-0.4	0.064	0.9	0.059	1.0	0.262	-0.1	0.036	-0.7	0.358	-0.5	0.72	0.1	0.049	0.0
66	0.087	0.2	0.434	0.3	0.051	-0.1	0.05	0.2	0.263	-0.1	0.053	0.8	0.439	0.3	0.709	0.0	0.058	0.7
67	0.069	-0.6	0.735	3.3	0.086	2.5	0.03	-1.5	0.326	0.9	0.035	-0.8	0.542	1.3	1.24	3.0	FN	-3.2
68	0.065	-0.8	FN	-3.9	FN	-3.2	0.045	-0.2	FN	-3.9	0.045	0.1	0.33	-0.8	0.05	-3.7	0.45	>5
70																	0.042	-0.6
71	0.09	0.4	0.401	0.0	0.053	0.0	0.048	0.0	0.308	0.6	0.04	-0.4	0.441	0.3	0.708	0.0	0.047	-0.2
72	0.078	-0.2	0.295	-1.1	0.041	-0.9	0.051	0.3	0.179	-1.3	0.050	0.6	0.307	-1.0	0.616	-0.5		
73	0.101	0.9	0.429	0.2	0.041	-0.9	0.04	-0.7	0.247	-0.3	0.034	-0.9	0.349	-0.6	0.709	0.0	0.051	0.1
74	0.045	-1.8	0.402	0.0			0.035	-1.1	0.273	0.1	FN	-3.1	0.454	0.5	0.708	0.0	0.03	-1.6
75																	0.065	1.3
76	0.081	0.0	0.405	0.0	0.052	0.0	0.049	0.2	0.275	0.1	0.050	0.6	0.502	0.9	0.698	0.0	0.053	0.3
77							0.055	0.6									FN	-3.2
78					0.103	3.8	0.049	0.1			FN	-3.1	0.279	-1.3	0.798	0.5	0.044	-0.5
79																	0.037	-1.0
80	0.075	-0.3	0.317	-0.9	0.04	-1.0	0.034	-1.2	0.289	0.3	0.037	-0.6	0.394	-	0.695	-0.1		
81							0.054	0.6							0.6	-0.6	0.053	0.3
82	0.051	-1.5	0.345	-0.6			0.041	-0.6					0.237	-1.7	0.4	-1.7		
83	0.1	0.9	0.522	1.2			0.031	-1.4					0.366	-0.4	0.742	0.2	FN	-3.2
84	0.086	0.2	0.431	0.3	0.052	0.0	0.058	0.9	0.251	-0.3	0.040	-0.3	0.402	0.0	0.681	-0.1	0.039	-0.9
85	0.109	1.3	0.538	1.3	0.054	0.1	0.043	-0.4	0.347	1.2	0.041	-0.3	0.562	1.5	0.86	0.9	0.051	0.1
86	0.098	0.8	FN	-3.9			0.05	0.2							0.826	0.7		

Table 11a. Results for azoxystrobin, carbendazim, chlorpyrifos, cyproconazole, cyprodinil, endosulfan, alpha-, fenbuconazole, fenpropimorph and fenvalerate in mg/kg, and the corresponding z scores, MRRLs and the assigned values..

Laboratory code	Azoxystrobin	Z score (FFP RSD (25%)	Carbendazim	Z score (FFP RSD (25%)	Chlorpyrifos	Z score (FFP RSD (25%)	Cyproconazole	Z score (FFP RSD (25%)	Cyprodinil	Z score (FFP RSD (25%)	Endosulfan, alpha-	Z score (FFP RSD (25%)	Fenbuconazole	Z score (FFP RSD (25%)	Fenpropimorph	Z score (FFP RSD (25%)	Fenvalerate	Z scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.097**		**0.274**		**0.555**		**0.251**		**0.075**		**0.055**		**0.275**		**0.065**		**0.057**	
87	0.0988	0.1	0.18	-1.4	0.591	0.3	0.22	-0.5	0.084	0.5	0.0629	0.6	0.282	0.1	0.06	-0.3	0.0372	-1.4
88	0.0696	-1.1	0.356	1.2	0.585	0.2	0.236	-0.2	0.063	-0.6	0.052	-0.2	0.288	0.2	0.055	-0.6	0.0629	0.4
89	0.15	2.2	0.519	3.6	0.63	0.5	0.44	3.0	0.079	0.2	0.061	0.4	0.514	3.5	0.093	1.7	0.063	0.4
90	0.112	0.6	0.788	>5	0.6	0.3	0.272	0.3	0.071	-0.2	0.06	0.4	0.315	0.6	0.073	0.5	0.059	0.2
92	0.103	0.3			0.57	0.1	0.257	0.1	0.077	0.2	0.0563	0.1	0.253	-0.3	0.072	0.4	0.0539	-0.2
93											0.0541	-0.1						
94	0.0859	-0.4	0.27	-0.1	0.545	-0.1	0.215	-0.6	0.063	-0.6	0.036	-1.4	0.248	-0.4	0.074	0.6	0.047	-0.7
95	0.078	-0.8	0.241	-0.5	0.497	-0.4	0.258	0.1	0.073	-0.1	0.052	-0.2	0.245	-0.4	0.052	-0.8	0.058	0.1
96	0.106	0.4	0.279	0.1	0.568	0.1	0.24	-0.2	0.076	0.1	0.0583	0.2	0.274	0.0	0.062	-0.2	0.0645	0.6
97	0.106	0.4	0.912	>5	0.573	0.1	FN	-3.8	0.069	-0.3			0.249	-0.4	0.061	-0.3	0.053	-0.3
98	FN	-3.6	0.136	-2.0	FN	-3.9	0.191	-1.0	0.096	1.2	0.0524	-0.2	0.27	-0.1	FN	-3.4	FN	-3.3
100	0.079	-0.7	0.32	0.7	0.44	-0.8	0.27	0.3	0.076	0.1			0.27	-0.1				
101	0.0752	-0.9	0.331	0.8	0.52	-0.3	0.243	-0.1	0.068	-0.4	0.0357	-1.4	0.274	0.0	0.077	0.7	0.0546	-0.1
102	0.107	0.4	0.285	0.2	0.511	-0.3	0.278	0.4	0.073	-0.1	0.049	-0.4	0.372	1.4	0.068	0.2	0.0467	-0.7
104	0.098	0.1	0.353	1.2	0.583	0.2	0.261	0.2	0.105	1.6	0.058	0.2	0.331	0.8	0.07	0.3	0.058	0.1
105	0.0966	0.0			0.567	0.1	0.255	0.1	0.072	-0.1	0.0527	-0.2	0.266	-0.1	0.071	0.4	0.0549	-0.1
106	0.108	0.5	0.282	0.1	0.633	0.6	0.255	0.1	0.073	-0.1	0.0525	-0.2	0.301	0.4	0.059	-0.3	0.0578	0.1
107	0.085	-0.5	0.2922	0.3	0.586	0.2	0.235	-0.2	0.082	0.4	0.065	0.7	0.236	-0.6	0.065	0.0	0.1003	3.1
108	0.115	0.8	0.292	0.3	0.523	-0.2	0.254	0.1	0.083	0.5	0.07	1.1	0.214	-0.9	0.082	1.0	0.061	0.3
109	0.0859	-0.4	0.18	-1.4	0.58	0.2	0.231	-0.3	0.055	-1.0	0.0578	0.2	0.245	-0.4	0.053	-0.7	0.0574	0.0
110	0.147	2.1	0.207	-1.0	0.73	1.3	FN	-3.8	0.095	1.1	0.056	0.1	0.37	1.4	0.088	1.4	0.127	5.0
111	0.097	0.0	0.32	0.7	0.62	0.5	0.24	-0.2	0.081	0.3	0.087	2.3	0.3	0.4	0.076	0.7	0.059	0.2
112	FN	-3.6	FN	-3.9	0.644	0.6	0.22	-0.5	0.050	-1.3	0.0302	-1.8	0.243	-0.5	FN	-3.4	FN	-3.3
114	0.094	-0.1			0.396	-1.1	0.242	-0.1	0.061	-0.7	FN	-3.3	0.25	-0.4			0.048	-0.6
115	0.0812	-0.6	0.41	2.0	0.629	0.5	0.267	0.3	0.111	2.0	0.0532	-0.1	0.277	0.0	0.044	-1.2	0.0541	-0.2
116	0.107	0.4			0.544	-0.1	0.266	0.2	0.073	-0.1	0.0509	-0.3	0.283	0.1	0.066	0.1	0.054	-0.2
118	0.0107	-3.6	0.0209	-3.7	0.352	-1.5	0.051	-3.2	0.026	-2.6					0.020	-2.8		
119	0.0791	-0.7	0.197	-1.1	0.612	0.4	0.286	0.6	0.057	-0.9	0.129	>5	0.223	-0.8	0.052	-0.8	0.0594	0.2
120			0.46	2.7	0.548	0.0												
121																		
122	0.0965	0.0			0.661	0.8	0.257	0.1	0.077	0.1	0.0507	-0.3	0.241	-0.5	0.069	0.3	0.074	1.2
123	0.111	0.6	0.261	-0.2	0.822	1.9	0.371	1.9	0.084	0.5			0.301	0.4	0.072	0.4		
124	0.104	0.3	0.287	0.2	0.597	0.3	0.263	0.2	0.081	0.4	0.0676	0.9	0.291	0.2	0.057	-0.5	0.065	0.6
125																		
126	0.0902	-0.3			0.449	-0.8	FN	-3.8	0.060	-0.8	0.0514	-0.3	0.0235	-3.7	0.060	-0.3	0.0543	-0.2
127			0.275	0.0	0.558	0.0	0.193	-0.9	0.064	-0.6	0.06	0.4	0.185	-1.3	0.057	-0.5	0.039	-1.2
128	0.104	0.3	0.318	0.6	0.658	0.7	0.274	0.4	0.083	0.5			0.303	0.4	0.068	0.2		
129	0.043	-2.2	0.139	-2.0	0.299	-1.8	0.12	-2.1	FN	-3.5	0.021	-2.5	0.142	-1.9	FN	-3.4	FN	-3.3
130	0.862	>5	2.845	>5	FN	-3.9	7.856	>5	0.722	>5	FN	-3.3	1.459	>5	FN	-3.4	FN	-3.3
131	FN	-3.6			FN	-3.9			0.087	0.7	FN	-3.3					FN	-3.3
132	0.0886	-0.3	0.187	-1.3	0.653	0.7	0.235	-0.2	0.059	-0.8	0.042	-1.0	0.227	-0.7	0.042	-1.4		

Table 11b. Results for fludioxonil, flusilazole, flutolanil, lambda-cyhalothrin, metconazole , prothiofos, pyraclostrobin, tebuconazole and chlordane, trans in mg/kg, and the corresponding z scores, MRRLs and the assigned values.

Laboratory code	Fludioxonil	Z score (FFP RSD (25%)	Flusilazole	Z score (FFP RSD (25%)	Flutolanil	Z score (FFP RSD (25%)	Lambda-cyhalothrin	Z score (FFP RSD (25%)	Metconazole	Z score (FFP RSD (25%)	Prothiofos	Z score (FFP RSD (25%)	Pyraclostrobin	Z score (FFP RSD (25%)	Tebuconazole	Z score (FFP RSD (25%)	Chlordane, trans	Z score (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.082**		**0.405**		**0.053**		**0.048**		**0.268**		**0.044**		**0.406**		**0.706**		**0.050**	
87	0.086	0.2	0.367	-0.4	0.057	0.4	0.040	-0.6	0.237	-0.5	0.039	-0.4	0.374	-0.3	0.558	-0.8	0.067	1.4
88	0.084	0.1	0.427	0.2	0.053	0.0	0.057	0.8	0.269	0.0	0.041	-0.3	0.344	-0.6	0.672	-0.2	0.047	-0.2
89	0.088	0.3	0.691	2.8	0.062	0.7	0.056	0.7	0.605	5.0	0.05	0.5	1.075	>5	1.138	2.4	0.052	0.2
90	0.081	0.0	0.513	1.1	0.073	1.6	0.049	0.1	0.287	0.3	0.052	0.7	0.543	1.3	0.956	1.4	0.047	-0.2
92	0.089	0.3	0.399	-0.1			0.051	0.3							0.71	0.0	0.047	-0.2
93																	0.049	0.0
94	0.074	-0.4	0.376	-0.3	0.046	-0.5	0.043	-0.4	0.225	-0.6	0.039	-0.4	0.384	-0.2	0.692	-0.1	0.04	-0.8
95	0.063	-0.9	0.331	-0.7	0.049	-0.3	0.043	-0.4	0.234	-0.5	0.044	0.0	0.325	-0.8	0.635	-0.4	0.051	0.1
96	0.078	-0.2	0.37	-0.3	0.050	-0.2	0.049	0.1	0.255	-0.2	0.049	0.5	0.429	0.2	0.614	-0.5	0.052	0.3
97	0.079	-0.1	FN	-3.9	0.064	0.9	0.048	0.0	0.255	-0.2			0.42	0.1	0.726	0.1		
98	FN	-3.5	0.448	0.4			FN	-3.2					0.248	-1.6	0.686	-0.1		
100			0.43	0.2					0.27	0.0			0.35	-0.6	0.72	0.1		
101	0.115	1.6	0.411	0.1	0.054	0.2	0.051	0.3	0.269	0.0	0.039	-0.4	0.39	-0.2	0.655	-0.3	0.035	-1.2
102	0.099	0.8	0.497	0.9	0.044	-0.7	0.045	-0.2	0.306	0.6	0.036	-0.6	0.438	0.3	0.765	0.3	0.048	-0.1
104	0.086	0.2	0.484	0.8	0.055	0.2	0.053	0.4	0.289	0.3	0.023	-1.9	0.381	-0.2	0.8	0.5	0.052	0.2
105	0.076	-0.3	0.363	-0.4			0.046	-0.1	0.312	0.7					0.709	0.0	0.045	-0.4
106	0.095	0.6	0.418	0.1	0.059	0.5	0.054	0.5	0.249	-0.3	0.050	0.6	0.429	0.2	0.69	-0.1	0.053	0.3
107	0.074	-0.4	0.365	-0.4	0.061	0.7	0.063	1.3	0.267	0.0	0.061	1.6	0.381	-0.2	0.65	-0.3	0.059	0.8
108	0.09	0.4	0.44	0.3	0.049	-0.3	0.055	0.6	0.248	-0.3	0.047	0.3	0.461	0.5	0.63	-0.4	0.03	-1.6
109	0.078	-0.2	0.373	-0.3	0.036	-1.2	0.049	0.2	0.233	-0.5	0.038	-0.5	0.387	-0.2	0.683	-0.1	0.041	-0.7
110	0.108	1.3	0.804	3.9	0.059	0.5	0.114	>5	0.229	-0.6	0.053	0.8	FN	-3.9	0.79	0.5	FN	-3.2
111	0.09	0.4	0.46	0.5	0.06	0.6	0.048	0.0	0.31	0.6	0.05	0.5	0.46	0.5	0.73	0.1		
112	0.092	0.5	0.371	-0.3	0.053	0.0	FN	-3.2	0.279	0.2	0.036	-0.7	0.394	-0.1	0.785	0.4		
114	0.094	0.6	0.528	1.2			0.036	-1.0					0.472	0.6	0.649	-0.3		
115	0.096	0.7	0.318	-0.9	0.060	0.6	0.050	0.3	0.298	0.4	0.042	-0.2	0.37	-0.4	0.78	0.4	0.052	0.2
116	0.088	0.3	0.445	0.4			0.049	0.1							0.705	0.0		
118											0.050	0.6	0.264	-1.4	0.159	-3.1		
119	0.117	1.7	0.446	0.4	0.043	-0.7	0.06	1.0	0.162	-1.6	0.033	-0.9	0.346	-0.6	0.78	0.4	0.431	>5
120															0.514	-1.1		
121																		
122	0.073	-0.4	0.361	-0.4			0.043	-0.4					0.326	-0.8	0.624	-0.5	0.044	-0.4
123			0.451	0.5			0.050	0.2					0.44	0.3	0.77	0.4		
124	0.085	0.1	0.41	0.0	0.053	0.0	0.050	0.2	0.28	0.2	0.052	0.8	0.479	0.7	0.709	0.0	0.053	0.3
125																		
126	0.090	0.4	0.472	0.7			0.033	-1.2					0.43	0.2	0.625	-0.5		
127	0.065	-0.8					0.051	0.3	0.158	-1.6			0.57	1.6	0.566	-0.8	0.048	-0.1
128	0.087	0.3	0.458	0.5	0.055	0.2			0.251	-0.3	0.046	0.2	0.482	0.7	0.718	0.1	0.044	-0.4
129	0.038	-2.1	0.196	-2.1	0.024	-2.2	0.025	-1.9	0.118	-2.2	0.019	-2.3	0.17	-2.3	0.322	-2.2	0.025	-2.0
130	1.02	>5	2.901	>5	0.541	>5	FN	-3.2	0.958	>5	FN	-3.1	2.67	>5	4.102	>5	FN	-3.2
131	FN	-3.5					FN	-3.2			FN	-3.1			0.794	0.5		
132	FN	-3.5	0.319	-0.9	0.044	-0.6	0.041	-0.5					0.364	-0.4	0.661	-0.3		

Table 12a. Results for azoxystrobin, carbendazim, chlorpyrifos, cyproconazole, cyprodinil, endosulfan, alpha-, fenbuconazole, fenpropimorph and fenvalerate in mg/kg, and the corresponding z scores, MRRLs and the assigned values.

Laboratory code	Azoxystrobin	Z score (FFP RSD (25%)	Carbendazim	Z score (FFP RSD (25%)	Chlorpyrifos	Z score (FFP RSD (25%)	Cyproconazole	Z score (FFP RSD (25%)	Cyprodinil	Z score (FFP RSD (25%)	Endosulfan, alpha-	Z score (FFP RSD (25%)	Fenbuconazole	Z score (FFP RSD (25%)	Fenpropimorph	Z score (FFP RSD (25%)	Fenvalerate	Z scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.097**		**0.274**		**0.555**		**0.251**		**0.075**		**0.055**		**0.275**		**0.065**		**0.057**	
133	0.103	0.3	0.269	-0.1	0.54	-0.1	0.251	0.0	0.0674	-0.4	0.0521	-0.2	0.279	0.1	0.073	0.5	0.0514	-0.4
134	0.072	-1.0	0.16	-1.7	0.389	-1.2	0.21	-0.6	0.048	-1.4	0.035	-1.5	0.17	-1.5	0.05	-0.9	0.05	-0.5
135	0.0905	-0.3			0.355	-1.4	0.247	-0.1	0.0599	-0.8	FN	-3.3	0.263	-0.2	0.0482	-1.0	FN	-3.3
136	0.072	-1.0	0.251	-0.3	0.589	0.2	0.179	-1.1	0.057	-0.9	0.034	-1.5	0.214	-0.9	0.077	0.7	0.068	0.8
137	0.0707	-1.1	0.257	-0.2	0.498	-0.4	0.25	0.0	0.0792	0.2	0.0396	-1.1	0.203	-1.0	0.0591	-0.4	0.0436	-0.9
138	0.094	-0.1	0.255	-0.3	0.51	-0.3	0.273	0.4	FN	-3.5	0.058	0.2	0.163	-1.6	FN	-3.4	0.056	0.0
139	0.0918	-0.2			0.642	0.6	0.231	-0.3	0.0779	0.2	0.0542	-0.1	0.251	-0.3	0.0693	0.3	0.0498	-0.5
140	0.084	-0.5			0.605	0.4	FN	-3.8	0.087	0.7	FN	-3.3	FN	-3.9			FN	-3.3
141																		
142	0.11	0.6			0.593	0.3	0.229	-0.3	0.07	-0.2					0.071	0.4		
143	0.123	1.1	0.22	-0.8	0.888	2.4	0.364	1.8	0.0697	-0.3			0.333	0.8				
144							0.15	-1.6	0.067	-0.4							0.053	-0.3
145	0.091	-0.2	0.134	-2.0	0.503	-0.4	0.229	-0.3	0.058	-0.9	0.043	-0.9	0.238	-0.5	0.061	-0.3	0.051	-0.4
146	0.0991	0.1	0.282	0.1	0.542	-0.1	0.273	0.4	0.0736	-0.1	0.053	-0.2	0.292	0.2	0.0633	-0.1	0.057	0.0
147	0.0889	-0.3	0.225	-0.7	0.478	-0.6	0.254	0.1	0.0709	-0.2	0.0543	-0.1	0.245	-0.4	0.0669	0.1	0.0482	-0.6
148																		
149	0.086	-0.4	1.03	>5	0.0501	-3.6	0.43	2.9	0.094	1.0	0.054	-0.1	0.328	0.8	0.1	2.1	0.05	-0.5
151	0.118	0.9	0.27	-0.1	0.593	0.3	0.259	0.1	0.0744	0.0	0.0633	0.6	0.292	0.2	0.0854	1.2	0.0562	0.0
152	0.166	2.9	1.02	>5	0.501	-0.4	0.315	1.0	0.136	3.3	0.0517	-0.2	0.476	2.9	0.106	2.5	0.0648	0.6
153	0.0733	-1.0	0.427	2.2	0.516	-0.3	0.268	0.3	0.055	-1.1	0.0468	-0.6					0.0553	-0.1
154	0.127	1.3	0.123	-2.2	0.502	-0.4	0.168	-1.3	0.0802	0.3	0.0571	0.1	0.151	-1.8	0.0668	0.1	0.0169	-2.8
155	0.1	0.1	0.24	-0.5	0.518	-0.3	0.26	0.2	0.071	-0.2					FN	-3.4		
156	0.1	0.1	0.261	-0.2	0.573	0.1	0.257	0.1	0.078	0.2	0.049	-0.4	0.285	0.1	0.076	0.7	0.051	-0.4
157	0.0775	-0.8	0.243	-0.5	0.504	-0.4	0.199	-0.8	0.067	-0.4	0.0497	-0.4	0.223	-0.8	0.0463	-1.2	0.0664	0.7
158					0.618	0.5												
159	0.104	0.3	0.577	4.4	0.762	1.5	0.247	-0.1	0.098	1.3	0.078	1.7	0.29	0.2	0.104	2.4	0.065	0.6
160	0.102	0.2	0.31	0.5	0.551	0.0	0.252	0.0	0.0736	-0.1	0.0628	0.6	0.257	-0.3	0.0552	-0.6	0.0652	0.6
161	0.0848	-0.5	0.229	-0.7	0.659	0.8	0.176	-1.2	0.0647	-0.5	0.0365	-1.4	0.234	-0.6	0.0692	0.2	0.06	0.2
162	0.101	0.2	0.198	-1.1	0.709	1.1	FN	-3.8	0.0894	0.8	0.0633	0.6	0.31	0.5			0.024	-2.3
163	0.083	-0.6	0.418	2.1	0.456	-0.7	FN	-3.8	0.06	-0.8	0.067	0.9	0.321	0.7	0.06	-0.3		
164	0.095	-0.1			0.769	1.5	0.241	-0.2	0.085	0.6	0.051	-0.3	0.27	-0.1	0.066	0.0	0.076	1.4
165											0.0447	-0.8						
166	0.11	0.6	0.22	-0.8	0.563	0.1	0.27	0.3	0.083	0.5	0.112	4.1	0.303	0.4	0.062	-0.2	0.092	2.5
167	0.083	-0.6	0.242	-0.5	0.51	-0.3	0.24	-0.2	0.08	0.3	0.048	-0.5	0.26	-0.2	0.066	0.0	0.049	-0.5
168											0.0743	1.4						
169	0.1	0.1	0.419	2.1	0.637	0.6	FN	-3.8	0.104	1.6	0.077	1.6	0.305	0.4	0.075	0.6	0.037	-1.4
170	0.104	0.3	0.25	-0.3	0.411	-1.0	0.299	0.8	0.093	1.0	0.015	-2.9	0.311	0.5	0.073	0.5	0.046	-0.8
171	0.059	-1.6	0.26	-0.2	0.39	-1.2	0.045	-3.3	0.1	1.4	0.061	0.4	0.28	0.1	0.077	0.7	0.061	0.3
172									0.0719	-0.1	0.0712	1.2					0.0657	0.6
173	0.112	0.6	0.256	-0.3	0.629	0.5	0.256	0.1	0.072	-0.1	0.0374	-1.3	0.287	0.2	0.064	-0.1		
174	0.096	0.0	FN	-3.9	0.504	-0.4	0.277	0.4	0.09	0.8	FN	-3.3	0.334	0.9	0.13	4.0	0.14	>5

