Proficiency Test on pesticide residues in maize flour

EU Reference Laboratory on Cereals & Feeding stuff

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DTU Food
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Pesticide Residues in Maize Flour

Final Report

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PREFACE

Regulation 882/2004/EC [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 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 396/2005/EC 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 ninth organized within the frame of the EURL activities with cereal or feed matrix as Test Items. The previous PTs were EUPT-C1/SRM2 on wheat (2007), EUPT-C2 on wheat (2008), EUPT-C3/SRM4 on oats (2009), EUPT-C4 on rye (2010), EUPT-C5/SRM6 on rice (2011), EUPT-C6 on barley, EUPT-CF7 on animal feed and EUPT-CF8 on wheat. 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-CF9 on maize (2015) was only focused on MRM-pesticides. The maize Test Item used for EUPT-CF9 was treated with formulations in the field and post-harvest in the laboratory.

Participation in EUPT-CF9 was compulsory for all National Reference Laboratories (NRLs) and Official Laboratories (OfLs) within the EU involved in the determination of pesticide residues in cereal 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. All NRLs and OfLs that were supposed to participate in this exercise, but decided not to take part, were asked to state the reasons for their non-participation.

DG-SANCO 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 21 January 2015 the announcement of the 9th European Commission's Proficiency Test on cereals and feed (EUPT-CF9) was published on the EURL homepage, 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 116 individual compounds 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-CF9, were provided via the homepage. Laboratories were able to register on-line from the 10 February to 20 March 2015. In total 141 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 maize 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 maize was a feed variety. The Test Item contained 18 compounds in total. Danish Centre for Food and Agriculture at Aarhus University grew the maize and performed the field treatments in 2014. The pesticides employed for field 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. However, no residues were found of any of the pesticides included in Pesticide Target List. The harvested maize kernels were spiked with 18 pesticides post-harvest, and then checked for homogeneity before shipping to participants. Furthermore, the stabilities of the pesticides in the Test Item were checked several times during the period of time allowed for laboratories to complete the PT exercise.

The participating laboratories were provided with 125 g portions of the Treated maize Test Item and 125 g of untreated Blank maize Test Item. Both Test Items were shipped to participants on 20 April 2015 and the deadline for submission of results to the Organiser was the 19 May 2015. 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 and the sample shaken again 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 (maize). 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. Because the maize grains on the cobs were covered with leaves at the time the pesticides were applied in the field, only systemic pesticides were used. Additionally, only pesticides that are appropriate for the treatment of maize were chosen. Twelve pesticides were selected (10 formulations) and the field spraying, at double the prescribed application rate, was performed by the Danish Centre for Food and Agriculture at Aarhus University. Approximately, 70 kg of the harvested maize grain was delivered for preparation of the Test Item. The preliminary analysis of the material showed that no residues were found in the maize except for one degradation product of an active substance that was not included in the target list. Consequently, the test material had to be spiked in the laboratory. However, 8 of the pesticides used for the treatment in the field were either, not included multiannual programme, or had been used several times in the previous EUPT cereals/feed, and were therefore not chosen for the spiking treatment. The remaining 4 pesticides were then supplemented with 14 other pesticides so the test material was spiked with a total of 18 pesticides. Both the pesticides used for treatment in the field and those used for spiking in the laboratory can be seen in Table 1. Spiking in the laboratory was performed using formulations or pure analytical standards. One kilogram of the field treated maize was spiked and subsequently mixed with 44 kg of field treated maize and homogenised thoroughly. The resulting 45 kg of mixed maize grain was milled separately as 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 twenty-five gram portions were weighed out into screw-capped polyethylene plastic bottles, sealed, numbered, and stored in a freezer at about -20 °C prior to homogeneity testing and distribution to participants.

1.4 Preparation of the 'blank' Test Item

The maize 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. One hundred and twenty-five gram portions were weighed out into screw-capped polyethylene plastic bottles, sealed, and stored in a freezer at about -20 °C prior to distribution to 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 Item were sufficiently homogenous for the proficiency test was that: $S_s^2 < c$ where S_s is the between-bottle sampling 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-CF9.

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

Pesticide	Application in field	Spike in laboratory	Formulation
Bifenazate	x		Floramite
Cypermethrin	x		Cyperb
Epoxiconazole	x	x	Opera
Foramsulfuron	x		MaisTer
Iodosulfurum	x		MaisTer
Lambda-cyhalothrin	x	x	Karate
Pendimethalin	x	x	Stomp
Propiconazole	x	x	Bumper/Tilt
Pyraclostrobin	x		Opera
Spirotetramat	x		Movento
Tebuconazole	x		Folicur
Thifensulfuron-methyl	x		Harmony
Azoxystrobin		x	Amistar
Carbendazim		x	Bavistin FL
Chlorfenvinphos		x	
Chlorpyrifos-methyl		x	Reldan
Clothianidin		x	
Fluopyram		x	
Isocarbophos		x	
Lindane		x	
Metolachlor		x	
Metribuzin		x	
Spiromesifen		x	
Terbuthylazine		x	
Thiacloprid		x	
Triticonazole		x	

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. Two different storage temperatures were used; room temperature and -18 °C. Six sub-samples (analytical portions) was 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: 21 April 2015

Day 2: 5 May 2015

Day 3: 19 May 2015

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	0.050	0.00001	0.0000	Pass
Carbendazim	0.399	0.00097	0.0018	Pass
Chlorfenvinphos	0.041	0.00000	0.0001	Pass
Chlorpyrifos-methyl	0.043	0.00000	0.0000	Pass
Clothianidin	0.489	0.00116	0.0036	Pass
Epoxiconazole	0.050	0.00001	0.0000	Pass
Fluopyram	0.089	0.00005	0.0001	Pass
Isocarbofos	0.068	0.00001	0.0001	Pass
Lambda-cyhalothrin	0.100	0.00001	0.0003	Pass
Lindane	0.059	0	0.0002	Pass
Metolachlor	0.071	0.00000	0.0002	Pass
Metribuzin	0.351	0.00057	0.0016	Pass
Pendimethalin	0.043	0.00001	0.0000	Pass
Propiconazole	0.148	0.00013	0.0003	Pass
Spiromesifen	0.073	0.00000	0.0002	Pass
Terbuthylazine	0.079	0.00002	0.0001	Pass
Thiacloprid	0.086	0.00003	0.0001	Pass
Triticonazole	0.081	0.00003	0.0001	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	0.049	0.003	0.004	Pass
Carbendazim	0.435	0.012	0.034	Pass
Chlorfenvinphos	0.050	0.002	0.004	Pass
Chlorpyrifos-methyl	0.051	0.002	0.004	Pass
Clothianidin	0.534	0.022	0.032	Pass
Epoxiconazole	0.054	0.001	0.004	Pass
Fluopyram	0.089	0.006	0.007	Pass
Isocarbofos	0.079	0.005	0.006	Pass
Lambda-cyhalothrin	0.095	0.003	0.007	Pass
Lindane	0.074	0.002	0.005	Pass
Metolachlor	0.086	0.004	0.006	Pass
Metribuzin	0.386	0.001	0.027	Pass
Pendimethalin	0.038	0.001	0.003	Pass
Propiconazole	0.147	0.003	0.011	Pass
Spiromefesin	0.075	0.003	0.005	Pass
Terbuthylazine	0.088	0.004	0.006	Pass
Thiacloprid	0.094	0.005	0.007	Pass
Triticonazole	0.079	0.002	0.006	Pass

The results of the stability test for storage temperature -18 are given in Table 3. All pesticides passed the test at -18 °C. However, spiromefesin did not pass when stored for 4 weeks at room temperature and this compound had clearly degraded. Additionally, carbendazim, fluopyram, lindane, metribuzin, pendimethalin and triticonazole did not pass the test. This, however, was this was not due to degradation of the pesticides, but caused by changes in the maize matrix. During storage at room temperature the fatty acid peak eluting from 16-18 min increased dramatically, see Figure 1. All stability test was analysed in the same run and calculated from matrix matched calibration curve where the matrix was maize stored at -18 degree. Consequently, the differences in the coextracted compound was the most likely cause of the higher result measured after 4 weeks storage. See the individual figures for all pesticides in Appendix 4.

The laboratories were instructed to store the test item at -18 degree and the result for spiromefesin was very good with 22% robust RSD. The stability of the test material was consequently acceptable for all pesticides.

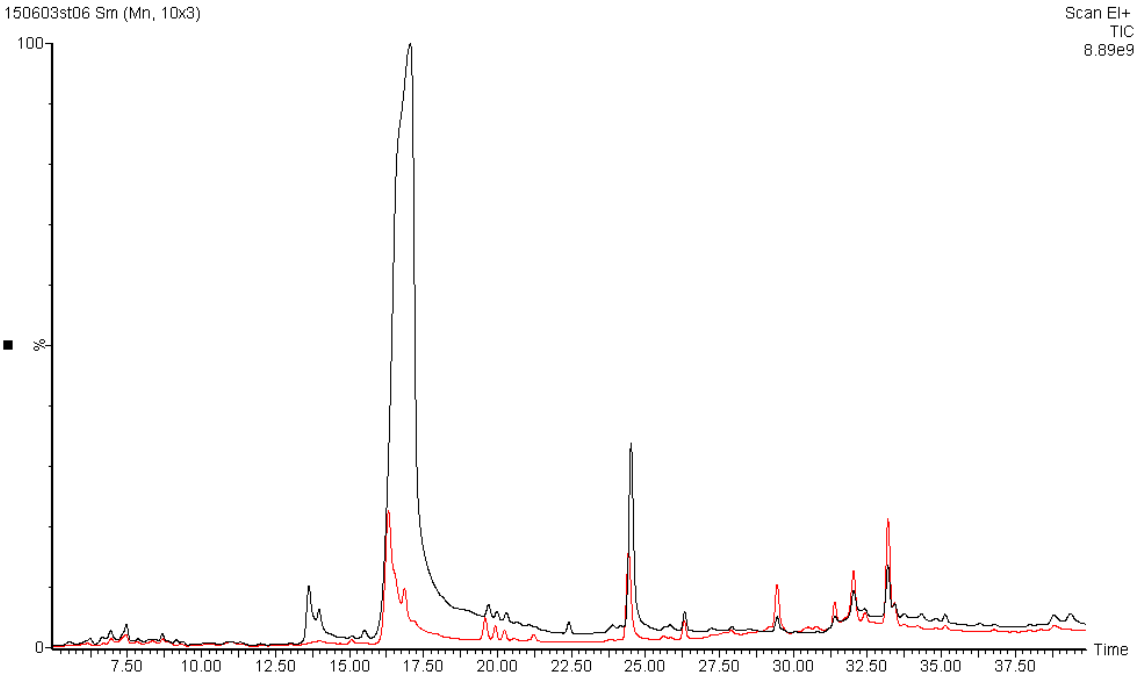

Figure 1. GC-MS chromatograms (scan mode) of the maize stored at 4 week at -18 degrees (red) or at room temperature (black).

1.7 Organisational details

1.7.1 Access to documents, registration and confidentiality

In the invitation letter of 21 January 2015, all NRLs and OfLs were requested to register using the online registration link from 10 February to 20 March 2015. 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 20 April 2015, 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 (19 May 2015). 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 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.

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 4 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 (μ)

In order to minimise the influence of out-lying results on the statistical evaluation, the assigned value (= consensus concentration) will typically be estimated using robust statistics as described in ISO 13528:2009 [5]. The EUPT-Panel has decided that only results from laboratories that added water to the samples before extraction should be included in the calculation of the assigned values as this has repeatedly demonstrated good performance in the past.

2.3 Uncertainty of the assigned value

The uncertainty of the assigned values μ_i is calculated according to ISO 13528:2009 [5] as:

$$\mu_i = 1.25 \frac{s*}{\sqrt{n}}$$

Where:

- μ_i is the uncertainty in mg/kg
- s* is the robust standard deviation estimate
- n is the total number results

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

The target standard deviation (δ) of the assigned value will be calculated using a Fit-For-Purpose Relative Standard Deviation (FFP-RSD) approach, as follows:

$$\delta = b_i * \mu_i \qquad \text{with } b_i = 0.25 \text{ (25\% FFP-RSD)}$$

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

For informative purposes the robust relative standard deviation (CVs*) is calculated according to ISO 13528:2009-01; Chapter 5.6 (Consensus value from participants) following Algorithm A.

2.5 Z-scores

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

$$z_i = (x_i - \mu_i) / \delta_i$$

Where:

- x_i is the value reported by the laboratory
- μ_i the assigned value
- δ_i the standard deviation at that level for each pesticide (i).

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 typically be 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)

Laboratories that have detected at least 90% of the pesticides present in the Test Item and reported no false positives are classified into Category A. For evaluation of the overall performance of laboratories within Category A, the Average of the squared z-score (AZ^2), are calculated.

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

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, 147 EU and EFTA laboratories, from 30 different countries (27 EU member states), agreed to participate in this proficiency test. Malta was represented by UK NRL. One NRL did not register and four EU/EFTA participants did not submit results. Additionally, 16 Third Countries registered for the PT. Three of these laboratories did not submit results, two of them because the test items did not reach the laboratories due to difficulties with customs clearance. 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 pesticides and are shown in **Table 9a/b-12a/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 4-8** 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]	False negatives	% results [2]
Azoxystrobin	128	15	4	90
Carbendazim	108	35	0	76
Chlorfenvinphos	125	18	2	88
Chlorpyrifos-methyl	131	12	2	92
Clothianidin	102	41	1	72
Epoxiconazole	118	25	3	83
Fluopyram	87	56	1	61
Isocarbophos	88	55	7	62
Lambda-cyhalothrin	129	14	5	91
Lindane	133	10	3	94
Metolachlor	96	47	4	68
Metribuzin	102	41	2	72
Pendimethalin	127	16	0	89
Propiconazole	126	17	2	89
Spiromesifen	88	55	3	62
Terbuthylazine	109	34	0	77
Thiacloprid	108	35	0	76
Triticonazole	107	36	4	75

[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 = 143). False negatives are included in reported results.

Azoxystrobin, chlorpyrifos-methyl, lambda-cyhalothrin and lindane were the most frequently analysed compounds with 90 % or more of the labs submitting results for these compounds. Carbendazim, chlorfenvinphos, clothianidin, epoxiconazole, metribuzin, pendimethalin, propiconazole, terbuthylazine, thiacloprid and triticonazole were analysed and reported by 71-89% of the participants and fluopyram, isocarbophos, metolachlor and spiromesifen were only analysed and reported by 61-67% of participants.

3.1.1 False positives

Eight participants from EU and EFTA countries reported 9 results for 5 different additional pesticides above the MRRL that had not been used to treat the Test Item (**Table 5**). The pesticides were: bifenthrin, flonicamid, HCH-alpha, HCH-beta and thiamethoxam. 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.

One laboratory reported a result for pirimiphos-methyl below the MRRL but above their own Reporting Level. This results is, however, not considered to be false positive.

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
214	Bifenthrin	0.052	GC-MS/MS (QQQ)	[1]	0.01
226	Thiamethoxam	0.54	LC-MS/MS QQQ	0.02	0.01
247	HCH-alpha	0.01	GC-MS/MS (QQQ)	0.01	0.01
256	Flonicamid	0.08	LC-MS/MS QQQ	0.01	0.01
283	HCH-beta	0.0657	GC-MS/MS (QQQ)	0.01	0.01
334	HCH-alpha	0.0457	GC-MSD	0.01	0.01
374	HCH-alpha	0.059	GC-MS/MS (QQQ)	0.01	0.01
374	HCH-beta	0.081	GC-MS/MS (QQQ)	0.01	0.01
406	HCH-beta	0.036	GC-MSD	0.01	0.01

[1] No information received.

3.1.2 False negatives

Missing 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. Forty-two results were judged as false negatives, which represents 2 % of the total number of results. Around 20 % of the participants (27 laboratories) reported false negative results. This is similar to previous EUPT on cereals where 20-30% of the labs reported false negative results. No false negatives results were reported for carbendazim, pendimethalin, terbuthylazine and thiacloprid.

Table 6. False negative results (FN).

Lab code	Azoxystrobin	Chlorfenvinphos	Chlorpyrifos-methyl	Clothianidin	Epoxiconazole	Fluopyram	Isocarbophos	Lambda-cyhalothrin	Lindane	Metolachlor	Metribuzin	Propiconazole	Spiromesifen	Triticonazole
214							FN						FN	
226				FN										
235										FN				
242													FN	
249					FN									
250										FN				
254														FN
256							FN							
269	FN							FN				FN		FN
278			FN						FN					
279			FN											
280		FN						FN						
283									FN					
298													FN	
306											FN			
314							FN				FN			
319	FN													
329					FN		FN	FN						
334									FN					
336								FN						
338						FN	FN							
354	FN											FN		
372	FN				FN			FN						FN
373										FN				
377														FN
386							FN							
406		FN					FN							

[1] The laboratory reported after the deadline that the sample extract had not been injected correctly.

3.2 Assigned values and target standard deviations

The Assigned Values was 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. The results from this EUPT on maize did not show the same dependency of adding water, maybe because all pesticides were spiked. However, it was decided by the Advisory Group that the assigned value should still not include results from laboratories not adding water. 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 were in the range of 16-28 % but on average the Qn-RSD was 19 %, and thus close to 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 datapoint equal to the number of results used to calculate the assigned value (number of results in **Table 8**)

Table 7. Assigned values, the 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	0.050	0.001	25	19
Carbendazim	0.01	0.452	0.015	25	28
Chlorfenvinphos	0.01	0.048	0.001	25	18
Chlorpyrifos-methyl	0.01	0.054	0.001	25	18
Clothianidin	0.01	0.429	0.011	25	21
Epoxiconazole	0.01	0.050	0.001	25	18
Fluopyram	0.01	0.092	0.002	25	17
Isocarbophos	0.01	0.081	0.002	25	16
Lambda-cyhalothrin	0.01	0.088	0.002	25	20
Lindane	0.01	0.070	0.002	25	19
Metolachlor	0.01	0.085	0.002	25	18
Metribuzin	0.01	0.355	0.008	25	17
Pendimethalin	0.01	0.044	0.001	25	19
Propiconazole	0.01	0.141	0.003	25	19
Spiromesifen	0.01	0.072	0.002	25	22
Terbuthylazine	0.01	0.086	0.002	25	17
Thiacloprid	0.01	0.087	0.002	25	20
Triticonazole	0.01	0.082	0.002	25	18

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 10a-j** 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. The unacceptable z-scores include the false negatives.

	No. of reported results	Assigned values	Acceptable	Questionable	Unacceptable[1]	False negatives[1]
Azoxystrobin	128	0.050	120	3	5	4
Carbendazim	108	0.452	98	5	5	0
Chlorfenvinphos	125	0.048	120	2	3	2
Chlorpyrifos-methyl	131	0.054	120	8	3	2
Clothianidin	102	0.429	97	3	2	1
Epoxiconazole	118	0.050	114	1	3	3
Fluopyram	87	0.092	86	0	1	1
Isocarbophos	88	0.081	79	2	7	7
Lambda-cyhalothrin	129	0.088	121	2	6	5
Lindane	133	0.070	121	9	3	3
Metolachlor	96	0.085	89	1	6	4
Metribuzin	102	0.355	98	0	4	2
Pendimethalin	127	0.044	120	6	1	0
Propiconazole	126	0.141	121	1	4	2
Spiromesifen	88	0.072	83	1	4	3
Terbuthylazine	109	0.086	106	1	2	0
Thiacloprid	108	0.087	104	1	3	0
Triticonazole	107	0.082	98	4	5	4

[1] Unacceptable z-scores include the false negative results.

For epoxiconazole, fluopyram, isocarbophos, lambda-cyhalothrin, metolachlor, propiconazole and triticonazole acceptable results were obtained by 96-99% of the laboratories. For azoxystrobin, carbendazim, chlorfenvinphos, chlorpyrifos-methyl, clothianidin, lindane, metribuzin, pendimethalin, spiromesifen, terbuthylazine and thiacloprid acceptable results were obtained by 91-94% of the laboratories.

The Guidance document on analytical quality control and validation procedures for pesticide residues analysis in food and feed, SANCO/12571/2013 [7] recommends the addition of water to the samples prior to extraction to improve the extraction efficiency of low moisture containing commodities like cereals (paragraph C7). As described above, the result from this PT shows no correlation between adding water and improved extraction efficiency, probably because the pesticides residues were spiked and not incurred.

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 procedure 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) and the Acetate buffered (AOAC Official Method 2007.01). It was used for 5% and 4% of the results, respectively. The SweEt method (NMKL 195, 2013) was used for 3%, Mini Luke 7%, the German S-19 method 2% of the results. Finally 16% of the results were analysed by other methods and for 4% of the results no information was given by the laboratories.

Almost 90% of the results derived from a method where water was added before extraction, for 6% of the results no information was given. Likewise, no information was reported concerning the use of ISTD for 9% of the results, while 53% of the results was produced with the use of ISTD and 38% with nonuse. Finally, noo information on the instrument used was reported for 5% of the results. Of the rest 50% was analysed by GC and 50% by LC.

Table 9a. Results for azoxystrobin, carbendazim, chlorfenvinphos, chlorpyrifos-methyl, clothianidin, epoxiconazole, fluopyram, isocarbophos and lambda-cyhalothrin in mg/kg, the corresponding z-scores, MRRLs and the assigned values.

Laboratory code	Azoxystrobin	Z-scores (FFP RSD (25%)	Carbendazim	Z-scores (FFP RSD (25%)	Chlorfenvinphos	Z-scores (FFP RSD (25%)	Chlorpyrifos-methyl	Z-scores (FFP RSD (25%)	Clothianidin	Z-scores (FFP RSD (25%)	Epoxiconazole	Z-scores (FFP RSD (25%)	Fluopyram	Z-scores (FFP RSD (25%)	Isocarbophos	Z-scores (FFP RSD (25%)	Lambda-cyhalothrin	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	**0.050**		**0.452**		**0.048**		**0.054**		**0.429**		**0.050**		**0.092**		**0.081**		**0.088**	
212	0.0398	-0.8	0.293	-1.4	0.0412	-0.6	0.042	-0.9	0.328	-0.9	0.0392	-0.9	0.0807	-0.5			0.0756	-0.6
213	0.0485	-0.1	0.422	-0.3	0.0411	-0.6	0.045	-0.6	0.489	0.6	0.0509	0.0	0.0889	-0.1	0.074	-0.3	0.0823	-0.3
214	0.014	-2.9	0.292	-1.4	0.011	-3.1	0.045	-0.7	0.242	-1.7	0.028	-1.8			FN	-3.5		
215	0.042	-0.7	0.542	0.8	0.054	0.5	0.052	-0.1	0.41	-0.2	0.053	0.2	0.092	0.0	0.072	-0.4	0.087	0.0
216	0.0475	-0.2	0.368	-0.7	0.0492	0.1	0.052	-0.1	0.36	-0.6	0.0432	-0.6	0.0789	-0.6	0.073	-0.3	0.089	0.0
217	0.047	-0.3	0.317	-1.2	0.042	-0.5	0.056	0.1	0.32	-1.0	0.038	-1.0	0.079	-0.6	0.088	0.4	0.087	0.0
221	0.0489	-0.1			0.061	1.1	0.063	0.7			0.0538	0.3					0.109	1.0
222	0.05	0.0	0.49	0.3	0.054	0.5	0.054	0.0	0.53	0.9			0.093	0.0	0.085	0.2	0.09	0.1
223	0.0479	-0.2	0.375	-0.7	0.049	0.1	0.039	-1.1	0.406	-0.2	0.0519	0.1					0.0853	-0.1
224	0.054	0.3	0.39	-0.5	0.044	-0.3	0.052	-0.1	0.371	-0.5	0.05	0.0	0.065	-1.2	0.06	-1.0	0.085	-0.1
225	0.045	-0.4	0.355	-0.9	0.046	-0.2	0.05	-0.3	0.395	-0.3	0.045	-0.4	0.084	-0.3	0.062	-0.9	0.09	0.1
226	0.075	2.0	0.37	-0.7	0.039	-0.8	0.044	-0.7	FN	-3.9	0.042	-0.7	0.084	-0.3	0.069	-0.6	0.19	4.6
227																		
228	0.059	0.7	0.485	0.3	0.053	0.4	0.069	1.1	0.64	2.0	0.065	1.2	0.097	0.2	0.089	0.4	0.047	-1.9
229	0.0439	-0.5	0.396	-0.5	0.0392	-0.7	0.043	-0.8	0.513	0.8	0.0486	-0.1	0.114	1.0	0.079	-0.1	0.0943	0.3
230	0.0479	-0.2			0.0574	0.8	0.065	0.8			0.0495	-0.1					0.1056	0.8
231	0.056	0.5	0.422	-0.3	0.045	-0.3	0.05	-0.3	0.358	-0.7	0.046	-0.3	0.083	-0.4	0.088	0.4	0.08	-0.4
232	0.035	-1.2	0.3	-1.3	0.038	-0.8	0.037	-1.3	0.41	-0.2	0.042	-0.7					0.079	-0.4
233	0.047	-0.3	0.424	-0.2	0.053	0.4	0.057	0.2	0.402	-0.3	0.039	-0.9	0.079	-0.6	0.068	-0.6	0.073	-0.7
234	0.059	0.7	0.456	0.0	0.058	0.8	0.057	0.2	0.477	0.4	0.056	0.5	0.097	0.2	0.088	0.4	0.101	0.6
235	0.0405	-0.8	0.377	-0.7	0.0346	-1.1	0.045	-0.7	0.213	-2.0	0.043	-0.6	0.07	-1.0	0.061	-1.0	0.0777	-0.5
236	0.0506	0.0	0.831	3.4	0.0597	1.0	0.069	1.1	0.63	1.9	0.0675	1.4	0.119	1.2	0.136	2.7	0.0991	0.5
237	0.058	0.6	0.568	1.0	0.049	0.1	0.046	-0.6	0.544	1.1	0.056	0.5	0.082	-0.4	0.079	-0.1	0.092	0.2
238							0.039	-1.1										
239	0.0552	0.4			0.0608	1.1	0.059	0.4			0.0522	0.2					0.0937	0.3
240	0.06	0.8	0.398	-0.5	0.055	0.6	0.069	1.1	0.506	0.7	0.058	0.6	0.113	0.9	0.095	0.7	0.096	0.4
241	0.062	0.9	0.678	2.0	0.0599	1.0	0.060	0.5	0.435	0.1	0.0627	1.0	0.0925	0.0	0.096	0.8	0.124	1.6
242	0.062	0.9	0.54	0.8	0.061	1.1	0.054	0.0	0.505	0.7	0.066	1.2	0.109	0.7	0.074	-0.3	0.061	-1.2
243																		
244	0.041	-0.7	0.483	0.3	0.044	-0.3	0.038	-1.2	0.24	-1.8	0.048	-0.2	0.05	-1.8	0.059	-1.1	0.093	0.2
245	0.0563	0.5	0.386	-0.6	0.0377	-0.9	0.067	1.0	0.445	0.2	0.0577	0.6	0.103	0.5	0.096	0.8	0.0905	0.1
246	0.05	0.0	0.39	-0.5	0.04	-0.7	0.04	-1.0			0.05	0.0			0.07	-0.5	0.08	-0.4
247	0.057	0.5	0.275	-1.6	0.051	0.2	0.054	0.0	0.356	-0.7	0.062	0.9	0.1	0.3			0.075	-0.6
248																		
249	0.041	-0.7	0.391	-0.5	0.039	-0.8	0.042	-0.9	0.318	-1.0	FN	-3.2	0.088	-0.2	0.072	-0.4	0.064	-1.1
250	0.0609	0.8	0.672	2.0	0.0467	-0.1	0.046	-0.6	0.531	1.0	0.0545	0.3	0.121	1.3	0.061	-1.0	0.0744	-0.6
251																		
252	0.0519	0.1	0.401	-0.4	0.047	-0.1	0.052	-0.1	0.413	-0.1	0.0471	-0.3	0.0738	-0.8	0.072	-0.4	0.077	-0.5
253	0.0558	0.4	0.44	-0.1	0.0472	-0.1	0.055	0.1	0.43	0.0	0.0554	0.4	0.095	0.1	0.082	0.1	0.0928	0.2
254	0.052	0.1	0.516	0.6	0.038	-0.8	0.047	-0.5	0.492	0.6	0.042	-0.7	0.084	-0.3	0.094	0.7	0.08	-0.4
255	0.034	-1.3	0.201	-2.2	0.037	-0.9	0.043	-0.8	0.183	-2.3	0.05	0.0					0.083	-0.2

Table 9b. Results for lindane, metolachlor, metribuzin, pendimethalin,propiconazole, spiromesifen, terbuthylazine, thiacloprid and triticonazole in mg/kg, the corresponding z-scores, MRRLs and the assigned values.

