Proficiency Test on pesticide residues in hay flour

EU Reference Laboratory on Cereals & Feeding stuff

EUPT-CF12 2018

DTU Food
National Food Institute

EU PROFICIENCY TESTS
EUPT-CF12, 2018

Pesticide Residues in Hay Flour

Final Report

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PREFACE

Regulation (EC) No 882/2004 [1], defines the general tasks and duties of the European Union Reference Laboratories (EURLs) for Food, Feed and Animal Health including the organisation of comparative tests. These proficiency tests (PTs) are carried out on an annual basis, and aim to improve the quality, accuracy and comparability of the analytical results generated by EU Member States within the framework of the EU multi-annual co-ordinated control and national monitoring programmes. Participation in the proficiency test scheme "European Union Proficiency Tests (EUPTs) for pesticide residues" is mandatory according to Article 28 of Regulation (EC) No 396/2005 on maximum residue levels of pesticides in, or on, food and feed of plant and animal origin [2], as long as the analytical scope of the PT and the laboratory overlap.

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

Participation in EUPT-CF12 was compulsory for all National Reference Laboratories (NRLs) and Official Laboratories (OfLs) within the EU involved in the determination of pesticide residues in cereals for human or animal consumption using multiresidue methods for their national programmes. Official laboratories from EFTA countries (Iceland, Norway and Switzerland), as well as official laboratories from EU-candidate states, were invited to take part in this EUPT. Selected laboratories from Third Countries were also allowed to take part in this exercise, but their results, together with the EU-candidate state laboratories, were not used when establishing the Assigned Values for each pesticide.

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

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1. INTRODUCTION

In November 2017 the announcement of the 12th European Commission's Proficiency Test on cereals and feed (EUPT-CF12) was published on the EURL website, together with the Calendar and the Pesticide Target List including all compounds that could potentially be present in the Test Item. The Target Pesticides List included 153 individual compulsory compounds and 9 voluntary 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-CF12, were provided via the homepage. Laboratories were able to register on-line from 5 December 2017 to 12 January 2018. In total 119 laboratories from EU and EFTA countries agreed to participate in the test as well as 11 laboratories from EU-Candidate States and Third Countries (**Appendix 1**).

The present proficiency test was performed using hay flour of Danish origin, which originated from rye grass that has been treated in the field. The Test Item contained 9 compounds. The Danish Centre for Food and Agriculture at Aarhus University grew the rye grass and performed the field treatments. The pesticides employed for the treatment were selected by the EURL-CF and the EURL quality control group. The application rates and harvest intervals chosen were based on previous experience and data from supervised residue trials. Furthermore, the stabilities of the pesticides in the Treated Test Item were checked several times during the period of time allowed for laboratories to complete the PT exercise.

The participating laboratories were provided with 20 g portions of the treated hay Test Item and 50 g of untreated blank hay Test Item. Both Test Items were shipped to participants on 29 January 2018. The deadline for submission of results to the Organiser was the 1 March 2018. 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. The determination was performed by GC-MS/MS and LC-MS/MS.

- QuEChERS: Cold water was added to a milled portion of the test item and shaken. Acetonitrile was added immediately and the tube was shaken again. A salt and buffer mixture was then added together with ceramic homogenizers and the sample was shaken vigorously for 1 min. After centrifugation, an aliquot of the supernatant was cleaned by freezing out. After additional centrifugation of the cold extract, the supernatant was transferred to a tube containing PSA and $MgSO_4$. 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 (hay). The overall capacity and capability of the laboratories within the EU, as assessed from previous PTs and surveys, was also taken into account. The minimum required reporting level (MRRL) for all pesticides in the target list was set at 0.01 mg/kg.