Table 12b. Results for fludioxonil, flusilazole, flutolanil, lambda-cyhalothrin, metconazole , prothiofos, pyraclostrobin, tebuconazole and chlordane, trans in mg/kg, and the corresponding z scores, MRRLs and the assigned values.

Laboratory code	Fludioxonil	Z score (FFP RSD (25%)	Flusilazole	Z score (FFP RSD (25%)	Flutolanil	Z score (FFP RSD (25%)	Lambda-cyhalothrin	Z score (FFP RSD (25%)	Metconazole	Z score (FFP RSD (25%)	Prothiofos	Z score (FFP RSD (25%)	Pyraclostrobin	Z score (FFP RSD (25%)	Tebuconazole	Z score (FFP RSD (25%)	Chlordane, trans	Z score (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.082**		**0.405**		**0.053**		**0.048**		**0.268**		**0.044**		**0.406**		**0.706**		**0.050**	
133	0.093	0.6	0.42	0.1	0.053	0.0	0.051	0.3	0.237	-0.5	0.038	-0.5	0.446	0.4	0.698	0.0	0.050	0.1
134	0.054	-1.4	0.432	0.3	0.04	-1.0	0.04	-0.7	0.273	0.1	FN	-3.1	0.309	-1.0	0.46	-1.4		
135	0.078	-0.2	0.394	-0.1			0.034	-1.2	0.231	-0.6	FN	-3.1	0.388	-0.2	0.618	-0.5		
136	0.078	-0.2	0.367	-0.4	0.047	-0.4	0.055	0.6	0.326	0.9	0.052	0.7	0.379	-0.3	0.629	-0.4	0.043	-0.5
137	0.063	-0.9	0.328	-0.8			0.042	-0.4	0.246	-0.3	FN	-3.1	0.24	-1.6	0.528	-1.0	0.045	-0.4
138	0.093	0.5	0.315	-0.9	0.048	-0.3	0.05	0.2	0.21	-0.9	0.047	0.3	0.18	-2.2	0.7	0.0		
139	0.078	-0.2	0.41	0.0			0.046	-0.1	0.235	-0.5					0.671	-0.2	0.045	-0.3
140	FN	-3.5	FN	-3.9			FN	-3.2							FN	-3.9		
141																		
142	0.086	0.2					0.043	-0.4							0.624	-0.5		
143			0.474	0.7									0.53	1.2	0.787	0.5	0.303	>5
144	0.062	-1.0					0.068	1.7					0.38	-0.3	0.61	-0.5	0.056	0.5
145	0.077	-0.2	0.426	0.2	0.051	-0.1	0.04	-0.7	0.225	-0.6					0.696	-0.1	0.041	-0.7
146	0.093	0.6	0.423	0.2	0.055	0.3	0.053	0.4	0.255	-0.2	0.041	-0.2	0.432	0.3	0.72	0.1	0.043	-0.5
147	0.082	0.0	0.461	0.5	0.056	0.3	0.045	-0.2	0.301	0.5	0.049	0.5	0.379	-0.3	0.832	0.7		
148																		
149	0.053	-1.4	0.423	0.2	0.057	0.4	0.046	-0.2	0.253	-0.2			0.262	-1.4	0.896	1.1	0.052	0.2
151	0.104	1.1	0.438	0.3			0.050	0.2	0.284	0.2	0.042	-0.1	0.428	0.2	0.702	0.0	0.074	2.0
152	0.055	-1.3	0.56	1.5	0.104	3.9	0.042	-0.4	0.322	0.8	0.042	-0.1	FN	-3.9	1.09	2.2	0.06	0.8
153	0.081	0.0	0.349	-0.6			0.042	-0.4							0.654	-0.3		
154	0.078	-0.2	0.314	-0.9	0.052	0.0	0.033	-1.2	0.169	-1.5			0.449	0.4	0.657	-0.3	0.048	-0.1
155	0.115	1.6	0.331	-0.7	0.051	-0.1	0.042	-0.5					0.182	-2.2	0.62	-0.5	0.175	>5
156	0.09	0.4	0.431	0.3	0.06	0.6	0.045	-0.2	0.227	-0.6	0.047	0.3	0.433	0.3	0.689	-0.1	0.049	-0.1
157	0.064	-0.8	0.34	-0.6	0.042	-0.8	0.050	0.2	0.238	-0.5	0.043	0.0	0.337	-0.7	0.558	-0.8	0.051	0.1
158																		
159	0.099	0.8	0.427	0.2	0.06	0.6	0.077	2.4	0.304	0.5			0.486	0.8	0.735	0.2	0.066	1.3
160	0.112	1.5	0.393	-0.1	0.051	-0.1	0.060	1.1	0.278	0.1	0.044	0.0	0.386	-0.2	0.622	-0.5	0.075	2.1
161	0.066	-0.8	0.311	-0.9	0.052	0.0	0.045	-0.2	0.146	-1.8	0.048	0.4	0.424	0.2	0.299	-2.3	0.049	0.0
162	FN	-3.5	0.464	0.6	0.050	-0.2	0.057	0.8	0.338	1.0	0.052	0.8	0.437	0.3	0.866	0.9		
163	0.077	-0.2	0.472	0.7	0.04	-1.0	0.025	-1.8	0.329	0.9	0.044	0.0	0.412	0.1	0.767	0.3	0.053	0.3
164	0.074	-0.4	0.391	-0.1			0.053	0.4							0.7	0.0	0.044	-0.5
165																	0.044	-0.4
166	0.091	0.4	FN	-3.9	FN	-3.2	0.065	1.4	0.285	0.2	0.063	1.7	0.4	-0.1	1.23	3.0		
167	0.076	-0.3	FN	-3.9	0.054	0.1	0.044	-0.3	0.28	0.2	0.085	3.7	0.33	-0.8	0.74	0.2		
168																	0.053	0.3
169	0.099	0.8	FN	-3.9			0.061	1.1	0.413	2.2	0.031	-1.2	0.481	0.7	0.844	0.8	0.064	1.2
170	FN	-3.5	0.43	0.2	0.072	1.5	FN	-3.2	0.296	0.4			0.39	-0.2	0.72	0.1	FN	-3.2
171	0.066	-0.8	0.36	-0.4	0.07	1.3	0.058	0.9	0.15	-1.8	0.047	0.3	0.28	-1.2	0.31	-2.2	0.044	-0.5
172	0.081	0.0	0.485	0.8			0.041	-0.5			0.036	-0.7			0.738	0.2	0.061	1.0
173	0.148	3.2	0.479	0.7	0.051	-0.1	FN	-3.2	0.294	0.4			0.465	0.6	0.697	-0.1		
174	FN	-3.5	0.444	0.4	0.044	-0.7	0.04	-0.7	0.356	1.3	0.058	1.3	0.328	-0.8	0.7	0.0		

Table 13a. Results for azoxystrobin, carbendazim, chlorpyrifos, cyproconazole, cyprodinil, endosulfan, alpha-, fenbuconazole, fenpropimorph and fenvalerate in mg/kg, and the corresponding z scores, MRRLs and the assigned values.

Laboratory code	Azoxystrobin	Z score (FFP RSD (25%)	Carbendazim	Z score (FFP RSD (25%)	Chlorpyrifos	Z score (FFP RSD (25%)	Cyproconazole	Z score (FFP RSD (25%)	Cyprodinil	Z score (FFP RSD (25%)	Endosulfan, alpha-	Z score (FFP RSD (25%)	Fenbuconazole	Z score (FFP RSD (25%)	Fenpropimorph	Z score (FFP RSD (25%)	Fenvalerate	Z scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.097**		**0.274**		**0.555**		**0.251**		**0.075**		**0.055**		**0.275**		**0.065**		**0.057**	
175	0.115	0.8	0.105	-2.5	0.384	-1.2	0.151	-1.6	0.0489	-1.4	0.0237	-2.3	0.217	-0.8	0.0518	-0.8	0.0581	0.1
176	0.118	0.9	0.311	0.5	0.532	-0.2					0.052	-0.2					0.076	1.4
177	0.103	0.3	0.627	>5	0.585	0.2					0.056	0.1					0.065	0.6
178	0.102	0.2			0.508	-0.3	0.217	-0.5	0.0643	-0.6	0.0396	-1.1	0.267	-0.1	0.0665	0.1	0.1135	4.0
179	0.039	-2.4	0.049	-3.3	FN	-3.9	0.082	-2.7	0.044	-1.6	FN	-3.3	0.128	-2.1	0.11	2.7	FN	-3.3
180					0.67	0.8	0.28	0.5	0.075	0.0	0.06	0.4					0.068	0.8
186	0.101	0.2	0.228	-0.7	0.667	0.8	0.212	-0.6	0.074	0.0	0.067	0.9	0.26	-0.2	0.056	-0.6	0.061	0.3

Table 13b. Results for fludioxonil, flusilazole, flutolanil, lambda-cyhalothrin, metconazole , prothiofos, pyraclostrobin, tebuconazole and chlordane, trans in mg/kg, and the corresponding z scores, MRRLs and the assigned values.

Laboratory code	Fludioxonil	Z score (FFP RSD (25%)	Flusilazole	Z score (FFP RSD (25%)	Flutolanil	Z score (FFP RSD (25%)	Lambda-cyhalothrin	Z score (FFP RSD (25%)	Metconazole	Z score (FFP RSD (25%)	Prothiofos	Z score (FFP RSD (25%)	Pyraclostrobin	Z score (FFP RSD (25%)	Tebuconazole	Z score (FFP RSD (25%)	Chlordane, trans	Z score (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.082**		**0.405**		**0.053**		**0.048**		**0.268**		**0.044**		**0.406**		**0.706**		**0.050**	
175	0.048	-1.6	0.427	0.2	0.039	-1.0	0.051	0.3	0.172	-1.4	0.035	-0.8	0.432	0.3	0.667	-0.2	0.043	-0.5
176			0.48	0.7			0.065	1.4	0.365	1.4			0.455	0.5			0.241	>5
177			0.42	0.1	0.051	-0.1	0.044	-0.3	0.255	-0.2			0.393	-0.1	0.636	-0.4	0.252	>5
178	0.084	0.1	0.385	-0.2	0.057	0.4	0.031	-1.4			0.033	-1.0	0.404	0.0	0.634	-0.4		
179	0.19	>5	FN	-3.9			FN	-3.2	0.126	-2.1	FN	-3.1	0.366	-0.4	0.095	-3.5		
180	0.07	-0.6	0.46	0.5			0.061	1.1							0.69	-0.1		
186	0.08	-0.1	0.381	-0.2	0.053	0.0	0.05	0.2	0.235	-0.5	0.042	-0.2	0.491	0.8	0.736	0.2	0.048	-0.1

3.3.2 Sum of Weighted Z scores (AZ^2) – 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 (≥15 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 65 EU and EFTA laboratories in Category A (45%), the results were additionally evaluated by calculating the Average of the Squared -Score (AZ^2). Of the 65 participants, 60 participants (92%) obtained AZ^2 values at or below 2 (good) and 1 participant (1.5%) obtained AZ^2 values between 2-3 (satisfactory) and 4 participants (6.1%) obtained AZ^2 values ≥3 (unsatisfactory). Additional one laboratory from Third Countries were evaluated and classified into Category A. The AZ^2 scores achieved by the labs can be seen in **Table 14.**

Table 14 Sum of Weighted z scores (AZ^2) for laboratories in Category A, the number of pesticide analysed by the laboratory, the number of false negatives reported and the classification as good, satisfactory and unsatisfactory. The table include data for both EU and non-EU participants

Lab code	No. of detected compulsory pesticides	No. of detected volutary pesticides	AZ^2	No. Of False negative	Classification	NRL
8	16	1	1.8	1	Good	NRL
9	17	1	0.3	0	Good	
11	17	1	0.1	0	Good	
12	16	1	1.0	1	Good	
19	17	1	0.1	0	Good	NRL
22	16	1	1.5	1	Good	
23	17	1	0.7	0	Good	NRL
24	17	1	1.8	0	Good	NRL
29	17	1	0.2	0	Good	
32	17	1	0.2	0	Good	
34	16	0	0.5	0	Good	NRL
38	17	1	0.3	0	Good	NRL
40	17	1	0.3	0	Good	
41	17	1	1.0	0	Good	
42	17	1	1.0	0	Good	
43	17	1	0.2	0	Good	
53	17	0	0.3	0	Good	
55	17	1	0.3	0	Good	NRL
58	17	1	0.5	0	Good	
59	17	1	4.0	0	Unsatisfactory	
60	17	0	0.4	0	Good	
61	15	1	1.9	2	Good	NRL
62	17	1	0.5	0	Good	
63	17	1	3.4	0	Unsatisfactory	
64	17	1	0.4	0	Good	NRL
66	16	1	0.3	0	Good	
71	17	1	0.2	0	Good	
72	17	0	0.6	0	Good	NRL

Lab code	No. of detected compulsory pesticides	No. of detected volutary pesticides	AZ[2]	No. Of False negative	Classification	NRL
73	17	1	0.3	0	Good	
74	15	1	2.9	1	Satisfactory	
76	17	1	0.1	0	Good	
80	16	0	1.3	1	Good	
84	15	1	0.8	1	Good	
85	17	1	0.8	0	Good	
87	17	1	0.4	0	Good	
88	17	1	0.3	0	Good	
89	17	1	>5	0	Unsatisfactory	
90	17	1	2.0	0	Good	NRL
94	17	1	0.3	0	Good	NRL
95	17	1	0.3	0	Good	NRL
96	17	1	0.1	0	Good	
101	17	1	0.4	0	Good	NRL
102	17	1	0.4	0	Good	
104	17	1	0.6	0	Good	NRL
106	17	1	0.1	0	Good	NRL
107	17	1	0.9	0	Good	NRL
108	17	1	0.3	0	Good	
109	17	1	0.4	0	Good	
111	17	0	0.5	0	Good	
115	17	1	0.7	0	Good	
124	17	1	0.2	0	Good	NRL
133	17	1	0.1	0	Good	
136	17	1	0.6	0	Good	
137	15	1	1.2	1	Good	
146	17	1	0.1	0	Good	
147	17	0	0.2	0	Good	
151	16	1	0.3	0	Good	
156	17	1	0.1	0	Good	
157	17	1	0.4	0	Good	NRL
160	17	1	0.3	0	Good	
161	17	1	0.9	0	Good	NRL
166	15	0	3.8	2	Unsatisfactory	
167	16	0	1.8	1	Good	
171	17	1	1.8	0	Good	
175	17	1	1.5	0	Good	
186	17	1	0.2	0	Good	NRL

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 15.** Nine participants were moved from Category A to B, due to false positive results (marked with *). Sixteen 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 or had false positive results. The reverse was the case for two participants. Fifty-eight participants (25%) analysed and detected less than 70% of the pesticides present in the Test Item.

Table 15 Number of pesticides analysed, number of compulsory and voluntary compounds analysed from the Target List, number of acceptable z scores, false negative and positive results for the laboratories in Category B.

Lab code	No. of compulsory pesticides detected	Compulsory pesticides detected, %	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
1	6	35	41	0	1	0	2	
2	14	82	72	1	14	0	0	
3*	16	94	97	1	14	0	2	
5	15	88	82	1	13	0	0	
6	17	100	83	0	16	0	0	NRL
10*	17	100	100	1	14	0	1	
13	14	88	72	1	15	1	1	
14	13	82	63	1	13	1	0	
15	10	100	100	0	10	7	0	NRL
16	14	82	67	1	14	0	0	
18	15	88	79	0	13	0	0	
20	17	100	88	1	16	0	0	
21	15	88	79	1	11	0	1	
25	3	18	20	1	3	0	0	
26	12	76	50	1	12	1	0	
27*	16	100	97	1	16	1	1	NRL
28	1	6	7	1	1	0	0	
31	16	94	80	1	13	0	0	
33	14	100	100	1	15	3	0	
35	16	100	78	0	16	1	0	
36	8	47	42	0	8	0	0	
37	15	88	73	1	15	0	0	
39*	17	100	99	0	17	0	1	NRL
44	1	6	7	1	0	0	0	
45	15	88	79	1	15	0	0	
46	12	71	39	0	12	0	1	
47*	16	100	100	1	17	1	1	
48*	15	100	99	1	14	2	1	
49	11	65	69	0	11	0	0	

Lab code	No. of compulsory pesticides detected	Compulsory pesticides detected, %	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
50	9	82	66	0	8	5	0	
51	9	65	64	0	9	2	0	
54	0	0	0	1	0	0	0	
56	4	24	21	1	4	0	0	
67*	16	100	97	1	13	1	1	
68	13	100	100	1	10	4	2	
70	3	18	20	1	3	0	0	
75	3	18	18	1	3	0	0	
77	2	18	21	1	3	1	0	
78	6	53	43	1	4	3	0	
79	1	6	7	1	1	0	0	
81	7	41	33	1	6	0	0	
82	13	76	63	0	12	0	0	
83	12	76	67	1	13	1	1	
86	11	71	61	0	11	1	0	
92	12	71	39	1	12	0	0	
93	1	6	7	1	1	0	0	
97	13	88	87	0	12	2	1	NRL
98	8	82	71	0	7	6	0	NRL
100	10	59	62	0	10	0	0	
105	13	76	48	1	13	0	0	
110	14	100	88	1	11	3	0	
112	12	100	100	0	12	5	1	
114	11	71	65	0	11	1	0	
116	12	71	39	0	12	0	1	
118	9	53	52	0	3	0	0	
119*	17	100	99	1	16	0	1	
120	3	18	29	0	2	0	0	
121	0	0	13	0	0	0	4	
122	13	76	44	1	13	0	0	
123	11	65	62	0	11	0	0	
126	12	76	48	0	11	1	0	
127	13	76	90	1	13	0	0	
128	14	82	100	1	14	0	0	
129	14	100	92	1	4	3	0	
130	10	100	77	1	0	7	1	
131	2	53	38	0	2	7	0	
132	13	82	68	0	13	1	0	

Lab code	No. of compulsory pesticides detected	Compulsory pesticides detected, %	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
134	16	100	88	0	16	1	0	
135	12	88	77	0	12	3	1	
138	15	100	88	0	14	2	3	NRL
139	13	76	44	1	13	0	0	
140	3	65	35	0	3	8	0	
141	0	0	25	0	0	0	2	
142	8	47	37	0	8	0	0	
143	9	53	50	1	8	0	0	
144	7	41	76	1	7	0	0	
145	15	88	71	1	14	0	0	
149*	16	94	92	1	12	0	1	NRL
152	16	100	89	1	9	1	0	
153	11	65	56	0	10	0	0	NRL
154	16	94	89	1	14	0	0	NRL
155	11	71	57	1	10	1	1	
158	1	6	18	0	1	0	0	
159	16	94	87	1	13	0	0	
162	14	94	68	0	13	2	0	
163	15	94	77	1	14	1	0	
164	12	71	38	1	12	0	0	
165	1	6	7	1	1	0	0	
168	1	6	7	1	1	0	0	
169	14	94	86	1	12	2	0	
170	13	94	96	1	13	3	1	
172	8	47	34	1	8	0	0	
173	14	88	79	0	13	1	0	
174	14	100	75	0	12	3	1	NRL
176	9	53	80	1	9	0	1	
177	11	65	80	1	10	0	0	
178	15	88	63	0	14	0	0	
179	10	94	83	0	2	6	1	NRL
180	9	53	42	0	9	0	0	

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. In EUPT-C5 in 2009 133 labs participated and in the latest EUPT-CF11 149 labs participated (**Table 16**). The number of pesticides included in the Target Pesticide List has also increased during this 7-year period, from 103 to 153 individual mandatory compounds and 9 voluntary compounds. The number of spiked or incurred pesticides contained in the Test Items has been between 16 and 23. 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. Of the laboratories submitting results, 25 % submitted results for less than 70% of pesticides present in the Test Item.