Laboratory code	Lindane	Z-scores (FFP RSD (25%)	Metolachlor	Z-scores (FFP RSD (25%)	Metribuzin	Z-scores (FFP RSD (25%)	Pendimethalin	Z-scores (FFP RSD (25%)	Propiconazole	Z-scores (FFP RSD (25%)	Spiromesifen	Z-scores (FFP RSD (25%)	Terbuthylazine	Z-scores (FFP RSD (25%)	Thiacloprid	Z-scores (FFP RSD (25%)	Triticonazole	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	**0.070**		**0.085**		**0.355**		**0.044**		**0.141**		**0.072**		**0.086**		**0.087**		**0.082**	
212	0.062	-0.4	0.066	-0.9	0.333	-0.3	0.034	-0.9	0.115	-0.7	0.0584	-0.8	0.0658	-0.9	0.069	-0.8	0.0676	-0.7
213	0.0699	0.0	0.083	0.0	0.338	-0.2	0.039	-0.4	0.157	0.5	0.0658	-0.3	0.0639	-1.0	0.101	0.6	0.101	0.9
214			0.021	-3.0			0.029	-1.4	0.082	-1.7	FN	-3.4	0.048	-1.8	0.032	-2.5	0.045	-1.8
215	0.062	-0.4	0.103	0.9	0.196	-1.8	0.041	-0.3	0.13	-0.3	0.071	-0.1	0.083	-0.1	0.087	0.0	0.081	0.0
216			0.086	0.1	0.29	-0.7	0.047	0.3	0.143	0.1	0.0602	-0.7	0.0783	-0.3	0.101	0.6	0.0743	-0.4
217	0.072	0.1	0.086	0.1	0.341	-0.2	0.051	0.6	0.142	0.0	0.074	0.1	0.088	0.1	0.078	-0.4	0.082	0.0
221	0.0548	-0.8					0.046	0.2	0.136	-0.1							0.0482	-1.6
222	0.055	-0.8	0.089	0.2	0.294	-0.7	0.051	0.6	0.159	0.5	0.075	0.2	0.09	0.2	0.069	-0.8	0.07	-0.6
223	0.0621	-0.4	0.083	-0.1	0.384	0.3			0.161	0.6	0.0793	0.4	0.788	32.8	0.081	-0.3	0.081	0.0
224	0.075	0.3	0.054	-1.5	0.337	-0.2	0.046	0.2	0.121	-0.6			0.08	-0.3	0.061	-1.2	0.09	0.4
225	0.067	-0.1	0.092	0.3	0.333	-0.3	0.042	-0.2	0.125	-0.5	0.056	-0.9	0.079	-0.3	0.081	-0.3	0.074	-0.4
226	0.068	-0.1	0.067	-0.8	0.33	-0.3	0.043	-0.1	0.096	-1.3	0.065	-0.4	0.07	-0.7	0.088	0.1	0.075	-0.3
227	0.06	-0.5																
228	0.083	0.8	0.095	0.5	0.499	1.6	0.051	0.6	0.204	1.8	0.1	1.5	0.103	0.8	0.101	0.6	0.082	0.0
229	0.112	2.4	0.069	-0.7	0.325	-0.3	0.033	-0.9	0.12	-0.6	0.0913	1.1	0.0865	0.0	0.093	0.3	0.084	0.1
230	0.0616	-0.5					0.048	0.4	0.132	-0.2							0.051	-1.5
231	0.062	-0.4	0.083	-0.1	0.367	0.1	0.042	-0.2	0.128	-0.4	0.075	0.2	0.076	-0.5	0.084	-0.1	0.072	-0.5
232	0.042	-1.6	0.075	-0.5	0.24	-1.3	0.032	-1.1	0.124	-0.5	0.027	-2.5	0.075	-0.5	0.071	-0.7	0.065	-0.8
233	0.091	1.2	0.076	-0.4	0.33	-0.3	0.048	0.4	0.141	0.0	0.067	-0.3	0.066	-0.9	0.086	0.0	0.078	-0.2
234	0.076	0.4	0.095	0.5	0.396	0.5	0.05	0.5	0.16	0.5	0.067	-0.3	0.086	0.0	0.093	0.3	0.089	0.4
235	0.0618	-0.4	FN	-3.5	0.265	-1.0	0.036	-0.7	0.108	-0.9	0.0564	-0.9	0.084	-0.1	0.076	-0.5	0.0646	-0.8
236	0.0952	1.5	0.13	2.1	0.48	1.4	0.064	1.9	0.138	-0.1	0.102	1.7	0.15	3.0	0.107	0.9	0.115	1.6
237	0.067	-0.1	0.098	0.6	0.366	0.1	0.043	-0.1	0.159	0.5	0.074	0.1	0.089	0.2	0.093	0.3	0.087	0.3
238	0.0516	-1.0																
239	0.0534	-0.9					0.048	0.4	0.145	0.1							0.0454	-1.8
240	0.092	1.3	0.107	1.1	0.385	0.3	0.055	1.0	0.206	1.8	0.095	1.3	0.105	0.9	0.109	1.0	0.099	0.8
241	0.08	0.6	0.088	0.2	0.417	0.7	0.048	0.4	0.187	1.3	0.0833	0.6	0.099	0.6	0.106	0.9	0.104	1.1
242	0.086	0.9	0.107	1.1	0.819	>5	0.058	1.3	0.175	1.0	FN	-3.4	0.094	0.4	0.1	0.6	0.099	0.8
243																		
244	0.07	0.0	0.082	-0.1	0.326	-0.3	0.036	-0.7	0.033	-3.1	0.1	1.5	0.08	-0.3	0.082	-0.2	0.081	0.0
245	0.0794	0.6	0.094	0.5	0.399	0.5	0.048	0.4	0.148	0.2	0.0825	0.6	0.1	0.7	0.062	-1.1	0.0873	0.3
246	0.07	0.0	0.07	-0.7	0.22	-1.5	0.04	-0.4	0.12	-0.6	0.07	-0.1	0.08	-0.3			0.08	-0.1
247	0.076	0.4	0.091	0.3	0.27	-1.0	0.032	-1.1	0.143	0.1	0.067	-0.3	0.089	0.2	0.064	-1.1	0.077	-0.2
248	0.05	-1.1																
249	0.059	-0.6	0.073	-0.6	0.298	-0.6	0.038	-0.6	0.12	-0.6	0.036	-2.0	0.071	-0.7	0.07	-0.8	0.071	-0.5
250	0.0581	-0.7	FN	-3.5	0.314	-0.5	0.042	-0.1	0.124	-0.5	0.0817	0.5	0.0965	0.5	0.095	0.4	0.0916	0.5
251	0.0609	-0.5																
252	0.0518	-1.0	0.067	-0.8	0.372	0.2	0.040	-0.3	0.121	-0.6	0.0626	-0.5	0.0593	-1.2	0.072	-0.6	0.0701	-0.6
253	0.0584	-0.6	0.092	0.4	0.381	0.3	0.040	-0.4	0.156	0.4	0.0835	0.6	0.0897	0.2	0.096	0.4	0.0866	0.2
254	0.063	-0.4	0.086	0.1	0.356	0.0	0.038	-0.6	0.112	-0.8	0.058	-0.8	0.085	0.0	0.091	0.2	FN	-3.5
255	0.062	-0.4	0.059	-1.2	0.137	-2.5	0.056	1.1	0.163	0.6					0.024	-2.9	0.071	-0.5

Table 10a. Results for azoxystrobin, carbendazim, chlorfenvinphos, chlorpyrifos-methyl, clothianidin, epoxiconazole, fluopyram, isocarbophos and lambda-cyhalothrin in mg/kg, the corresponding z-scores, MRRLs and the assigned values.

Laboratory code	Azoxystrobin	Z-scores (FFP RSD (25%)	Carbendazim	Z-scores (FFP RSD (25%)	Chlorfenvinphos	Z-scores (FFP RSD (25%)	Chlorpyrifos-methyl	Z-scores (FFP RSD (25%)	Clothianidin	Z-scores (FFP RSD (25%)	Epoxiconazole	Z-scores (FFP RSD (25%)	Fluopyram	Z-scores (FFP RSD (25%)	Isocarbophos	Z-scores (FFP RSD (25%)	Lambda-cyhalothrin	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	**0.050**		**0.452**		**0.048**		**0.054**		**0.429**		**0.050**		**0.092**		**0.081**		**0.088**	
256	0.045	-0.4	0.458	0.1	0.049	0.1	0.051	-0.2	0.323	-1.0	0.049	-0.1	0.087	-0.2	FN	-3.5	0.087	0.0
257	0.0409	-0.7			0.0413	-0.6	0.044	-0.7			0.043	-0.6					0.114	1.2
258	0.0482	-0.2	0.544	0.8	0.0494	0.1	0.052	-0.1	0.423	-0.1	0.0467	-0.3	0.0853	-0.3	0.094	0.7	0.0509	-1.7
259	0.0426	-0.6	0.354	-0.9	0.0423	-0.5	0.049	-0.3	0.395	-0.3	0.0502	0.0	0.0806	-0.5	0.067	-0.6	0.0823	-0.3
260	0.052	0.1	0.4	-0.5	0.047	-0.1	0.062	0.6	0.418	-0.1	0.05	0.0	0.086	-0.3	0.08	0.0	0.093	0.2
261																	0.09	0.1
262	0.055	0.4	0.538	0.8	0.0602	1.0	0.063	0.7	0.438	0.1	0.058	0.6	0.1	0.3	0.093	0.6	0.0751	-0.6
263	0.0375	-1.0	0.72	2.4	0.0415	-0.5	0.042	-0.9	0.372	-0.5	0.038	-1.0	0.071	-0.9	0.06	-1.0	0.07	-0.8
265	0.059	0.7	0.504	0.5	0.046	-0.2	0.067	1.0	0.468	0.4	0.049	-0.1	0.092	0.0	0.062	-0.9	0.112	1.1
267	0.038	-1.0	0.537	0.8	0.0485	0.0	0.054	0.0	0.496	0.6	0.045	-0.4	0.0745	-0.8			0.0855	-0.1
268	0.0524	0.2	0.595	1.3	0.0557	0.6	0.062	0.7	0.52	0.9	0.0543	0.3	0.0959	0.2	0.104	1.2	0.107	0.9
269	FN	-3.2	0.48	0.3	0.07	1.8	0.16	>5	0.36	-0.6	0.043	-0.6					FN	-3.5
271	0.066	1.3	0.538	0.8	0.063	1.2	0.063	0.7	0.606	1.7	0.062	0.9	0.105	0.6	0.095	0.7	0.082	-0.3
272	0.062	0.9	0.406	-0.4	0.06	1.0	0.058	0.3	0.494	0.6	0.058	0.6	0.116	1.0			0.091	0.1
273	0.038	-1.0	0.297	-1.4	0.054	0.5	0.044	-0.7	0.561	1.2	0.051	0.1	0.085	-0.3	0.102	1.1	0.069	-0.9
274	0.0508	0.0	0.65	1.8	0.0482	0.0	0.055	0.1	0.602	1.6	0.0496	-0.1	0.125	1.4	0.087	0.4	0.0846	-0.2
275	0.042	-0.7	0.39	-0.5	0.0459	-0.2	0.048	-0.4	0.376	-0.5	0.0433	-0.6	0.0769	-0.7	0.065	-0.8	0.085	-0.1
276	0.056	0.5	0.315	-1.2	0.039	-0.8	0.042	-0.9	0.16	-2.5	0.037	-1.1					0.064	-1.1
277	0.0664	1.3	0.426	-0.2	0.044	-0.3	0.052	-0.1	0.523	0.9	0.0593	0.7					0.073	-0.7
278	0.0638	1.1	0.293	-1.4	0.0413	-0.6	FN	-3.3										
279	0.0429	-0.6	0.375	-0.7	0.0417	-0.5	FN	-3.3	0.361	-0.6	0.0403	-0.8	0.0702	-0.9	0.064	-0.8	0.0704	-0.8
280	0.048	-0.2	0.37	-0.7	FN	-3.2	0.039	-1.1	0.12	-2.9	0.052	0.1			0.087	0.3	FN	-3.5
281	0.0533	0.2	0.502	0.4	0.0449	-0.3	0.052	-0.1	0.461	0.3	0.0535	0.3	0.0911	0.0	0.070	-0.5	0.072	-0.7
282	0.044	-0.5	0.467	0.1	0.045	-0.3	0.047	-0.5	0.416	-0.1	0.048	-0.2	0.082	-0.4	0.073	-0.4	0.117	1.3
283	0.0481	-0.2	0.597	1.3	0.0491	0.1	0.053	0.0	0.41	-0.2	0.0477	-0.2	0.0897	-0.1	0.081	0.1	0.0872	0.0
284	0.052	0.1	0.415	-0.3	0.0499	0.2	0.053	-0.1	0.4	-0.3	0.0478	-0.2	0.0834	-0.4	0.072	-0.4	0.0799	-0.4
285	0.0512	0.1	0.581	1.1	0.0417	-0.5	0.058	0.3	0.453	0.2	0.0511	0.1	0.0986	0.3	0.082	0.1	0.0922	0.2
287	0.052	0.1					0.061	0.5			0.046	-0.3					0.104	0.7
288	0.058	0.6	0.237	-1.9	0.049	0.1	0.06	0.4	0.382	-0.4			0.085	-0.3			0.095	0.3
289																		
290	0.0545	0.3	0.489	0.3	0.0479	0.0	0.059	0.4	0.579	1.4	0.0565	0.5	0.125	1.4	0.097	0.8	0.0894	0.1
291	0.064	1.1	0.97	4.6	0.053	0.4	0.065	0.8	0.404	-0.2	0.053	0.2	0.092	0.0	0.088	0.4	0.095	0.3
293	0.0396	-0.8	0.298	-1.4	0.0396	-0.7	0.04	-1.0	0.317	-1.0	0.0376	-1.0					0.0723	-0.7
294	0.0555	0.4	0.53	0.7	0.0528	0.4	0.061	0.5	0.491	0.6	0.0582	0.6	0.102	0.4	0.084	0.2	0.0997	0.5
295					0.052	0.3	0.050	-0.3									0.101	0.6
296					FN	-3.2	0.038	-1.2									0.092	0.2
297	0.0515	0.1			0.0589	0.9	0.059	0.4			0.0523	0.2					0.0984	0.5
298	0.051	0.1	0.443	-0.1	0.054	0.5	0.057	0.2	0.598	1.6	0.044	-0.5	0.088	-0.2	0.071	-0.5	0.113	1.1
299	0.048	-0.2					0.056	0.1									0.068	-0.9
300	0.0487	-0.1	0.331	-1.1	0.0404	-0.6	0.045	-0.7	0.416	-0.1	0.0451	-0.4	0.0886	-0.1	0.076	-0.2	0.128	1.8
301	0.0399	-0.8	0.845	3.5	0.0351	-1.1	0.047	-0.5	0.384	-0.4	0.0441	-0.5	0.0848	-0.3	0.081	0.0	0.0974	0.4

Table 10b. Results for lindane, metolachlor, metribuzin, pendimethalin,propiconazole, spiromesifen, terbuthylazine, thiacloprid and triticonazole in mg/kg, the corresponding z-scores, MRRLs and the assigned values.

Laboratory code	Lindane	Z-scores (FFP RSD (25%)	Metolachlor	Z-scores (FFP RSD (25%)	Metribuzin	Z-scores (FFP RSD (25%)	Pendimethalin	Z-scores (FFP RSD (25%)	Propiconazole	Z-scores (FFP RSD (25%)	Spiromesifen	Z-scores (FFP RSD (25%)	Terbuthylazine	Z-scores (FFP RSD (25%)	Thiacloprid	Z-scores (FFP RSD (25%)	Triticonazole	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	**0.070**		**0.085**		**0.355**		**0.044**		**0.141**		**0.072**		**0.086**		**0.087**		**0.082**	
256	0.066	-0.2	0.079	-0.3	0.32	-0.4	0.041	-0.3	0.131	-0.3			0.082	-0.2	0.083	-0.2	0.084	0.1
257	0.0605	-0.5	0.068	-0.8	0.285	-0.8	0.038	-0.5	0.118	-0.7			0.105	0.9			0.0818	0.0
258	0.0703	0.0	0.078	-0.3	0.31	-0.5	0.054	0.9	0.137	-0.1	0.0671	-0.3	0.0861	0.0	0.078	-0.4	0.0798	-0.1
259	0.0695	0.0	0.078	-0.3	0.298	-0.6	0.040	-0.4	0.137	-0.1	0.0657	-0.4	0.0757	-0.5	0.084	-0.1	0.0895	0.4
260	0.079	0.5			0.344	-0.1	0.053	0.8	0.143	0.1	0.058	-0.8	0.081	-0.2	0.092	0.2	0.09	0.4
261	0.081	0.7											0.043	-2.0				
262	0.0838	0.8	0.097	0.6	0.388	0.4	0.048	0.4	0.161	0.6	0.1	1.5	0.092	0.3	0.19	4.7	0.091	0.5
263	0.062	-0.4	0.050	-1.6	0.307	-0.5	0.030	-1.2	0.122	-0.5	0.039	-1.8	0.087	0.1	0.07	-0.8	0.058	-1.2
265	0.082	0.7	0.069	-0.7	0.288	-0.8	0.049	0.4	0.147	0.2	0.089	0.9	0.095	0.4	0.094	0.3	0.076	-0.3
267	0.079	0.5	0.079	-0.2	0.429	0.8	0.047	0.3	0.135	-0.2	0.0825	0.6	0.0698	-0.7	0.090	0.2	0.0733	-0.4
268	0.0659	-0.2	0.097	0.6	0.424	0.8	0.056	1.1	0.149	0.2	0.0858	0.8	0.0929	0.3	0.099	0.6	0.0927	0.5
269							0.06	1.4	FN	-3.7					0.074	-0.6	FN	-3.5
271	0.056	-0.8	0.094	0.4	0.389	0.4	0.048	0.4	0.159	0.5	0.094	1.2	0.081	-0.2	0.108	1.0	0.098	0.8
272	0.081	0.7	0.112	1.3			0.053	0.8	0.159	0.5	0.093	1.2	0.106	0.9	0.107	0.9	0.102	1.0
273	0.067	-0.1	0.075	-0.5	0.404	0.5	0.039	-0.5	0.139	-0.1	0.07	-0.1	0.077	-0.4	0.082	-0.2	0.101	0.9
274	0.0651	-0.3	0.078	-0.3	0.342	-0.1	0.045	0.1	0.126	-0.4	0.0725	0.0	0.0854	0.0	0.122	1.6	0.0868	0.3
275	0.066	-0.2	0.076	-0.4	0.339	-0.2	0.042	-0.2	0.129	-0.3	0.069	-0.2	0.096	0.5	0.080	-0.3	0.0684	-0.6
276	0.06	-0.5	0.075	-0.5	0.369	0.2	0.035	-0.8	0.096	-1.3			0.061	-1.2	0.069	-0.8	0.053	-1.4
277	0.066	-0.2			0.367	0.1	0.041	-0.3	0.194	1.5			0.102	0.8	0.114	1.2		
278	FN	-3.4	0.095	0.5	0.358	0.0	0.049	0.5	0.21	2.0			0.101	0.7	0.104	0.8		
279	0.0632	-0.4	0.069	-0.7	0.325	-0.3	0.046	0.2	0.125	-0.5	0.068	-0.2	0.0751	-0.5	0.076	-0.5	0.0646	-0.8
280							0.041	-0.3	0.149	0.2	0.0626	-0.5	0.0656	-0.9	0.083	-0.2	0.082	0.0
281	0.072	0.1	0.069	-0.7	0.341	-0.2	0.045	0.1	0.14	0.0	0.0653	-0.4	0.0839	-0.1	0.094	0.3	0.0809	0.0
282	0.07	0.0	0.08	-0.2	0.349	-0.1	0.047	0.3	0.122	-0.5	0.078	0.3	0.08	-0.3	0.082	-0.2	0.076	-0.3
283	FN	-3.4	0.088	0.2	0.307	-0.5	0.047	0.3	0.127	-0.4	0.0846	0.7	0.0794	-0.3	0.074	-0.6	0.0695	-0.6
284	0.053	-1.0	0.069	-0.7	0.364	0.1	0.040	-0.3	0.128	-0.4	0.0602	-0.7	0.0749	-0.5	0.079	-0.4	0.0694	-0.6
285	0.0754	0.3	0.087	0.1	0.353	0.0	0.043	0.0	0.161	0.6	0.0796	0.4	0.0827	-0.1	0.085	-0.1	0.0867	0.2
287	0.078	0.5	0.083	-0.1			0.054	0.9	0.139	-0.1			0.084	-0.1				
288	0.075	0.3			0.371	0.2	0.045	0.1	0.178	1.1			0.11	1.1	0.081	-0.3		
289																		
290	0.0737	0.2	0.085	0.0	0.411	0.6	0.057	1.2	0.148	0.2	0.0685	-0.2	0.0881	0.1	0.11	1.1	0.0886	0.3
291	0.091	1.2	0.095	0.5	0.371	0.2	0.05	0.5	0.158	0.5	0.075	0.2	0.085	0.0	0.087	0.0	0.084	0.1
293	0.0611	-0.5	0.068	-0.8	0.304	-0.6	0.031	-1.2	0.109	-0.9			0.065	-1.0	0.057	-1.4	0.0572	-1.2
294	0.0773	0.4	0.104	0.9	0.403	0.5	0.043	-0.1	0.171	0.9	0.0848	0.7	0.098	0.6	0.102	0.7	0.0952	0.7
295	0.0679	-0.1					0.037	-0.6										
296	0.0395	-1.7			0.211	-1.6												
297	0.0521	-1.0					0.051	0.6	0.142	0.0							0.0511	-1.5
298	0.115	2.6					0.043	-0.1	0.13	-0.3	FN	-3.4	0.078	-0.4	0.098	0.5	0.081	0.0
299	0.053	-1.0			0.373	0.2	0.036	-0.7	0.28	3.9								
300	0.0715	0.1	0.083	0.0	0.32	-0.4	0.037	-0.6	0.13	-0.3	0.0737	0.1	0.0745	-0.5	0.081	-0.3	0.0799	-0.1
301	0.0783	0.5	0.071	-0.6	0.483	1.4	0.034	-0.9	0.137	-0.1	0.065	-0.4	0.0835	-0.1	0.068	-0.9	0.0722	-0.5

Table 11a. Results for azoxystrobin, carbendazim, chlorfenvinphos, chlorpyrifos-methyl, clothianidin, epoxiconazole, fluopyram, isocarbophos and lambda-cyhalothrin in mg/kg, the corresponding z-scores, MRRLs and the assigned values.