1.3 Preparation of the treated Test Item

Before preparing the Test Item, the pesticides and suitable target residue levels for the study were selected. The application rates and harvest intervals for the pesticides used for treatment in the field were chosen based on experience from previous PTs and data from supervised residue trials. The doses used did not exceed the recommended dose for rye grass. However, because the formulation are sprayed directly on the grass that are harvested as hay, then the pesticide residue levels will be relatively higher. Cereal kernels are covered by

husk and are not in the same extent exposed to pesticides. The field spraying was performed by the Danish Center for Food and Agriculture at Aarhus University in 2017. Approximately, 3.5 kg of the harvested hay was delivered for preparation of the Test Item. The hay was sprayed with nine formulations (containing nine active substances) that was included in the Target List, see **Table 1**. Additionally, the hay was sprayed with six formulations which were not included in the Target List. The hay was milled to a particle size ≤ 1mm and mixed thoroughly by hand. Twenty 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.

1.4 Preparation of the 'blank' Test Item

The hay used to prepare the blank Test Item was obtained from organically produced rye grass from Germany. The hay was milled by same equipment as the incurred Test Item. Fifty gram portions were weighed out into screw-capped polyethylene plastic bottles, sealed, and stored in freezer until it was distributed to the participants.

1.5 Homogeneity test

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

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

The homogeneity test is to show that the between bottle variance is not greater than the within bottle variance. The acceptance criteria to show that the Test Items were sufficiently homogeneous for the proficiency test was that: $S_s^2 < c$ where S_s is the between-bottle sample standard deviation and $c = F_1 \times \sigma_{all}^2 + F_{2\,x}\, s_{an}^2$; F1 and F2 being constants with values of 1.83 and 0.93, respectively, from the 11 samples taken, σ_{all}^2 = 0.3 x FFP RSD (25%) x the analytical sampling mean for all pesticides, and s_{an} is the estimate of the analytical standard deviation, FFP RSD is the Fit For Purpose Relative Standard Deviation.

As all pesticides passed the homogeneity test, the treated Test Item was considered to be sufficiently homogenous and suitable for the EUPT-CF12.

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

Pesticides	Included in the Target List	Formulation	g.ai/ha
Azoxystrobin	x	Amisiar	250
Boscalid	x	Viverda	350
Epoxiconazole	x	Viverda	125
Fluxapyroxad	x	Imtrex	125
Metrafenone	x	Flexity	150
Pirimicarb	x	Pirimir G	100
Proquinazid	x	Talius	50
Pyraclostrobin	x	Viverda	150
Tau-Fluvalinate	x	Mavrik	48
Chlormequat [1]		Cycocel extra	1518
Chlorotalonil [1]		Bravo	1000
Fluroxypyr [1]		Starane 333 HL	133
Glyphosate [1]		Roundup Bio	1440
MCPA [1]		Metaxon	750
Trinexapac-ethyl [1]		Modus M	200

[1] With the intention of using the hay for other purposes, the rye grass was additionally sprayed with chlormequat, chlorotalonil, fluroxypyr, glyphosat, MCPA and trinexapac-ethyl. Especially, the use of glyphosate resulted in a more brownish colour of the treated hay compared to the untreated Test Item. However, no differences in the co-extracted compounds were observed in a GC-MS scan.

1.6 Stability tests

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

The stability test was performed according to ISO 13528, Annex B [5]. Two different storage temperatures were used; room temperature and -18 °C. Six sub-samples (analytical portions) were analysed on each test day. A pesticide is considered to be adequately stable if $| x1 - yi | \leq 0.3 \times \sigma$, where x1 is the mean value of the first stability test, yi the mean value of the last stability test and σ the standard deviation used for proficiency assessment (25% of the assigned value):

The dates of testing were as follows:

Day 1: 30 January 2018
Day 2: 16 February 2018
Day 3: 05 March 2018

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

Pesticides	Mean, mg/kg	S_s^2	c	$S_s^2 < c$
Azoxystrobin	0.991	0.00108	0.0127	Pass
Boscalid	11.9	0.00000	2.387	Pass
Epoxiconazole	2.90	0.02887	0.1177	Pass
Metrafenone	1.030	0.00314	0.0140	Pass
Fluxapyroxad	1.48	0.00055	0.0280	Pass
Proquinazad	0.871	0.00077	0.0093	Pass
Pyraclostrobin	6.25	0.00912	0.5877	Pass
Tau-fluvalinate	0.812	0.00219	0.0086	Pass