Table 16. Overall trends in participation of laboratories, pesticides in the target list and test item, and performance of laboratories in the 7 latest EUPTs cereals and feed.

PT and type of test item	EUPT-C5 Rice flour	EUPT-C6 Barley flour	EUPT-C7 Feed[1)]	EUPT-CF8 Wheat flour	EUPT-CF9 Maize flour[2)]	EUPT-CF10 Rye flour	EUPT-CF11 Oat flour
Participants submitting results (EU and EFTA)	133	127	106	142	143	160	149
MRM pesticides in the Target Pesticide List	103	107	116	111	117	134/7	153/9
MRM pesticides in the test material	16	18	23	17	18	16	18
No. of results for MRM pesticides	1521	1741	1932	1893	2012	2012	2172
Average of 'reported results', %	71	76	83	78	78	79	83
Range of 'reported results', %	41-95	50-95	60-97	49-93	61-94	58-90	65-93
Acceptable z scores, %	87	90	90	90	89	95	89
Questionable z scores, %	4	5	4	6	2	2	3
Unacceptable z scores, %	9	4	5	4	3	2	8
False negatives, %	3	1	2	3	2	2	4
Number of false positives	16	2	9	4	9	0	19
Category A, % of participating laboratories	44	55	58	50	57	53	45
Good SWZ/AZ^2, %	80	79	82	85	96	93	92
Satisfactory SWZ/AZ^2, %	15	14	8	10	1	5	1.5
Unsatisfactory SWZ/AZ^2, %	5	7	10	4	3	2	6.2

[1)] Feed for laying hens.
[2)] Feed variety

The test material in EUPT-CF11 was relatively difficult to analyse due to the high fat content in oat combined with the storage at room temperature (see **Chapter 3.3**) and the analytical scope was relative high compared to the previous PTs. Consequently, the overall analytical performance (accuracy of measurement) has decreased a bit if looking at the percentage of acceptable, questionable and unacceptable z scores. The average % of reported results has been between 71-83%. This was because a lot of participants analysed for less than 50% of the pesticide residues present in the test Item. The number of false negative results has been very high (4%) and also the number of false positives has been high. Again this was most likely due to the difficult matrix.

The percentage of Category A laboratories has varied during the years and for the last 7 EUPTs, no improvement can be seen. Due to the many false positive in EUPT-CF11 only 45% of the laboratories were evaluated as Category A. For Category A labs an improvement in AZ^2 was seen in EUPT-CF9 where 96% of the results were good, and in EUPT-CF10 still 93% and EUPT-CF11 92% 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 numbers of pesticides in the Test item as well as the number of laboratories participating in the PTs have increased significantly. Furthermore, the matrix varies and thereby the performance can change.

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

The EUPT-CF11 Test Item consisted of oat flour containing incurred and spiked pesticides. The oat crop had been sprayed in the field with commercially available pesticide formulations. The final Test Item contained the following pesticides: azoxystrobin, carbendazim, chlordane-trans, chlorpyrifos, cyproconazole, cyprodinil, endosulfan, alpha-, fenbuconazole, fenpropimorph, fenvalerate, fludioxonil, flusilazole, flutolanil, lambda-cyhalothrin, metconazole, prothiofos, pyraclostrobin and tebuconazole. One hundred forty-five laboratories, representing 31 EU and EFTA countries submitted results. Four more laboratories registered, but did not submit any results. All NRLs, except one, participated in the PT. Malta was represented in the PT by the NRL for the UK. An additional 22 laboratories from EU candidate states and Third Countries registered for the PT and 20 submitted results. The Target Pesticide List distributed to the laboratories prior to the test contained 153 individual compulsory and 9 voluntary compounds..

The number of false positives and false negatives has varied between the EUPTs. However, due to the relatively difficult matrix (oat partially stored at room temperature) many false negatives and false positives was seen. The number of false negatives represented 5% of the total number of results and 19 false positive results were reported. The average Alg A-RSD was at 17%, well below the FFP-RSD of 25% with a range from 14-23% for the individual compounds.

For azoxystrobin, chlorpyrifos, cyprodinil, fenbuconazole, flutolanil, metconazole pyraclostrobin and tebuconazole acceptable results were obtained by 90-95% of the laboratories. For carbendazim, chlordane-trans, cyproconazole, endosulfan, alpha-, fenpropimorph, fenvalerate, fludioxonil, flusilazole, lambda-cyhalothrin, and prothiofos acceptable results were obtained by 81-89% of the laboratories.

The EUPT-CF12 will be a feed commodity, hay, as test item, which will be shipped to the laboratories in January 2018. 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

The Organisers wish to thank the members of the EURL Quality Control and Advisory Groups for their valuable advice.

5. REFERENCES

[1] Regulation (EC) No 882 /2004 of the European Parliament and of the Council on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules. Published at OJ of the EU L191 of 28.05.2004

[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

[7] Guidance document on analytical quality control and validation procedures for pesticide residues analysis in food and feed. SANTE 11945/2015.
http://www.crl-pesticides.eu/docs/public/tmplt_article.asp?CntID=727&LabID=100&Lang=EN

APPENDICES

Appendix 1 List of laboratories registered to participate in the EUPT-CF11

Participating labs from EU and EFTA member states

Country	Institution	City	NRL-CF	Report data
Austria	Austrian Agency for Health and Food Safety, Institute for Food Safety Innsbruck - Department for Pesticide and Food Analytics	Innsbruck	NRL	Yes
Austria	MA 38 - LUA	Vienna		Yes
Belgium	Scientific Institute of Public Health	Brussels	NRL	Yes
Belgium	PRIMORIS - Belgium, Gent (Zwijnaarde)	Gent - Zwijnaarde		Yes
Belgium	Federal Laboratory for Safety of Food Chain, Tervuren	Tervuren		Yes
Bulgaria	Fytolab - Bulgaria, Plovdiv	Plovdiv		Yes
Bulgaria	Central Laboratory for Chemical Testing and Control, Sofia	Sofia	NRL	Yes
Croatia	Bioinstitut d.o.o., Cakovec	Cakovec		Yes
Croatia	Institute of Public Health, Primorje-Gorski	Kotar County, Rijeka		Yes
Croatia	INSPECTO d.o.o. Laboratorij - Pesticide Lab	Osijek		Yes
Croatia	Croatian Veterinary Institute - Zagreb	ZAGREB	NRL	No
Croatia	Faculty of Food Technology and Biotechnology, Food Control Center - Croatia, Zagreb	Zagreb		Yes
Croatia	Euroinspekt - Croatiakontrola d.o.o.	Zagreb		Yes
Croatia	Institute of Public Health, Dr. Andrija štampar	Zagreb		Yes
Croatia	Croatian National Institute of Public Health	Zagreb	NRL	Yes
Cyprus	Animal Feeds and Feed Additives Laboratory of the Analytical Laboratories of the Department of Agriculture	Nicosia	NRL	Yes
Cyprus	Laboratory of Pesticide Residues Analysis, State General Laboratory	Nicosia	NRL	Yes
Czech Republic	Central Institute for Supervising and Testing in Agriculture1)	Brno	NRL	Yes
Czech Republic	University of Chemical Technology, Dept. of Food Chemistry and Analysis - Prague	Praha		Yes
Czech Republic	Czech Agriculture and Food Inspection Authority	Praha	NRL	Yes
Denmark	Danish Veterinary and Food Administration, Department of Residues, Ringsted	Ringsted	NRL	Yes
Estonia	Agricultural Research Centre, Saku, Lab for Residues and Contaminants	Saku	NRL	Yes
Finland	Finnish Customs Laboratory	Espoo	NRL	Yes
Finland	Finnish Food Safety Authority - Helsinki	Helsinki	NRL	Yes
France	GIRPA - FREDON Pays de la Loire	Beaucouzé		Yes
France	CERECO SUD	GARONS		Yes
France	Capinov	Landerneau		Yes
France	INOVALYS Le Mans	Le Mans		Yes
France	Service Commun des Laboratoires / Laboratoire Ile de France - Massy	Massy Cedex	NRL	Yes
France	SCL (Montpellier)	Montpellier		Yes
France	Phytocontrol - France, Nimes	Nimes		Yes

Country	Institution	City	NRL-CF	Report data
France	CAMP Méditerrannée (Perpignan)	PERPIGNAN		Yes
Germany	ILAU GmbH	Anzing		No
Germany	Federal Office of Consumer Protection and Food Safety, NRL for Pesticide Residues	Berlin	NRL	Yes
Germany	State Investigation Institute of Health and Veterinary Saxony	Dresden		Yes
Germany	Amt für Verbraucherschutz Düsseldorf - 39/2 Chemische und Lebensmitteluntersuchung	Düsseldorf		Yes
Germany	Bavarian Health and Food Safety Authority Office Erlangen	Erlangen		Yes
Germany	Chemical and Veterinary Investigations Office, Stuttgart (CVUAS), (Residues)	Fellbach		Yes
Germany	Landesamt für Verbraucherschutz - Sachsen-Anhalt	Halle/Saale		Yes
Germany	Landesanstalt für Landwirtschaft, Forsten und Gartenbau, Halle	Halle/Saale		Yes
Germany	Eurofins - Dr. Specht Laboratorien GmbH	Hamburg		Yes
Germany	Thuringian Institute of Agriculture	Jena		Yes
Germany	Landwirtschaftliches Technologiezentrum Augustenberg, Karlsruhe	Karlsruhe		Yes
Germany	LUFA-ITL GmbH	Kiel		Yes
Germany	Chemical and Veterinary Analytical Institute Rhine-Ruhr-Wupper	Krefeld		Yes
Germany	State Laboratory Schleswig-Holstein	Neumünster		Yes
Germany	State Department of Environmental and Agricultural Operations in Saxony	Nossen		Yes
Germany	Food and Veterinary Institute Oldenburg	Oldenburg		Yes
Germany	Berlin-Brandenburg State Laboratory, Potsdam	Potsdam		Yes
Germany	Landesamt für Landwirtschaft, Lebensmittelsicherheit und Fischerei Mecklenburg-Vorpommern	Rostock		Yes
Germany	Landwirtschaftliche Untersuchungs- und Forschungsanstalt Speyer	Speyer		Yes
Germany	Landesuntersuchungsamt Institut für Lebensmittelchemie Speyer	Speyer		Yes
Germany	Niedersächsisches Landesamt für Verbraucherschutz und Lebensmittelsicherheit, Stade	Stade		Yes
Germany	Landesbetrieb Hessisches Landeslabor, Wiesbaden	Wiesbaden		Yes
Greece	General Chemical State Laboratory, D Division, Pesticide Residues Laboratory	Athens		Yes
Greece	Benaki Phytopathological Institute, Pesticide Residues Laboratory	Kifissia	NRL	Yes
Greece	Regional Center of Plant Protection and Quality Control of Thessaloniki, Pesticide Residues Laboratory	Thessaloniki		Yes
Hungary	National Food Chain Safety Office, Directorate of Plant Protection, Soil Conservation and Agri-Environment, Pesticide Residue Analytical Laboratory, Hódmezovásárhely	Hódmezovásárhely		Yes
Hungary	National Food Chain Safety Office, Directorate of Plant Protection, Soil Conservation and Agri-Environment, Pesticide Residue Analytical Laboratory, Miskolc	Miskolc		Yes
Hungary	National Food Chain Safety Office Directorate of Plant Protection, Soil Conservation and Agri-environment, Pesticide Residue Analytical Laboratory, Szolnok	Szolnok		Yes
Hungary	National Food Chain Safety Office, Directorate of Plant Protection, Soil Conservation and Agri-environment - Pesticide	Velence	NRL	Yes

Country	Institution	City	NRL-CF	Report data
	Analytical Laboratory, Velence			
Iceland	Matís - Icelandic Food Research	Reykjavík		Yes
Ireland	Pesticide Control Laboratory, Department of Agriculture, Fisheries and Food	Co. Kildare	NRL	Yes
Italy	ARPA Puglia - Dipartimento di Bari	Bari		Yes
Italy	Laboratorio di Sanità Pubblica ASL BERGAMO	Bergamo		Yes
Italy	APPA Bolzano	Bolzano		Yes
Italy	Istituto Zooprofilattico Sperimentale Lombardia ed Emilia Romagna	Brescia		Yes
Italy	ARPA Ferrara Eccellenza Fitofarmaci	Ferrara		Yes
Italy	Public Health Laboratory - Florence	Firenze		Yes
Italy	Istituto Zooprofilattico Sperimentale Puglia e Basilicata	Foggia		Yes
Italy	Centro di referenza nazionale per la sorveglianza ed il controllo degli alimenti per animali	Genova	NRL	Yes
Italy	ARPAL Sez. di La Spezia	La Spezia		Yes
Italy	ARPALAZIO SEZIONE P.LE DI LATINA - Servizio Laboratorio Ambiente e Salute, Unita di Chemica Inorganica	Latina		Yes
Italy	Istituto Zooprofilattico Sperimentale delle Venezie	Legnaro (Padova)		Yes
Italy	Agenzia della Tutela della Salute (ATS) della Città Metropolitana di Milano	Milano		Yes
Italy	Istituto Zooprofilattico Sperimentale Umbria e Marche, PERUGIA	Perugia		Yes
Italy	ARPA FVG Settore Laboratorio Unico - Laboratorio di Pordenone	Pordenone		Yes
Italy	Istituto Zooprofilattico Sperimentale Lazio e Toscana - Roma	Roma		Yes
Italy	Istituto Superiore di Sanità, Pesticide Section	Roma		No
Italy	Environement protection regional agency Aosta Valley	Saint Christophe		Yes
Italy	IZS LT (sezione Firenze)	San Martino alla Palma Scandicci (FI)		Yes
Italy	Istituto Zooprofilattico Sperimentale Sardegna	Sassari		Yes
Italy	Istituto Zooprofilattico Sperimentale Abruzzo e Molise	Teramo		Yes
Italy	APPA Trento Settore Laboratorio e Controlli	Trento		Yes
Italy	ARPA Veneto Dip.reg.lab, S.L. Verona	Verona		Yes
Latvia	Institute of Food Safety, Animal Health and Environment (BIOR) - Riga	Riga	NRL	Yes
Lithuania	National Food and Veterinary Risk Assessment Institute (Lithuania, Vilnius)	Vilnius	NRL	Yes
Luxembourg	National Health Laboratory Luxembourg (Food Laboratory)	Dudelange	NRL	Yes
Netherlands	Eurofins Lab Zeeuws-Vlaanderen (LZV) B.V.	Graauw		Yes
Netherlands	Handelslaboratorium Dr. Verwey	Rotterdam		Yes
Netherlands	RIKILT – Wageningen UR (Natural Toxins & Pesticides)	Wageningen		Yes
Netherlands	NVWA - Netherlands Food and Consumer Product Safety Authority (Wageningen, The Netherlands)	Wageningen	NRL	Yes
Norway	National Institute of Nutrition and Seafood Research - Norway, Bergen	Bergen		No
Norway	Norwegian Institute of Bioeconomy Research, Dep. of Pesticide Chemistry and Natural Bioactive Compounds	Ås	NRL	Yes

Country	Institution	City	NRL-CF	Report data
Poland	Regional Veterinary Laboratory in Bialystok	Bialystok		Yes
Poland	Institute of Plant Protection Pesticide Residue Laboratory, Bialystok	Bialystok		Yes
Poland	Regional Veterinary Laboratory in Gdansk (Kartuska)	Gdansk		Yes
Poland	J.S. Hamilton Poland S.A.	Gdynia		Yes
Poland	Regional Veterinary Laboratory in Katowice	Katowice		Yes
Poland	Wojewódzka Stacja Sanitarno-Epidemiologiczna w Opolu, Oddział Laboratoryjny w Kluczborku	Opole		Yes
Poland	Voievodship Sanitary - Epidemiological Station in Opole	Opole		Yes
Poland	Institute of Plant Protection, Department of Pesticide Residue Research - Poznan	Poznan	NRL	Yes
Poland	Provincial Veterinary Inspectorate Establishment of Veterinary Hygiene, Poznan	Poznan		Yes
Poland	Institute of Horticulture, Food Safety Laboratory (Skierniewice)	Skierniewice		Yes
Poland	Institute of Plant Protection - National Research Institute, Branch Sosnicowice	Sosnicowice		Yes
Poland	Regional Veterinary Laboratory in Szczecin	Szczecin		Yes
Poland	Voivodeship Sanitary-Epidemiological Station in Warsaw	Warsaw	NRL	Yes
Poland	Regional Veterinary Laboratory in Warsaw	Warszaw		Yes
Poland	Regional Veterinary Laboratory Wroclaw	Wroclaw		Yes
Portugal	Regional Laboratory of Veterinary and Food Safety - Madeira Island	Funchal - Madeira Island		Yes
Portugal	INIAV- Pesticide Residues Laboratory	Oeiras	NRL	Yes
Portugal	Vairão - Pesticide Lab (Plant Origin Products)	Vairão - Vila do Conde		Yes
Romania	Zonal Laboratory for pesticides Residues in feed - Bistrita	Bistrita		Yes
Romania	Institute for Hygiene and Veterinary Public Health - Bucharest	Bucharest		Yes
Romania	Central Laboratory for Pesticides Residues Control in Plants and Vegetable Products - Bucharest	Bucharest	NRL	Yes
Romania	Sanitary Veterinary and Food Safety Directorate, Bucharest	Bucharest		Yes
Romania	Sanitary Veterinary and Food Safety Directorate Cluj, Gas-Chromatography Laboratory	Cluj Napoca		Yes
Romania	Regional Laboratory for Pesticide Residues Control in Plant and Plant Products Mures	Targu Mures, jud. Mures		Yes
Slovakia	State Veterinary and Food Institute - Veterinary and Food Institute in Bratislava	Bratislava	NRL	Yes
Slovakia	Public Health Authority of the Slovak Republic	Bratislava		Yes
Slovenia	National Laboratory for Health, Environment and Food - Maribor (Location Ljubljana)	Ljubljana		Yes
Slovenia	National Laboratory of Health, Environment and Foodstuffs - Maribor	Maribor	NRL	Yes
Spain	Agricultural and Phytopathological Laboratory of Galicia	Abegondo. A Coruña		Yes
Spain	Instituto Tecnologico de Canarias, División de Investigación y Desarrollo Tecnológico - Laboratorio de Residuos	Agüimes, Gran Canaria		Yes
Spain	Analytica Alimentaria GmbH Sucursal España	Almeria		Yes
Spain	Laboratorio de Salud Pública de Badajoz	Badajoz		Yes

Country	Institution	City	NRL-CF	Report data
Spain	Laboratory of Barcelona Public Health Agency	Barcelona		Yes
Spain	Laboratorio Agrario Regional - Junta de Castilla y Leon	Burgos		Yes
Spain	Labs & Technological Services AGQ, S.L. - Spain, Burguillos	Burguillos		Yes
Spain	Laboratori Agroalimentari de la Generalitat de Catalunya	Cabrils		Yes
Spain	Laboratorio Agroalimentario de Extremadura (Cáceres)	CÁCERES		Yes
Spain	Laboratorio de Salud Pública de Cuenca	Cuenca		Yes
Spain	Laboratorio Regional CCAA La Rioja	Logroño		Yes
Spain	Laboratorios Ecosur, S.A.L.	Lorquí (Murcia)		Yes
Spain	Laboratorio Arbitral Agroalimentario, Madrid	Madrid	NRL	Yes
Spain	Laboratorio de Salud Pública Madrid Salud Ayuntamiento de Madrid	Madrid		Yes
Spain	National Centre for Food - Spain, Majadahonda	Majadahonda (Madrid)	NRL	Yes
Spain	Laboratorio KUDAM S.L.	Pilar de la Horadada (Alicante)		Yes
Spain	National Centre for Technology and Food Safety (CNTA) - Spain, San Adrián	San Adrián (Navarra)		Yes
Spain	Servicio de Laboratorio y Control de Santander	Santander		Yes
Spain	Laboratorio Regional Agroalimentario y Ambiental de Castilla la Mancha (LARAGA), Toledo	Toledo		Yes
Spain	Agrofood Laboratory of the Comunidad Valenciana	Valencia		Yes
Spain	Navarra de Servicios y Tecnologias, S.A.	Villava		Yes
Spain	Laboratorio Agroalimentario de Zaragoza	Zaragoza		Yes
Sweden	Eurofins - Food&Agro Sweden, Lidköping	Lidköping		Yes
Sweden	National Food Agency, Science Department, Chemistry Division 1	Uppsala	NRL	Yes
Switzerland	Kantonales Laboratorium Zürich	Zürich		Yes
United Kingdom	Agri-Food and Biosciences Institute	Belfast		Yes
United Kingdom	The Food and Environment Research Agency - York	York	NRL	Yes