Laboratory code	Azoxystrobin	Z-scores (FFP RSD (25%)	Carbendazim	Z-scores (FFP RSD (25%)	Chlorfenvinphos	Z-scores (FFP RSD (25%)	Chlorpyrifos-methyl	Z-scores (FFP RSD (25%)	Clothianidin	Z-scores (FFP RSD (25%)	Epoxiconazole	Z-scores (FFP RSD (25%)	Fluopyram	Z-scores (FFP RSD (25%)	Isocarbophos	Z-scores (FFP RSD (25%)	Lambda-cyhalothrin	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	**0.050**		**0.452**		**0.048**		**0.054**		**0.429**		**0.050**		**0.092**		**0.081**		**0.088**	
302	0.0691	1.5	0.666	1.9	0.0643	1.4	0.074	1.5	0.582	1.4	0.064	1.1					0.13	1.9
303	0.032	-1.5	0.9	4.0	0.023	-2.1	0.03	-1.8	0.5	0.7	0.035	-1.2					0.06	-1.3
304	0.045	-0.4	0.38	-0.6	0.046	-0.2	0.051	-0.2	0.496	0.6	0.043	-0.6	0.082	-0.4	0.08	0.0	0.085	-0.1
305	0.038	-1.0	0.561	1.0	0.037	-0.9	0.049	-0.4	0.454	0.2	0.037	-1.1	0.099	0.3	0.055	-1.3	0.072	-0.7
306	0.067	1.3	0.34	-1.0	0.06	1.0	0.06	0.4	0.432	0.0	0.055	0.4	0.113	0.9	0.105	1.2	0.082	-0.3
307	0.059	0.7	1.032	>5	0.056	0.7	0.065	0.8	0.601	1.6	0.056	0.5	0.11	0.8	0.095	0.7	0.097	0.4
308																		
309	0.069	1.5	0.49	0.3	0.059	0.9	0.061	0.5	0.519	0.8	0.062	0.9	0.108	0.7	0.1	1.0	0.081	-0.3
310	0.0538	0.3	0.442	-0.1	0.0398	-0.7	0.056	0.2	0.452	0.2	0.0556	0.4	0.0935	0.1	0.095	0.8	0.1	0.5
311	0.0435	-0.5			0.0536	0.5	0.056	0.2			0.0417	-0.7						
312	0.0467	-0.3	0.57	1.0	0.0508	0.2	0.054	0.1	0.421	-0.1	0.0526	0.2						
313																	0.097	0.4
314	0.0529	0.2	0.692	2.1	0.0497	0.1	0.074	1.5	0.396	-0.3	0.0401	-0.8	0.0949	0.1	FN	-3.5	0.123	1.6
315							0.054	0.1									0.0894	0.1
317																		
318	0.0372	-1.0	0.385	-0.6	0.0391	-0.7	0.046	-0.6	0.305	-1.2	0.0371	-1.0	0.0643	-1.2	0.067	-0.6	0.059	-1.3
319	FN	-3.2			0.0466	-0.1	0.053	0.0									0.0851	-0.1
320	0.043	-0.6	0.347	-0.9	0.04	-0.7	0.044	-0.7	0.329	-0.9	0.057	0.5	0.087	-0.2	0.073	-0.4	0.096	0.4
321	0.0451	-0.4	0.488	0.3	0.0431	-0.4	0.042	-0.9	0.415	-0.1	0.054	0.3	0.0851	-0.3			0.103	0.7
323			0.616	1.5														
324	0.0483	-0.2			0.0489	0.1	0.038	-1.1			0.0491	-0.1	0.0958	0.2	0.078	-0.1	0.0713	-0.8
325	0.0547	0.4			0.0602	1.0	0.061	0.6			0.0527	0.2					0.105	0.8
326	0.0518	0.1			0.0433	-0.4	0.058	0.3									0.101	0.6
327	0.0548	0.4	0.522	0.6	0.0508	0.2	0.058	0.3	0.466	0.3	0.0567	0.5	0.0937	0.1	0.082	0.1	0.103	0.7
328	0.0559	0.5	1.108	>5	0.0451	-0.2	0.041	-0.9	0.547	1.1	0.0568	0.5	0.0854	-0.3	0.081	0.0	0.158	3.2
329	0.055	0.4	0.146	-2.7	0.03	-1.5	0.082	2.1	0.512	0.8	FN	-3.2	0.116	1.0	FN	-3.5	FN	-3.5
330	0.051	0.1	0.45	0.0	0.05	0.2	0.054	0.0			0.048	-0.2	0.093	0.0	0.072	-0.4	0.132	2.0
331	0.0396	-0.8	0.318	-1.2	0.0231	-2.1	0.043	-0.8	0.325	-1.0	0.0353	-1.2	0.0657	-1.1	0.035	-2.2	0.0466	-1.9
332	0.0331	-1.4	0.41	-0.4	0.0275	-1.7			0.274	-1.4	0.0347	-1.2	0.0614	-1.3	0.082	0.1	0.0532	-1.6
333							0.015	-2.9										
334	0.054	0.3	0.567	1.0			0.050	-0.2	0.481	0.5	0.054	0.3						
335	0.0688	1.5			0.0402	-0.7	0.062	0.6			0.0645	1.1					0.0966	0.4
336	0.051	0.1			0.055	0.6	0.066	0.9			0.062	0.9					FN	-3.5
337	0.046	-0.3			0.061	1.1	0.05	-0.3			0.048	-0.2					0.113	1.1
338	0.038	-1.0	0.558	0.9	0.046	-0.2	0.051	-0.2	0.511	0.8	0.049	-0.1	FN	-3.6	FN	-3.5	0.06	-1.3
339	0.06	0.8	0.535	0.7	0.063	1.2	0.066	0.9	0.389	-0.4	0.064	1.1	0.099	0.3	0.081	0.0	0.099	0.5
340	0.0555	0.4	0.445	-0.1	0.072	2.0	0.071	1.3	0.343	-0.8	0.0625	1.0	0.0929	0.0	0.088	0.4	0.106	0.8
341	0.05	0.0	0.538	0.8			0.077	1.7	0.324	-1.0	0.0432	-0.6					0.122	1.5
342	0.046	-0.3			0.051	0.2	0.048	-0.4			0.043	-0.6					0.098	0.5
343	0.0469	-0.3	0.413	-0.3	0.0459	-0.2	0.058	0.3	0.362	-0.6	0.0488	-0.1	0.0708	-0.9	0.092	0.6	0.0815	-0.3
344	0.0602	0.8	0.489	0.3	0.0443	-0.3	0.058	0.3	0.494	0.6	0.06	0.8	0.103	0.5	0.082	0.1	0.112	1.1

Table 11b. Results for lindane, metolachlor, metribuzin, pendimethalin,propiconazole, spiromesifen, terbuthylazine, thiacloprid and triticonazole in mg/kg, the corresponding z-scores, MRRLs and the assigned values.

Laboratory code	Lindane	Z-scores (FFP RSD (25%)	Metolachlor	Z-scores (FFP RSD (25%)	Metribuzin	Z-scores (FFP RSD (25%)	Pendimethalin	Z-scores (FFP RSD (25%)	Propiconazole	Z-scores (FFP RSD (25%)	Spiromesifen	Z-scores (FFP RSD (25%)	Terbuthylazine	Z-scores (FFP RSD (25%)	Thiacloprid	Z-scores (FFP RSD (25%)	Triticonazole	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	**0.070**		**0.085**		**0.355**		**0.044**		**0.141**		**0.072**		**0.086**		**0.087**		**0.082**	
302	0.0763	0.4																
303	0.039	-1.8			0.3	-0.6	0.024	-1.8	0.091	-1.4					0.086	0.0		
304	0.07	0.0	0.082	-0.1	0.349	-0.1	0.046	0.2	0.15	0.3	0.06	-0.7	0.085	0.0	0.098	0.5	0.078	-0.2
305	0.064	-0.3	0.093	0.4	0.331	-0.3	0.036	-0.7	0.112	-0.8	0.07	-0.1	0.099	0.6	0.09	0.1	0.081	0.0
306	0.09	1.2	0.11	1.2	FN	-3.9	0.05	0.5	0.07	-2.0	0.066	-0.3	0.11	1.1	0.106	0.9		
307	0.083	0.8	0.119	1.6	0.51	1.7	0.07	2.3	0.18	1.1	0.134	3.4	0.096	0.5	0.156	3.2	0.098	0.8
308																		
309	0.052	-1.0	0.102	0.8	0.421	0.7	0.052	0.7	0.17	0.8	0.098	1.4	0.1	0.7	0.101	0.6	0.108	1.3
310	0.0808	0.6	0.095	0.5	0.365	0.1	0.051	0.7	0.164	0.7	0.0968	1.4	0.1	0.7	0.103	0.7	0.086	0.2
311	0.0714	0.1					0.037	-0.6							0.074	-0.6		
312	0.0754	0.3							0.154	0.4					0.094	0.4	0.0781	-0.2
313	0.053	-1.0																
314	0.0812	0.7			FN	-3.9	0.043	0.0	0.136	-0.1	0.068	-0.2	0.109	1.1	0.098	0.5	0.0849	0.2
315	0.0609	-0.5																
317																		
318	0.0591	-0.6	0.061	-1.1	0.286	-0.8	0.032	-1.0	0.12	-0.6	0.0628	-0.5	0.0692	-0.8	0.064	-1.0	0.0615	-1.0
319	0.064	-0.3			0.352	0.0			0.191	1.4			0.077	-0.4				
320	0.059	-0.6	0.077	-0.4	0.393	0.4	0.036	-0.7	0.154	0.4	0.064	-0.4	0.073	-0.6	0.083	-0.2	0.074	-0.4
321	0.0793	0.6	0.086	0.1	0.332	-0.3	0.035	-0.8	0.152	0.3			0.0756	-0.5	0.070	-0.8	0.0884	0.3
323																		
324	0.0662	-0.2	0.087	0.1	0.316	-0.4	0.037	-0.6	0.134	-0.2	0.0614	-0.6						
325	0.0521	-1.0					0.048	0.4	0.146	0.1							0.0451	-1.8
326	0.0838	0.8			0.436	0.9	0.051	0.7	0.126	-0.4			0.104	0.9				
327	0.0775	0.5	0.088	0.2	0.411	0.6	0.047	0.3	0.158	0.5	0.0731	0.1	0.0928	0.3	0.096	0.4	0.0879	0.3
328	0.0747	0.3	FN	-3.5	0.501	1.6	0.046	0.2	0.144	0.1	0.0738	0.1	0.0886	0.1	0.098	0.5	0.0985	0.8
329	0.085	0.9	0.128	2.0	0.425	0.8	0.075	2.8	0.133	-0.2	0.061	-0.6	0.138	2.4	0.12	1.5	0.127	2.2
330	0.071	0.1	0.081	-0.2	0.334	-0.2	0.028	-1.5	0.137	-0.1			0.08	-0.3	0.07	-0.8	0.07	-0.6
331	0.0323	-2.1	0.075	-0.4	0.281	-0.8	0.038	-0.6	0.103	-1.1	0.0384	-1.9	0.0685	-0.8	0.074	-0.6	0.0665	-0.7
332	0.0523	-1.0	0.061	-1.1	0.275	-0.9	0.035	-0.8	0.104	-1.0	0.0442	-1.5	0.0647	-1.0	0.059	-1.3	0.0329	-2.4
333	0.019	-2.9																
334	FN	-3.4					0.045	0.1	0.144	0.1	0.056	-0.9			0.098	0.5	0.081	0.0
335					0.37	0.2	0.047	0.3	0.204	1.8			0.066	-0.9				
336	0.094	1.4					0.053	0.8	0.186	1.3			0.094	0.4	0.109	1.0	0.097	0.8
337	0.06	-0.5					0.049	0.4	0.14	0.0							0.058	-1.2
338	0.061	-0.5	0.042	-2.0	0.394	0.4	0.027	-1.6	0.122	-0.5	0.051	-1.2	0.082	-0.2	0.076	-0.5	0.08	-0.1
339	0.09	1.2			0.4	0.5	0.049	0.4	0.19	1.4			0.103	0.8	0.091	0.2	0.091	0.5
340	0.0947	1.4	0.096	0.6	0.435	0.9	0.061	1.6	0.16	0.5	0.08	0.4	0.112	1.2	0.084	-0.1	0.103	1.0
341	0.0661	-0.2					0.035	-0.8	0.128	-0.4					0.066	-1.0		
342	0.047	-1.3					0.042	-0.2	0.11	-0.9								
343	0.0512	-1.1	0.067	-0.8	0.406	0.6	0.041	-0.2	0.117	-0.7	0.0551	-0.9	0.0579	-1.3	0.071	-0.7	0.0641	-0.9
344	0.0809	0.7	0.096	0.6	0.422	0.8	0.051	0.7	0.157	0.5	0.0848	0.7	0.0944	0.4	0.105	0.8	0.0927	0.5

Table 12a. Results for azoxystrobin, carbendazim, chlorfenvinphos, chlorpyrifos-methyl, clothianidin, epoxiconazole, fluopyram, isocarbophos and lambda-cyhalothrin in mg/kg, the corresponding z-scores, MRRLs and the assigned values.

Laboratory code	Azoxystrobin	Z-scores (FFP RSD (25%)	Carbendazim	Z-scores (FFP RSD (25%)	Chlorfenvinphos	Z-scores (FFP RSD (25%)	Chlorpyrifos-methyl	Z-scores (FFP RSD (25%)	Clothianidin	Z-scores (FFP RSD (25%)	Epoxiconazole	Z-scores (FFP RSD (25%)	Fluopyram	Z-scores (FFP RSD (25%)	Isocarbophos	Z-scores (FFP RSD (25%)	Lambda-cyhalothrin	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	**0.050**		**0.452**		**0.048**		**0.054**		**0.429**		**0.050**		**0.092**		**0.081**		**0.088**	
345					0.0488	0.1	0.052	-0.1									0.0861	-0.1
346	0.049	-0.1	0.416	-0.3	0.046	-0.2	0.054	0.0			0.052	0.1	0.089	-0.1	0.081	0.0	0.085	-0.1
347	0.0449	-0.4	0.333	-1.1	0.045	-0.3	0.045	-0.7									0.095	0.3
348					0.0504	0.2	0.059	0.4									0.0838	-0.2
349	0.053	0.2	0.517	0.6	0.06	1.0	0.08	1.9	0.449	0.2	0.051	0.1					0.092	0.2
350	0.0395	-0.9	0.341	-1.0	0.0347	-1.1	0.046	-0.6	0.307	-1.1	0.0454	-0.4	0.0712	-0.9	0.056	-1.2	0.0836	-0.2
351	0.051	0.1	0.424	-0.2	0.058	0.8	0.052	-0.1	0.458	0.3	0.051	0.1			0.068	-0.6	0.081	-0.3
352	0.046	-0.3	0.538	0.8	0.05	0.2	0.048	-0.4	0.46	0.3			0.082	-0.4	0.084	0.2	0.082	-0.3
353																		
354	FN	-3.2			0.0463	-0.1	0.082	2.1									0.0237	-2.9
355	0.0609	0.8	0.416	-0.3	0.0596	1.0	0.062	0.6	0.475	0.4	0.0594	0.7	0.0983	0.3	0.079	-0.1	0.102	0.6
356	0.0435	-0.5	0.334	-1.0	0.037	-0.9	0.046	-0.6	0.351	-0.7	0.0415	-0.7					0.114	1.2
360																		
361	0.058	0.6	0.15	-2.7	0.046	-0.2	0.059	0.4	0.392	-0.3	0.042	-0.7	0.081	-0.5	0.095	0.7	0.08	-0.4
362	0.0541	0.3	0.42	-0.3	0.0411	-0.6	0.048	-0.4	0.369	-0.6	0.0531	0.2	0.0932	0.1	0.079	-0.1	0.0941	0.3
363	0.0472	-0.2	0.485	0.3	0.0442	-0.3	0.056	0.2	0.485	0.5	0.0562	0.5	0.095	0.1	0.077	-0.2	0.0991	0.5
364																		
365	0.0351	-1.2	0.349	-0.9	0.0384	-0.8	0.042	-0.8	0.4	-0.3	0.0486	-0.1	0.127	1.5	0.054	-1.3	0.0547	-1.5
366	0.019	-2.5	0.35	-0.9	0.041	-0.6	0.04	-1.0									0.081	-0.3
367					0.0716	2.0												
368	0.0639	1.1			0.047	-0.1	0.055	0.1	0.41	-0.2	0.0642	1.1	0.122	1.3	0.067	-0.6	0.104	0.7
369			0.295	-1.4	0.064	1.3	0.097	3.2					0.278	>5			0.113	1.1
372	FN	-3.2			0.0518	0.3	0.048	-0.4			FN	-3.2	0.0711	-0.9	0.082	0.1	FN	-3.5
373	0.029	-1.7	0.204	-2.2	0.0496	0.1	0.054	0.1	0.272	-1.5	0.0528	0.2					0.0743	-0.6
374	0.108	4.6															0.06	-1.3
375																		
376	0.054	0.3			0.0713	1.9	0.065	0.9			0.0482	-0.2					0.0948	0.3
377			0.499	0.4					0.181	-2.3	0.0386	-0.9						
378	0.0511	0.1	0.536	0.7	0.0443	-0.3	0.039	-1.1	0.545	1.1	0.0525	0.2	0.0961	0.2	0.068	-0.6	0.1	0.5
379	0.0554	0.4	0.581	1.1	0.0427	-0.4	0.056	0.2	0.477	0.4	0.0564	0.5	0.101	0.4	0.083	0.1	0.0872	0.0
380																		
381	0.03	-1.6	0.36	-0.8	0.056	0.7	0.06	0.4			0.022	-2.3					0.086	-0.1
382							0.057	0.3										
384	0.0281	-1.8			0.04	-0.7	0.038	-1.2							0.08	0.0	0.131	2.0
385	0.0481	-0.2	0.628	1.6	0.051	0.2	0.067	1.0			0.0505	0.0			0.084	0.2	0.105	0.8
386	0.0478	-0.2	0.668	1.9	0.0512	0.3	0.054	0.0	0.491	0.6	0.0425	-0.6	0.068	-1.0	FN	-3.5	0.0761	-0.5
387	0.035	-1.2	0.29	-1.4	0.035	-1.1	0.044	-0.7	0.38	-0.5	0.03	-1.6			0.065	-0.8	0.075	-0.6
406	0.021	-2.3	0.396	-0.5	FN	-3.2			0.077	-3.3	0.031	-1.5	0.05	-1.8	FN	-3.5	0.031	-2.6
407	0.0356	-1.2																
408	0.0796	2.3					0.074	1.5	0.372	-0.5							0.0786	-0.4

Table 12b. Results for lindane, metolachlor, metribuzin, pendimethalin,propiconazole, spiromesifen, terbuthylazine, thiacloprid and triticonazole in mg/kg, the corresponding z-scores, MRRLs and the assigned values.

Laboratory code	Lindane	Z-scores (FFP RSD (25%)	Metolachlor	Z-scores (FFP RSD (25%)	Metribuzin	Z-scores (FFP RSD (25%)	Pendimethalin	Z-scores (FFP RSD (25%)	Propiconazole	Z-scores (FFP RSD (25%)	Spiromesifen	Z-scores (FFP RSD (25%)	Terbuthylazine	Z-scores (FFP RSD (25%)	Thiacloprid	Z-scores (FFP RSD (25%)	Triticonazole	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	**0.070**		**0.085**		**0.355**		**0.044**		**0.141**		**0.072**		**0.086**		**0.087**		**0.082**	
345	0.0609	-0.5					0.046	0.2										
346	0.054	-0.9					0.041	-0.3	0.137	-0.1	0.072	0.0	0.082	-0.2				
347	0.0582	-0.7			0.263	-1.0	0.043	-0.1	0.131	-0.3					0.081	-0.3		
348	0.0757	0.4					0.038	-0.5							0.092	0.2		
349	0.09	1.2	0.11	1.2	0.22	-1.5	0.045	0.1	0.151	0.3	0.078	0.3	0.085	0.0	0.091	0.2	0.074	-0.4
350	0.0575	-0.7	0.069	-0.7	0.341	-0.2	0.043	0.0	0.109	-0.9	0.0698	-0.1	0.08	-0.3	0.076	-0.5	0.0703	-0.6
351	0.089	1.1	0.066	-0.9	0.302	-0.6	0.043	-0.1	0.125	-0.5			0.07	-0.7	0.074	-0.6		
352	0.058	-0.7	0.084	0.0	0.31	-0.5	0.042	-0.2	0.14	0.0	0.082	0.5	0.08	-0.3	0.075	-0.5	0.078	-0.2
353	0.0794	0.6																
354	0.0842	0.8	0.052	-1.5	0.27	-1.0	0.108	>5	FN	-3.7			0.0613	-1.1				
355	0.0848	0.9	0.092	0.4	0.421	0.7	0.048	0.4	0.149	0.2	0.0828	0.6	0.096	0.5	0.11	1.1	0.09	0.4
356	0.0675	-0.1					0.033	-1.0	0.113	-0.8					0.067	-0.9	0.0655	-0.8
360																		
361	0.061	-0.5	0.084	0.0	0.299	-0.6	0.04	-0.4	0.138	-0.1	0.094	1.2	0.085	0.0	0.087	0.0	0.033	-2.4
362	0.075	0.3	0.081	-0.2	0.378	0.3	0.041	-0.3	0.149	0.2	0.0681	-0.2	0.0802	-0.3	0.088	0.1	0.0753	-0.3
363	0.0723	0.2	0.082	-0.1	0.3312	-0.3	0.049	0.5	0.157	0.5	0.0724	0.0	0.0752	-0.5	0.105	0.8	0.098	0.8
364																		
365	0.0637	-0.3	0.103	0.9	0.301	-0.6	0.047	0.3	0.134	-0.2	0.1	1.5	0.0739	-0.5	0.080	-0.3	0.0864	0.2
366	0.049	-1.2	0.067	-0.8	0.278	-0.9	0.028	-1.5	0.095	-1.3	FN	-3.4			0.075	-0.5		
367																		
368	0.0629	-0.4	0.087	0.1	0.379	0.3	0.042	-0.2	0.203	1.8			0.107	1.0	0.079	-0.3	0.0915	0.5
369	0.063	-0.4			0.255	-1.1	0.057	1.2			0.033	-2.2			0.081	-0.3		
372	0.0728	0.2	0.087	0.2	0.243	-1.3	0.042	-0.2	0.070	-2.0			0.0779	-0.4			FN	-3.5
373			FN	-3.5			0.039	-0.4	0.159	0.5			0.0779	-0.4	0.057	-1.4	0.049	-1.6
374	0.091	1.2	0.2	>5			0.055	1.0									0.198	>5
375																		
376	0.0581	-0.7							0.111	-0.9								
377							0.045	0.1	0.146	0.1			0.0724	-0.6	0.391	>5	FN	-3.5
378	0.105	2.0	0.053	-1.5	0.407	0.6	0.047	0.3	0.15	0.3	0.086	0.8	0.0711	-0.7	0.095	0.4	0.0798	-0.1
379	0.0784	0.5	0.054	-1.4	0.377	0.2	0.045	0.1	0.134	-0.2	0.0711	-0.1	0.0973	0.5	0.101	0.6	0.0955	0.7
380																		
381	0.061	-0.5			0.282	-0.8	0.028	-1.5	0.12	-0.6								
382	0.0569	-0.7																
384			0.084	0.0	0.176	-2.0			0.169	0.8	0.04	-1.8	0.126	1.9				
385	0.0797	0.6			0.962	>5	0.062	1.7	0.145	0.1	0.0837	0.6	0.0931	0.3	0.085	0.0	0.0857	0.2
386	0.0635	-0.3	0.087	0.1	0.414	0.7	0.042	-0.1	0.129	-0.3	0.0896	1.0	0.0995	0.6	0.061	-1.2	0.0946	0.6
387	0.06	-0.5	0.08	-0.2	0.3	-0.6	0.047	0.3	0.104	-1.0	0.08	0.4	0.075	-0.5	0.051	-1.7	0.08	-0.1
406	0.029	-2.3					0.023	-1.9	0.025	-3.3	0.047	-1.4	0.044	-1.9	0.05	-1.7	0.136	2.7
407					0.332	-0.3			0.118	-0.7								
408			0.103	0.9			0.060	1.5	0.184	1.2			0.096	0.5	0.103	0.7		

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 pesticides present in the Test Item (≥16 pesticide residues, exclusive of any false negatives results) and also report no false positive results. For the 80 EU and EFTA laboratories in Category A (57%), the results were additionally evaluated by calculating the Average of the Squared -Score (AZ^2). Of the 80 participants, 77 participants (96%) obtained AZ^2 values at or below 2 (good) and 1 participant (1%) obtained AZ^2 values between 2-3 (satisfactory) and 2 participants (3%) obtained AZ^2 values ≥3 (unsatisfactory). An additional four laboratories from Third Countries were evaluated and classified into Category A. The AZ^2 scores achieved by the labs can be seen in **Table 13.**

Table 13 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 pesticides	AZ^2	False negative	Classification	NRL
212	17	0.6	0	Good	
213	18	0.2	0	Good	
215	18	0.3	0	Good	
216	17	0.2	0	Good	
217	18	0.3	0	Good	
222	17	0.2	0	Good	NRL
224	17	0.4	0	Good	
225	18	0.2	0	Good	NRL
228	18	1.2	0	Good	
229	18	0.7	0	Good	NRL
231	18	0.1	0	Good	
232	16	1.2	0	Good	
233	18	0.3	0	Good	
234	18	0.2	0	Good	NRL
235	17	0.8	1	Good	NRL
236	18	3.1	0	Unsatisfactory	
237	18	0.2	0	Good	
240	18	0.9	0	Good	
241	18	0.9	0	Good	
242	17	2.2	1	Satisfactory	
244	18	1.2	0	Good	
245	18	0.4	0	Good	
249	17	0.7	1	Good	
250	17	0.6	1	Good	
252	18	0.3	0	Good	
253	18	0.1	0	Good	
254	17	0.3	1	Good	NRL
258	18	0.3	0	Good	NRL
259	18	0.2	0	Good	NRL
260	17	0.2	0	Good	
262	18	1.7	0	Good	

Lab code	No. of detected pesticides	AZ^2	False negative	Classification	NRL
263	18	1.2	0	Good	
265	18	0.4	0	Good	
267	17	0.3	0	Good	
268	18	0.5	0	Good	
271	18	0.7	0	Good	
272	16	0.7	0	Good	
273	18	0.5	0	Good	NRL
274	18	0.6	0	Good	NRL
275	18	0.2	0	Good	NRL
279	17	0.4	1	Good	NRL
281	18	0.1	0	Good	
282	18	0.2	0	Good	
284	18	0.2	0	Good	NRL
285	18	0.1	0	Good	
290	18	0.5	0	Good	NRL
291	18	1.4	0	Good	NRL
294	18	0.3	0	Good	
298	15	0.8	1	Good	
300	18	0.4	0	Good	
301	18	1.1	0	Good	NRL
304	18	0.1	0	Good	NRL
305	18	0.5	0	Good	
306	16	1.0	1	Good	
307	18	3.7	0	Unsatisfactory	NRL
309	18	0.8	0	Good	
310	18	0.3	0	Good	
314	15	0.8	2	Good	
318	18	0.8	0	Good	NRL
320	18	0.3	0	Good	NRL
321	16	0.2	0	Good	
327	18	0.2	0	Good	
328	17	2.5	1	Satisfactory	
329	15	2.9	3	Satisfactory	
330	16	0.5	0	Good	NRL
331	18	1.7	0	Good	
332	17	1.7	0	Good	
338	16	0.8	2	Good	
339	16	0.6	0	Good	
340	18	0.9	0	Good	
343	18	0.5	0	Good	
344	18	0.4	0	Good	
350	18	0.5	0	Good	NRL

Lab code	No. of detected pesticides	AZ^2	False negative	Classification	NRL
352	17	0.2	0	Good	
355	18	0.4	0	Good	
361	18	0.9	0	Good	
362	18	0.1	0	Good	
363	18	0.2	0	Good	NRL
365	18	0.8	0	Good	NRL
368	16	0.6	0	Good	
378	18	0.7	0	Good	NRL
379	18	0.3	0	Good	
386	17	0.6	1	Good	
387	17	0.8	0	Good	

The laboratories that did not fulfil the requirement described above, was evaluated to be classified in Category B. The number of reported results and acceptable z-scores as well as information on false negative and false positive results are show in **Table 14**. Of the 61 EU and EFTA participants 5 labs was moved to Category B because of false positive results. Forty-one participants (29%) analysed and detected less than 70% of the pesticides present in the Test Item.