Table 3. Statistical evaluation of the stability test data

Pesticides	Mean, mg/kg	| x1 - yi |	0.3×σ	| x1 - yi | ≤ 0.3×σ
Azoxystrobin	0.777	0.047	0.060	Pass
Boscalid	11.9	0.079	0.870	Pass
Epoxiconazole	2.49	0.125	0.185	Pass
Fluxapyroxad	1.01	0.020	0.101	Pass
Metrafenone	0.843	0.049	0.067	Pass
Proquinazid	0.754	0.044	0.060	Pass
Pyraclostrobin	4.04	0.195	0.444	Pass
Tau-Fluvalinate	0.586	0.006	0.046	Pass

The results of the stability test for storage temperature -18 °C are given in **Table 3**. All pesticides passed the test at -18 °C and at room temperature. See the individual stability figures for all pesticides in **Appendix 4**.

1.7 Organisational details

1.7.1 Access to documents, registration and confidentiality

In the invitation letter, all NRLs and OfLs were requested to register using the online registration link from 5 December 2017 to 12 January 2018. 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 29 January 2018, one bottle of treated Test Item (20 g) and one bottle of blank Test Item (50 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 a few days earlier due the often very time-consuming customs clearance 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 (1 March 2018). 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

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

2.1 False positives and negatives

2.1.1 False positives

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

2.1.2 False negatives

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

2.2 Estimation of the true concentration (x_{pt})

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

2.3 Uncertainty of the assigned value

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

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

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

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

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

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

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

The percentage FFP-RSD is set at 25% based on experience from results of previous EUPTs . The EUPT-Panel reserves the right to also employ other approaches on a case-by-case basis considering analytical difficulties and experience gained from previous proficiency tests.
For informative purposes the robust relative standard deviation (CV*) is calculated according to ISO 13528:2015; Chapter 7.7 (Consensus value from participant results) following Algorithm A in Annex C.

2.5 Z scores

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

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

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

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

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

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

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

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

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

For evaluation of the overall performance of laboratories within Category A, the Average of the Squared z Score (AZ^2) will be used. The AZ^2 is calculated as follows:

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

where "n" is the number of each laboratory's z scores that were considered in this formula. For the calculation, any z-score > 5 was set at "5". Based on the AZ^2 achieved, the laboratories are classified as follows:

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

The AZ^2 is considered being of lesser importance than the individual z scores.

Laboratories within Category B are ranked according to the total number of pesticides that they correctly reported to be present in the Test Item. The number of acceptable z scores achieved is listed as well.

3. RESULTS

3.1 Summary of reported results

In total, 119 EU and EFTA laboratories, from 29 different countries (26 EU member states), agreed to participate in this proficiency test. Additionally, Malta was represented by UK NRL. Eight EU/EFTA participants did not submit results, among these was one NRL. Furthermore, 11 Third Countries registered for the PT. One of these laboratories did not submit results. The participating laboratories are listed in **Appendix 1.**

An overview of results submitted by laboratories from the EU and EFTA can be seen in **Table 4.** All reported analytical results for the pesticide residues are shown in **Table 10-13** and in **Appendix 5**. The methods used are presented in a separate electronic file. However, only results submitted by laboratories from EU and EFTA countries are included in **Table 4, 7-9 and 16** and in the z scores histograms in **Appendix 5.**

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

Pesticides	No. of reported results	No. of NA [1]	No. of false negatives	% of labs reporting results [2]
Azoxystrobin	101	10	0	91
Boscalid	101	10	0	91
Epoxiconazole	96	15	0	86
Fluxapyroxad	67	44	3	60
Metrafenone	69	42	1	62
Pendimethalin [3]	99[3]	12	0	89
Proquinazid	46	65	1	41
Pyraclostrobin	89	22	1	80
Tau-Fluvalinate	87	24	1	78

[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 = 111). False negatives are included in reported results.