Participating labs from EU candidate state and other non EU countries

Country	Institution	City	Reported data
Argentina	Bolsa de Comercio de Rosario	Rosario	Yes
Australia	Symbio Laboratories Pty Ltd	Eight Mile Plains QLD	Yes
Brazil	Laboratorio Nacional Agropecário em Minas Gerais	PEDRO LEOPOLDO/MG	Yes
Brazil	Bioagri Análise de Alimentos LTDA - MXNS Brazil	São Paulo/SP	No
China	Shanghai Municipal Center For Disease Control and Prevention	Shanghai	Yes
Egypt	Central Lab of Residue Analysis of Pesticides and Heavy Metals in Foods	Giza	Yes
FYR Macedonia	Faculty of Veterinary Medicine, Food Institue - Skopje	Skopje	Yes
India	Pesticide Formulation and Residue Analytical Center	hyderabad	Yes
India	SGS India Pvt Ltd Chennai	Chennai	Yes
India	All India Network Project on Pesticide Residues	Hyderabad	Yes
India	TUV SUD South Asia PVT PVT LTD - Gurugram Lab	GURUGRAM	Yes
India	TUV SUD South Asia PVT LTD - Bangalore	Bangalore	Yes
India	CSIR-Indian Institute of Toxicology Research	LUCKNOW	No
Indonesia	Angler Biochemlab, PT.	SURABAYA	Yes
Mexico	Centro Nacional de Referencia de Plaguicidas y Contaminantes	TECAMAC, ESTADO DE MEXICO	Yes
Serbia	Center for Food Analysis, Belgrade	Belgrade	Yes
Serbia	SP LABORATORY	BECEJ	Yes
Serbia	MIPHEM d.o.o.	Belgrade	Yes
Serbia	Institute of Public Health of Belgrade	Belgrade	Yes
Singapore	Veterinary Public Health Laboratory	Singapore	Yes
Thailand	Central laboratory (Thailand) Co., Ltd.	Bangkok	Yes
Ukraine	Ukrainian Laboratory of Quality and Safety of Agricultural Products National University of Life and Environmental Sciences of Ukraine	Chabany village	Yes

Appendix 2 Target Pesticide List

Pesticides	MRRL (mg/kg)
Compulsory Compounds considered in Category A/B classification)	
2-phenylphenol	0.01
Acephate	0.01
Acetamiprid	0.01
Acrinathrin	0.01
Aldrin	0.01
Azinphos-methyl	0.01
Azoxystrobin	0.01
Bifenthrin	0.01
Biphenyl	0.01
Bixafen	0.01
Boscalid	0.01
Bromuconazole	0.01
Buprofezin	0.01
Cadusafos	0.01
Carbaryl	0.01
Carbendazim	0.01
Carbofuran	0.01
Carbofuran, 3-hydroxy-	0.01
Carboxin	0.01
Chlorantraniliprole	0.01
Chlorfenapyr	0.01
Chlorfenvinphos	0.01
Chlorpropham (parent compound only)	0.01
Chlorpyrifos	0.01
Chlorpyrifos-methyl	0.01
Clothianidin	0.01
Cyfluthrin (sum of isomers)	0.01
Cymoxanil	0.01
Cypermethrin (sum of isomers)	0.01
Cyproconazole	0.01
Cyprodinil	0.01
DDD, p,p'-	0.01
DDE, p,p'-	0.01
DDT, o,p'-	0.01
DDT, p,p'-	0.01
Deltamethrin, cis-	0.01
Demeton-S-methylsulfone	0.01
Diazinon	0.01
Dichlorvos	0.01
Dieldrin	0.01

Pesticides	MRRL (mg/kg)
Difenoconazole	0.01
Diflubenzuron	0.01
Dimethoate	0.01
Dimethomorph	0.01
Diniconazole	0.01
Endosulfan, alpha-	0.01
Endosulfan, beta-	0.01
Endosulfan-sulfate	0.01
Epoxiconazole	0.01
Ethion	0.01
Ethirimol	0.01
Ethoprophos	0.01
Famoxadone	0.01
Fenbuconazole	0.01
Fenhexamid	0.01
Fenitrothion	0.01
Fenpropathrin	0.01
Fenpropidin	0.01
Fenpropimorph	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 and Esfenvalerate (Sum of RR/SS and RS/SR isomers)	0.01
Fipronil (parent compound only)	0.01
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
Hexaconazole	0.01
Imazalil	0.01
Imidacloprid	0.01
Indoxacarb	0.01

Pesticides	MRRL (mg/kg)
Iprodione	0.01
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	0.01
Methacrifos	0.01
Methamidophos	0.01
Methomyl	0.01
Metolachlor	0.01
Metrafenone	0.01
Metribuzin	0.01
Omethoate	0.01
Oxydemeton-methyl	0.01
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	0.01
Propiconazole	0.01
Prothioconazole-desthio	0.01
Prothiofos	0.01
Pyraclostrobin	0.01
Pyridaben	0.01
Pyrimethanil	0.01
Pyriproxyfen	0.01
Quinoxyfen	0.01

Pesticides	MRRL (mg/kg)
Spirodiclofen	0.01
Spiromesifen	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	0.01
Tricyclazole	0.01
Trifloxystrobin	0.01
Trifluralin	0.01
Triticonazole	0.01
Vinclozolin (parent compound only)	0.01
Zoxamide	0.01
Voluntary Compounds	
Chlordane, cis-	0.01
Chlordane, oxy-	0.01
Chlordane, trans-	0.01
Endrin	0.01
HCH, alpha-	0.01
HCH, beta-	0.01
Heptachlor	0.01
Heptachlorepoxid, cis-	0.01
Heptachlorepoxid, trans-	0.01

Only individual compounds are included in the pesticide target list. except for pyrethroids where the sum of isomers should be reported, unless other is specified in the list.

Appendix 3 Homogeneity data

	Azoxystrobin, mg/kg		Carbendazim, mg/kg		Chlordane, trans, mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
12	0.098	0.110	0.245	0.257	0.032	0.034
55	0.109	0.091	0.242	0.276	0.034	0.032
68	0.103	0.097	0.284	0.265	0.034	0.033
80	0.102	0.101	0.254	0.276	0.031	0.033
101	0.101	0.100	0.267	0.236	0.038	0.034
120	0.097	0.100	0.239	0.235	0.034	0.036
140	0.106	0.095	0.279	0.270	0.035	0.035
160	0.100	0.103	0.258	0.245	0.035	0.038
180	0.109	0.100	0.289	0.258	0.039	0.036
200	0.109	0.101	0.240	0.264	0.032	0.034
220	0.115	0.107	0.302	0.283	0.036	0.037

	Chlorpyrifos, mg/kg		Cyproconazole, mg/kg		Cyprodinil, mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
12	0.528	0.496	0.257	0.244	0.068	0.080
55	0.522	0.511	0.247	0.248	0.067	0.078
68	0.535	0.512	0.263	0.255	0.078	0.078
80	0.499	0.500	0.260	0.268	0.071	0.078
101	0.538	0.527	0.273	0.261	0.077	0.072
120	0.553	0.533	0.261	0.266	0.070	0.069
140	0.559	0.494	0.282	0.258	0.079	0.077
160	0.536	0.563	0.263	0.276	0.075	0.071
180	0.584	0.552	0.281	0.269	0.081	0.079
200	0.560	0.552	0.274	0.272	0.072	0.077
220	0.585	0.562	0.300	0.276	0.085	0.086

	Endosulfan, alpha-, mg/kg		Fenbuconazole, mg/kg		Fenpropimorph, mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
12	0.050	0.049	0.283	0.299	0.084	0.081
55	0.049	0.055	0.301	0.269	0.082	0.084
68	0.057	0.059	0.294	0.277	0.088	0.083
80	0.051	0.058	0.282	0.284	0.082	0.085
101	0.050	0.051	0.289	0.283	0.089	0.084
120	0.056	0.053	0.285	0.284	0.088	0.085
140	0.054	0.059	0.300	0.273	0.088	0.085
160	0.052	0.052	0.285	0.299	0.087	0.090
180	0.057	0.052	0.312	0.288	0.091	0.088
200	0.056	0.059	0.296	0.289	0.090	0.090
220	0.050	0.053	0.321	0.299	0.092	0.090

	Fenvalerate, mg/kg		Fludioxonil, mg/kg		Flusilazole, mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
12	0.048	0.058	0.080	0.083	0.421	0.440
55	0.061	0.044	0.086	0.083	0.402	0.457
68	0.047	0.044	0.084	0.083	0.480	0.446
80	0.045	0.040	0.083	0.090	0.413	0.457
101	0.044	0.051	0.088	0.089	0.457	0.399
120	0.048	0.049	0.083	0.085	0.402	0.403
140	0.052	0.030	0.093	0.092	0.460	0.447
160	0.047	0.050	0.089	0.088	0.426	0.412
180	0.056	0.054	0.097	0.089	0.494	0.433
200	0.058	0.041	0.087	0.093	0.395	0.450
220	0.058	0.052	0.101	0.093	0.516	0.479

	Flutolanil, mg/kg		Lambda-cyhalothrin, mg/kg		Metconazole, mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
12	0.053	0.050	0.043	0.048	0.247	0.244
55	0.052	0.051	0.051	0.039	0.250	0.241
68	0.055	0.052	0.043	0.038	0.266	0.248
80	0.055	0.056	0.040	0.037	0.248	0.251
101	0.056	0.055	0.039	0.046	0.262	0.245
120	0.053	0.055	0.044	0.044	0.256	0.256
140	0.058	0.053	0.047	0.042	0.262	0.260
160	0.053	0.058	0.045	0.044	0.257	0.264
180	0.059	0.054	0.049	0.048	0.275	0.254
200	0.055	0.058	0.046	0.044	0.255	0.272
220	0.060	0.059	0.054	0.044	0.286	0.268

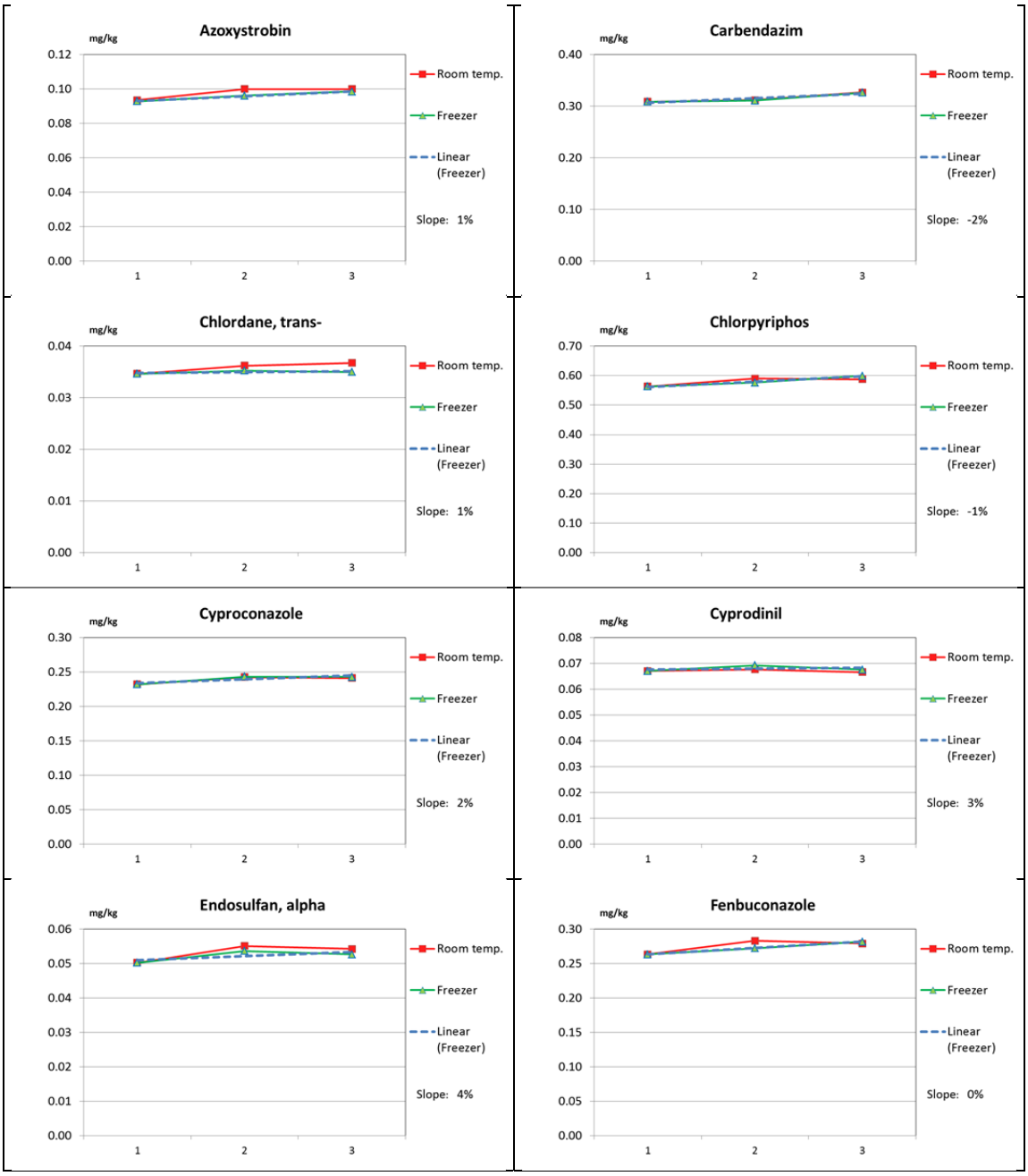
	Prothiofos, mg/kg		Pyraclostrobin, mg/kg		Tebuconazole, mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 1
12	0.040	0.042	0.326	0.353	0.750	0.725
55	0.039	0.040	0.331	0.363	0.740	0.721
68	0.040	0.040	0.381	0.352	0.787	0.742
80	0.040	0.043	0.327	0.360	0.760	0.785
101	0.043	0.042	0.375	0.326	0.794	0.752
120	0.046	0.039	0.336	0.315	0.775	0.769
140	0.046	0.040	0.371	0.360	0.811	0.799
160	0.042	0.045	0.348	0.327	0.785	0.812
180	0.047	0.043	0.386	0.357	0.827	0.778
200	0.046	0.043	0.321	0.353	0.792	0.823
220	0.045	0.048	0.396	0.402	0.873	0.806

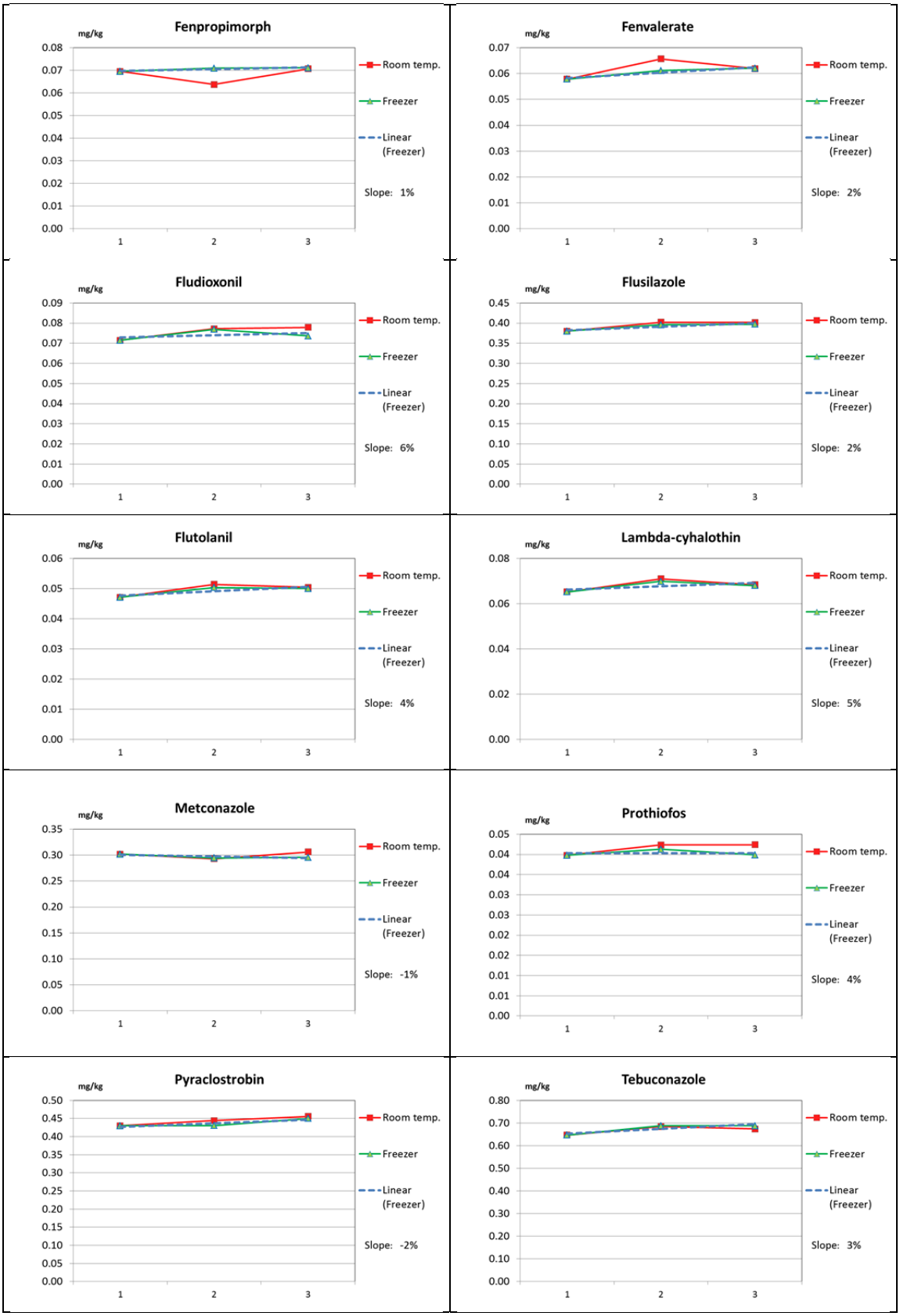
Appendix 4 Stability figures

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

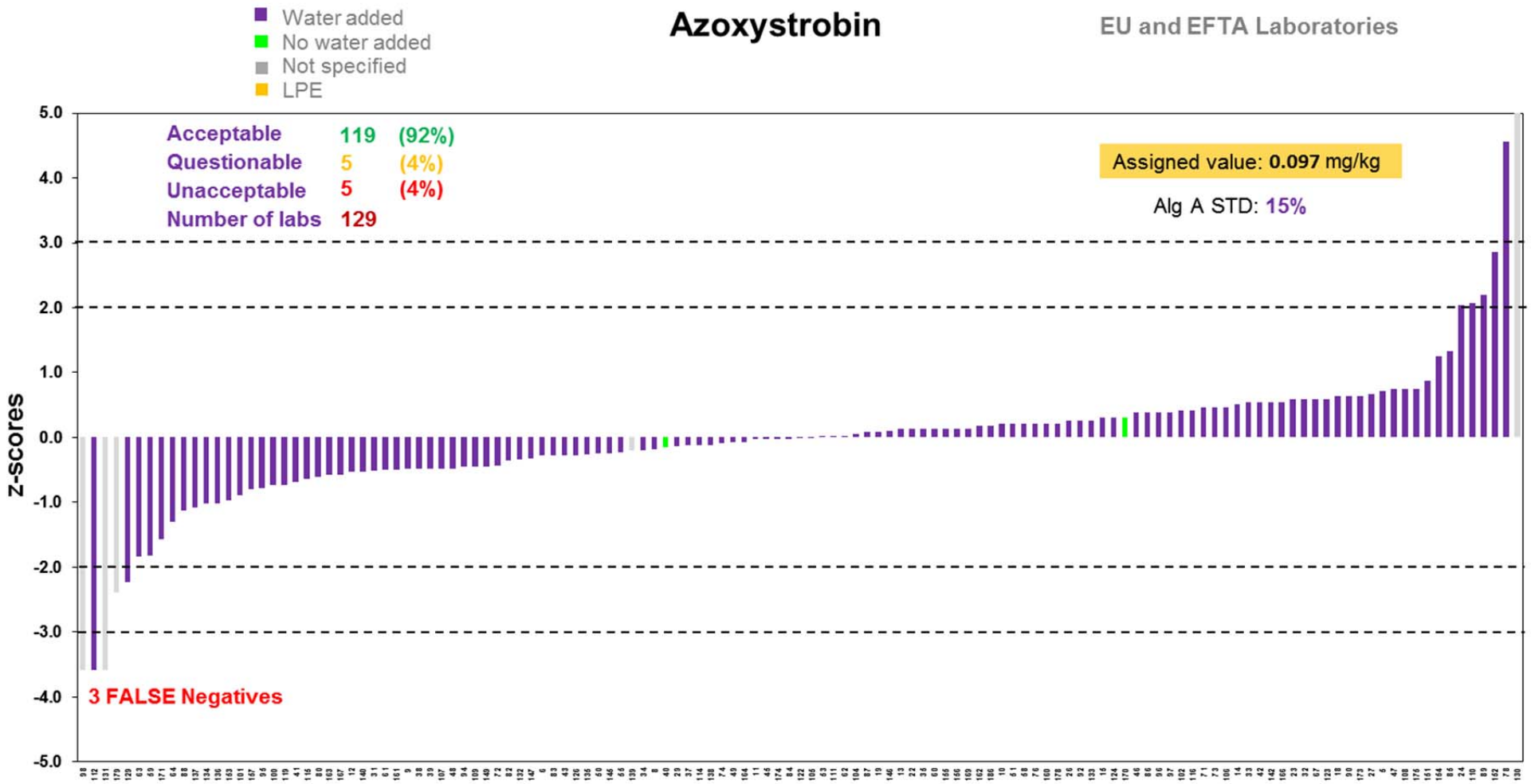
The dates of testing were as follows:
Day 1: 9 May 2017
Day 2: 23 May 2017
Day 3: 06 June 2017