Table 14 Number of pesticides analysed, number of acceptable z-scores, false negative and positive for the laboratories in Category B.

Lab code	No. of detected pesticides	No. of acceptable	False negative	False positive	NRL
214	12	11	2	1	
221	9	9	0	0	
223	15	14	0	0	
226[1]	17	16	1	1	
227	1	1	0	0	
230	9	9	0	0	
238	2	2	0	0	
239	9	9	0	0	
243	0	0	0	0	
246	15	15	0	0	
247[1]	17	17	0	1	
248	1	1	0	0	
251	1	1	0	0	
255	14	14	0	0	
256[1]	16	16	1	1	NRL
257	12	12	0	0	
261	3	3	0	0	
269	7	6	4	0	NRL
276	15	15	0	0	
277	13	13	0	0	
278	9	9	2	0	
280	12	12	2	0	
283[1]	17	17	1	1	NRL
287	9	9	0	0	
288	13	13	0	0	
293	15	15	0	0	NRL
295	5	5	0	0	
296	4	4	1	0	
297	9	9	0	0	
299	7	6	0	0	
302	8	8	0	0	NRL
303	12	11	0	0	
311	7	7	0	0	
312	10	10	0	0	
313	2	2	0	0	
315	3	3	0	0	

Lab code	No. of detected pesticides	No. of acceptable	False negative	False positive	NRL
319	7	7	1	0	
323	1	1	0	0	
324	13	13	0	0	
325	9	9	0	0	
326	9	9	0	0	
333	2	2	0	0	
334	10	10	1	1	
335	9	9	0	0	
336	10	10	1	0	
337	9	9	0	0	
341	10	10	0	0	
342	8	8	0	0	
345	5	5	0	0	NRL
346	13	13	0	0	
347	10	10	0	0	NRL
348	6	6	0	0	
349[1]	16	16	0	1	
351	15	15	0	0	
353	1	1	0	0	
354	8	7	2	0	
356	12	12	0	0	
366	11	11	1	0	
367	1	1	0	3	
369	10	8	0	2	
372	10	10	4	0	
373	12	12	1	0	
374	6	3	0	2	
376	7	7	0	0	
377	7	6	1	0	NRL
381	10	10	0	0	NRL
382	2	2	0	1	
384	10	10	0	1	
385	15	14	0	0	
406	13	11	2	1	NRL
407	3	3	0	0	
408	9	9	0	0	

[1] Laboratory moved from Category A to Category B due to a false positive result.

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-C2 in 2008 72 labs participated and in the latest EUPT-CF9 143 labs participated. (**Table 15**). The number of pesticides included in the Target Pesticide List has also increased during this 8-year period, from 43 to 117 individual compounds. The number of spiked or incurred pesticides contained in the Test Items has in the same period increased from 13 to 18 (two of them not evaluated in this PT). Thus the demands put on the participating laboratories has increased every year. Many laboratories have a limited scope and are therefore is not able to cover all pesticides in the PT. Of the laboratories submitting results 30 % submitted results for less than 70% of pesticides present in the Test Item.

Table 15. Overall trends in participation, pesticides and performance of laboratories in the 7 latest EUPTs cereals focusing on MRM pesticides.

PT and type of test item	EUPT-C2 Wheat flour	EUPT-C3 Oat flour	EUPT-C4 Rye flour	EUPT-C5 Rice Flour	EUPT-C6 Barley flour	EUPT-CF8 Wheat flour	EUPT-CF9 Maize flour
Participants submitting results (EU and EFTA)	72	102	115	133	127	142	143
MRM pesticides in the Target Pesticide List	43	51	64	103	107	111	117
MRM pesticides in the test material	13	14	16	16	18	17	18
No. of results for MRM pesticides	830	981	1624	1521	1741	1893	2012
Average of 'reported results', %	89	69	88	71	76	78	78
Range of 'reported results', %	60 - 96	48 - 95	55 - 95	41-95	50-95	49-93	61-94
Acceptable z-scores, %	85	87	87	87	90	90	94
Questionable z-scores, %	12	8	6	4	5	6	3
Unacceptable z-scores, %	3	5	7	9	4	4	3
False negatives, %	3	3	4	3	1	3	2
Number of false positives	2	3	17	16	2	4	9
Catagory A, % of participating laboratories	60	46	46	44	55	50	57
Good SWZ/AZ^2, %	70	72	77	80	79	85	96
Satisfactory SWZ/AZ^2, %	9	15	8	15	14	10	1
Unsatisfactory SWZ/AZ^2, %	21	13	15	5	7	4	3

Although improvement was seen in the overall analytical performance (accuracy of measurement) if looking at the percentage of acceptable, questionable, unacceptable z-scores, this is not the case for the analytical scope . The average % of reported results has in the last four EUPT-C/CF been between 70-80%. This is caused by a lot of participants only analysing for less than 50% of the pesticide residues present in the test Item. The false negative results has been relatively low, for the last PT only 2% of the results. The number of false positive results varies from year to year but is <0.5%.

The percentage of Category A laboratories has varied but a general increase is indicated, with 44% in EUPT-C5 to 55% and 50% in EUPT-C6 to EUPT-CF8, respectively and now 57% for EUPT-CF9. For the Category A improvement in AZ^2 was seen in EUPT-CF9 where 96% of the results were Good. However, it is difficult to assess any improvement/deterioration in laboratory performance between the six Proficiency Tests, because the pesticides in the Test item and the laboratories participating in the PTs have both significantly increased.

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

EUPT-CF9 consisted of maize containing spiked pesticides. Although the maize had been sprayed in the field with commercially available pesticide formulations, no residues were found in the test item.

Consequently, the maize were spiked in the laboratory and the final Test Item contained the following pesticides: azoxystrobin, carbendazim, chlorfenvinphos, chlorpyrifos-methyl, clothianidin, epoxiconazole, fluopyram, isocarbophos, lambda-cyhalothrin, lindane, metolachlor, metribuzin, pendimethalin propiconazole, spiromesifen, terbuthylazine, thiacloprid and triticonazole. One hundred and forty-three laboratories, representing 30 EU and EFTA countries submitted results. Four further laboratories registered, but did not submit any results. All NRLs, except one of the two Finish, participated in the PT. Malta was represented in the PT by the NRL for the UK. An additional 16 laboratories from EU candidate states and Third Countries registered for the PT and 13 submitted results. The Target Pesticide List distributed to the laboratories prior to the test contained 117 individual compounds.

The number of false positives and false negatives has varied between the EUPTs. The 9 false positive results in EUPT-CF9 were: bifenthrin, flonicamid, HCH-alpha (3), HCH-beta (3) and thiamethoxam. This relatively small number is in good agreement with the majority of the previous EUPTs. The number of false negatives represented only % of the total number of results. This is also in good agreement with the percentage of false negatives reported in the previous EUPTs. The average Qn-RSD (robust RSD) was at 19 %, close to the FFP-RSD of 25 % with a range from 16 to 28 % for the individual compounds.

For epoxiconazole, fluopyram, isocarbophos, lambda-cyhalothrin, metolachlor, propiconazole and triticonazole acceptable results were obtained by 96-99% of the laboratories. For azoxystrobin, carbendazim, chlorfenvinphos, chlorpyrifos-methyl, clothianidin, lindane, metribuzin, pendimethalin, spiromesifen, terbuthylazine and thiacloprid acceptable results were obtained by 91-94% of the laboratories.

The EUPT-CF10 will be on rye and will be shipped to the laboratories in March 2016. The selection of pesticides will continue to focus on pesticides included in the scope of the EU multi-annual coordinated control programme as well as additional pesticides of relevance to feed and/or cereal production in Europe and in other parts of the world from where significant quantities of feed and cereals are imported.

4. ACKNOWLEDGEMENTS

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5. REFERENCES

[1] Regulation (EC) N° 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 13528:2005 – 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. SANCO/12571/2013. http://www.eurl-pesticides.eu/library/docs/allcrl/AqcGuidance_Sanco_2013_12571.pdf

APPENDICES

Appendix 1 List of laboratories registered to participate in the EUPT-CF8

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	Fytolab - Belgium, Gent (Zwijnaarde)	Gent - Zwijnaarde		Yes
Belgium	Federal Laboratory for Safety of Food Chain, Tervuren	Tervuren		Yes
Bulgaria	SGS - Bulgaria Ltd., Varna	Varna		Yes
Bulgaria	Fytolab - Bulgaria, Plovdiv	Plovdiv		Yes
Bulgaria	Central Laboratory for Chemical Testing and Control, Sofia	Sofia	NRL	Yes
Croatia	Euroinspekt - Croatiakontrola d.o.o.	Zagreb		Yes
Croatia	Institute of Public Health, Split	Dalmatia County, Split		Yes
Croatia	Teaching Institute of Public Health, Dr. Andrija štampar	Zagreb		Yes
Croatia	Croatian National Institute of Public Health	Zagreb	NRL	Yes
Cyprus	Laboratory of Pesticide Residues Analysis, State General Laboratory	Nicosia	NRL	Yes
Cyprus	Animal Feeds and Feed Additives Laboratory of the Analytical Laboratories of the Department of Agriculture	Nicosia	NRL	Yes
Czech Republic	University of Chemical Technology, Dept. of Food Chemistry and Analysis - Prague	Praha		Yes
Czech Republic	Central Institute for Supervising and Testing in Agriculture	Brno	NRL	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
France	Analysis Center Mediterranean Pyrenees	perpignan		Yes
France	Capinov	Landerneau		Yes
France	INOVALYS Le Mans	Le Mans		Yes
France	Service Commun des Laboratoires / Laboratoire de Montpellier	Montpellier		Yes
France	CERECO SUD	GARONS		Yes
France	Laboratoire Départemental d`Analyses de la Drome	Valence		No
France	Service Commun des Laboratoires / Laboratoire Ile de France - Massy	Massy Cedex	NRL	Yes
France	GIRPA - FREDON PAYS DE LA LOIRE	BEAUCOUZE		Yes
Germany	Niedersächsisches Landesamt für Verbraucherschutz und Lebensmittelsicherheit, Stade	Stade		Yes
Germany	Landesamt für Landwirtschaft, Lebensmittelsicherheit und Fischerei Mecklenburg-Vorpommern	Rostock		Yes

Country	Institution	City	NRL-CF	Report data
Germany	Federal Office of Consumer Protection and Food Safety, NRL for Pesticide Residues	Berlin	NRL	Yes
Germany	Chemisches und Veterinäruntersuchungsamt Ostwestfalen-Lippe, Detmold	Detmold		Yes
Germany	LUFA-ITL GmbH	Kiel		Yes
Germany	Food and Veterinary Institute Oldenburg	Oldenburg		Yes
Germany	Bavarian Health and Food Safety Authority Office Erlangen	Erlangen		Yes
Germany	Chemical and Veterinary Analytical Institute Rhine-Ruhr-Wupper	Krefeld		Yes
Germany	Landwirtschaftliches Technologiezentrum Augustenberg, Karlsruhe	Karlsruhe		Yes
Germany	Landesbetrieb Hessisches Landeslabor, Wiesbaden	Wiesbaden		Yes
Germany	Chemisches und Veterinäruntersuchungsamt Rheinland, Standort Bonn	Bonn		Yes
Germany	Thuringian Institute of Agriculture	Jena		Yes
Germany	Landesamt für Verbraucherschutz - Sachsen-Anhalt	Halle/Saale		Yes
Germany	Eurofins - Dr. Specht Laboratorien GmbH	Hamburg		Yes
Germany	Berlin-Brandenburg State Laboratory, Potsdam	Potsdam		Yes
Germany	Landesanstalt für Landwirtschaft, Forsten und Gartenbau, Halle	Halle/Saale		Yes
Germany	Chemical and Veterinary Investigations Office, Stuttgart (CVUAS), (Residues)	Fellbach		Yes
Germany	Landwirtschaftliche Untersuchungs- und Forschungsanstalt Speyer	Speyer		Yes
Germany	State Department of Environmental and Agricultural Operations in Saxony	Nossen		Yes
Germany	State Investigation Institute of Health and Veterinary Saxony	Dresden		Yes
Germany	Landesuntersuchungsamt Institut für Lebensmittelchemie Speyer	Speyer		Yes
Germany	Chemical and Veterinary Analytical Institute Muensterland-Emscher Lippe	Münster		Yes
Germany	State Laboratory Schleswig-Holstein	Neumünster		Yes
Greece	Regional Center of Plant Protection and Quality Control of Ioannina, Pesticide Residues Laboratory	Ioannina		Yes
Greece	Benaki Phytopathological Institute, Pesticide Residues Laboratory	Kifissia	NRL	Yes
Greece	General Chemical State Laboratory, D Division, Pesticide Residues Laboratory	Athens	NRL	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 Analytical Laboratory, Velence	Velence	NRL	Yes
Hungary	Agricultural 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
Ireland	Pesticide Control Laboratory, Department of Agriculture, Fisher-	Co. Kildare	NRL	Yes

Country	Institution	City	NRL-CF	Report data
	ies and Food			
Italy	ARPA Puglia - Dipartimento di Bari	Bari		Yes
Italy	ARPALAZIO, SEZIONE DI RIETI	Rieti		Yes
Italy	ARPAL Sez. di La Spezia	La Spezia		Yes
Italy	APPA Bolzano	Bolzano		Yes
Italy	Laboratorio di Sanità Pubblica ASL BERGAMO	Bergamo		Yes
Italy	Centro di referenza nazionale per la sorveglianza ed il controllo degli alimenti per animali	Genova	NRL	Yes
Italy	Istituto Zooprofilattico Sperimentale Puglia e Basilicata	Foggia		Yes
Italy	Istituto Zooprofilattico dell'Abruzzo e del Molise- Bromatologia	Teramo		Yes
Italy	ARPA Piemonte POLO ALIMENTI	La Loggia (Torino)		Yes
Italy	Istituto Zooprofilattico Sperimentale Lazio e Toscana - Roma	Roma		Yes
Italy	Istituto Zooprofilattico Sperimentale Umbria e Marche, PERUGIA	Perugia		Yes
Italy	Environmental Regional Protection Agency - Laboratory of Pordenone	Pordenone		Yes
Italy	Istituto Zooprofilattico Sperimentale delle Venezie	Legnaro (Padova)		Yes
Italy	APPA Trento Settore Laboratorio	Trento		Yes
Italy	Istituto Zooprofilattico Sperimentale Lombardia ed Emilia Romagna	Brescia		Yes
Italy	ARPA LAZIO	ROME		No
Italy	ARPA Ferrara Eccellenza Fitofarmaci	Ferrara		Yes
Italy	Istituto Superiore di Sanità, Pesticide Section	Roma		No
Italy	Arpa Valle d'Aosta	Saint Christophe		Yes
Italy	Laboratorio di Prevenzione - ASL Provincia di Milano	Milano		Yes
Italy	ARPAB, Dipartimento Provinciale di Potenza - Laboratorio Strumentale	POTENZA		No
Italy	Public Health Laboratory - Florence	Firenze		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	Groen Agro Control	Delfgauw		Yes
Netherlands	Handelslaboratorium Dr. Verwey	Rotterdam		Yes
Netherlands	NVWA - Netherlands Food and Consumer Product Safety Authority (Wageningen, The Netherlands)	Wageningen	NRL	Yes
Netherlands	RIKILT Institute of Food Safety (Natural Toxins & Pesticides)	Wageningen		Yes
Netherlands	NofaLab	Schiedam		Yes
Netherlands	Eurofins Lab Zeeuws-Vlaanderen (LZV) B.V.	Graauw		Yes
Norway	Norwegian Institute for Agricultural and Environmental Research, Plant Health and Plant Protection Division, Pesticide Chemistry Section	Aas	NRL	Yes

Country	Institution	City	NRL-CF	Report data
Poland	Regional Veterinary Laboratory in Gdansk (Kartuska)	Gdansk		Yes
Poland	Voievodship Sanitary - Epidemiological Station in Warsaw	Warsaw	NRL	Yes
Poland	Regional Veterinary Laboratory in Warsaw	Warszaw		Yes
Poland	Regional Veterinary Laboratory in Opole	Opole		Yes
Poland	Institute of Plant Protection - National Research Institute, Branch Sosnicowice	Sosnicowice		Yes
Poland	Institute of Plant Protection - National Research Institute, Regional Experimental Station in Rzeszow	Rzeszow		Yes
Poland	Institute of Plant Protection, Department of Pesticide Residue Research - Poznan	Poznan	NRL	Yes
Poland	Regional Veterinary Laboratory in Bialystok	Bialystok		Yes
Poland	Institute of Horticulture, Food Safety Laboratory (Skierniewice)	Skierniewice		Yes
Poland	Regional Veterinary Laboratory in Szczecin	Szczecin		Yes
Poland	Regional Veterinary Laboratory Wroclaw	Wroclaw		Yes
Poland	Regional Veterinary Laboratory in Katowice	Katowice		Yes
Poland	Voievodship Sanitary - Epidemiological Station in Opole	Opole		Yes
Poland	Provincial Veterinary Inspectorate Establishment of Veterinary Hygiene, Poznan	Poznan		Yes
Poland	Institute of Plant Protection Pesticide Residue Laboratory, Bialystok	Bialystok		Yes
Portugal	Regional Laboratory of Veterinary and Food Safety - Madeira Island	Funchal - Madeira Island		Yes
Portugal	INIAV- Pesticide Residues Laboratory	Oeiras	NRL	Yes
Romania	Central Laboratory for Pesticides Residues Control in Plants and Vegetable Products - Bucharest	Bucharest	NRL	Yes
Romania	Zonal Laboratory for pesticides Residues in feed - Bistrita	Bistrita		Yes
Romania	Sanitary Veterinary and Food Safety Directorate Cluj, Gas-Chromatography Laboratory	Cluj Napoca		Yes
Romania	Institute for Hygiene and Veterinary Public Health - Bucharest	Bucharest	NRL	Yes
Romania	Sanitary Veterinary and Food Safety Directorate, Bucharest	Bucharest		Yes
Romania	National Institute of Public Health, Regional Centre of Public Health Cluj-Napoca	Cluj-Napoca		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 for Health, Environment and Food - Dep. for chem. anal. Maribor	Maribor	NRL	Yes
Slovenia	Agricultural Institute of Slovenia, Central Laboratories	Ljubljana	NRL	Yes
Spain	National Centre for Technology and Food Safety - Laborytory of Ebro	San Adrián (Navarra)		Yes
Spain	Agrofood Laboratory of the Comunidad Valenciana	Burjassot-Valencia		Yes
Spain	Laboratorio de Salud Pública de Badajoz	Badajoz		Yes

Country	Institution	City	NRL-CF	Report data
Spain	National Centre for Food - Spain, Majadahonda	Majadahonda	NRL	Yes
Spain	Laboratorio Agroalimentario de Zaragoza	Zaragoza		Yes
Spain	Laboratorio Agroalimentario de Extremadura (Cáceres)	Cáceres		Yes
Spain	Analytica Alimentaria GmbH Sucursal España	Almeria		Yes
Spain	Laboratorio Agrario Regional - Junta de Castilla y Leon	Burgos		Yes
Spain	Laboratorio Regional CCAA La Rioja	Logroño		Yes
Spain	Laboratory of Barcelona Public Health Agency	Barcelona		Yes
Spain	Navarra de Servicios y Tecnologias, S.A.	Villava		Yes
Spain	Labs & Technological Services AGQ, S.L.	Burguillos (Sevil-la)		Yes
Spain	Agricultural and Phytopathological Laboratory of Galicia	Abegondo. A Coruña		Yes
Spain	Laboratorio de los servicios perifericos de la consejeria de sani-dad y asuntos sociales de Cuenca	Cuenca		Yes
Spain	Laboratorios Ecosur S.A.	Lorqui-Murcia		Yes
Spain	Laboratori Agroalimentari de Cabrils	Cabrils		Yes
Spain	Laboratorio de Producción y Sanidad Vegetal de Jaén	Mengibar (Jaén)		Yes
Spain	Laboratorio Arbitral Agroalimentario, Madrid	Madrid	NRL	Yes
Spain	Toledo, Laboratorio Regional Agroalimentario y Ambiental de Castilla la Mancha (LARAGA)	Toledo		Yes
Spain	Laboratorio de Residuos-Instituto Tecnologico de Canarias, S. A., Departamento de Analisis Ambiental	Agüimes-Las Palmas		Yes
Spain	Laboratorio Kudam, S.L	Torre de la Horadada		Yes
Sweden	Eurofins Food&Feed Testing Sweden AB	Lidköping		Yes
Sweden	National Food Agency, Science Department, Chemistry Division 1	Uppsala	NRL	Yes
Switzerland	Kantonales Laboratorium Zürich	Zürich		Yes
UK	Agri-Food and Biosciences Institute	Belfast		Yes
UK	The Food and Environment Research Agency - York	York	NRL	Yes
UK	Laboratory of the Government Chemist - Teddington	Teddington		Yes

Participating labs from EU candidate state and the 3rd countries

Country	Institution	City	Report data
Albania	Pesticides Residues, Food Safety and Veterinary Institute	Tirana	Yes
Argentina	Complejo de Laboratorios de la Bolsa de Comercio de Rosario	Rosario	No
Australia	Symbio Alliance	Eight Mile Plains, QLD	Yes
Brazil	Eurofins do Brasil Análises de Alimentos Ltda	Indaiatuba	Yes
Brazil	Bioagri Analises de Alimentos Ltda	Sao Paulo	No
Egypt	Central Lab of Residue Analysis of Pesticides and Heavy Metals in Foods	Giz	Yes
Iceland	Matís - Icelandic Food Research	Reykjavík	Yes
India	Pesticide Management Division, National Institute of Plant Health Management	Hyderabad	Yes
India	Pesticide Residues Laboratory	Hyderabad	Yes
Indonesia	Angler BioChemLab, PT.	Surabaya	Yes
Jamaica	University of the West Indies Mona	Kingston	Yes
New Zealand	AsureQuality Ltd - Wellington	Wellington	Yes
Serbia	SP Laboratory	Becej	Yes
Serbia	Center for Food Analysis, Belgrade	Belgrade	Yes
Singapore	Agri-Food & Veterinary Authority of Singapore, Veterinary Public Health Center	Singapore	Yes
Tanzania	Government Chemist Laboratory Agency	Dar es salaam	Yes
Tanzania	Tropical Pesticides Research Institute (TPRI),	Arusha	No
Albania	Pesticides Residues, Food Safety and Veterinary Institute	Tirana	Yes
Argentina	Complejo de Laboratorios de la Bolsa de Comercio de Rosario	Rosario	No
Australia	Symbio Alliance	Eight Mile Plains, QLD	Yes