[3] Pendimetalin is included for informative purposes only. See below

Azoxystrobin and boscalid was the most frequently analysed compound with 91% of the labs submitting results for these compounds. Epoxiconazole, pendimethalin, pyraclostrobin and tau-fluvalinate were analysed by 78-89% of the participating laboratories. Less than 60% of the laboratories analysed for fluxapyroxad and metrafenone and the voluntary compound proquinazid.

Pendimethalin was not intentionally sprayed at the rye grass. However, 77 of the 99 laboratories that analysed for pendimethalin reported a result proving that pendimethalin was present in the Test Item. The finding of pendimethalin is considered as drift from spraying of fields nearby. The level was too low (0.049 mg/kg) to access the data, so the data and z scores are shown only for informative reasons. Accordingly, the 22 laboratories that analysed for pendimethalin but didn't report any results are not judge as False Negative results.

3.1.1 False positives

Seven participants from EU and EFTA countries reported 7 results for 7 different additional pesticides above the MRRL that had not been used to treat the Test Item (**Table 5**). The pesticides were: cadusafos, lambda-cyhalothrin, thiophanate-methyl, biphenyl, metolachlor, fenpropathrin and 2-phenylphenol. In all cases the compounds were not detected either by the Organizer, or by the other participating laboratories. The reported results were therefore considered to be false positives.

Table 5. False positive results at or above 0.05 mg/kg, the concentration detected in mg/kg, the determination technique used, the reporting level and the MRRL in mg/kg.

Lab code	Pesticides	Concentration mg/kg	Determination technique	RL, mg/kg	MRRL, mg/kg
17	Cadusafos	0.05	LC-MS/MS QQQ	0.01	0.05
28	Lambda-cyhalothrin	13.3	GC-MS/MS (QQQ)		0.05
43	Thiophanate-methyl	0.17	LC-MS/MS QQQ	0.01	0.05
60	Biphenyl	0.357	GC-MS/MS (QQQ)	0.02	0.05
75	Metolachlor	0.54	1)	0.01	0.05
95	Fenpropathrin	0.07	GC-MS/MS (QQQ)	0.05	0.05
97	2-phenylphenol	0.185	GC-MS/MS (QQQ)	0.05	0.05

1) No information received.

3.1.2 Findings of compounds below 0.05 mg/kg

Apart from the false positive results above and the results for the pesticides listed in **Table 4**, the findings of pendimethalin (see above), the participants reported results for nine other pesticides, see **Table 6**. These results are not evaluated as false positives as the concentrations are below the MRRL at 0.05. The individual results for pirimicarb, pirimicarb-desmethyl, prosulfocarb, prothioconazole-desthio and tebuconazole are shown in **Appendix 6**.

The hay was field treated with pirimicarb, which explain the findings of pirimicarb and pirimicarb-desmethyl. Prosulfocarb is known to be transported by evaporation, wind and rain, so it seems to be the reason for these results. The findings of prothioconazole-desthio and tebuconazole are also considered as drift from spraying of fields nearby. The results for fluopyram, lindane, pirimiphos-methyl and zoxamide however, might be false positives but will not be evaluated as this because the results are below the MRRLs.

Table 6. Reported results in mg/kg at or below the MRRL at 0.05 mg/kg .

Pesticides	No. of results	Minimum concentration mg/kg	Maximum concentration mg/kg
Fluopyram	1	0.0014	
Lindane	1	0.0012	
Pirimicarb	7	0.0058	0.0123
Pirimicarb-desmethyl	11	0.0058	0.015
Pirimiphos-methyl	1	0.0013	
Prosulfocarb	21	0.0095	0.028
Prothioconazole-desthio	14	0.0065	0.019
Tebuconazole	5	0.0054	0.0248
Zoxamide	1	0.0248	

3.1.3 False negatives

Not reported results for pesticides actually present in the treated Test Item were judged as false negatives. **Table 7** summarizes the number of reported false negatives for each pesticide. Five laboratories submitted 8 false negatives results for the 5 different pesticides, which represents more than 1% of the total number of results. In previous PTs, typically 20-30% of the labs reported false negative results, except from the last years PT (EUPT-CF12 on hay flour) where around 35% of the participants (51 laboratories) reported false negative results. This year only 4% of the laboratories reported false negative results. This is probably due to the relatively high pesticide residues levels present in the hay flour.