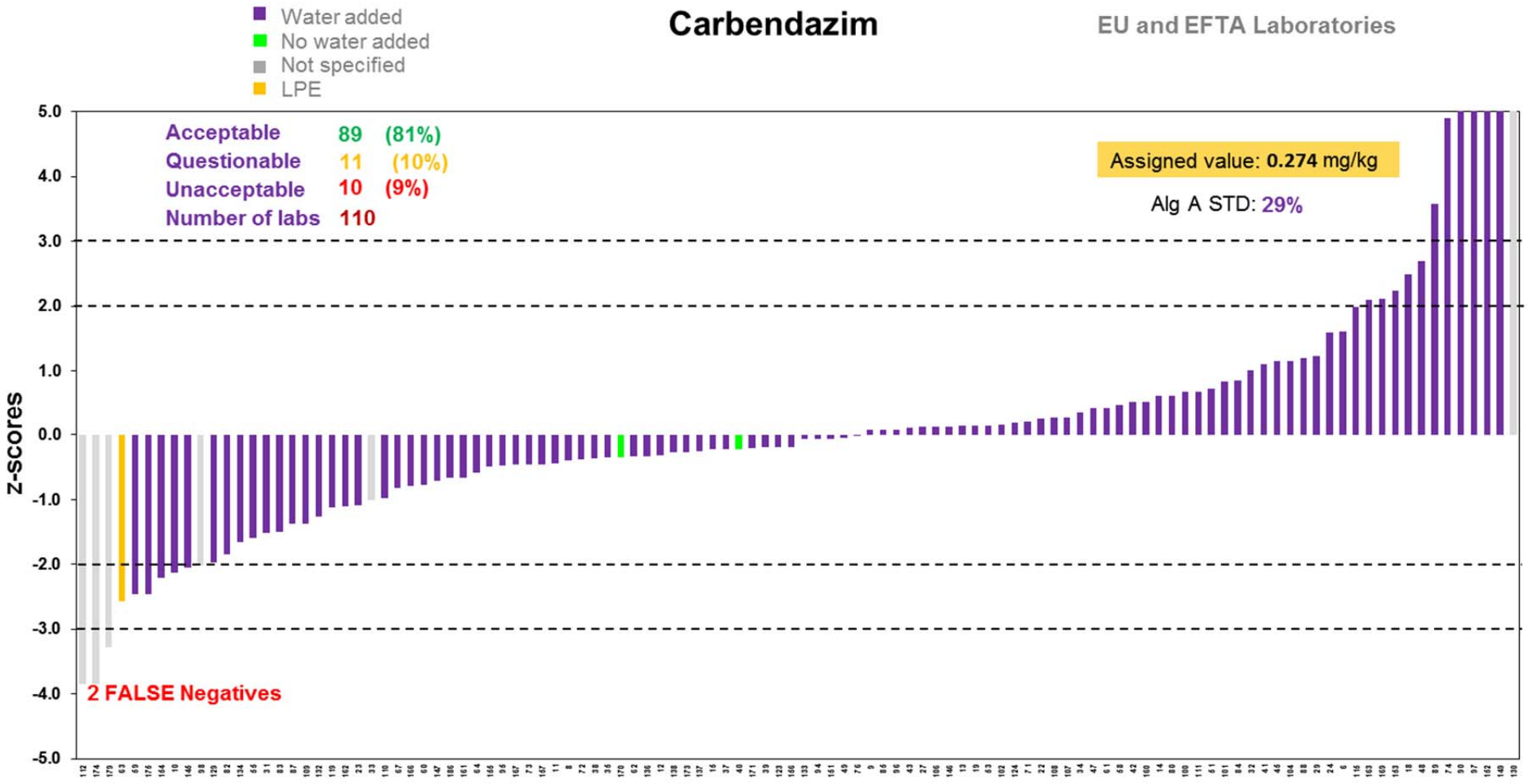
All pesticides passed the test at -18 °C as well as room temperature.

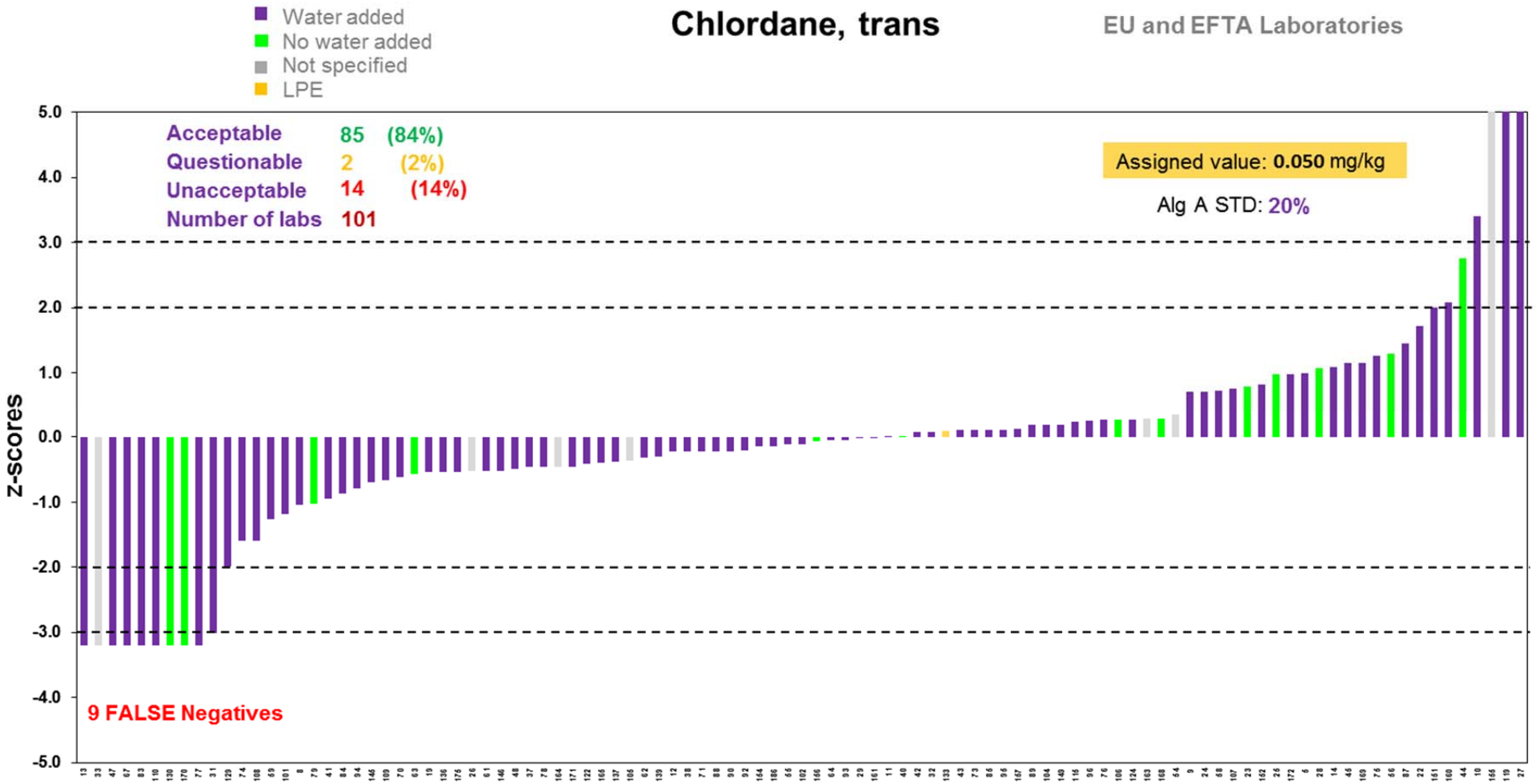


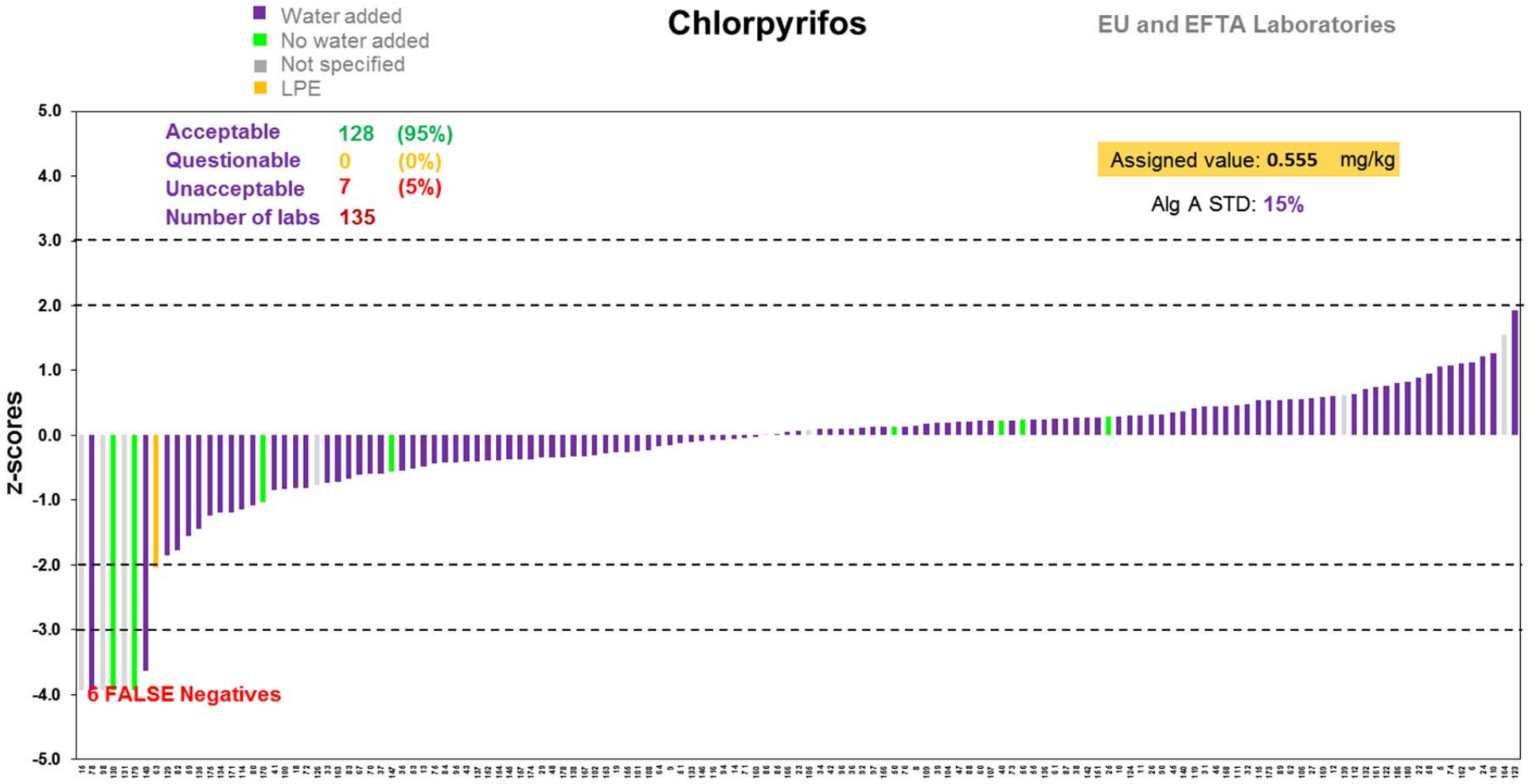

Fenpropimorph
mg/kg
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: 1%
Fenvalerate
mg/kg
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: 2%
Fludioxonil
mg/kg
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: 6%
Flusilazole
mg/kg
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: 2%
Flutolanil
mg/kg
0.06
0.05
0.04
0.03
0.02
0.01
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: 4%
Lambda-cyhalothin
mg/kg
0.08
0.06
0.04
0.02
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: 5%
Metconazole
mg/kg
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: -1%
Prothiofos
mg/kg
0.05
0.04
0.04
0.03
0.03
0.02
0.02
0.01
0.01
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: 4%
Pyraclostrobin
mg/kg
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: -2%
Tebuconazole
mg/kg
0.80
0.70
0.60
0.50
0.40
0.30
0.20
0.10
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: 3%

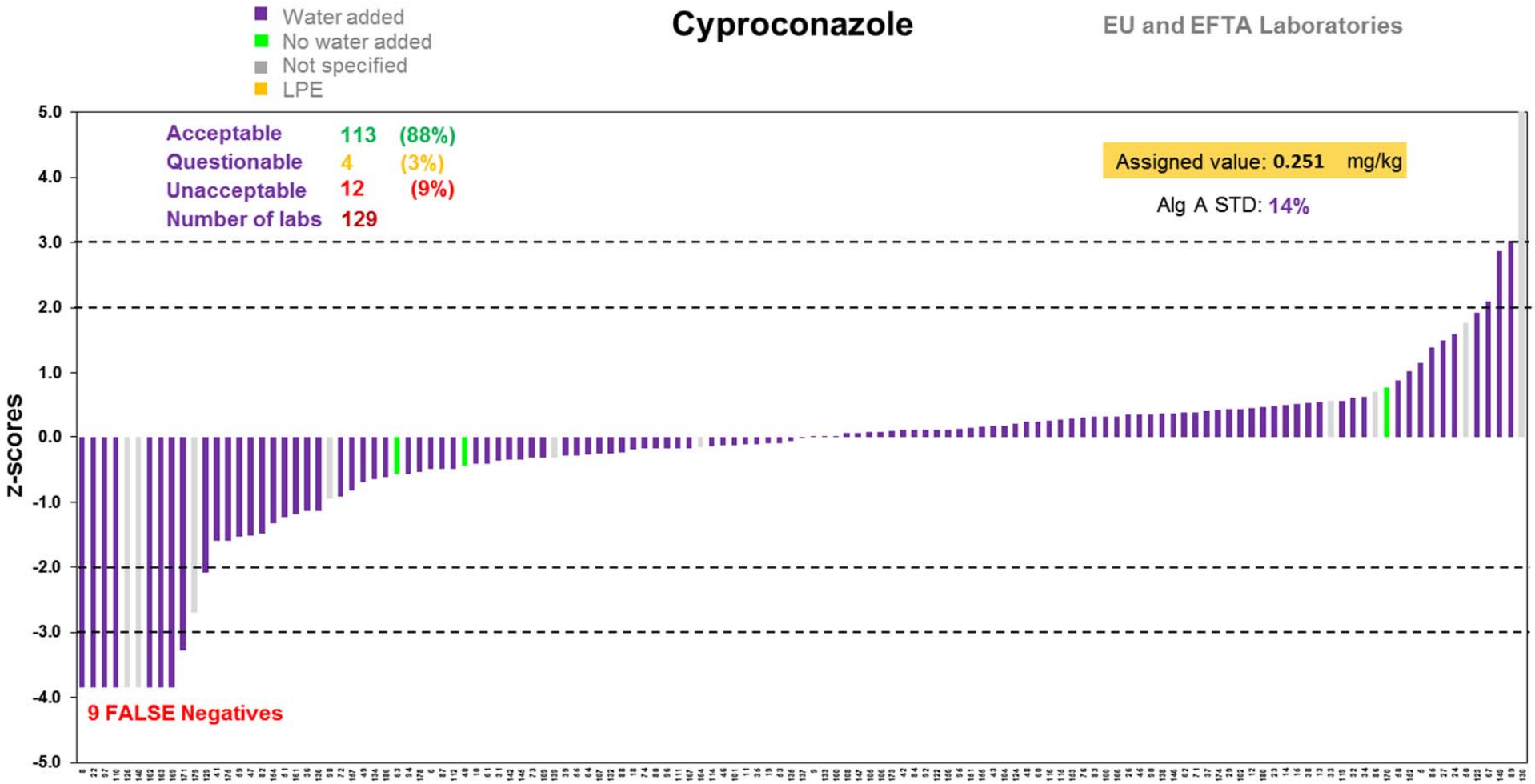
Appendix 5 Graphical presentation of z-scores