Appendix 2 Target Pesticide List

Pesticides	MRRL (mg/kg)
Acephate	0.01
Azinphos-methyl	0.01
Azoxystrobin	0.01
Bifenthrin	0.01
Bixafen	0.01
Boscalid	0.01
Carbaryl	0.01
Carbendazim	0.01
Carbofuran	0.01
Carbofuran, 3-hydroxy	0.01
Carboxin	0.01
Chlorfenvinphos	0.01
Chlorpropham (parent compound only)	0.01
Chlorpyrifos	0.02
Chlorpyrifos-methyl	0.02
Clothianidin	0.02
Cyfluthrin (sum of isomers)	0.01
Cypermethrin (sum of isomers)	0.01
Cyproconazole	0.01
Cyprodinil	0.01
Deltamethrin-cis	0.01
Demeton-S-methylsulfone	0.01
Diazinon	0.01
Dichlorvos	0.01
Difenoconazole	0.01
Diflubenzuron	0.01
Dimethoate	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
Fenbuconazole	0.01
Fenhexamid	0.01
Fenitrothion	0.01
Fenpropidin	0.01
Fenpropimorph	0.01
Fenthion	0.01
Fenthion-oxon	0.01

Pesticides	MRRL (mg/kg)
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
Fludioxonil	0.01
Fluopyram	0.01
Fluquinconazole	0.01
Flusilazole	0.01
Flutriafol	0.01
Fluxapyroxad	0.01
HCH-alpha	0.01
HCH-beta	0.01
Hexaconazole	0.01
Imazalil	0.01
Imidacloprid	0.01
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
Metconazole	0.01
Methacrifos	0.01
Methomyl	0.01
Metolachlor	0.01
Metrafenone	0.01
Metribuzin	0.01
DDD- p,p'	0.01
DDE- p,p'	0.01
DDT- o,p'	0.01
DDT- p,p'	0.01
Omethoate	0.01
Oxydemeton-methyl	0.01
Paclobutrazol	0.01
Parathion	0.01

Pesticides	MRRL (mg/kg)
Penconazole	0.01
Pendimethalin	0.01
Permethrin (sum of isomers)	0.01
Phenylphenol-ortho	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
Propiconazole	0.01
Prothioconazole-desthio	0.01
Pyraclostrobin	0.01
Pyrimethanil	0.01
Quinoxyfen	0.01
Spiromesifen	0.01
Spiroxamine	0.01
Tebuconazole	0.01
Tebufenozide	0.01
Terbuthylazine	0.01
Thiabendazole	0.01
Thiacloprid	0.01
Thiamethoxam	0.01
Thiodicarb	0.01
Thiophanate-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

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		Chlorfenvinphos, mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
12	0.049	0.043	0.370	0.358	0.034	0.042
44	0.051	0.044	0.395	0.371	0.038	0.039
73	0.052	0.046	0.398	0.378	0.030	0.045
96	0.054	0.056	0.452	0.434	0.039	0.041
116	0.044	0.052	0.356	0.393	0.043	0.050
140	0.051	0.042	0.360	0.360	0.041	0.037
181	0.057	0.059	0.456	0.452	0.055	0.043
213	0.056	0.056	0.424	0.425	0.036	0.045
260	0.046	0.052	0.398	0.394	0.043	0.032
263	0.053	0.046	0.412	0.402	0.039	0.044

	Chlorpyrifos-methyl, mg/kg		Clothianidin, mg/kg		Epoxiconazole, mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
12	0.034	0.049	0.463	0.454	0.046	0.043
44	0.036	0.046	0.514	0.421	0.052	0.045
73	0.030	0.046	0.508	0.459	0.051	0.045
96	0.034	0.046	0.518	0.536	0.054	0.054
116	0.048	0.060	0.438	0.495	0.043	0.051
140	0.042	0.049	0.440	0.408	0.048	0.042
181	0.059	0.044	0.555	0.584	0.057	0.056
213	0.030	0.050	0.542	0.496	0.054	0.055
260	0.046	0.030	0.462	0.496	0.048	0.049
263	0.039	0.044	0.508	0.476	0.051	0.047

	Fluopyram, mg/kg		Isocarbofos, mg/kg		Lambda-cyhalothrin, mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
12	0.083	0.079	0.058	0.064	0.087	0.092
44	0.087	0.087	0.064	0.070	0.104	0.086
73	0.085	0.082	0.051	0.074	0.072	0.104
96	0.098	0.097	0.061	0.070	0.098	0.092
116	0.079	0.087	0.072	0.086	0.096	0.123
140	0.084	0.080	0.067	0.060	0.112	0.095
181	0.105	0.101	0.090	0.075	0.134	0.108
213	0.095	0.099	0.062	0.074	0.093	0.103
260	0.087	0.090	0.072	0.050	0.098	0.094
263	0.090	0.089	0.063	0.070	0.100	0.099

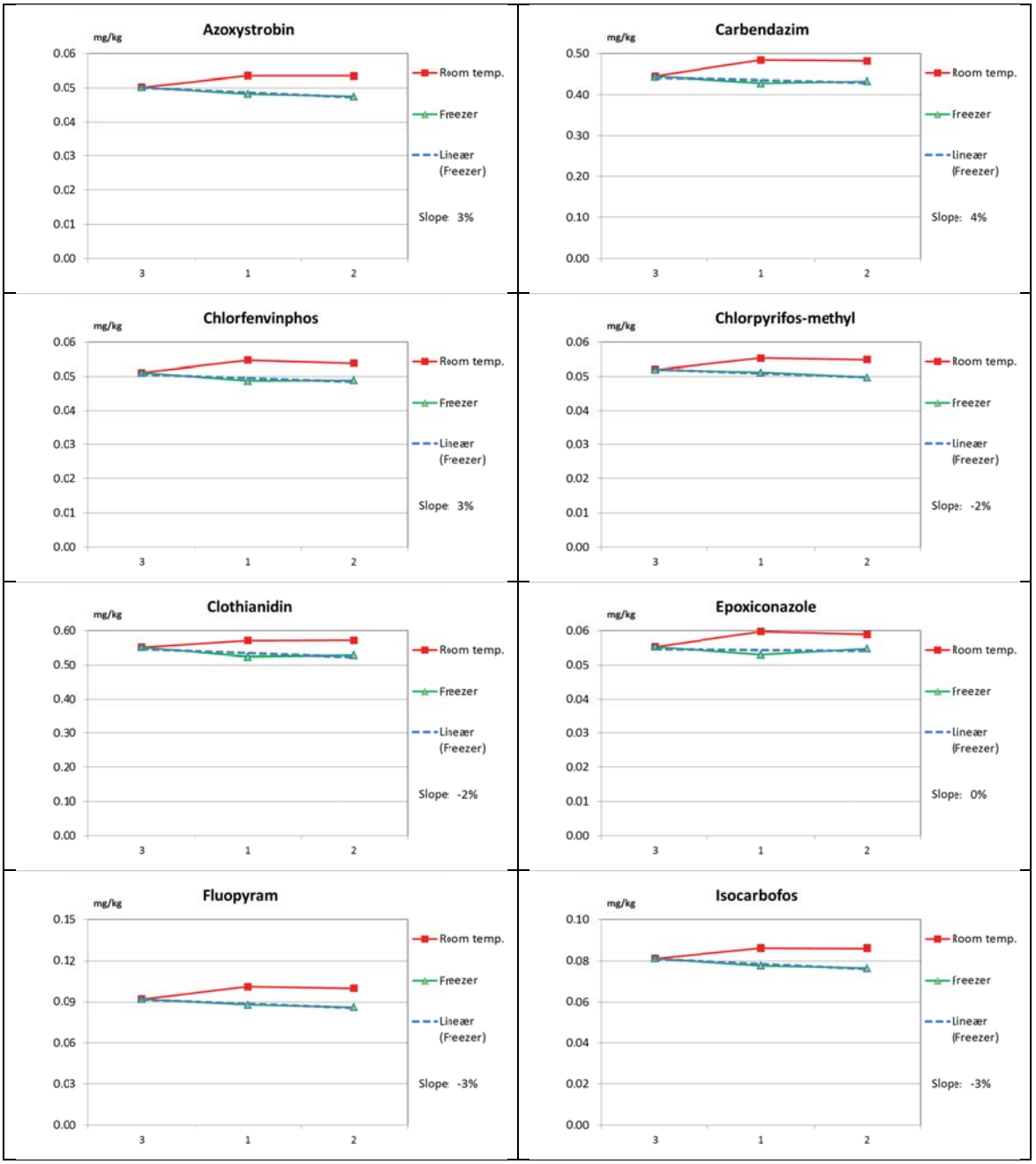
	Lindane, mg/kg		Metolachlor, mg/kg		Metribuzin,, mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
12	0.051	0.064	0.061	0.071	0.344	0.312
44	0.045	0.054	0.063	0.065	0.353	0.323
73	0.043	0.067	0.049	0.078	0.350	0.316
96	0.052	0.063	0.062	0.075	0.366	0.382
116	0.064	0.082	0.077	0.092	0.318	0.360
140	0.053	0.071	0.071	0.075	0.328	0.314
181	0.085	0.064	0.097	0.076	0.423	0.388
213	0.038	0.069	0.053	0.077	0.385	0.376
260	0.059	0.040	0.073	0.053	0.336	0.345
263	0.053	0.064	0.067	0.075	0.353	0.347

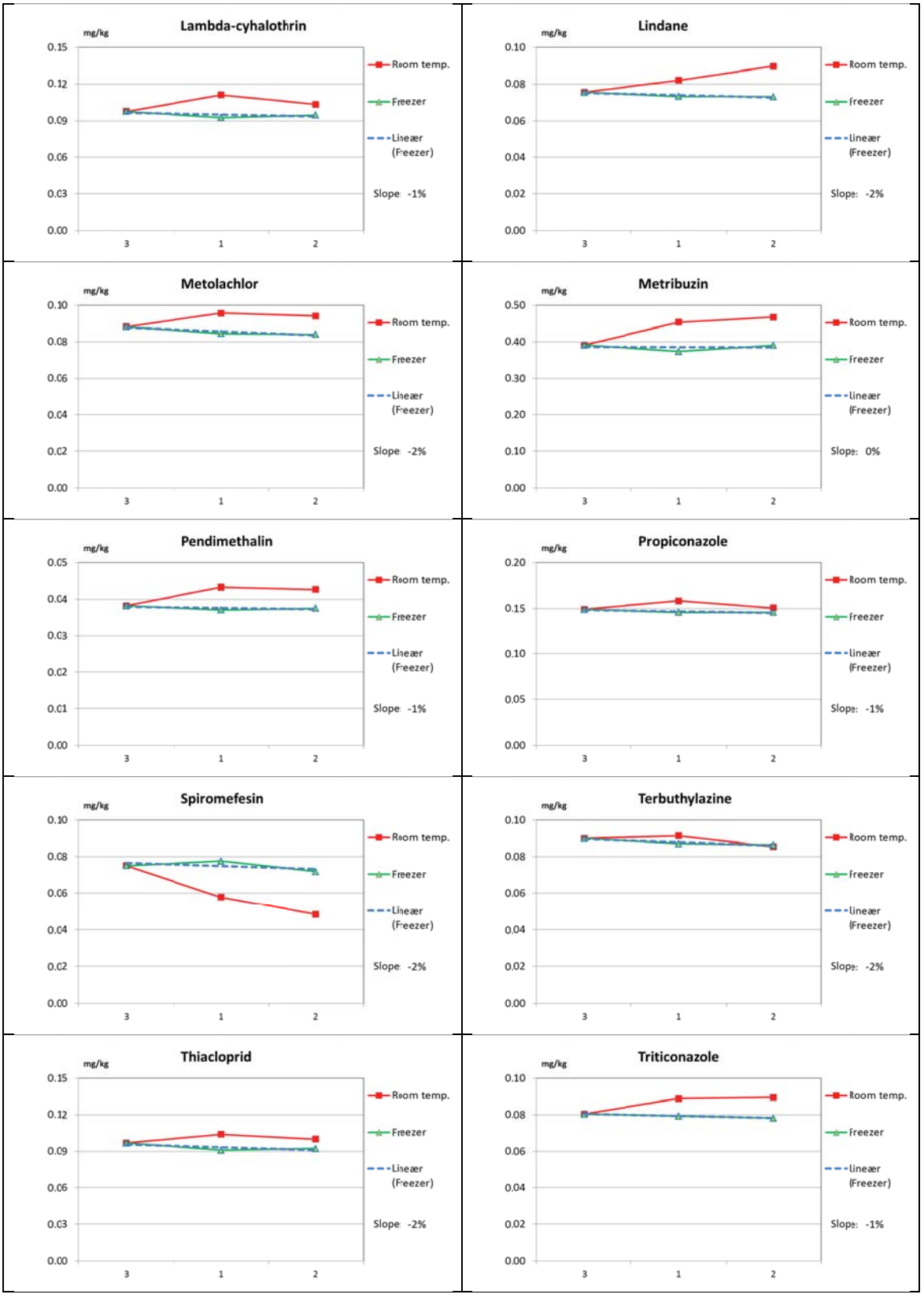
	Pendimethalin, mg/kg		Propiconazole, mg/kg		Spiromesifen, mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
12	0.042	0.035	0.141	0.129	0.045	0.054
44	0.044	0.040	0.143	0.136	0.056	0.056
73	0.042	0.041	0.144	0.135	0.047	0.066
96	0.048	0.048	0.161	0.165	0.052	0.057
116	0.040	0.043	0.134	0.147	0.064	0.070
140	0.041	0.038	0.140	0.133	0.063	0.051
181	0.047	0.050	0.170	0.164	0.082	0.061
213	0.046	0.048	0.157	0.166	0.054	0.060
260	0.038	0.042	0.145	0.150	0.067	0.050
263	0.041	0.040	0.151	0.144	0.059	0.066

	Terbuthylazine, mg/kg		Thiacloprid, mg/kg		Triticonazole, mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
12	0.078	0.070	0.082	0.079	0.080	0.070
44	0.082	0.072	0.088	0.075	0.083	0.072
73	0.082	0.074	0.090	0.079	0.084	0.073
96	0.082	0.088	0.088	0.095	0.090	0.091
116	0.072	0.079	0.079	0.087	0.070	0.083
140	0.076	0.067	0.081	0.074	0.077	0.068
181	0.090	0.086	0.096	0.100	0.089	0.097
213	0.086	0.088	0.099	0.091	0.088	0.090
260	0.077	0.082	0.078	0.084	0.078	0.084
263	0.080	0.076	0.090	0.086	0.082	0.079

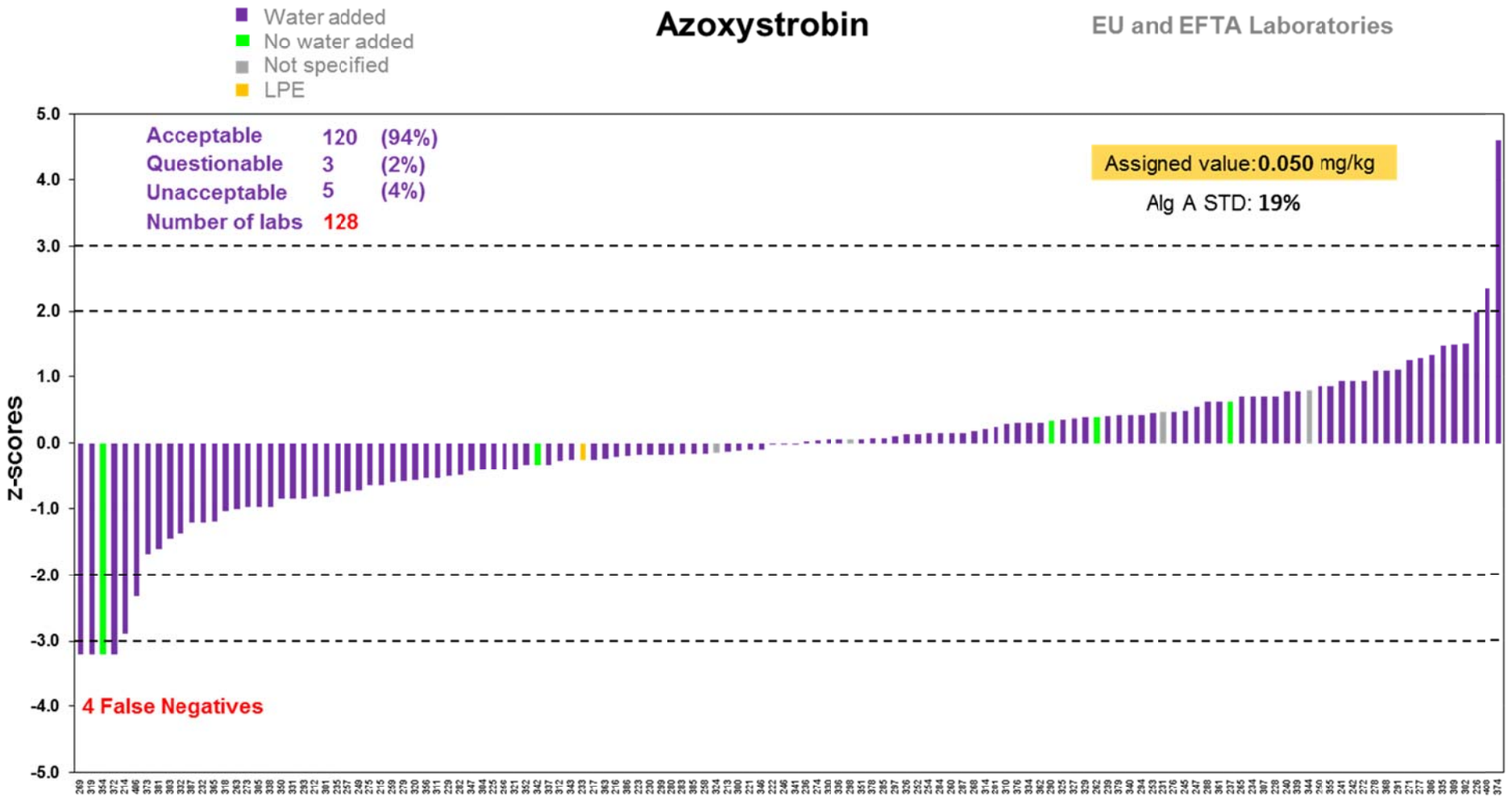
Appendix 4 Stability figures

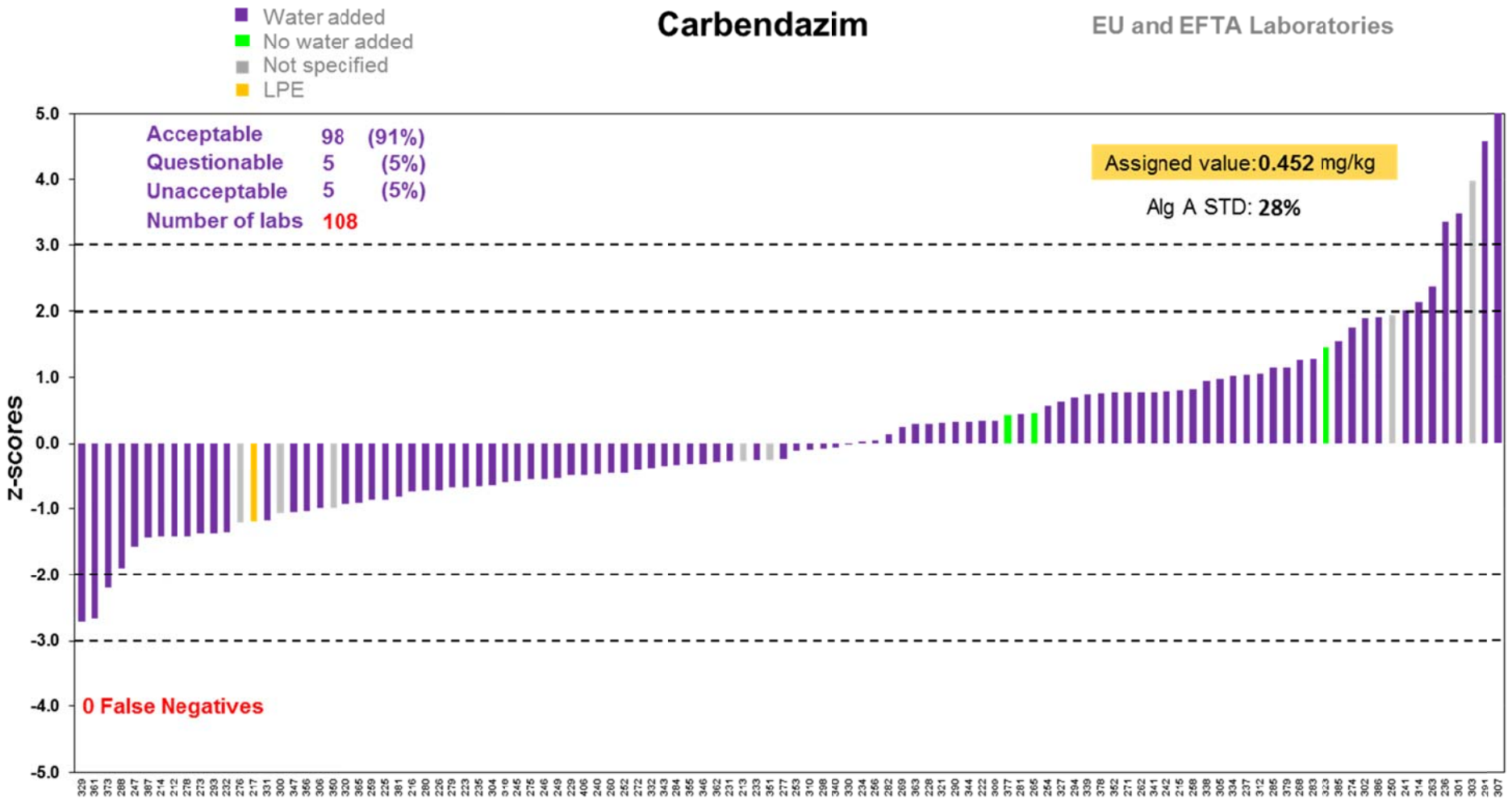
All pesticides passed the test at -18 °C. However, spiromefesin did not pass when stored 4 week at room temperature and this compound was clearly degraded. Additionally, carbendazim, fluopyram, lindane, metribuzin, pendimethalin and triticonazole did not pass the test. This, however, was evaluated not to be caused by degradation of the pesticides but changes the in maize matrix. During storage at room temperature the fatty acid peak eluting from 16-18 min increased dramatically, see Figure 1. All stability test was analysed in the same run and calculated from matrix matched calibration curve where the matrix was maize stored at -18 degree. Consequently, the differences in the coextracted compound has most likely caused the higher result measured after 4 weeks storage. See the statistical results in Table 3.