Table 7. False negative results (FN) for EU and EFTA country as well as for Candidate and Third Countries.

Lab code	Fluxapyroxad	Metrafenone	Pyraclostrobin	Tau-Fluvalinate	Proquinazid
5	FN				
44	FN		FN	FN	
87					FN
90	FN	FN			
102				FN	

3.2 Assigned values and target standard deviations

The Assigned Values were calculated as the Algorithm A mean, including the reported results submitted by laboratories from EU and EFTA countries. However, because of earlier experience with significantly biased results from laboratories not adding water to the sample before extraction (or using a mixture of water and extraction solvent) these results were not included in the calculation of the Algorithm A mean. Also results from laboratories that did not provide information about their extraction method were excluded from the calculations.

All Assigned Values for the pesticides can be seen in **Table 8**. The target standard deviation was obtained using a fixed FFP-RSD value of 25 %. In parallel, the Algorithm A standard deviation (Alg A-RSD) was calculated for informative purposes only. The range of Alg A-RSD values was 14-23 % but on average the Alg A-RSD was 16%, and thus below the 25% FFP-RSD used for the calculations.

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

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

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

The assigned values are high, for four of them > 1 mg/kg. However, as explained in **Chapter 1.3**, the doses used for the field treatment did not exceed the recommended doses for rye grass. However, because the formulation are sprayed directly on the grass that are harvested as hay, then the pesticide residue levels will be relatively higher, contrary cereal kernels that are covered by husk and not in the same extent exposed to the pesticides.

As explained in Chapter 1.3, the pesticide residue levels are high, for some of the pesticides >5mg/kg

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

Pesticides	MRRL, mg/kg	Assigned value, mg/kg	Uncertainty, mg/kg	FFP RSD, %	Alg A RSD, %
Azoxystrobin	0.05	0.799	0.020	25	20
Boscalid	0.05	11.6	0.290	25	20
Epoxiconazole	0.05	2.47	0.069	25	22
Fluxapyroxad	0.05	1.34	0.041	25	20
Metrafenone	0.05	0.897	0.021	25	18
Pendimethalin [3]	0.05	0.049	0.001	25	20
Proquinazid	0.05	0.805	0.018	25	13
Pyraclostrobin	0.05	5.94	0.183	25	23
Tau-Fluvalinate	0.05	0.618	0.022	25	26

[3] Informative purpose only

3.3 Assessment of laboratory performance

3.3.1 Z scores

Z scores have been calculated for all the quantified pesticides using the FFP RSD of 25 %. **Table 9** shows an overview of the acceptable, questionable and unacceptable z scores and **Tables 10-13** 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 5**.

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

Pesticides	No. of reported results	Assigned values	Acceptable z scores	Questionable z scores	Unacceptable z scores [1]	False negatives [1]
Azoxystrobin	101	0.794	95	3	3	0
Boscalid	101	11.5	96	2	3	0
Epoxiconazole	96	2.48	92	2	2	0
Fluxapyroxad	67	1.35	62	2	3	3
Metrafenone	69	0.894	67	1	1	1
Pendimethalin [2]	77	0.049	73	2	2	0
Proquinazid	46	0.803	44	1	1	1
Pyraclostrobin	89	5.94	78	5	6	1
Tau-Fluvalinate	87	0.617	77	7	3	1

[1] Unacceptable z scores include those for false negative results.
[2] Informative purpose only

For metrafenone, epoxiconazole and proquinazid acceptable z scores were obtained by 96-97% of the laboratories. For boscalid, pendimethalin, azoxystrobin and fluxapyroxad acceptable z scores were obtained by 93-95% of the laboratories, and finally 88-89% of the laboratories obtained acceptable z scores for tau-fluvalinate and pyraclostrobin.