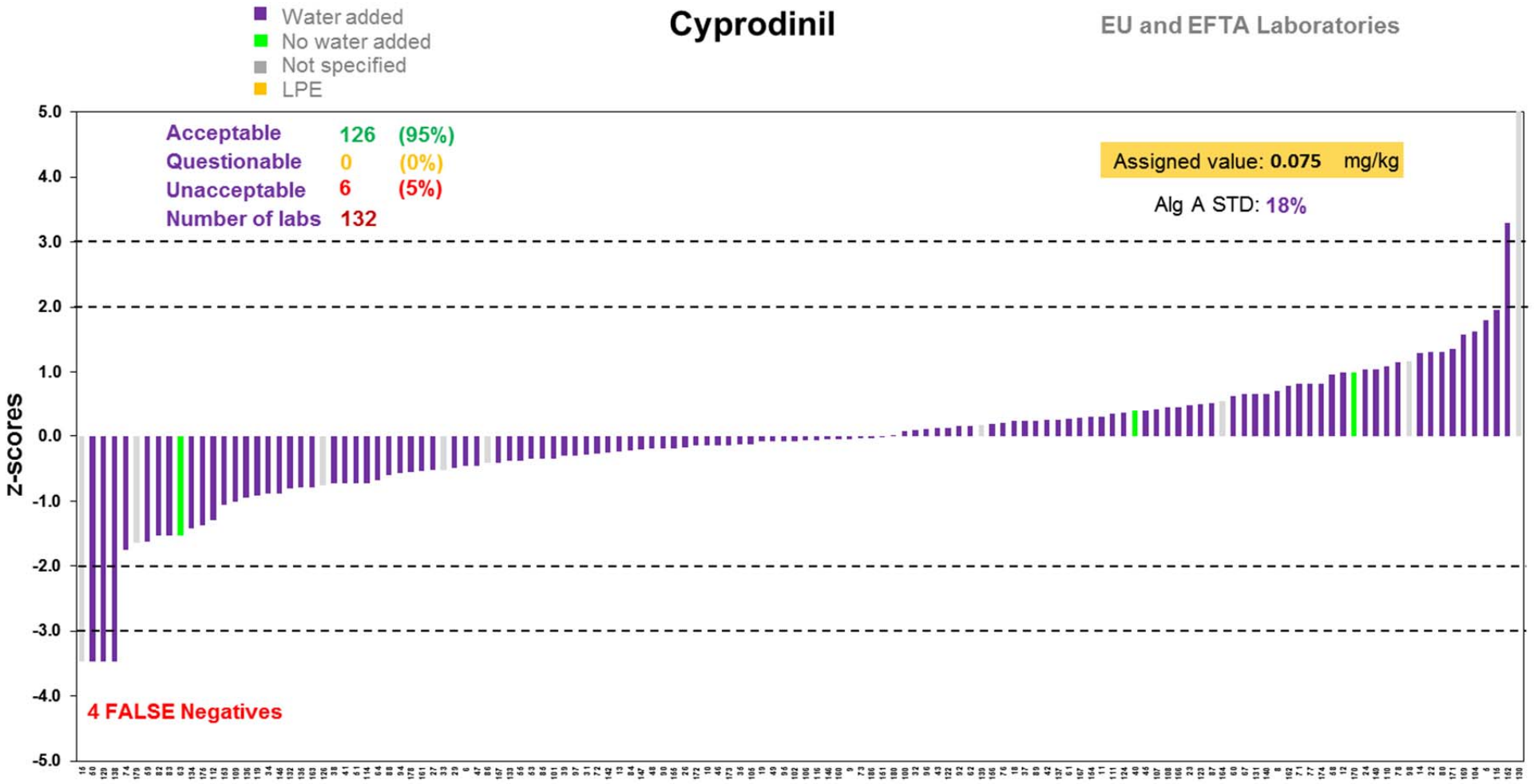


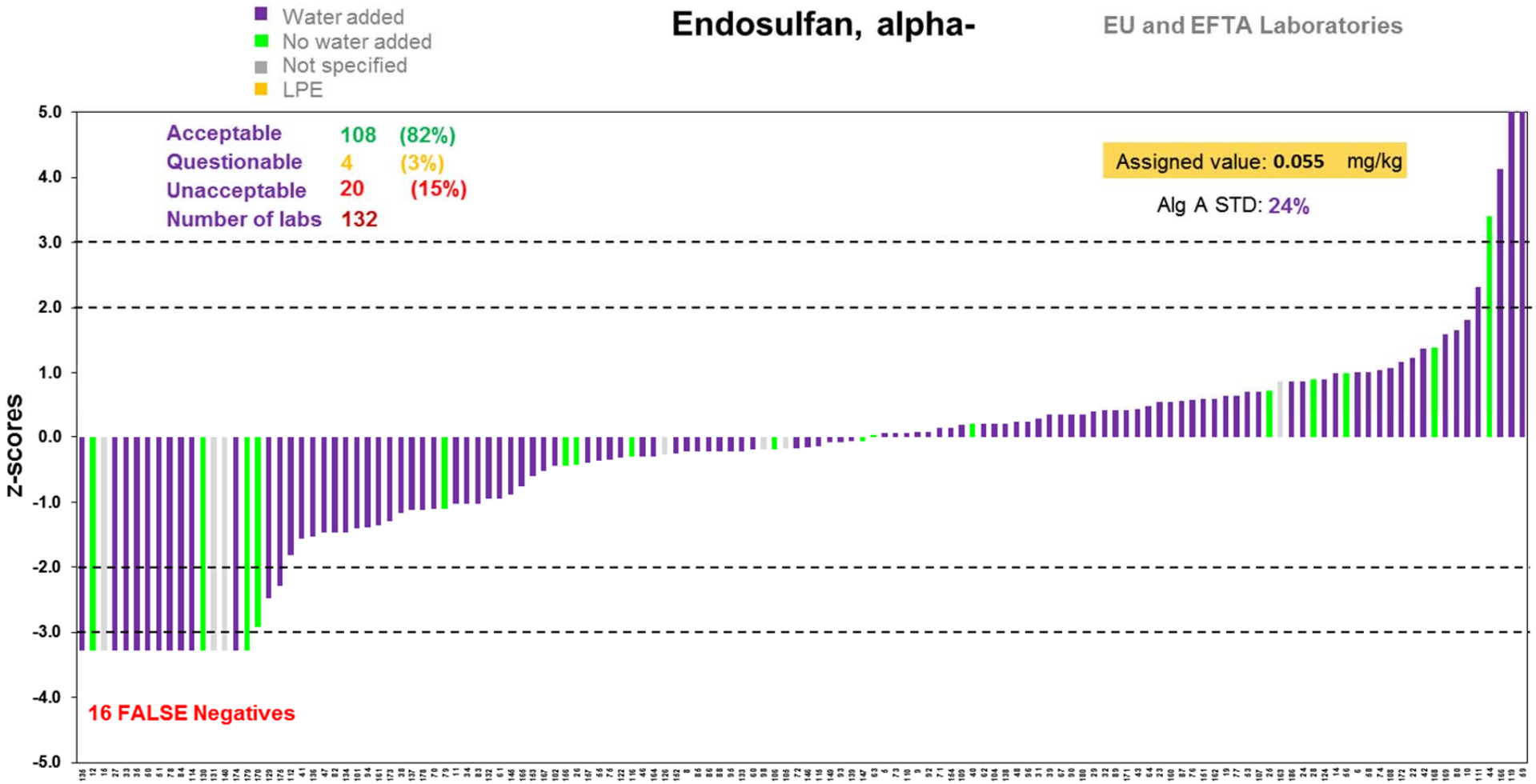

Water added
No water added
Not specified
LPE
Carbendazim
EU and EFTA Laboratories
Acceptable 89 (81%)
Questionable 11 (10%)
Unacceptable 10 (9%)
Number of labs 110
Assigned value: 0.274 mg/kg
Alg A STD: 29%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
2 FALSE Negatives

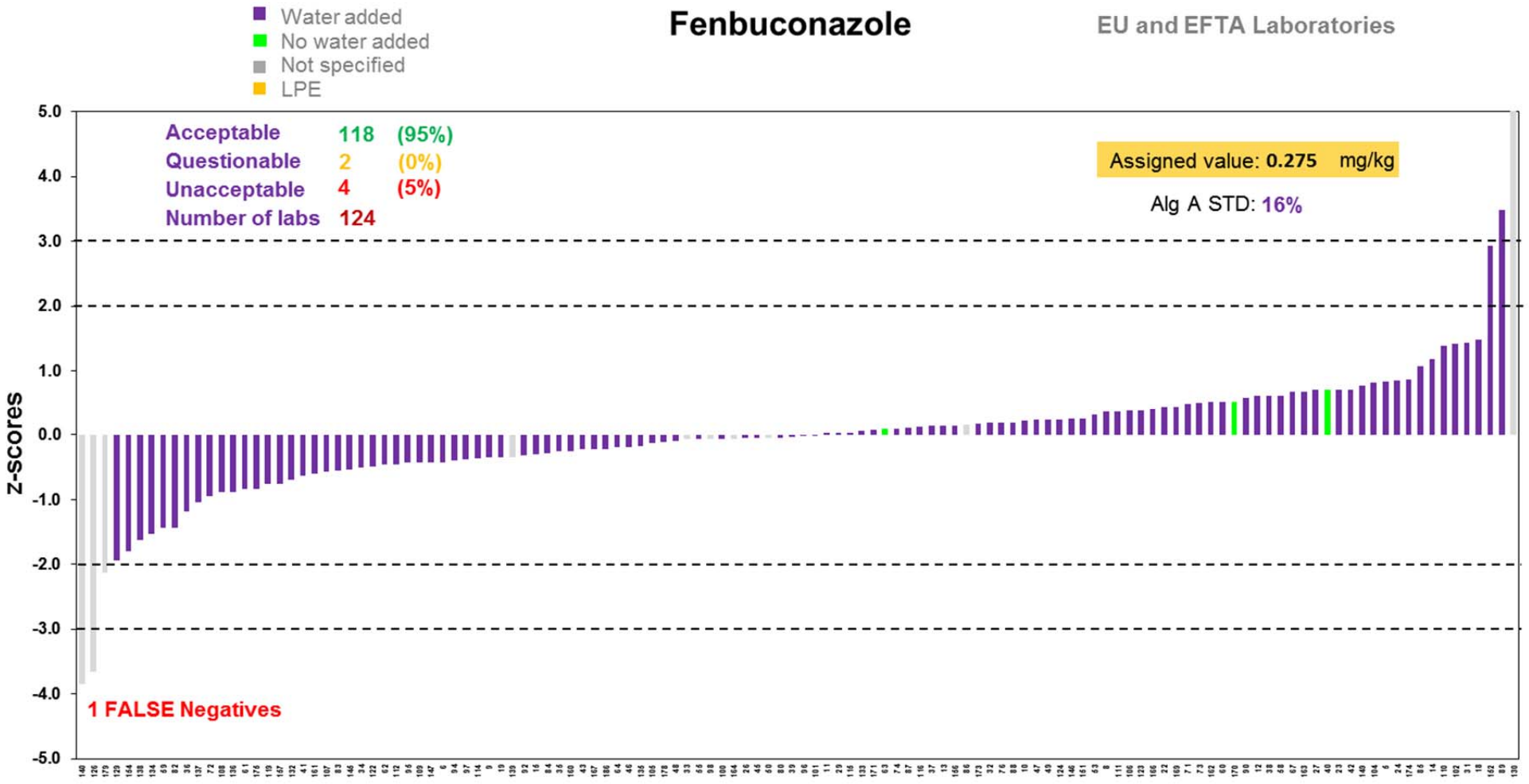

Water added
No water added
Not specified
LPE
Chlordane, trans
EU and EFTA Laboratories
Acceptable 85 (84%)
Questionable 2 (2%)
Unacceptable 14 (14%)
Number of labs 101
Assigned value: 0.050 mg/kg
Alg A STD: 20%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
9 FALSE Negatives

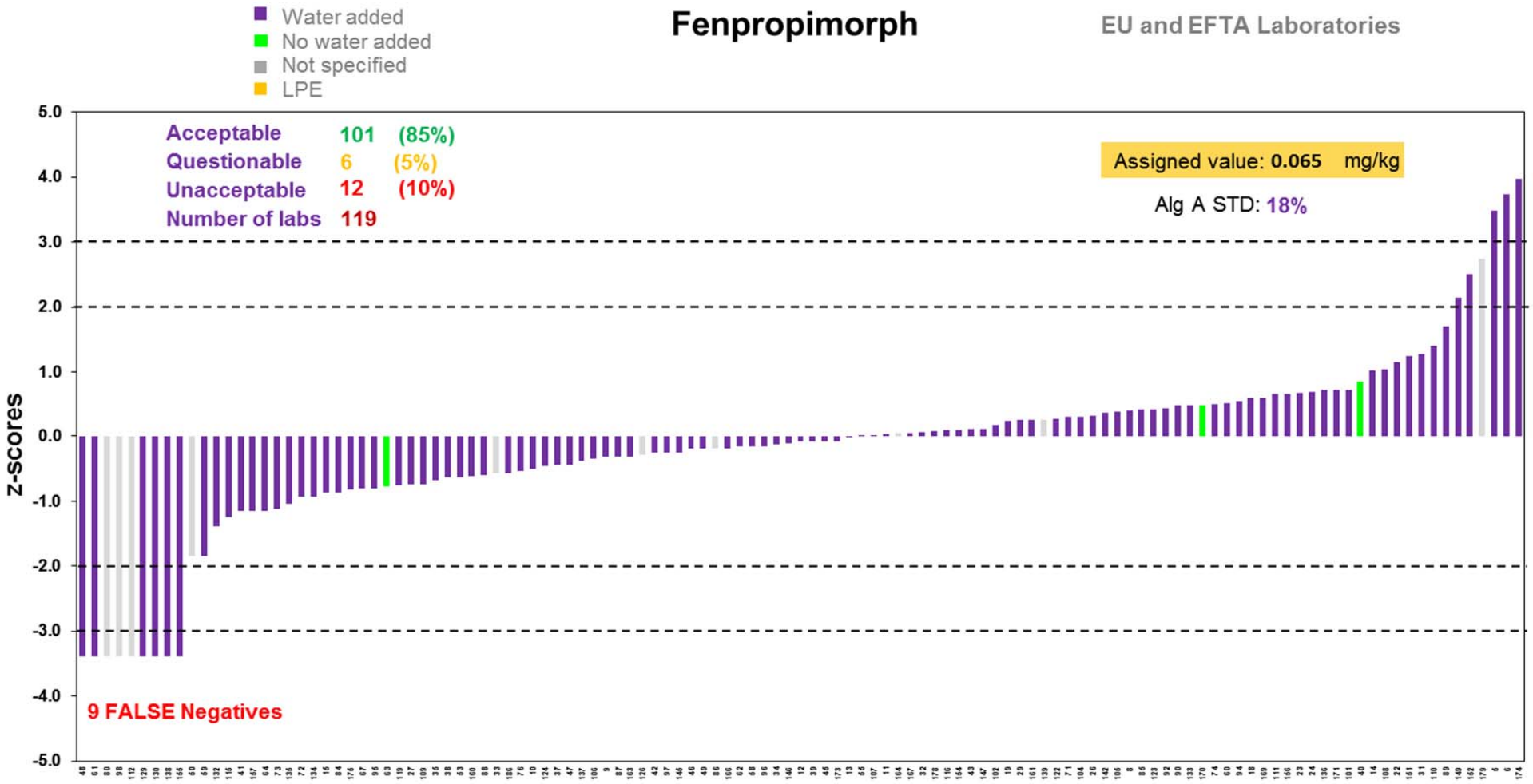

Water added
No water added
Not specified
LPE
Chlorpyrifos
EU and EFTA Laboratories
Acceptable 128 (95%)
Questionable 0 (0%)
Unacceptable 7 (5%)
Number of labs 135
Assigned value: 0.555 mg/kg
Alg A STD: 15%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
6 FALSE Negatives

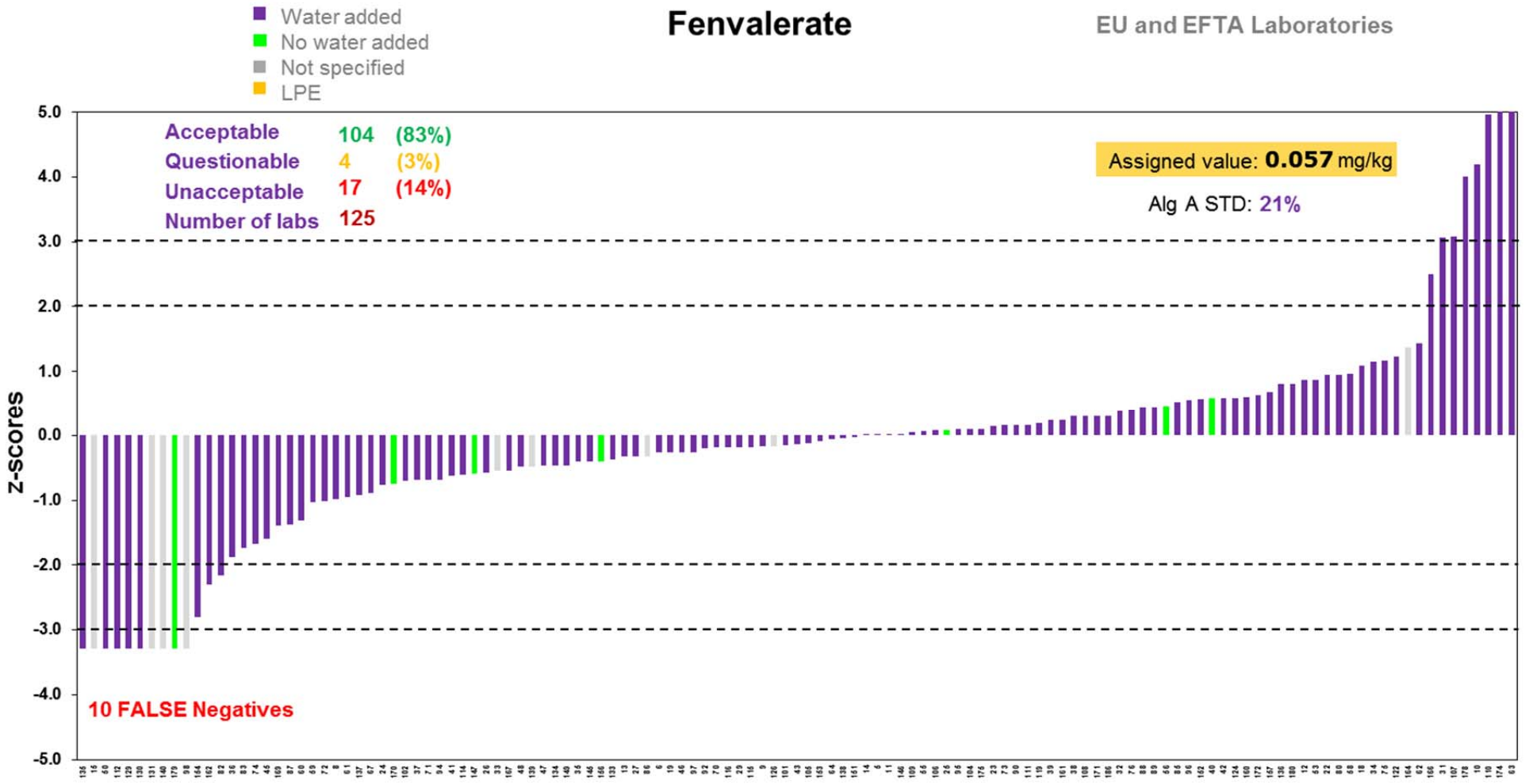

Water added
No water added
Not specified
LPE
Cyproconazole
EU and EFTA Laboratories
Acceptable 113 (88%)
Questionable 4 (3%)
Unacceptable 12 (9%)
Number of labs 129
Assigned value: 0.251 mg/kg
Alg A STD: 14%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
9 FALSE Negatives

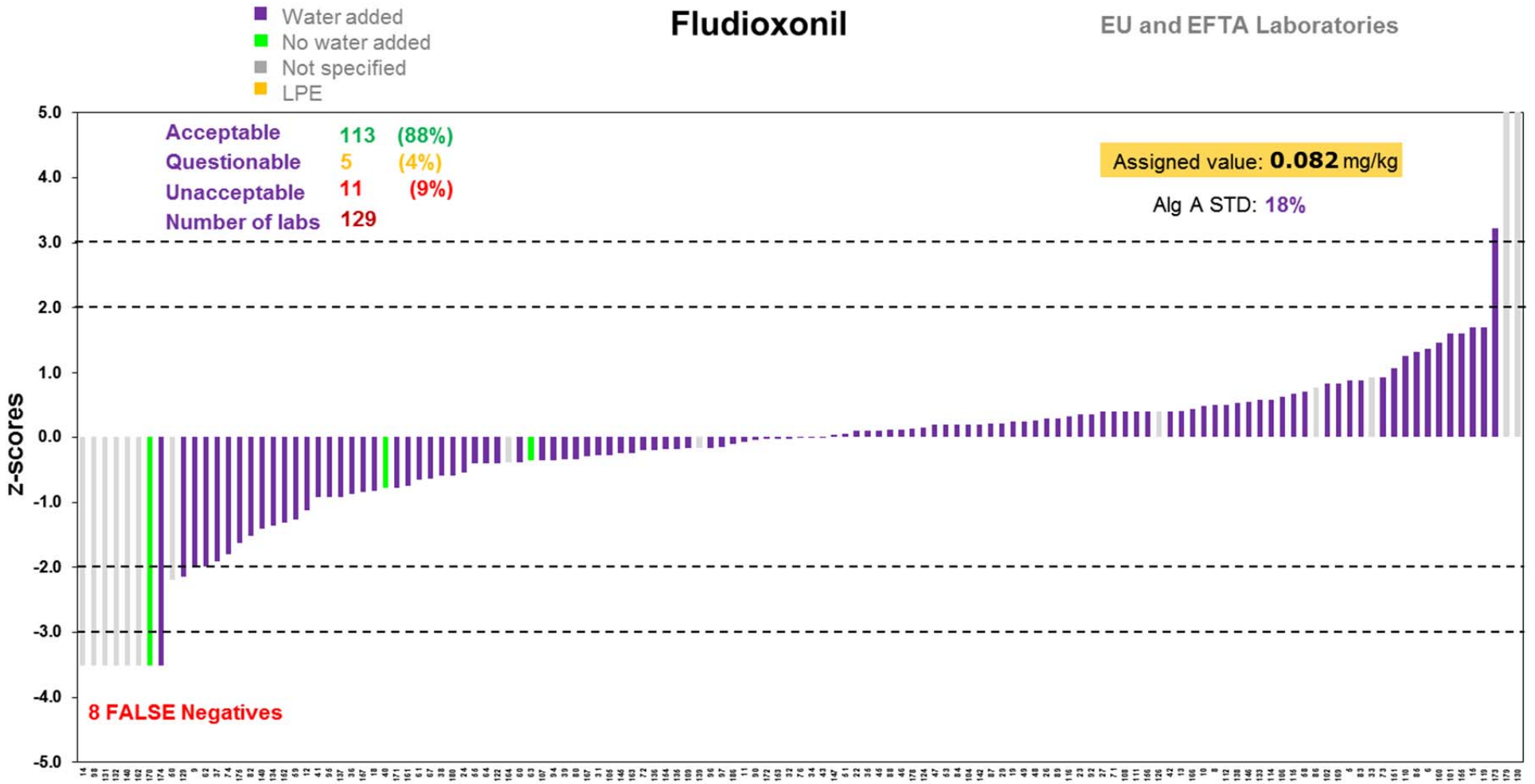

Water added
No water added
Not specified
LPE
Cyprodinil
EU and EFTA Laboratories
Acceptable 126 (95%)
Questionable 0 (0%)
Unacceptable 6 (5%)
Number of labs 132
Assigned value: 0.075 mg/kg
Alg A STD: 18%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
4 FALSE Negatives