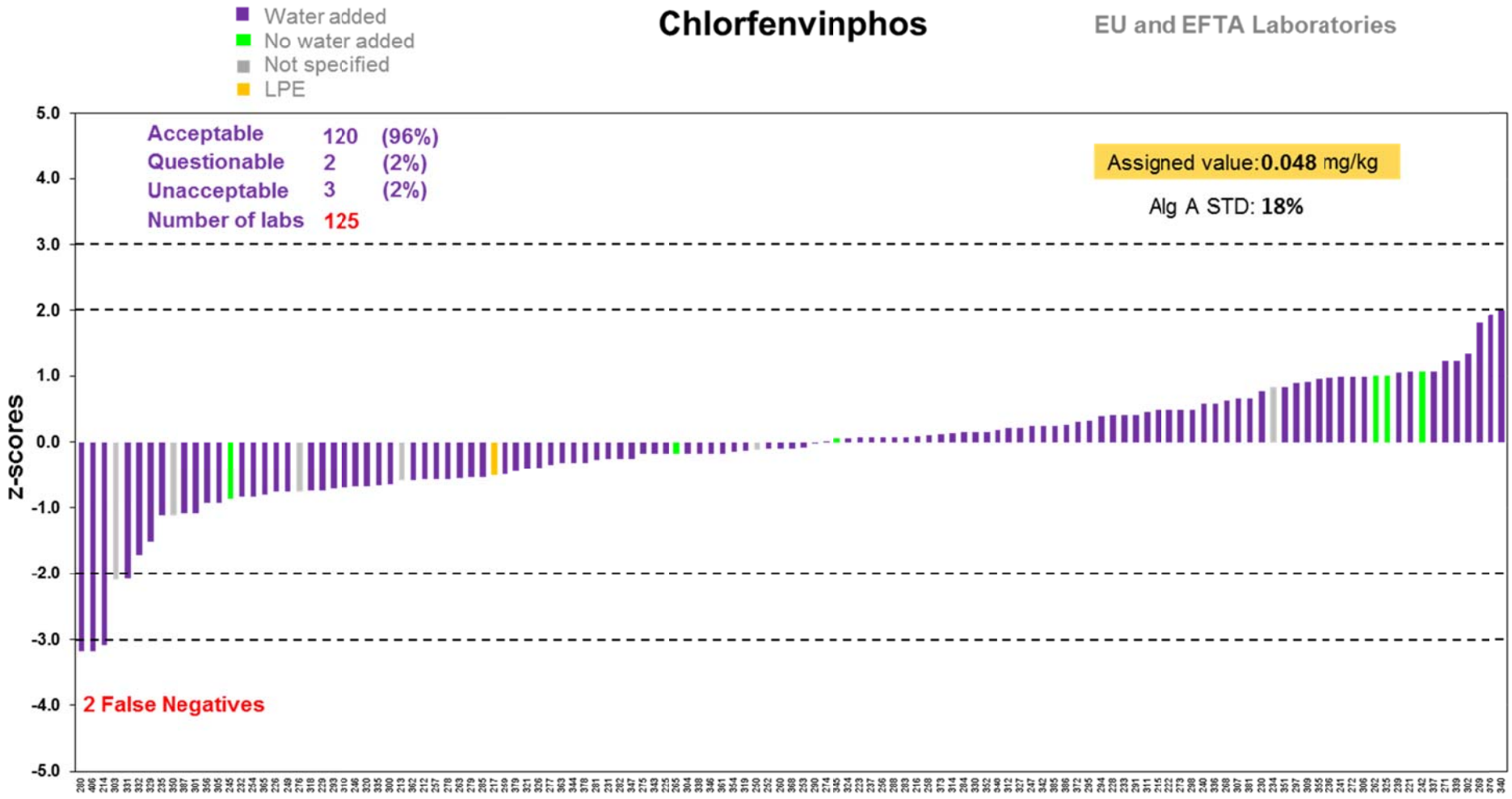


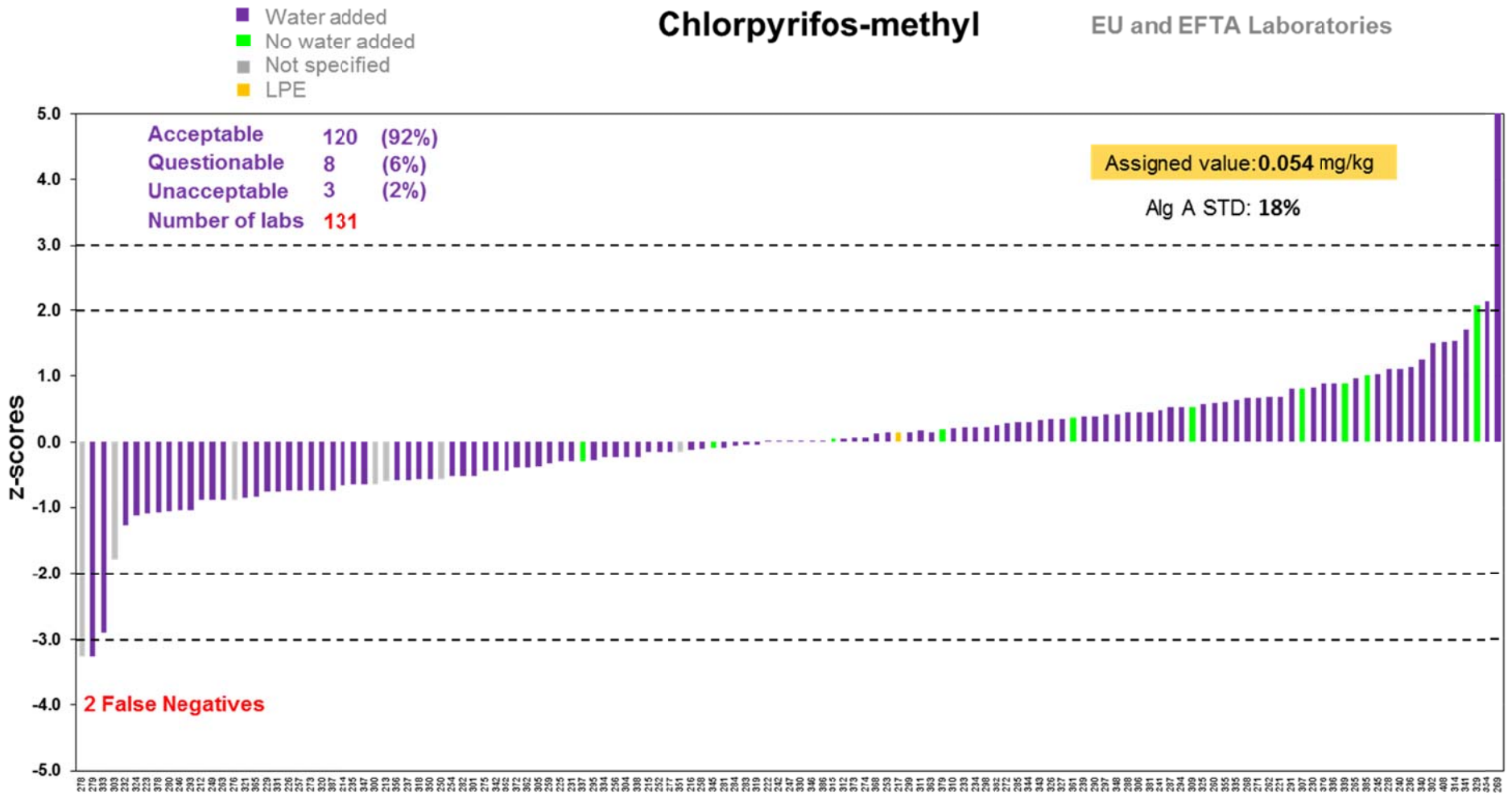

Lambda-cyhalothrin
mg/kg
0.15
0.12
0.09
0.06
0.03
0.00
3
1
2
Room temp.
Freezer
Lineær (Freezer)
Slope: -1%
Lindane
mg/kg
0.10
0.08
0.06
0.04
0.02
0.00
3
1
2
Room temp.
freezer
lineær (Freezer)
Slope: -2%
Metolachlor
mg/kg
0.10
0.08
0.06
0.04
0.02
0.00
3
1
2
Room temp.
Freezer
Lineær (Freezer)
Slope: -2%
Metribuzin
mg/kg
0.50
0.40
0.30
0.20
0.10
0.00
3
1
2
Room temp.
freezer
lineær (Freezer)
Slope: 0%
Pendimethalin
mg/kg
0.05
0.04
0.03
0.02
0.01
0.00
3
1
2
Room temp.
Freezer
Lineær (Freezer)
Slope: -1%
Propiconazole
mg/kg
0.20
0.15
0.10
0.05
0.00
3
1
2
Room temp.
freezer
lineær (Freezer)
Slope: -1%
Spiromefesin
mg/kg
0.10
0.08
0.06
0.04
0.02
0.00
3
1
2
Room temp.
Freezer
Lineær (Freezer)
Slope: -2%
Terbuthylazine
mg/kg
0.10
0.08
0.06
0.04
0.02
0.00
3
1
2
Room temp.
freezer
lineær (Freezer)
Slope: -2%
Thiacloprid
mg/kg
0.15
0.12
0.09
0.06
0.03
0.00
3
1
2
Room temp.
Freezer
Lineær (Freezer)
Slope: -2%
Triticonazole
mg/kg
0.10
0.08
0.06
0.04
0.02
0.00
3
1
2
Room temp.
freezer
lineær (Freezer)
Slope: -1%

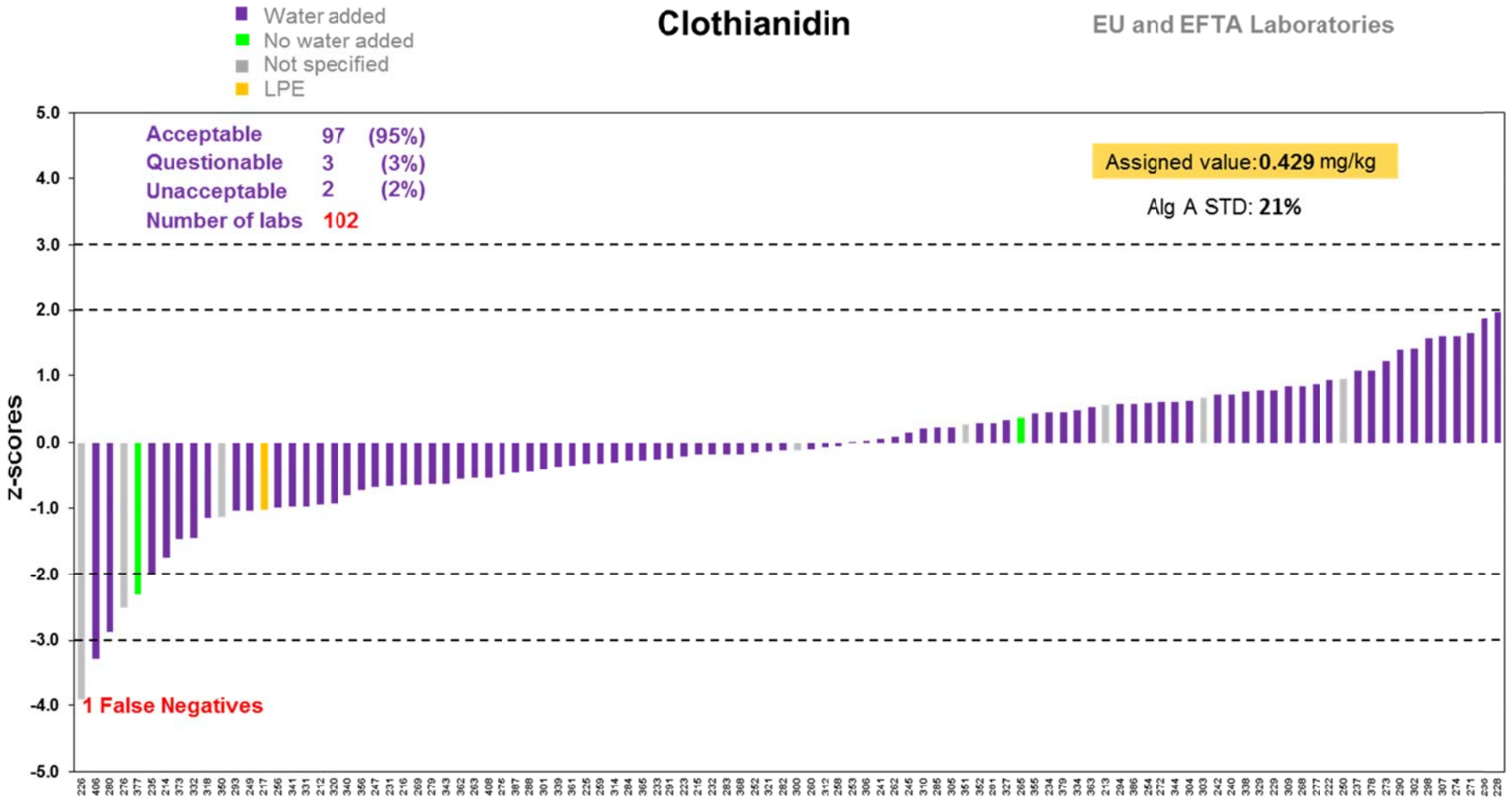
Appendix 4 Graphical presentation of z-scores

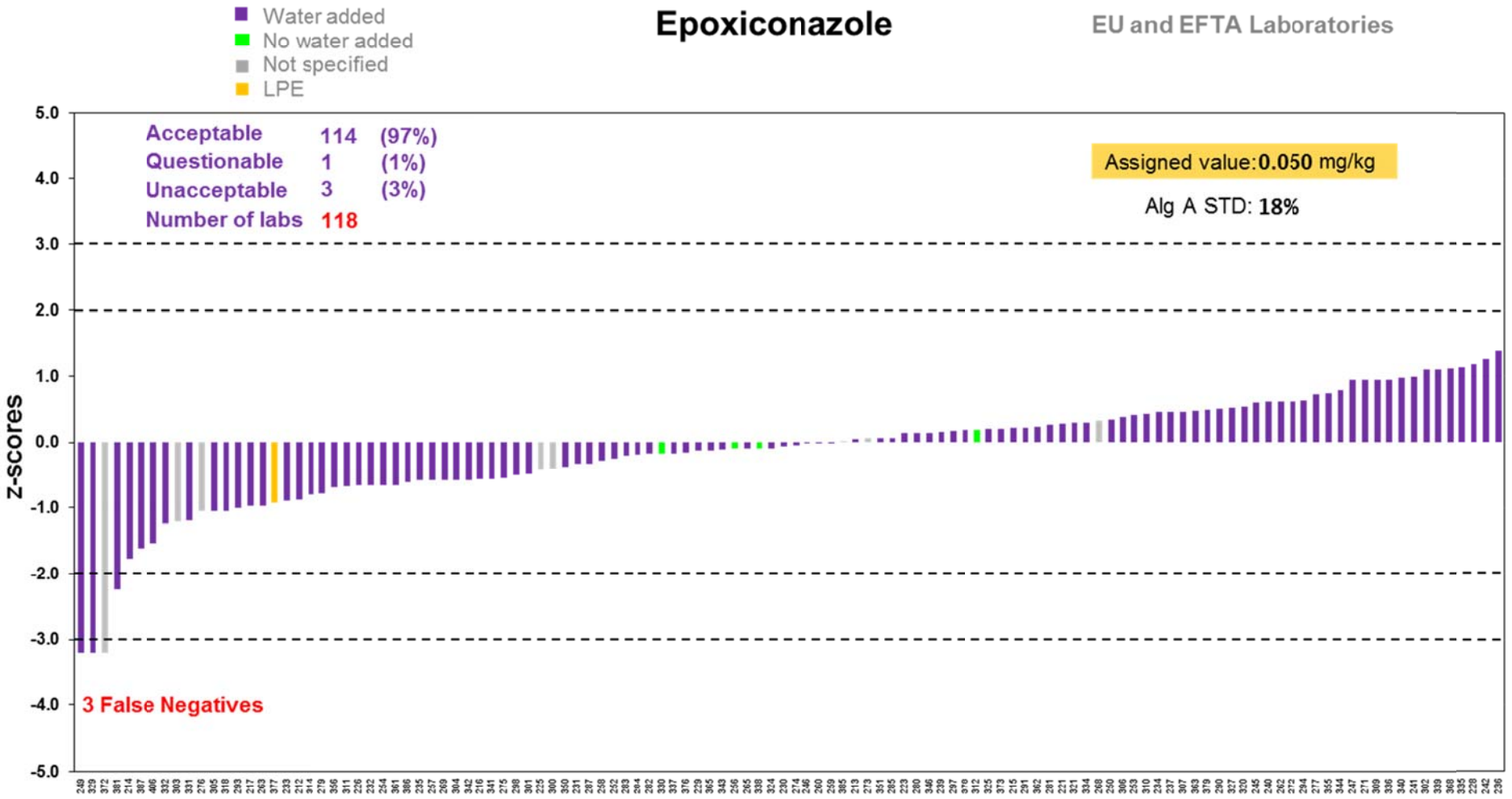


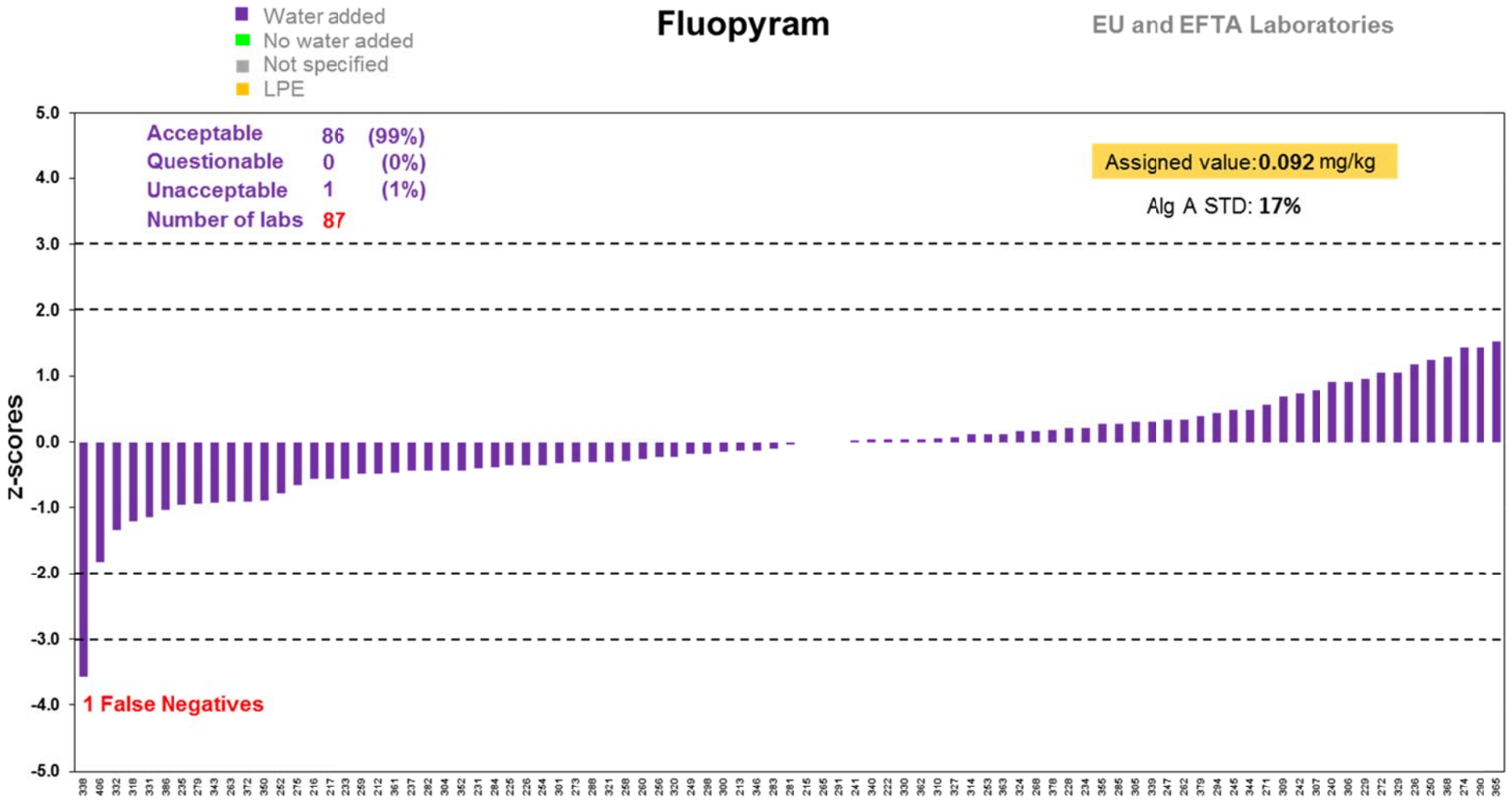

Water added
No water added
Not specified
LPE
Carbendazim
EU and EFTA Laboratories
Acceptable 98 (91%)
Questionable 5 (5%)
Unacceptable 5 (5%)
Number of labs 108
Assigned value: 0.452 mg/kg
Alg A STD: 28%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
0 False Negatives

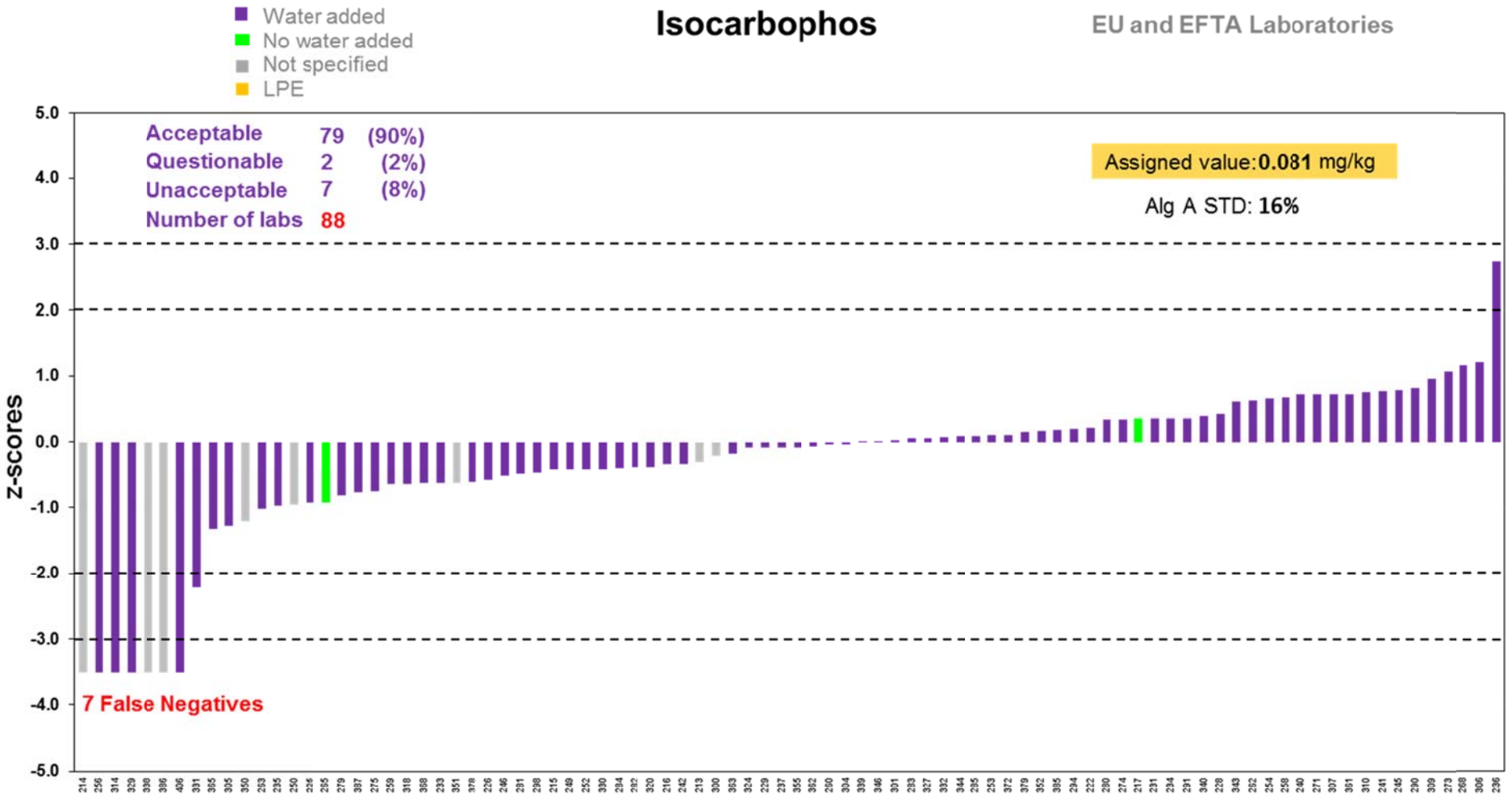

Water added
No water added
Not specified
LPE
Chlorfenvinphos
EU and EFTA Laboratories
Acceptable 120 (96%)
Questionable 2 (2%)
Unacceptable 3 (2%)
Number of labs 125
Assigned value: 0.048 mg/kg
Alg A STD: 18%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
2 False Negatives

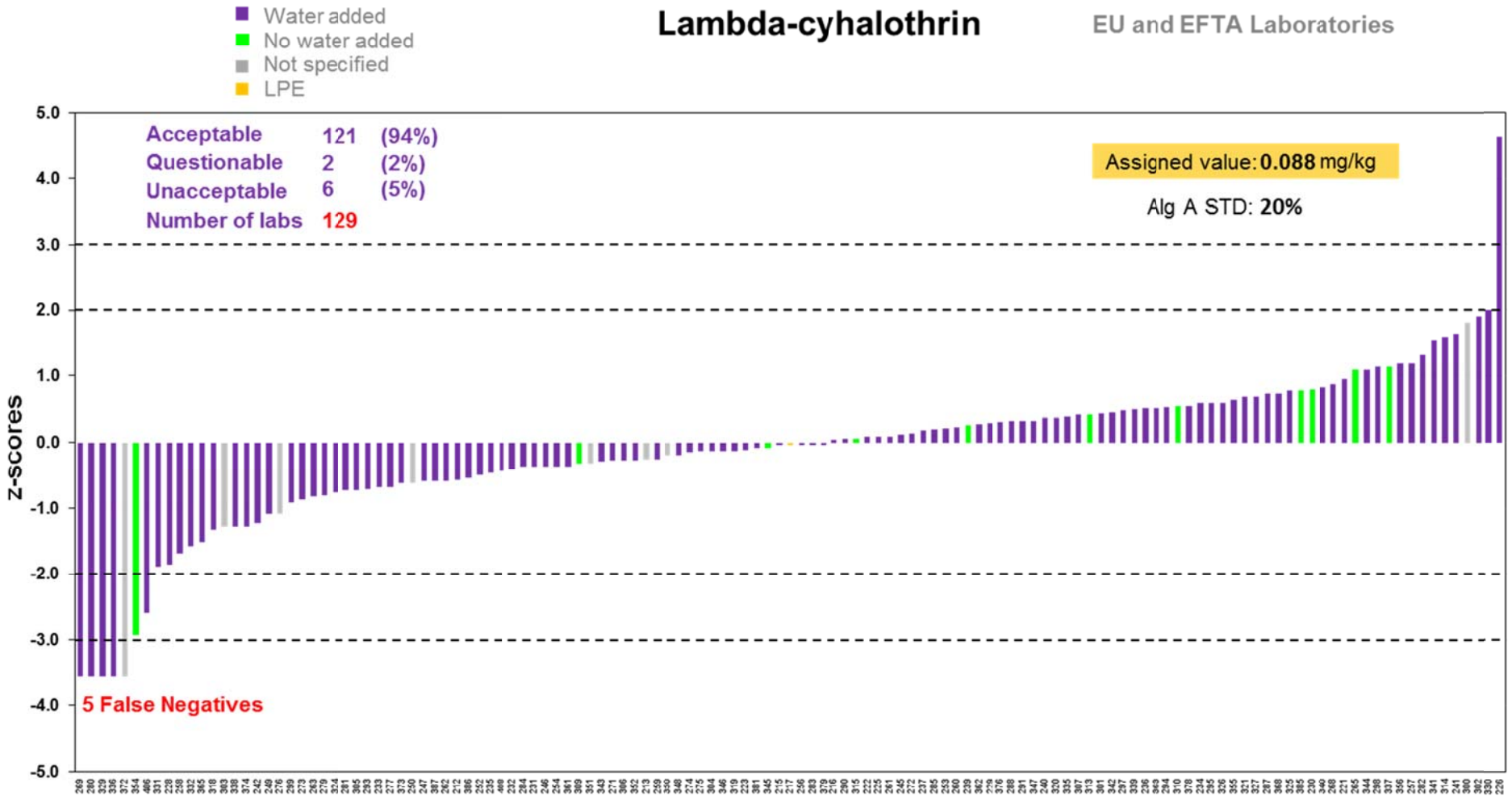

Water added
No water added
Not specified
LPE
Chlorpyrifos-methyl
EU and EFTA Laboratories
Acceptable 120 (92%)
Questionable 8 (6%)
Unacceptable 3 (2%)
Number of labs 131
Assigned value: 0.054 mg/kg
Alg A STD: 18%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
2 False Negatives