Several different analytical methods have been used by the laboratories. QuEChERS Citrate buffered (EN 151662) was used for 71% of the reported results. However, variations in the clean-up procedures were reported by the labs, e.g. some used a freezing out step, some used PSA, others PSA/C18 or PSA/ODS or PSA/GCB. So it is not one specific method. Two other QuEChERS methods were used, the Original Version (J. AOAC 86, (2003) 412) and the Acetate buffered (AOAC Official Method 2007.01). These were used for 6% and 8% of the results, respectively. The SweEt method (NMKL 195, 2013) was used for 5%, Mini Luke 3%. Finally

2% of the results were analysed by other methods (e.g. in house method) and for 3% of the results no information on the reference method was given by the laboratories.

Exactly 96% of the reported results derived from a method where water was added before extraction and for 1% of the results no information was given. Likewise, no information was given concerning the use of an ISTD for 8% of the results, while 57% of the results were produced with the use of an ISTD and 44% without an ISTD. Around 62% of the results were based on a mechanical agitation. Finally, GC instruments was used for 35% of the results, mainly GC-MS/MS and GC-MSD (32% and 4%). Only 1% used GC-iontrap and the rest (3%) used GC with specific detectors, mainly ECD but also NPD. LC instruments was used for 63% of the reported results, mainly LC-MS/MS (49%) but 4% used high resolution instrument like LC-Orbitrap or LC-Q-TOF.

Table 10. Results for azoxystrobin, boscalid, epoxiconazole, fluxapyroxad, metrafenone, pendimethalin, proquinazid, pyraclostrobin and tau-Fluvalinate in mg/kg, and the corresponding z scores, MRRLs and the assigned values.