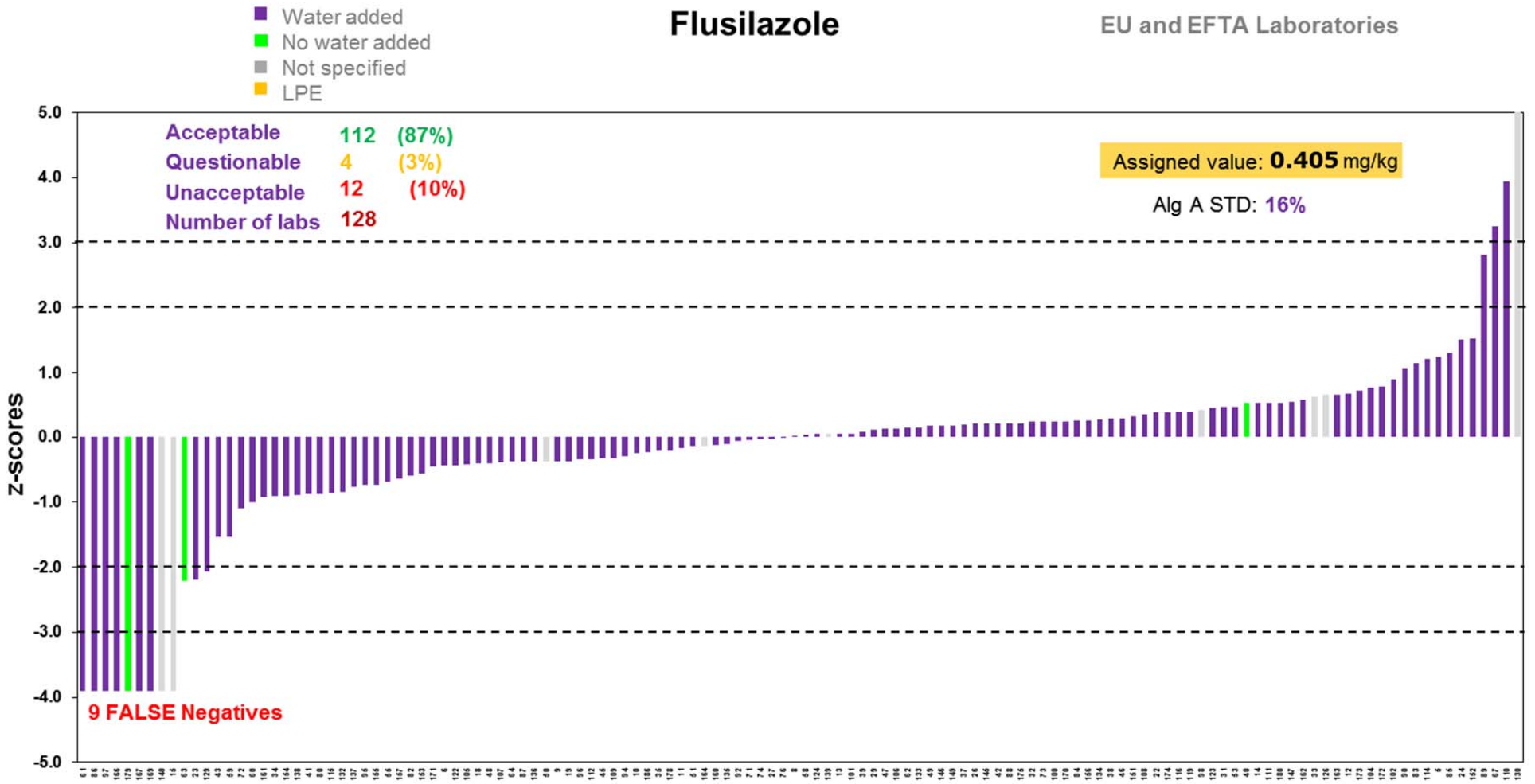

Water added
No water added
Not specified
LPE
Endosulfan, alpha-
EU and EFTA Laboratories
Acceptable 108 (82%)
Questionable 4 (3%)
Unacceptable 20 (15%)
Number of labs 132
Assigned value: 0.055 mg/kg
Alg A STD: 24%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
16 FALSE Negatives

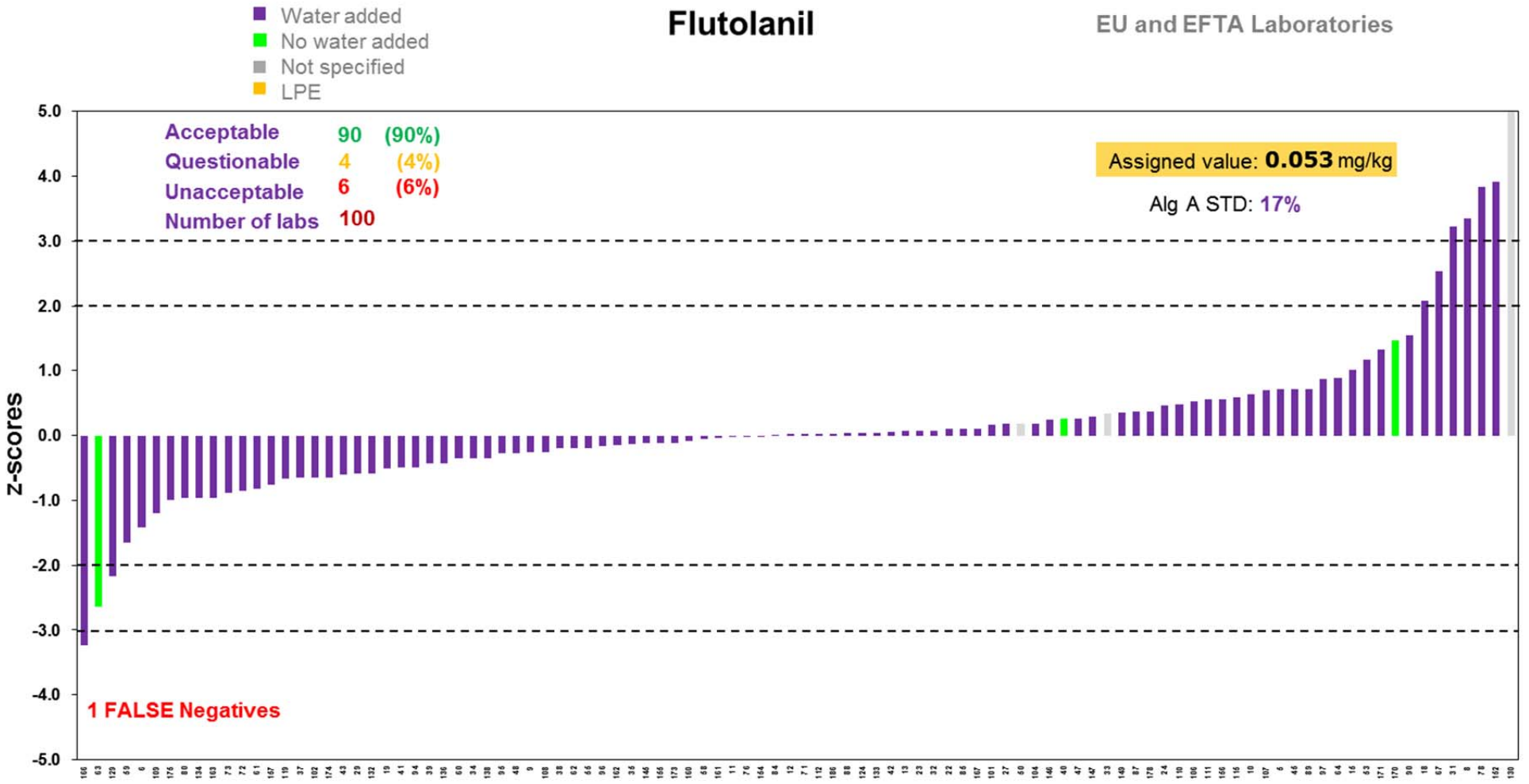

Water added
No water added
Not specified
LPE
Fenbuconazole
EU and EFTA Laboratories
Acceptable 118 (95%)
Questionable 2 (0%)
Unacceptable 4 (5%)
Number of labs 124
Assigned value: 0.275 mg/kg
Alg A STD: 16%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
1 FALSE Negatives

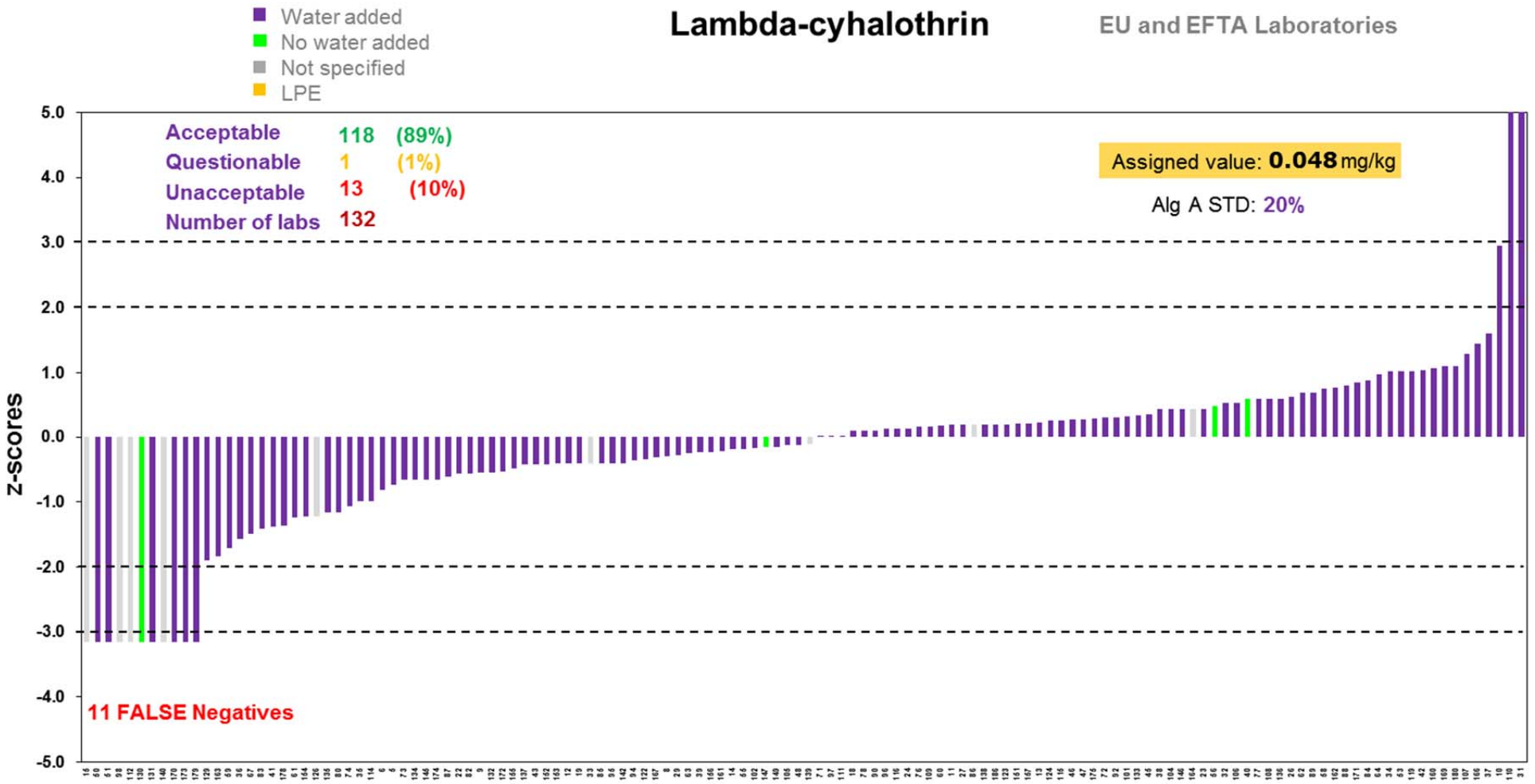

Water added
No water added
Not specified
LPE
Fenpropimorph
EU and EFTA Laboratories
Acceptable 101 (85%)
Questionable 6 (5%)
Unacceptable 12 (10%)
Number of labs 119
Assigned value: 0.065 mg/kg
Alg A STD: 18%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
9 FALSE Negatives

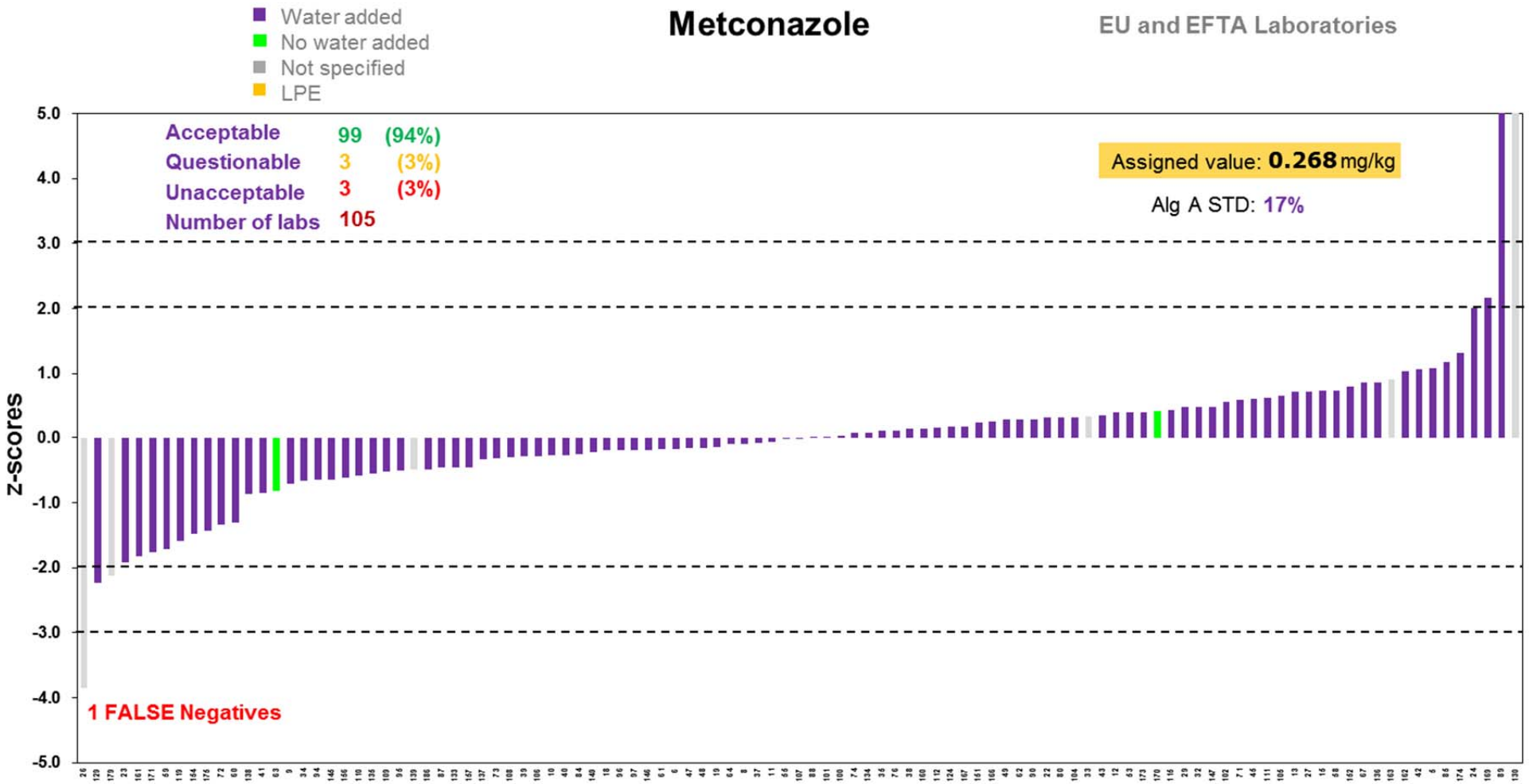

Water added
No water added
Not specified
LPE
Fenvalerate
EU and EFTA Laboratories
Acceptable 104 (83%)
Questionable 4 (3%)
Unacceptable 17 (14%)
Number of labs 125
Assigned value: 0.057 mg/kg
Alg A STD: 21%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
10 FALSE Negatives

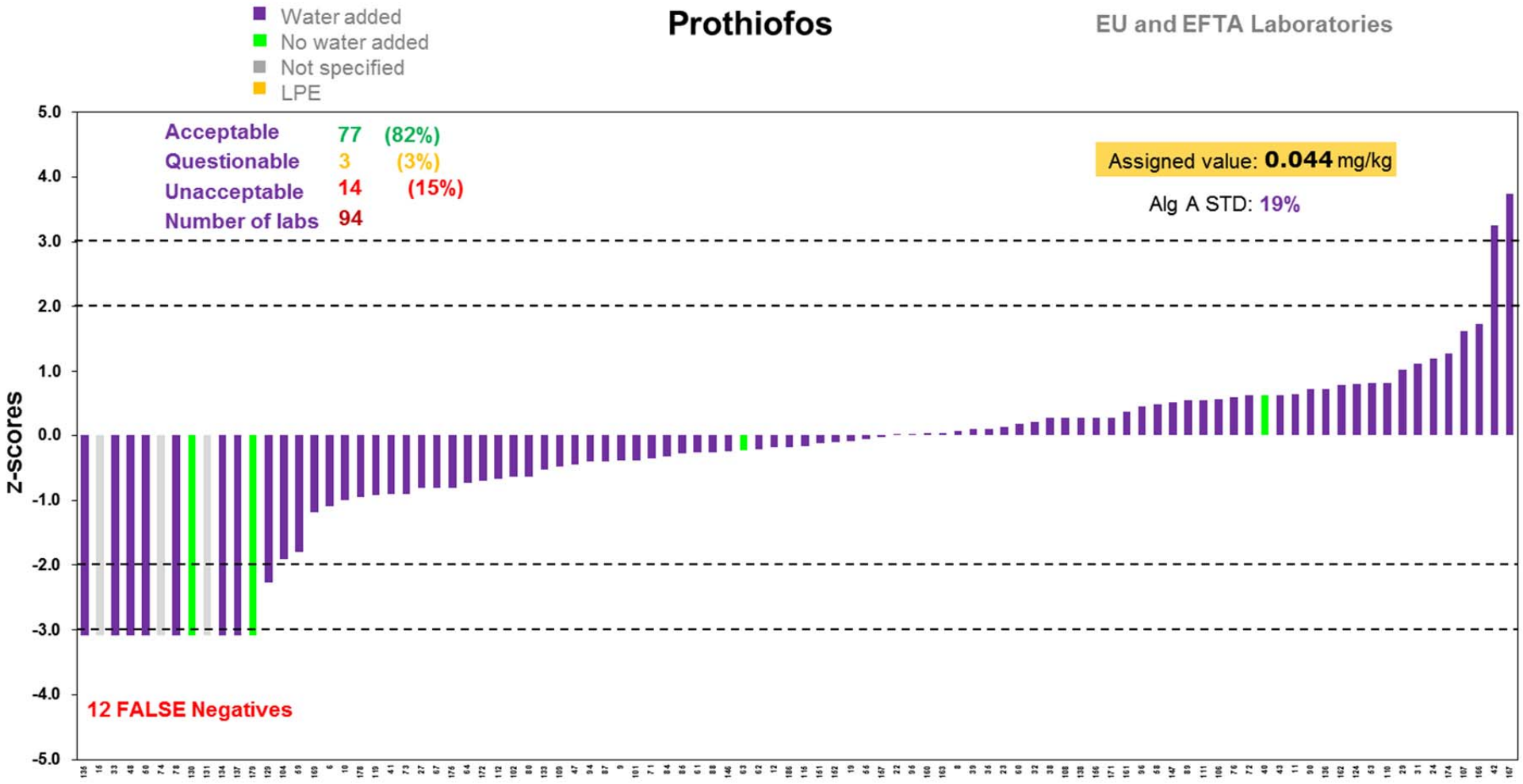

Water added
No water added
Not specified
LPE
Fludioxonil
EU and EFTA Laboratories
Acceptable 113 (88%)
Questionable 5 (4%)
Unacceptable 11 (9%)
Number of labs 129
Assigned value: 0.082 mg/kg
Alg A STD: 18%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
8 FALSE Negatives

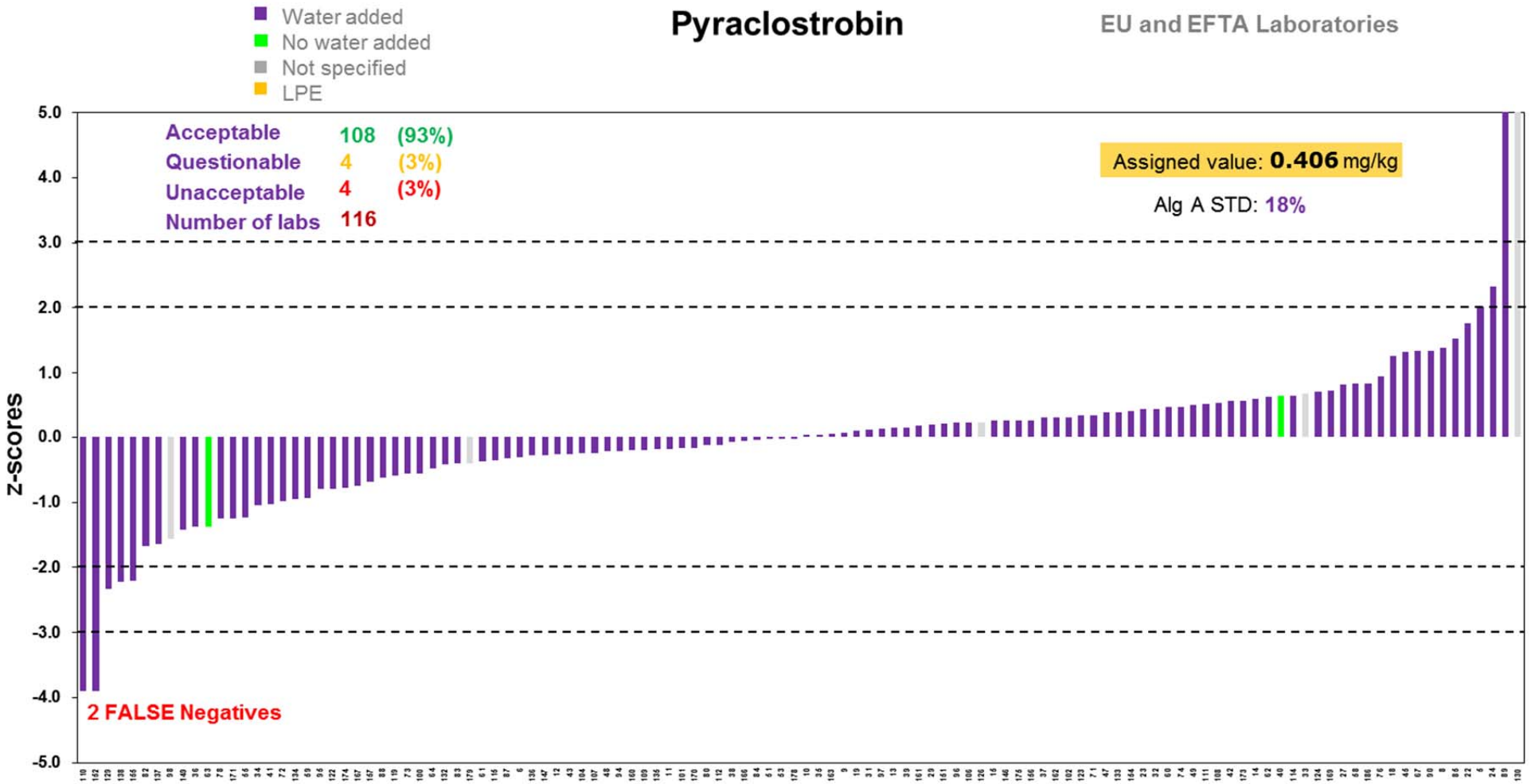

Water added
No water added
Not specified
LPE
Flusilazole
EU and EFTA Laboratories
Acceptable 112 (87%)
Questionable 4 (3%)
Unacceptable 12 (10%)
Number of labs 128
Assigned value: 0.405 mg/kg
Alg A STD: 16%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
9 FALSE Negatives