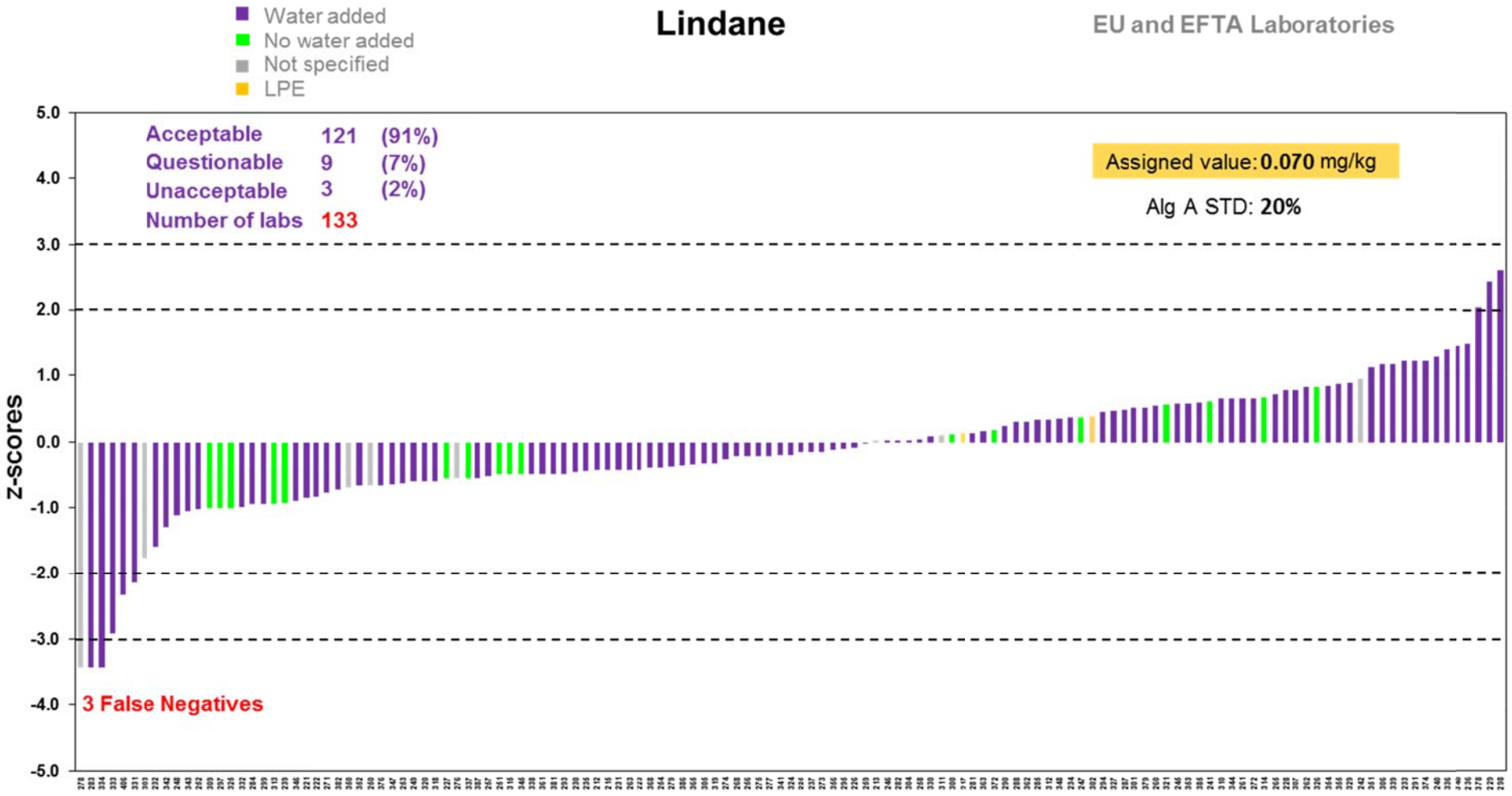

Water added
No water added
Not specified
LPE
Clothianidin
EU and EFTA Laboratories
Acceptable 97 (95%)
Questionable 3 (3%)
Unacceptable 2 (2%)
Number of labs 102
Assigned value: 0.429 mg/kg
Alg A STD: 21%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
1 False Negatives

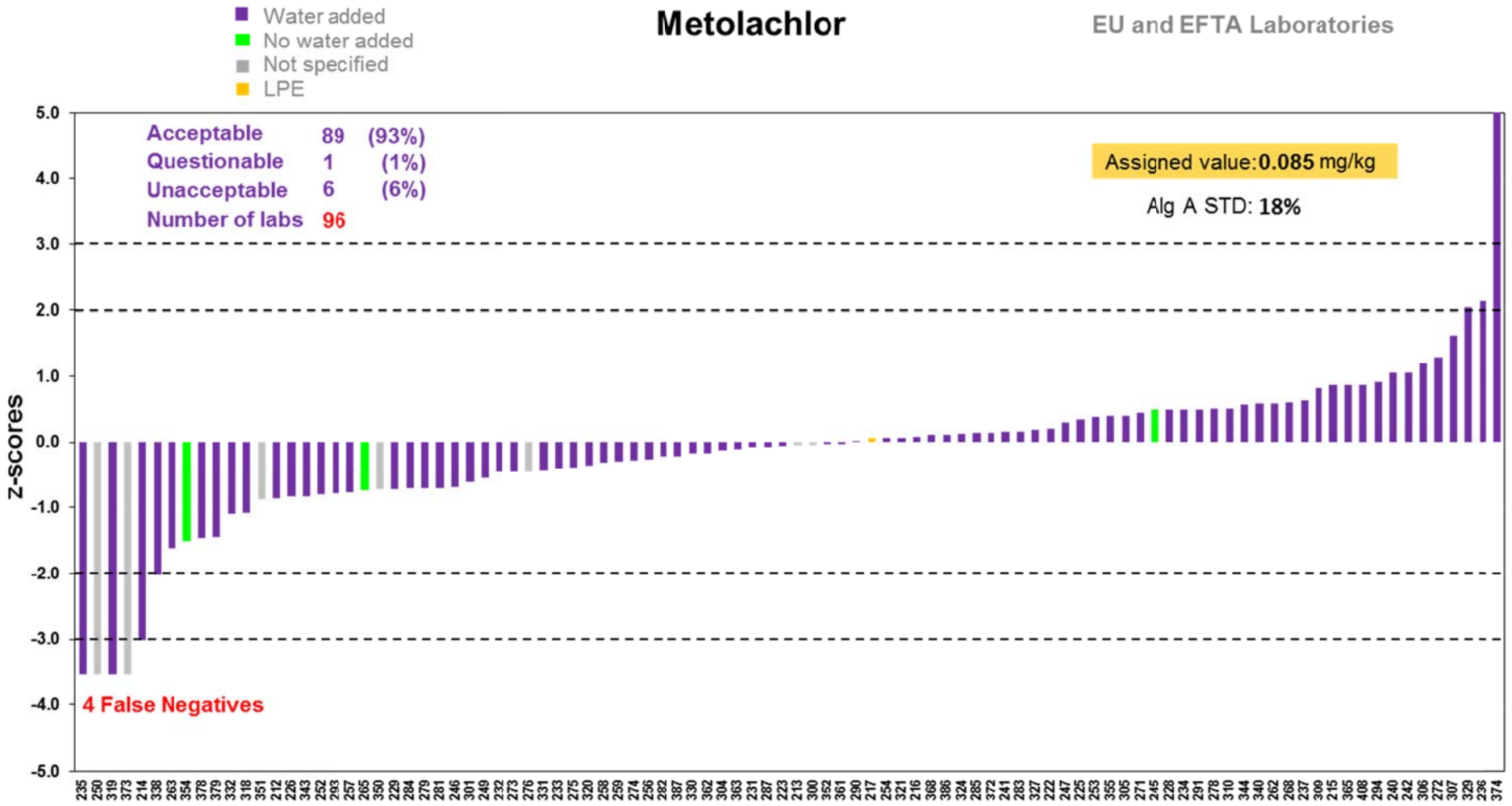

Water added
No water added
Not specified
LPE
Epoxiconazole
EU and EFTA Laboratories
Acceptable 114 (97%)
Questionable 1 (1%)
Unacceptable 3 (3%)
Number of labs 118
Assigned value: 0.050 mg/kg
Alg A STD: 18%
Z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
3 False Negatives

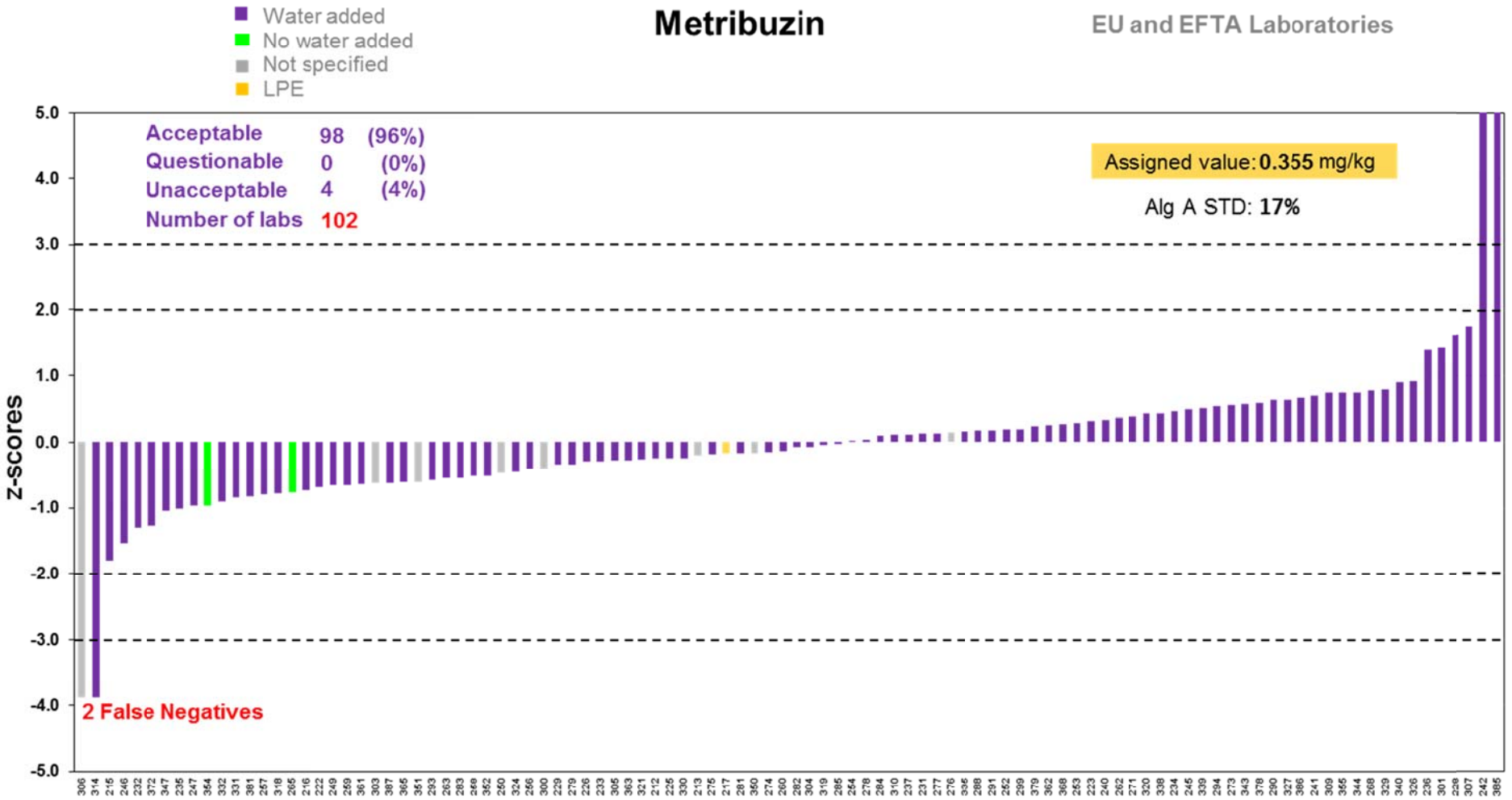

Water added
No water added
Not specified
LPE
Fluopyram
EU and EFTA Laboratories
Acceptable 86 (99%)
Questionable 0 (0%)
Unacceptable 1 (1%)
Number of labs 87
Assigned value: 0.092 mg/kg
Alg A STD: 17%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
1 False Negatives
338 406 332 318 331 386 235 279 343 263 372 350 252 275 216 217 233 259 212 361 237 282 304 352 231 284 225 226 254 301 273 288 321 258 260 256 320 249 298 300 213 346 283 281 215 265 291 241 340 222 330 362 310 327 314 253 363 324 268 378 228 234 355 285 305 339 247 262 379 294 245 344 271 309 242 307 240 306 229 272 329 236 250 368 274 290 365

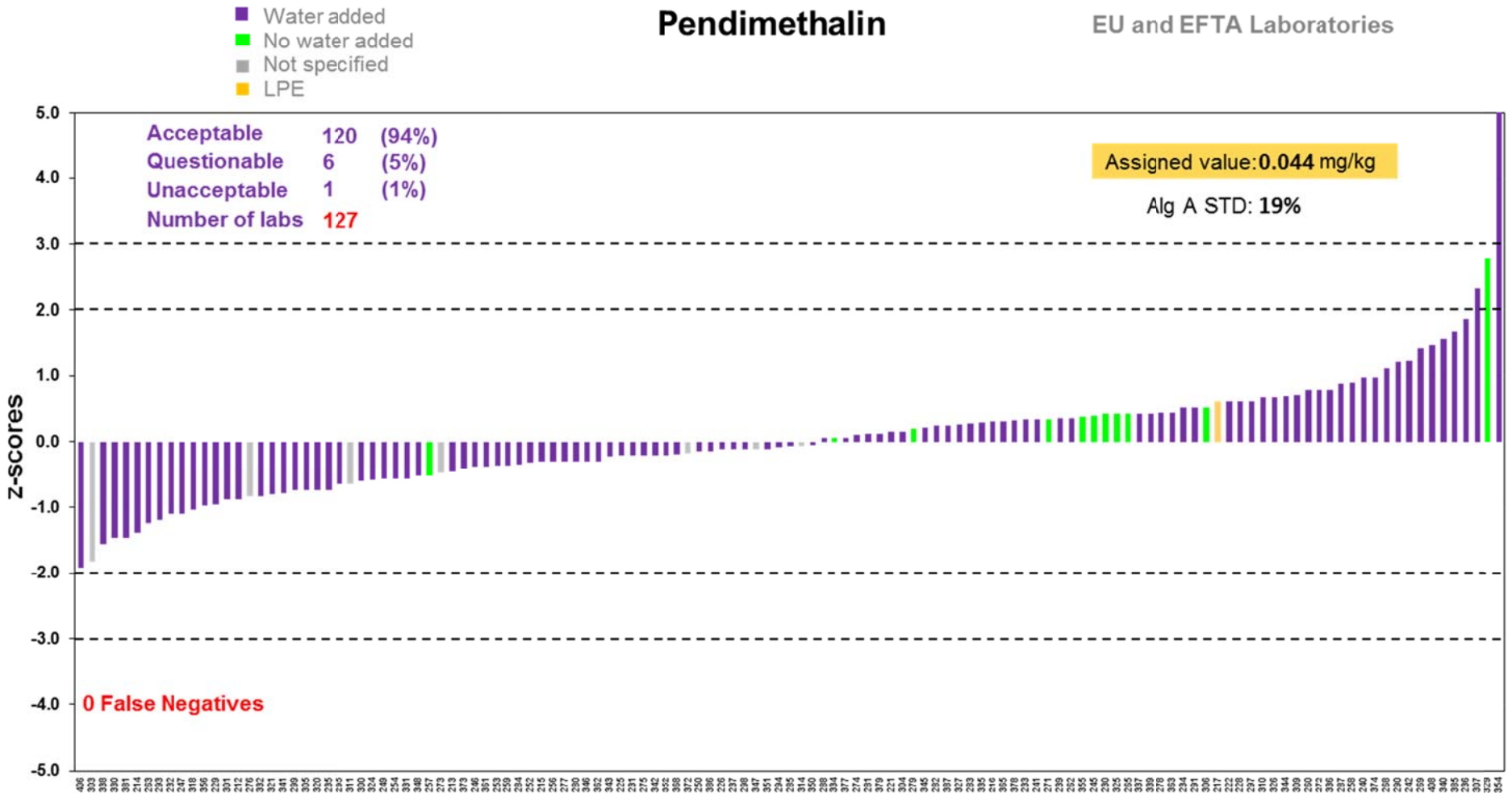

Water added
No water added
Not specified
LPE
Isocarbophos
EU and EFTA Laboratories
Acceptable 79 (90%)
Questionable 2 (2%)
Unacceptable 7 (8%)
Number of labs 88
Assigned value: 0.081 mg/kg
Alg A STD: 16%
Z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
7 False Negatives

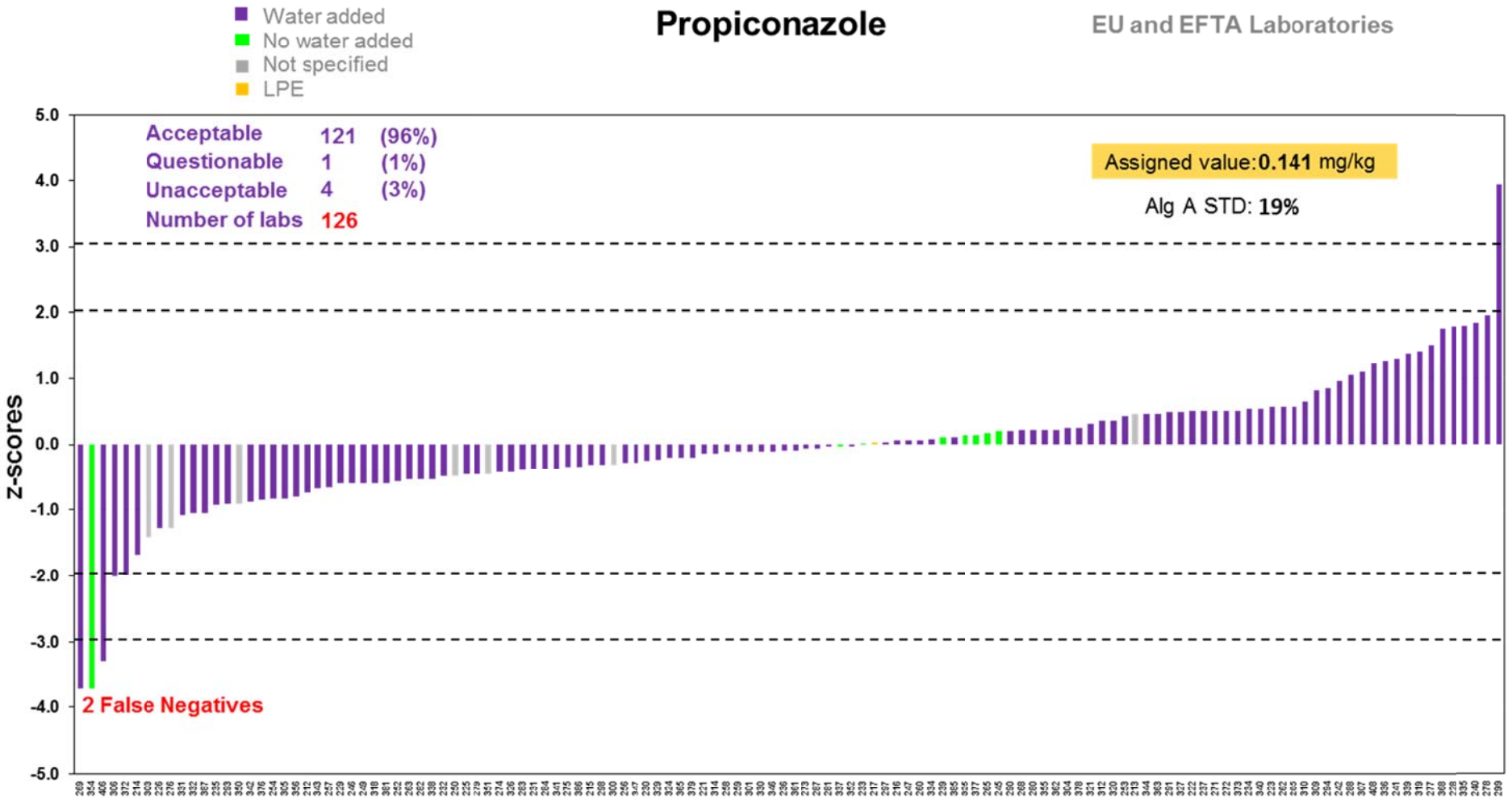

Water added
No water added
Not specified
LPE
Lambda-cyhalothrin
EU and EFTA Laboratories
Acceptable 121 (94%)
Questionable 2 (2%)
Unacceptable 6 (5%)
Number of labs 129
Assigned value: 0.088 mg/kg
Alg A STD: 20%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
5 False Negatives

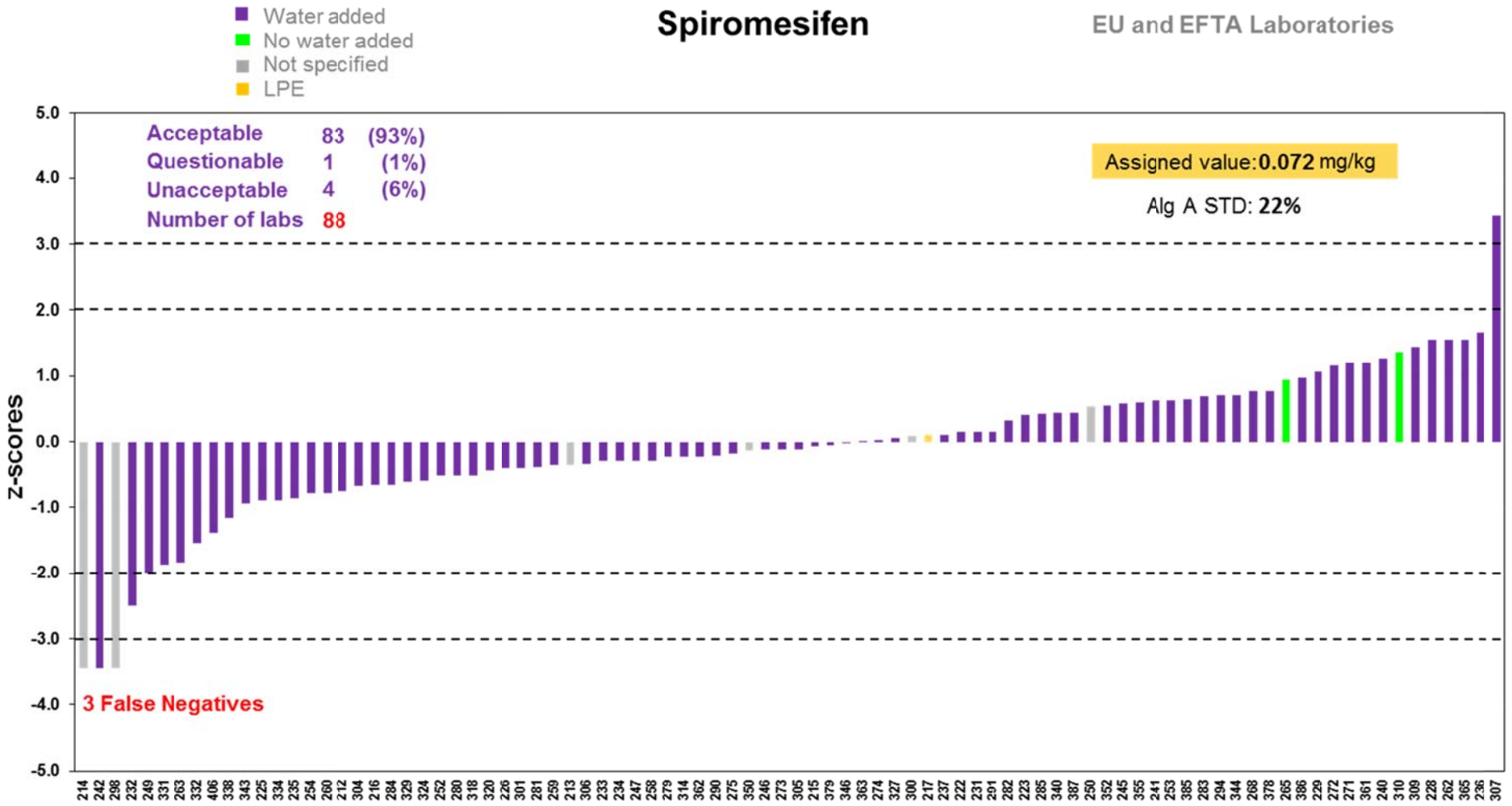

Water added
No water added
Not specified
LPE
Lindane
EU and EFTA Laboratories
Acceptable 121 (91%)
Questionable 9 (7%)
Unacceptable 3 (2%)
Number of labs 133
Assigned value: 0.070 mg/kg
Alg A STD: 20%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
3 False Negatives