Laboratory code	Azoxystrobin	Z score (FFP RSD (25%)	Boscalid	Z score (FFP RSD (25%)	Epoxiconazole	Z score (FFP RSD (25%)	Fluxapyroxad	Z score (FFP RSD (25%)	Metrafenone	Z score (FFP RSD (25%)	Pendimethalin [1]	Z score (FFP RSD (25%)	Proquinazid [2]	Z score (FFP RSD (25%)	Pyraclostrobin	Z score (FFP RSD (25%)	Tau-Fluvalinate	Z scores (FFP RSD (25%)
MRRL	**0.05**		**0.05**		**0.05**		**0.05**		**0.05**		**0.05**		**0.05**		**0.05**		**0.05**	
Assigned value	**0.794**		**11.5**		**2.48**		**1.35**		**0.894**		**0.049**		**0.803**		**5.94**		**0.617**	
1	0.839	0.2	12.9	0.5	2.41	-0.1	1.35	0.0	0.952	0.3	0.0508	0.1	0.728	-0.4	5.86	-0.1	0.745	0.8
2			9	-0.9	2.13	-0.6					0.0485	-0.1					0.398	-1.4
3	1.01	1.1	14	0.9	2.44	-0.1	1.08	-0.8	1.03	0.6	0.063	1.1	0.94	0.7	7.48	1.0	0.775	1.0
4	0.911	0.6	13.5	0.7	3.15	1.1	1.7	1.0	1.07	0.8	0.053	0.3	0.955	0.8	6.7	0.5	0.799	1.2
5	0.888	0.5	16.32	1.7	3.268	1.3	FN	-3.9	0.852	-0.2	0.055	0.4	0.752	-0.3	10.43	3.0	0.463	-1.0
6	0.846	0.3	12.1	0.2	2.31	-0.3	1.3	-0.1	0.93	0.2	0.046	-0.3	0.681	-0.6	6.55	0.4	0.567	-0.3
7	0.788	0.0	9.67	-0.6	2.25	-0.4	1.34	0.0	0.747	-0.7			0.841	0.2	4.67	-0.9	0.781	1.1
8	0.72	-0.4	10.2	-0.5	2.34	-0.2	1.22	-0.4	0.74	-0.7			0.68	-0.6	5.5	-0.3	0.62	0.0
9																		
10	0.7	-0.5	10.5	-0.4	2.29	-0.3	1.24	-0.3	0.862	-0.1	0.049	0.0	0.798	0.0	5.46	-0.3	0.887	1.7
11	0.538	-1.3	9.59	-0.7	1.61	-1.4					0.0823	2.7			4.62	-0.9	0.459	-1.0
12																		
13	0.946	0.8	12.61	0.4	3.07	1.0									7.26	0.9	0.837	1.4
14																		
15	0.817	0.1	12.6	0.4	2.57	0.1			0.978	0.4	0.0556	0.5			6.49	0.4	0.664	0.3
16	0.951	0.8	13.3	0.6	2.83	0.6	1.54	0.6	0.912	0.1	0.051	0.1	0.767	-0.2	6.88	0.6	0.732	0.7
17	1.05	1.3	14.1	0.9	1.71	-1.2	2.09	2.2	1.16	1.2	0.438	>5	1.024	1.1	5.353	-0.4	0.45	-1.1
18											0.0573	0.6						
19																		
20	0.841	0.2	11	-0.2	2.39	-0.1	1.75	1.2	1.05	0.7	0.0597	0.8	1.21	2.0	5.17	-0.5	0.646	0.2
21	0.714	-0.4	11.1	-0.2	1.96	-0.8	1.05	-0.9	0.68	-1.0	0.0432	-0.5	0.798	0.0	3.91	-1.4	1.01	2.5
22	0.85	0.3	13.1	0.5	2.45	0.0					0.046	-0.3			5.81	-0.1		
23	0.786	0.0	10.9	-0.2	2.36	-0.2	1.31	-0.1	0.997	0.5	0.042	-0.6	0.86	0.3	6.22	0.2		
24	0.862	0.3	14.4	1.0	2.1	-0.6			0.986	0.4	0.0621	1.0	0.853	0.2	7.41	1.0	0.651	0.2
25	0.659	-0.7	8.21	-1.2	2.04	-0.7	0.85	-1.5	0.726	-0.8	0.0508	0.1	0.609	-1.0	4.98	-0.6	0.551	-0.4
26																		
27	0.062	-3.7																
28	0.916	0.6	13.5	0.7	2.62	0.2									6.61	0.5	0.712	0.6
29	0.61	-0.9	10.88	-0.2	1.64	-1.4					0.03	-1.6					0.52	-0.6
30	0.908	0.6	14.6	1.1							0.0343	-1.2						
31	0.226	-2.9	5.28	-2.2	0.608	-3.0	0.4	-2.8	0.060	-3.7	0.0292	-1.6			0.303	-3.8	0.244	-2.4
32	1.09	1.5	17.3	2.0	3.4	1.5			1.22	1.5					7.56	1.1		
33	0.96	0.8	12.81	0.4	2.3	-0.3									6.41	0.3	0.386	-1.5
34																		
35	0.697	-0.5	11.4	-0.1	2.47	0.0	1.43	0.2	0.898	0.0	0.0439	-0.4			5.97	0.0	0.899	1.8
36																		
37	0.774	-0.1	8.88	-0.9	1.98	-0.8	1.17	-0.5	0.864	-0.1	0.053	0.3	0.783	-0.1	8.7	1.9	0.545	-0.5
38																		
39	0.896	0.5	13.1	0.5	2.68	0.3	1.37	0.1	0.974	0.4	0.0483	-0.1	0.768	-0.2	7.03	0.7	0.667	0.3
40	0.685	-0.5	13.2	0.6	2.81	0.5	1.05	-0.9	0.828	-0.3	0.05	0.0			8.52	1.7	0.685	0.4
41	0.919	0.6	12.86	0.5													0.5	-0.8

Table 11. Results for azoxystrobin, boscalid, epoxiconazole, fluxapyroxad, metrafenone, pendimethalin, proquinazid, pyraclostrobin and tau-Fluvalinate in mg/kg, and the corresponding z scores, MRRLs and the assigned values.