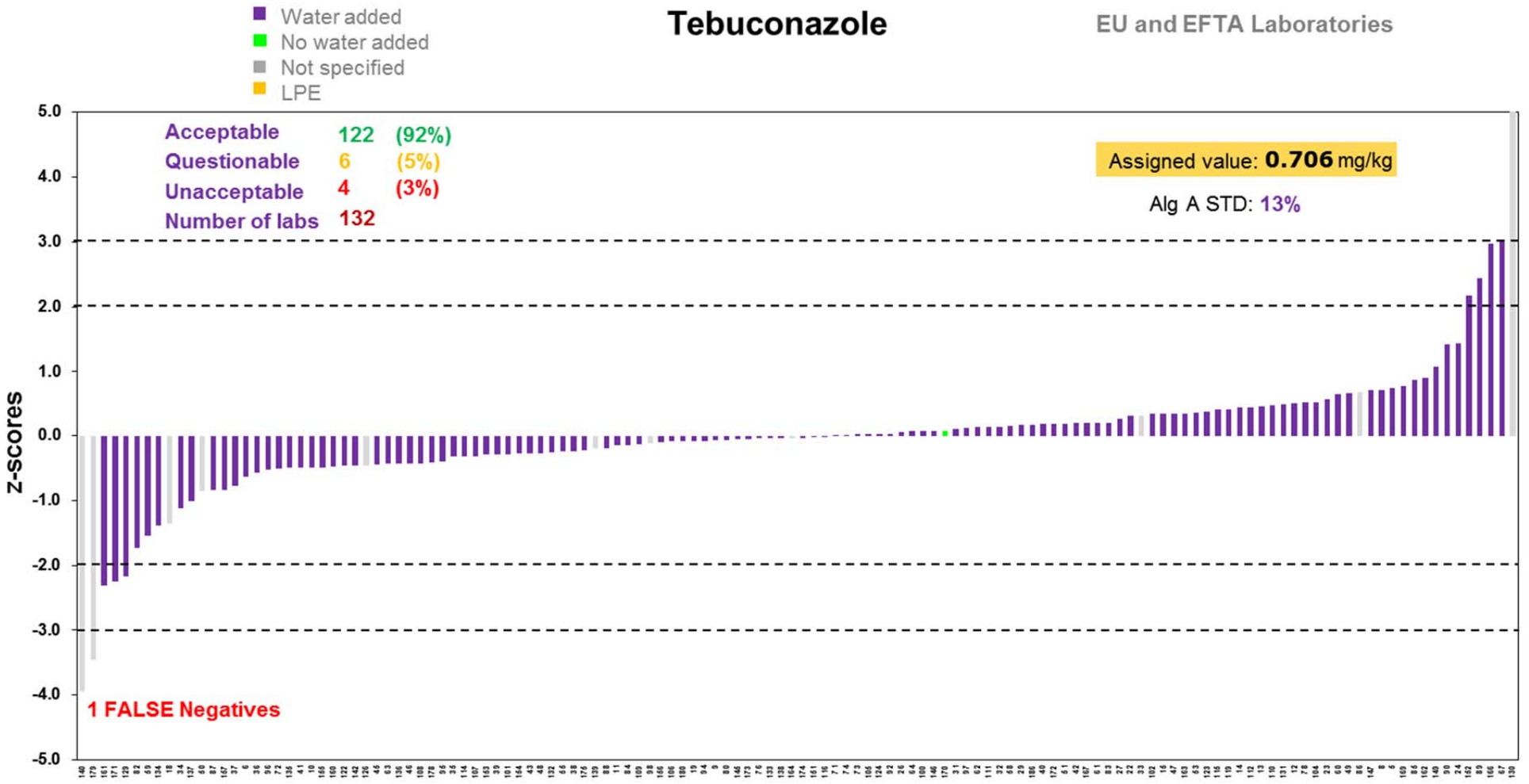

Water added
No water added
Not specified
LPE
Flutolanil
EU and EFTA Laboratories
Acceptable 90 (90%)
Questionable 4 (4%)
Unacceptable 6 (6%)
Number of labs 100
Assigned value: 0.053 mg/kg
Alg A STD: 17%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
1 FALSE Negatives


Water added
No water added
Not specified
LPE
Lambda-cyhalothrin
EU and EFTA Laboratories
Acceptable 118 (89%)
Questionable 1 (1%)
Unacceptable 13 (10%)
Number of labs 132
Assigned value: 0.048 mg/kg
Alg A STD: 20%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
11 FALSE Negatives


Water added
No water added
Not specified
LPE
Metconazole
EU and EFTA Laboratories
Acceptable 99 (94%)
Questionable 3 (3%)
Unacceptable 3 (3%)
Number of labs 105
Assigned value: 0.268 mg/kg
Alg A STD: 17%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
1 FALSE Negatives


Water added
No water added
Not specified
LPE
Prothiofos
EU and EFTA Laboratories
Acceptable 77 (82%)
Questionable 3 (3%)
Unacceptable 14 (15%)
Number of labs 94
Assigned value: 0.044 mg/kg
Alg A STD: 19%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
12 FALSE Negatives


Water added
No water added
Not specified
LPE
Pyraclostrobin
EU and EFTA Laboratories
Acceptable 108 (93%)
Questionable 4 (3%)
Unacceptable 4 (3%)
Number of labs 116
Assigned value: 0.406 mg/kg
Alg A STD: 18%
Z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
2 FALSE Negatives


Water added
No water added
Not specified
LPE
Tebuconazole
EU and EFTA Laboratories
Acceptable 122 (92%)
Questionable 6 (5%)
Unacceptable 4 (3%)
Number of labs 132
Assigned value: 0.706 mg/kg
Alg A STD: 13%
Z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
1 FALSE Negatives

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 joint collaboration.

An **Organising Team** is appointed by the EURL(s) in charge. This team is responsible for all administrative and technical matters concerning the organisation of the PT, e.g. the PT-announcement, production of Test Item and Blank Material, the undertaking of homogeneity and stability tests, packing and shipment of the Test Item and Blank Material, handling and evaluation of the results and method information submitted by the participants and the drafting of the preliminary and final reports.

To complement the internal expertise of the EURLs, a group of external consultants that form the **EUPT-Scientific Committee** (EUPT-SC)[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, the affiliation of each member is shown on the EURL-Website. The members of the EUPT-SC will also be listed in the Specific Protocol and the Final Report of each EUPT.

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

a) An independent **Quality Control Group** (EUPT-QCG) and

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

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

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

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

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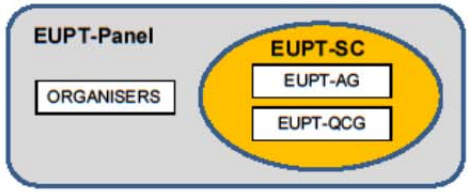
www.eurl-pesticides.eu

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

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



The decisions of the EUPT-Panel will be documented.

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

EUPT Participants

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

- Art. 28 of Reg. 396/2005/EC[6] (for all OfLs analysing for pesticide residues within the framework of official controls[7] of food or feed)
- Art. 33 of Reg. 882/2004/EC (for all NRLs)

The four EURLs will annually issue and distribute, via the EURL-website, a joint list of all OfLs that must participate in each of the EUPTs to be conducted within a given year. The list of obliged labs will be updated every year to take account of any changes in the lab profiles. Interim updates will be issued to eliminate any possible errors.

[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. 882/2004/EC, This includes labs involved in controls within the framework of national and/or EU-controlled programmes as well as labs involved in import controls according to Regulation 669/2009/EC.

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NRLs are responsible for checking whether all relevant OfLs within their network are included in the list of obligated laboratories and whether the contact information and commodity-scopes are correct.

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

Labs that are obliged to participate in a given EUPT, and that are not able to participate, must provide the reasons for their non-participation without prejudice of any legal action taken against them for not participating. This also applies to any participating laboratories that then fail to report results.

Confidentiality and Communication

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

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

As laid down in Regulation 882/2004, NRLs are responsible for evaluating and improving their own OfL-Network. On request from the NRLs, the EURLs will provide them with the PT-codes of the participating OfLs belonging to their OfL-Network. This will allow NRLs to follow the participation and performance of the laboratories within their network.

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

For each EUPT the organising EURL prepares a specific EUPT-Website where all relevant documents in their latest version are linked.

The official language used in all EUPTs is English.

Announcement / Invitation Letter

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

Target Pesticide List

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

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

Specific Protocol

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

Homogeneity of the Test Item

The Test Item will be tested for homogeneity typically before distribution to participants. The homogeneity tests 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-SC considering all relevant aspects (e.g. the homogeneity results of other pesticides spiked at the same time, the overall distribution of the participants' results, the analytical difficulties faced during the test, knowledge of the analytical behaviour of the

pesticide question) may decide to overrule the test. The reasons of this overruling have to be transparently explained in the Final EUPT-Report.

Stability of the analytes contained in the Test Item

The Test Items will also be tested for stability - according to ISO 13528, Annex B. The time delay between the first and the last stability test must exceed the period of the EUPT-exercise. Typically the first analysis is carried out shortly before the shipment of the Test Items and the last one shortly after the deadline for submission of results. To better recognise trends and gain additional certainty one or more additional tests may be conducted by the Organisers. At least 6 sub-samples (analytical portions) should be analysed on each test day (e.g. 2 analytical portions withdrawn from three randomly chosen containers OR 6 portions withdrawn from a single container). In principle all pesticides contained in the Test Item should be checked for stability. However, in individual cases, where sufficient knowledge exists that the stability of a certain analyte is very unlikely to be significantly affected during storage (e.g. based on experience from past stability tests or knowledge of its physicochemical properties), the Organisers, after consultation with the EUPT-QCG, may decide to omit a specific stability test. The EUPT-SC will finally decide whether analytes for which the stability test was not undertaken will be included in the final report, considering all relevant aspects such as the distribution of the participant's results (CV*).

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

The results of all stability tests are presented to the EUPT-SC. In special cases where the above stability test criteria are not met, the EUPT-SC considering all relevant aspects (e.g. the past experience with the stability of the compound, the overall distribution the participants' results, the 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.

Considering knowledge about the expected susceptibility of pesticides in the Test Item to possible losses, the Organisers will chose the shipment conditions to be such that pesticide losses are minimised (e.g. shipment of frozen samples, addition of dry ice). As shipment time can differ between labs/countries it is recommended that the Organisers conduct additional stability tests at

conditions simulating shipment. Should critical losses be detected for certain pesticides the EUPT-SC will be informed (or the EUPT-QCG before or during the test). Case-by-case decisions may be taken considering all relevant aspects including the shipment time of the samples to each laboratory.

Methodologies to be used by the participants

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

General procedures for reporting results

Participating laboratories are responsible for reporting their own quantitative results to the Organiser within the stipulated deadline. Any pesticide that was targeted by a participating laboratory should be reported as "analysed". Each laboratory will be able to report only one result for each analyte detected in the Test Item. The concentrations of the pesticides detected should be expressed in 'mg/kg' unless indicated otherwise in the specific protocol.

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

Correction of results for recovery

According to the Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed[8], it is common practice that pesticide analysis results are not corrected for recovery if the recovery rates range between 70 and 120 %. Correction of results for recovery is recommended if the average recovery is significantly different from 100 % (typically if outside the 70 – 120 % range). Approaches for recovery correction explicitly stated in the DG-SANTE document are the use of recovery correction factors, the use of stable isotope labelled analogues of the target analytes as Internal Standards (ILISs), the 'procedural calibration' approach as well as

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

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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. No recovery data are required where correction for recovery is automatic by adding amounts of analytes to the test portion for using the 'standard addition' approach, or isotopically-labelled internal standards (in both cases with spiking into the Test Item at the beginning of the extraction procedures) or procedural calibration. In these cases, the laboratories should report the actual approach that was followed.

Methodology information

All laboratories are requested to provide information on the analytical method(s) they have used. A compilation of the methodology information submitted by all participants is presented in an Annex of the final report or in a separate report. Where necessary the methods are evaluated and discussed, especially in those cases where the result distribution is not unimodal or very broad (e.g. CV* > 35 %). If no sufficient information on the methodology used is provided, the Organiser reserves the right not to accept the analytical results reported by the participants concerned 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 robust estimate of the participant's mean (x^*) as described in ISO 13528:2015[9], 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 compound in the past.

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

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

[9] 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).

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inappropriate storage or transport conditions (in case of susceptible compounds), and the use of inappropriate procedures that demonstrably lead to significantly biased results (e.g. due to degradation or incomplete extraction). Where the Organisers (e.g. after the publication of the preliminary report) receive information of such gross errors, having a significant impact on a generated result, the affected results will be examined on a case-by-case basis to decide whether, or not, they should be excluded from the population used for robust statistics. Results may also be omitted e.g. if an inappropriate method has been used even if they are not outliers. All decisions to omit/exclude results will be discussed with the EUPT-SC and the reasoning for the omission of each result clearly stated in the final EUPT-Report. However, z scores will be calculated for all results irrespective of the fact that they were omitted from the calculation of the assigned value.

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

Uncertainty of the assigned value

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

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

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

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

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

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

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

The percentage FFP-RSD is set at 25% based on experience from results of previous EUPTs[10]. The EUPT-Panel reserves the right to also employ other approaches on a case-by-case basis considering analytical difficulties and experience gained from previous proficiency tests.

For informative purposes the robust relative standard deviation (CV*) is calculated according to ISO 13528:2015; Chapter 7.7 (Consensus value from participant results) following Algorithm A in Annex C.

– ***z scores***

This parameter is calculated using the following formula:

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

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

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

Z scores will be interpreted in the following way, as is set in the ISO 17043:2010[11]:

$\|z\| \leq 2.0$	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 the RL < MRRL. The EUPT-Panel will decide whether, or not, these values should appear in the z score histograms.

– ***Category A and B classification***

[10] Comparative Study of the Main Top-down Approaches for the Estimation of Measurement Uncertainty in Multiresidue Analysis of Pesticides in Fruits and Vegetables. J. Agric. Food Chem., 2011, 59(14), 7609-7619.

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

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

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

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

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

For evaluation of the overall performance of laboratories within Category A, the Average of the Squared z score (AZ^2)[12,13] (see below) will be used. The AZ^2 is calculated as follows:

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

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

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

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

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

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

Publication of results

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

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

Certificates of participation

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

Feedback

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

Correction of errors

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

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

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

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

Follow-up activities

Laboratories are expected to undertake follow-up activities to trace back the sources of erroneous or strongly deviating results (typically those with $|z| > 2.0$) - including all false positives. Even results within $|z| \leq 2.0$ may have to be checked if there is indications of a significant positive or negative bias.

EURL EU REFERENCE LABORATORIES FOR RESIDUES OF PESTICIDES

7th Edition: Revised 27th January, 2017

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 if they: a) haven't participated, or b) targeted less than 90% of the compulsory pesticides in the target lists (80% for SRM-compounds), or c) detected less than 90% of the compulsory compounds present in the test items (80% for SRM-compounds). Additionally, NRLs that obtained AZ^2 higher than 3 in two consecutive EUPTs of the last four EUPTs, will be considered as **underperforming in accuracy**. A two-step protocol established by DG-SANTE will be applied as soon as underperformance of an NRL is detected[14]:

Phase 1:

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

[14] Article 32 of the Regulation 882/2004

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-CF11 (2017)

(last updated: 10 March 2017)

Introduction

This protocol is complementary to the General Protocol for EU Proficiency Tests for Pesticide Residues in Food and Feed. 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-CF11.

Test Item (Test Material)

This proficiency test concerns the analysis of pesticide residues in oat flour. The oat has been grown in Denmark and pesticides were applied in the field. Following harvest, the oat kernels were spiked with additional pesticides.

The blank Test Item provided, can be used for recovery experiments as well as for the preparation of matrix-matched calibration standards. However, the blank Test Item must also be analysed and possible detected pesticides reported.

The Organizers will check the Test Items for sufficient homogeneity and for stability at conditions reproducing sample shipment and storage during the duration of the test. The blank Test Item will also be checked to prove that the target analytes are not contained at any relevant levels. All these tests will be conducted by the EURL-CF, which is ISO 17025 accredited.

Analytical Parameters

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

Laboratories should carefully read the Target Pesticides List, where important information about reporting of results, as well as the Minimum Required Reporting Levels (MRRLs), is given. The Target Pesticides List contains only individual compounds, and results should only be reported for individual compounds, no matter how the residue definitions have been set.

The MRRL values will be used to help identify false positive and false negative results and for the calculation of z-scores for false negatives.

Amount of Test Item

The participants will receive:

- approximately 100 g of oat flour Test Item with incurred and spiked pesticides and
- approximately 100 g of blank oat flour Test Item.

Shipment of Test Items

The Test Items are planned to be shipped on 8 May, 2017.

Test Items will be shipped frozen and packed in thermo-boxes together with a freezer block. The organisers 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 less) before analysis to avoid any possible deterioration/spoilage and to minimize pesticide losses. The Test Items should be mixed thoroughly, before taking the analytical portion(s).

All participants should use their own routine standard operating procedures for extraction, clean-up and analytical measurement and their own reference standards for identification and quantification purposes. Considering the available amount of Test Items, laboratories employing methods requiring large analytical portions are advised to scale them down.

The homogeneity tests will be conducted using 5 g of 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.

Results Submission Website and Deadlines

Sample receipt acknowledgement, analytical results and method information are to be submitted via the EUPT-CF11 Result Submission Website.

Relevant links and documents can be found on the EURL-CF webpage: EUPT–CF11 Website.

The Result Submission Website will be accessible from 9 May 2017. The webpage contains a link to specific instructions on how to enter the data in the result submission website.

To access the data submission forms, participants must use their unique login data (username and password). **Username and password are included in the registration confirmation mail.**

The labs can fill in the sub-pages at different stages/sessions. Remember to save the data of each page before leaving it.

The deadline for result submission is 6 June 2017 at 24.00 CEST.

Test Item Receipt and Acceptance - Subpage 0

Once the laboratory has received the Test Items it must report to the organiser, via the **EUPT-CF11 Result Submission Website**, the date of receipt, the condition of the Test Item, and its acceptance. The deadline for acceptance is the 11 May 2017. If the laboratory does not respond by this deadline, the Organisers will assume that the Test Items have been received and accepted.

If participants have not received the Test Items by the 11 May 2017 at noon, they must inform the Organiser immediately by e-mail to eurl-cf@food.dtu.dk.

Reporting Qualitative and Quantitative Results - Subpages 1 and 2

To report their results, laboratories must access the **EUPT-CF11 Result Submission Website**.

Deadline: All results must be reported on the online result submission website by 06 June 2017 at 24.00 CEST. The website will NOT be accessible for result submission after this date and time, and any results reported after the deadline will not be included in the statistical treatment, or in the final report.

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 ≥ 0.010 mg/kg;

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

Reporting Information on Analytical Methodology - Subpage 3

All laboratories are requested to provide information on the analytical method(s) they have used via the **EUPT-CF11 Result Submission Website**. The laboratories are asked to thoroughly fill in this important information in order to minimize the administrative burden of collecting this information at a later stage.

Reporting of supplementary information in case of false negative results – Subpage 4

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 be done by accessing subpage 4 on the **EUPT-CF11 Result Submission Website**. This subpage will be accessible from 7-9 June 2017.

If no sufficient information on the methodology used is provided, the Organiser reserves the right not to accept the analytical results reported by the participant.

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-CF11 can be found on **EUPT-CF11 Website**.

Calendar

Activity	Dates
Announcement Calendar Target Pesticide List	February 2017
EUPT-Registration Website open	13 March 2017
Deadline for registration	23 April 2017
Specific Protocol published	24 April 2017
Distribution of Test items	8 May 2017
Deadline for receipt and acceptance of Test Materials	within 24 hr on receipt
Deadline for Result Submission	6 June 2017 at 24.00 CEST
Deadline for submission of additional method information for false negative results	9 June 2017 at 13.00 CEST
Preliminary Report (only compilation of results) published	3 August 2017
Final Report published	December 2017

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 invoice will be sent by ordinary mail. 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 Peter Skovgaard Christensen, pschr@adm.dtu.dk at the financial department of DTU.

Contact information:

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ISBN: 978-87-93565-11-1