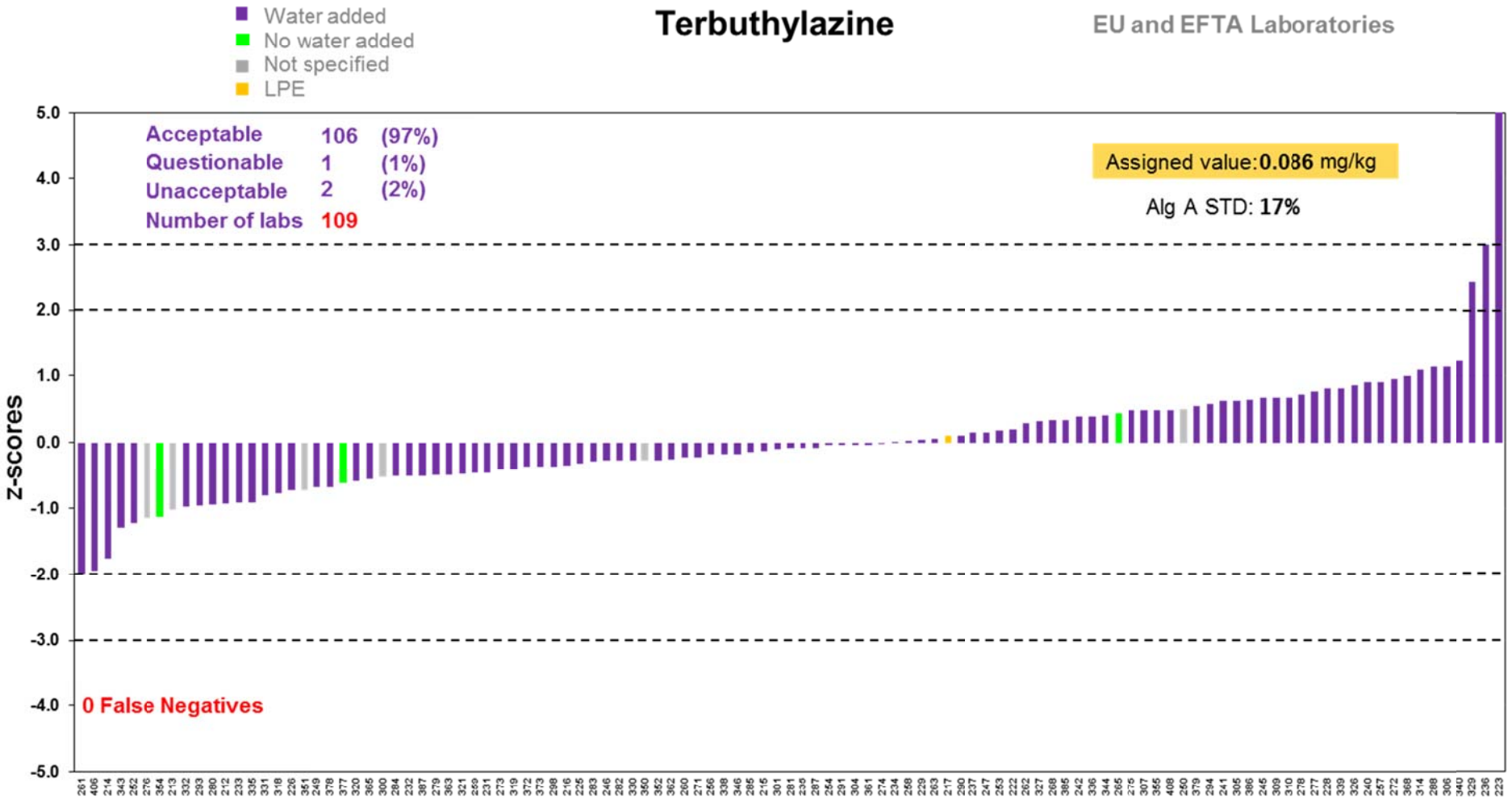

Water added
No water added
Not specified
LPE
Metolachlor
EU and EFTA Laboratories
Acceptable 89 (93%)
Questionable 1 (1%)
Unacceptable 6 (6%)
Number of labs 96
Assigned value: 0.085 mg/kg
Alg A STD: 18%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
4 False Negatives
235 250 319 373 214 338 263 354 378 379 332 318 351 212 226 343 252 293 257 265 350 229 284 279 281 246 301 249 232 273 276 331 233 275 320 258 259 274 256 282 387 330 362 304 363 231 287 223 213 300 352 361 290 217 254 321 216 368 386 324 285 372 241 283 327 222 247 225 253 355 305 271 245 228 234 291 278 310 344 340 262 268 237 309 215 365 408 294 240 242 306 272 307 329 236 374

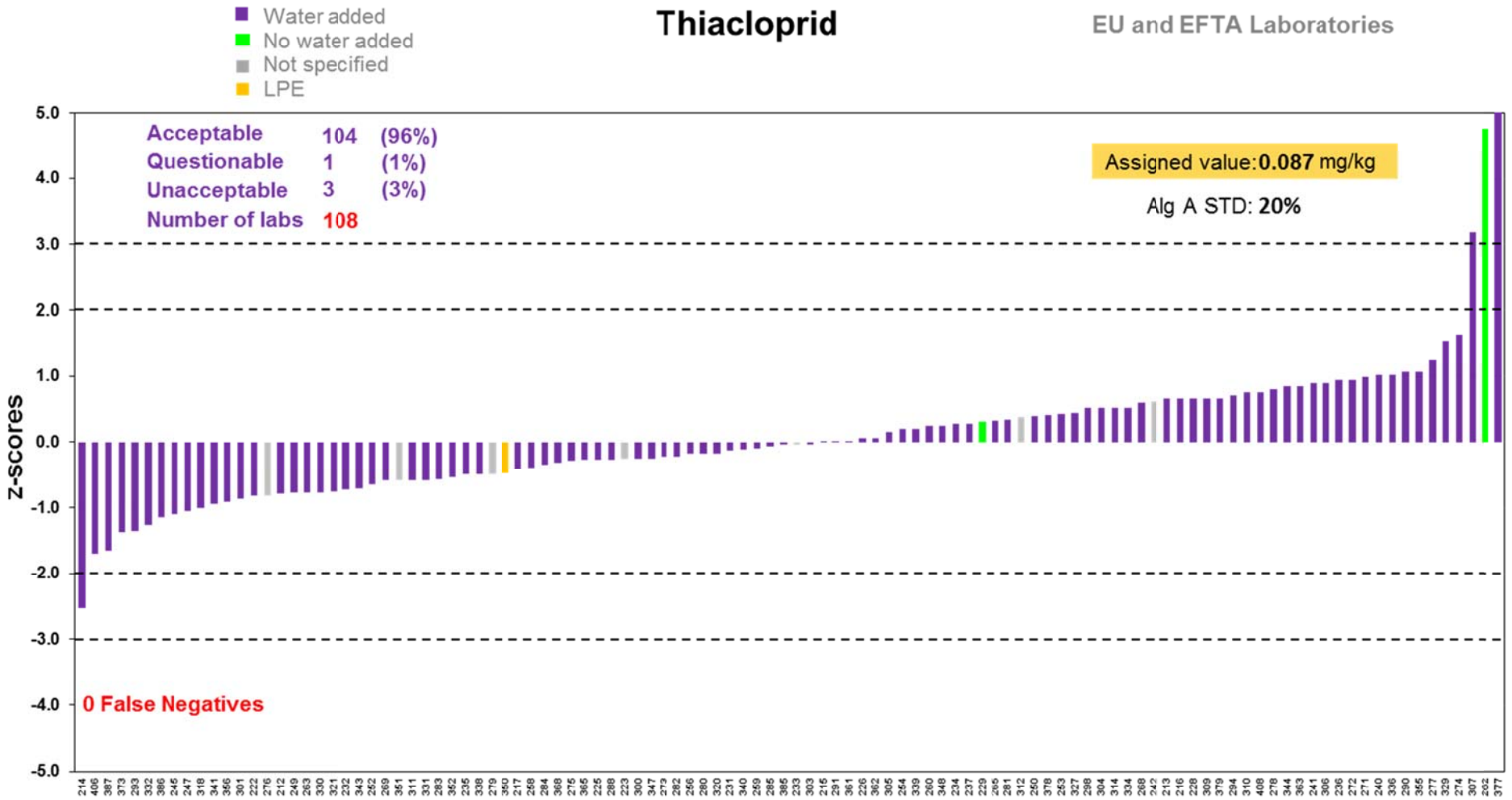

Water added
No water added
Not specified
LPE
Metribuzin
EU and EFTA Laboratories
Acceptable 98 (96%)
Questionable 0 (0%)
Unacceptable 4 (4%)
Number of labs 102
Assigned value: 0.355 mg/kg
Alg A STD: 17%
Z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
2 False Negatives

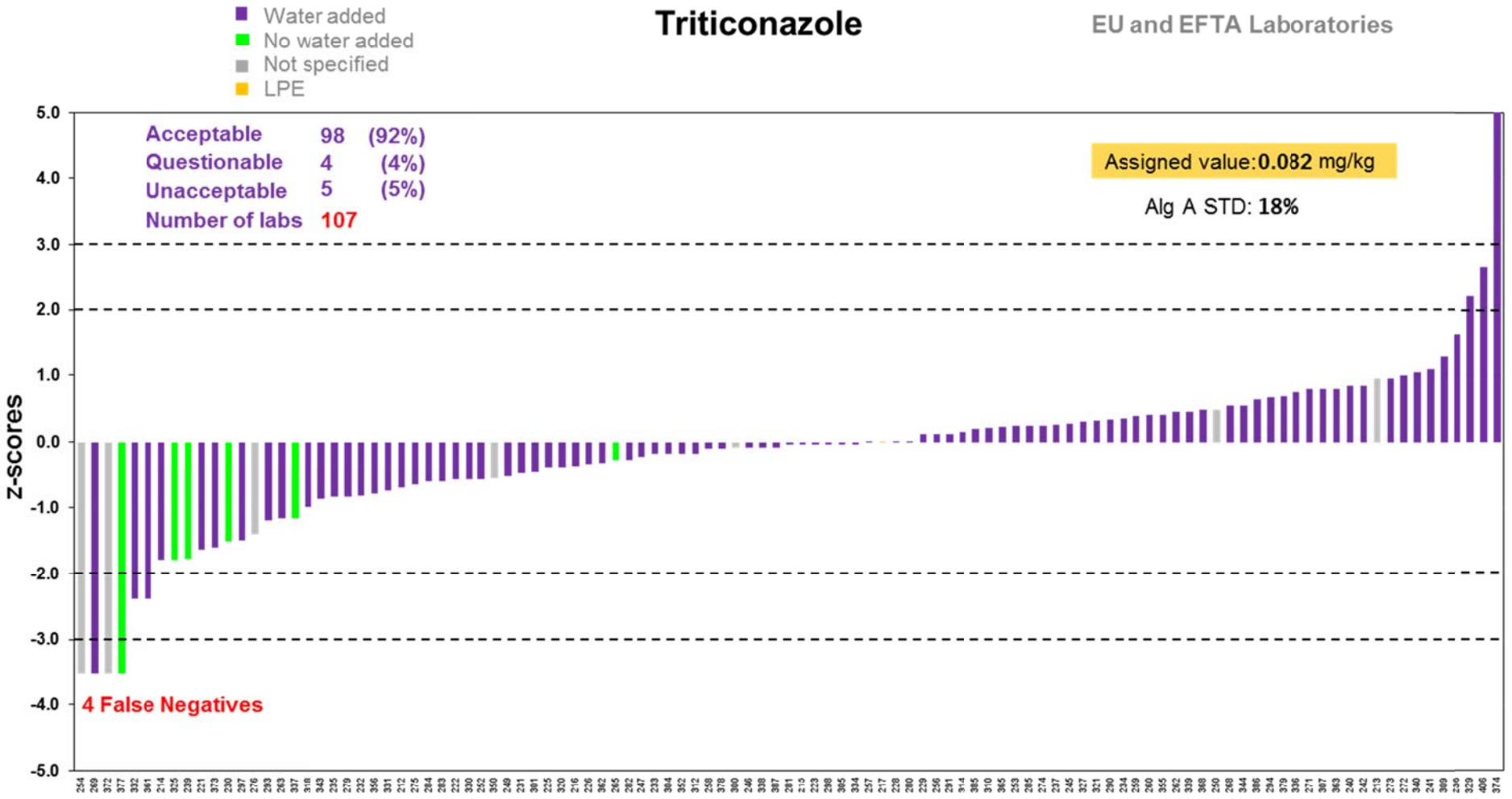

Water added
No water added
Not specified
LPE
Pendimethalin
EU and EFTA Laboratories
Acceptable 120 (94%)
Questionable 6 (5%)
Unacceptable 1 (1%)
Number of labs 127
Assigned value: 0.044 mg/kg
Alg A STD: 19%
Z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
0 False Negatives


Water added
No water added
Not specified
LPE
Propiconazole
EU and EFTA Laboratories
Acceptable 121 (96%)
Questionable 1 (1%)
Unacceptable 4 (3%)
Number of labs 126
Assigned value: 0.141 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
2 False Negatives


Water added
No water added
Not specified
LPE
Spiromesifen
EU and EFTA Laboratories
Acceptable 83 (93%)
Questionable 1 (1%)
Unacceptable 4 (6%)
Number of labs 88
Assigned value: 0.072 mg/kg
Alg A STD: 22%
Z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
3 False Negatives
214 242 298 232 249 331 263 332 406 338 343 225 334 235 254 260 212 304 216 284 329 324 252 280 318 320 226 301 281 259 213 306 233 234 247 258 279 314 362 290 275 350 246 273 305 215 379 346 363 274 327 300 217 237 222 231 291 282 223 285 340 387 250 352 245 355 241 253 385 283 294 344 268 378 265 386 229 272 271 361 240 310 309 228 262 365 236 307


Water added
No water added
Not specified
LPE
Terbuthylazine
EU and EFTA Laboratories
Acceptable 106 (97%)
Questionable 1 (1%)
Unacceptable 2 (2%)
Number of labs 109
Assigned value: 0.086 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
0 False Negatives


Water added
No water added
Not specified
LPE
Thiacloprid
EU and EFTA Laboratories
Acceptable 104 (96%)
Questionable 1 (1%)
Unacceptable 3 (3%)
Number of labs 108
Assigned value: 0.087 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
0 False Negatives


Water added
No water added
Not specified
LPE
Triticonazole
EU and EFTA Laboratories
Acceptable 98 (92%)
Questionable 4 (4%)
Unacceptable 5 (5%)
Number of labs 107
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
4 False Negatives

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GENERAL PROTOCOL

for EU Proficiency Tests on Pesticide Residues in Food and Feed

Introduction

This protocol contains general procedures valid for all European Union Proficiency Tests (EUPTs) organised on behalf of the European Commission, DG-SANTÉ[1] by the four European Union Reference Laboratories (EURLs) responsible for pesticide residues in food and feed. These EUPTs are directed at laboratories belonging to the Network[2] of National Reference Laboratories (NRLs) and Official Laboratories (OfLs) of the EU Member States. OfLs from EFTA countries and EU-Candidate countries are also welcome to participate in the EUPTs. OfLs from Third countries may be permitted to participate on a case-by-case basis.

The following four EURLs for pesticide residues were appointed by DG-SANTÉ based on regulation 882/2004/EC[3]:

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

The aim of these EUPTs is to obtain information regarding the quality, accuracy and comparability of pesticide residue data in food and feed reported to the European Union within the framework of the national control programmes and the EU multiannual co-ordinated control programme[4]. Participating laboratories will be provided with an assessment of their analytical performance that

[1] DG-SANTÉ = European Commission, Health and Consumer Protection Directorate-General

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

[3] Regulation (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

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

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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-SANTÉ. 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).

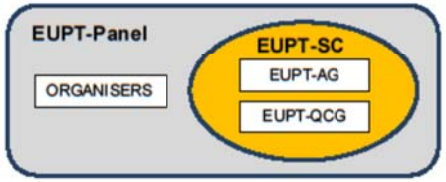
The EUPT-SC's role is to help the Organisers make decisions regarding the EUPT design: the selection of the commodity, the selection of pesticides to be included in the Target Pesticide List (see below), the establishment of the Minimum Required Reporting Levels (MRRLs), the statistical treatment and 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/allcrl/EUPT-SC.pdf

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-SANTÉ.

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

[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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will be updated every year to take account of any changes in the lab profiles. Interim updates will be issued to eliminate any possible errors.

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

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

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

Confidentiality and Communication

The proprietor of all EUPT data is DG-SANTÉ 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-SANTÉ, 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-SANTÉ.

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

Target Pesticide List

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

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

Specific Protocol

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

Homogeneity of the Test Item

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

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

with values of 1.88 and 1.01, respectively, if 10 samples are used. $\sigma_{all}^2 = 0.3 \times$ FFP-RSD[8] (25 %) × the analytical sampling mean for all pesticides, and s_{an} is the estimate of the analytical standard deviation.

The results of all homogeneity tests are presented to the EUPT-SC. In special cases where the above homogeneity test criteria are not met, the EUPT-SC considering all relevant aspects (e.g. the homogeneity results of other pesticides spiked at the same time, the overall distribution the participants' results, the analytical difficulties faced during the test, knowledge of the analytical behaviour of the pesticide question) may decide to overrule the test. The reasons of this overruling have to be transparently explained in the Final EUPT-Report.

Stability of the analytes contained in the Test Item

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

A pesticide is considered to be adequately stable if $| x_1 - y_1 | \leq 0.3 \times \sigma$, where x_1 is the mean value of the first stability test, y_1 the mean value of the last stability test and σ 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

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

analytical difficulties faced during the test, knowledge about the analytical behaviour of the pesticide question) may decide to overrule the test. The reasons of this overruling will be transparently explained in the Final EUPT-Report.

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.

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Correction of results for recovery

According to the Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed[9], it is common practice that pesticide analysis results are not corrected for recovery, but may be corrected if the average recovery is significantly different from 100 % (typically if outside the 70 – 120 % range, but also exhibiting good precision). Other approaches for recovery correction explicitly allowed in the SANCO document are the use of stable isotope labelled analogues of the target analytes used as Internal Standards (ISTDs), the 'procedural calibration' approach as well as the approach of 'standard addition' with additions of analyte(s) being made to analytical portions. Where reported residue data have been automatically adjusted for recovery by the method, or have subsequently been adjusted using a recovery factor, this must be indicated on the specific field of the 'Result Submission Form'. Results may be corrected for recovery only in cases where this correction is applied in routine practice (including cases of MRL-violations). Laboratories are required to report whether their results were adjusted for recovery and, if a recovery factor was used, the recovery (in percentage) must also be reported. No recovery data are required where correction for recovery is automatic by using the 'standard addition approach, or isotopically-labelled internal standards (in both cases with spiking of the Test Item at the beginning of the extraction procedures). In these cases, the laboratories should report the actual approach that was followed.

Methodology information

All laboratories are requested to provide information on the analytical method(s) they have used. A compilation of the methodology information submitted by all participants is presented in an Annex of the final report or in a separate report. Where necessary the methods are evaluated and discussed, especially in those cases where the result distribution is not unimodal or very broad (e.g. CVs* > 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.

[9] Document N° SANCO/12571/2013; Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed

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 4 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*)***

In order to minimise the influence of out-lying results on the statistical evaluation, the assigned value (= consensus concentration) will typically be estimated using robust statistics as described in ISO 13528:2009-01[10]. In special justifiable cases, the EUPT-Panel may decide to eliminate certain

[10] DIN ISO 13528:2009-01, 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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results traceably associated with gross errors (see "Omission or Exclusion of results" below) or to use only the results of a subgroup consisting of laboratories that have repeatedly demonstrated good performance for the specific compound in the past.

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

Before estimating the assigned value results associated with obvious mistakes have to be examined to decide whether they should be removed from the population. Such gross errors may include incorrect recording (e.g. due to transcription errors by the participant, decimal point faults or transposed digits, incorrect unit), calculation errors (e.g. missing factors), analysis of a wrong sample/extract (e.g. a spiked blank), use of wrong concentrations of standard solutions, incorrect data processing (e.g. integration of wrong peak), major deviations from the analytical procedure, inappropriate storage or transport conditions (in case of susceptible compounds), and the use of inappropriate procedures that demonstrably lead to significantly biased results (e.g. due to degradation or incomplete extraction). Where the Organisers (e.g. after the publication of the preliminary report) receive information of such gross errors, having a significant impact on a generated result, the affected results will be examined on a case-by-case basis to decide whether, or not, they should be excluded from the population used for robust statistics. Even results that cannot be specifically identified as outliers might be excluded. 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").

Any exclusion of results from the population is to be discussed within the EUPT-SC and the reasoning behind is to be revealed in the EUPT-final report.

Uncertainty of the assigned value

The uncertainty of the assigned values x_i is calculated according to ISO 13528:2009-01 as:

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

where s^* is the robust standard deviation and n 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 13528:2009-01 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 (FFP-s) of the assigned value will be calculated using a Fit-For-Purpose Relative Standard Deviation (FFP-RSD) approach, as follows:

$$\text{FFP-}s_i = b \cdot x^*_i \qquad \text{with } b = 0.25 \text{ (25\% FFP-RSD)}$$

The percentage FFP-RSD is set at 25% based on experience from results of previous EUPTs[11]. 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 (CVs*) is calculated according to ISO 13528:2009-01; Chapter 5.6 (Consensus value from participants) following Algorithm A in Annex C.

– ***z-scores***

This parameter is calculated using the following formula:

$$z_i = (x_i - x^*_i) / \text{FFP-}s_i$$

where x_i is the value reported by the laboratory, x^*_i the assigned value, and $\text{FFP-}s_i$ the standard deviation for each pesticide (i). 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.

[11] 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.

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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[12]:

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

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

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

The EUPT-Panel will decide if and how to classify the laboratories into two categories - A or B. Currently, laboratories that have detected and quantified a sufficiently high percentage of the pesticides present in the Test Item (e.g. at least 90 %) and reported no false positives will have demonstrated 'sufficient scope' and can therefore be classified into Category A. The 90 % criterion will be applied following Table 1.

Table 1. No. of pesticides needed to be detected to have sufficient scope.

No. of Pesticides Present in the Test Item (N)	90 %	No. of Pesticides needed to be reported 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	

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

No. of Pesticides Present in the Test Item (N)	90 %	No. of Pesticides needed to be reported to have sufficient scope (n)	n
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)[13,14] (see below) will be used. The AZ^2 is calculated as follows:

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

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

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

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

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

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

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

Publication of results

The EURLs will publish a preliminary report, containing tentative medians 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 annually by the EURLs in the following year, the final report may be published up to 10 months after the deadline for results submission.

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 with $|z| > 2.0$) - including all false positives and false negatives. Even results within $|z| \leq 2.0$ may have to be checked if there is indications of a significant positive or negative bias.

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

According to instructions from DG-SANTÉ, 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.

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-CF9 (2015)

(last updated: 23 April 2015)

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-CF9.

Test Item (Test Material)

This proficiency test concerns the analysis of pesticide residues in maize flour. The maize was grown in Denmark in 2014 and pesticides were applied in the field. Following harvest, the maize 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 125 g of maize flour Test Item with incurred and spiked pesticides and
- approximately 125 g of blank maize flour Test Item.

Shipment of Test Items

The Test Items are planned to be shipped on 20 April, 2015.

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 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-CF9 Result Submission Website**. Links to this can be found on the EURL-CF webpage: http://www.crl-pesticides.eu/docs/public/tmplt_article.asp?LabID=400&CntID=961&Theme_ID=1&Pdf=False&Lang=EN.

The website will be accessible from 21 April 2015. 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 will be email to the partidpants on 17 April.**

To access the data submission forms participants must use their unique login data (username and password), which will be sent by e-mail on 17 April 2015.

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 19 May 2015 at 13.00 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-CF9 Result Submission Website, the date of receipt, the condition of the Test Item, and its acceptance. The deadline for acceptance is the 24 April 2015. 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 24 April 2015 at noon, they must inform the Organiser immediately by e-mail (eurl-cf@food.dtu.dk).

Reporting Qualitative and Quantitative Results - Subpages 1 and 2

To report their results, laboratories must access the EUPT-CF9 Result Submission Website.

Deadline: All results must be reported on the online result submission website by 19 May 2015 at 13.00 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

c) below the MRRL.

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.

The following fields will be available for reporting the quantitative results:

- **"Concentration in mg/kg":** here you should fill in the results that you would report in your routine work. That means, the recovery-corrected result should be reported, if it reflects the normal procedure in your lab, otherwise the non-recovery-corrected result should be reported.
- **"Conc. in blank in mg/kg":** any concentration values of pesticides from the Target Pesticides List you will determine in the blank (even at levels below the MRRL), you can enter here.
- **"Experience with this compound".** Use the dropdown-menu to indicate how many years you have analysed for this compound using the method applied in this EUPT.
- **"Is your result recovery-corrected?":** Please specify whether the result was recovery-corrected and what kind of recovery-correction via the dropdown-menu.
- **"Recovery figure (in %)":** Here, labs can report any recovery figures (in %) obtained for the analyte in question. If a recovery factor was used to correct the result, the recovery figure (in %) used for the calculation MUST be reported.

Additional information on how each recovery figure was derived will be asked in separate fields.

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-CF9 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 missing information after result submission deadline – 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 within the EUPT-CF9 Result Submission Website. This subpage will be accessible from 25-27 May 2015.

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

According to 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 relating to EUPT-CF9 can be found in the EURL-Document Repository (CIRCA/FIS-VL). Links to the documents can be found in the EUPT–CF9 Website.

Calendar

Activity	Dates
Announcement Calendar Target Pesticide List	January 2015
EUPT-Registration Website	9 February 2015
Deadline for registration	20 March 2015
Release of Specific Protocol	7 April 2015
Distribution of Test items	20 April 2015
Deadline for Receipt and Acceptance of Test Materials	within 24 hr on receipt
Deadline for Result Submission	19 May 2015 at 13.00 CET
Deadline for submission of additional method information for false negative results	26 May 2015
Preliminary Report (only compilation of results)	26 June 2015
EUPT Evaluation Meeting	July 2015
Final Report	December 2015

Participation Fees

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

- 175 €

The participation fees for laboratories from third countries:

- 350 €

For further information, visit this website 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 Dahm-Jappe at the financial department pdahm@adm.dtu.dk.

Contact information:

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