Laboratory code	Azoxystrobin	Z score (FFP RSD (25%)	Boscalid	Z score (FFP RSD (25%)	Epoxiconazole	Z score (FFP RSD (25%)	Fluxapyroxad	Z score (FFP RSD (25%)	Metrafenone	Z score (FFP RSD (25%)	Pendimethalin [1]	Z score (FFP RSD (25%)	Proquinazid [2]	Z score (FFP RSD (25%)	Pyraclostrobin	Z score (FFP RSD (25%)	Tau-Fluvalinate	Z scores (FFP RSD (25%)
MRRL	**0.05**		**0.05**		**0.05**		**0.05**		**0.05**		**0.05**		**0.05**		**0.05**		**0.05**	
Assigned value	**0.794**		**11.5**		**2.48**		**1.35**		**0.894**		**0.049**		**0.803**		**5.94**		**0.617**	
42	1024	>5	13844	>5														
43	0.69	-0.5	16	1.5	2.5	0.0	1.2	-0.4	0.84	-0.2	0.056	0.5			5	-0.6	0.54	-0.5
44	0.101	-3.5	3.24	-2.9	0.993	-2.4	FN	-3.9	0.643	-1.1					FN	-4.0	FN	-3.7
45																		
46	0.57	-1.1	7.9	-1.3	1.8	-1.1					0.065	1.3			4.06	-1.3	0.69	0.5
47	0.938	0.7	14.4	1.0	2.49	0.0	1.52	0.5	0.442	-2.0	0.049	0.0	0.984	0.9	7.35	1.0	0.607	-0.1
48	0.703	-0.5	12.9	0.5	3.2	1.2	0.86	-1.4	0.982	0.4	0.058	0.7			3.2	-1.8	0.744	0.8
49	1.2	2.0			4.25	2.9												
50	0.756	-0.2	12.9	0.5	2.96	0.8	1.48	0.4	1.03	0.6	0.050	0.1	0.756	-0.2	6.12	0.1	0.725	0.7
51	0.833	0.2	9.672	-0.6	2.661	0.3	1.432	0.2	0.987	0.4	0.06	0.9			5.928	0.0		
52	0.946	0.8	14.7	1.1	2.75	0.4	1.76	1.2	1.16	1.2	0.048	-0.1			6.71	0.5	0.775	1.0
53																		
54	1.23	2.2	11.89	0.1	2.63	0.2			1.02	0.6	0.050	0.1			6.85	0.6	0.654	0.2
55	0.83	0.2	13.1	0.5							0.13	>5			5.81	-0.1	0.81	1.2
56	0.836	0.2	13.3	0.6	2.94	0.7	1.29	-0.2	1.15	1.1	0.053	0.3	0.557	-1.2	7.24	0.9	0.834	1.4
57	0.56	-1.2	10.8	-0.3	2.7	0.4	1.5	0.5	0.66	-1.0	0.053	0.3	0.73	-0.4	5.2	-0.5	0.69	0.5
58	0.589	-1.0	0.758	-3.7	0.542	-3.1					0.041	-0.7			1.13	-3.2	0.478	-0.9
59	0.440	-1.8	9.689	-0.6	1.640	-1.4	0.975	-1.1	0.487	-1.8	0.035	-1.2			4.085	-1.2	0.540	-0.5
60	0.874	0.4	10.1	-0.5	2.58	0.2	1.53	0.5	0.925	0.1			0.826	0.1	6.41	0.3	1.66	>5
61																		
62	0.84	0.2	10.6	-0.3	3.05	0.9	1.26	-0.3	0.706	-0.8			0.763	-0.2	6.82	0.6	1.11	3.2
63	0.866	0.4	13.5	0.7	2.01	-0.8	0.823	-1.6			0.048	0.0			6.02	0.1	0.623	0.0
64	0.825	0.2	11.05	-0.2	3.047	0.9					0.038	-0.9						
65	0.822	0.1	10.8	-0.3	2.88	0.7					0.039	-0.8						
66	0.91	0.6	9.89	-0.6	2.56	0.1					0.052	0.2						
67	0.78	-0.1	10.5	-0.4	2.9	0.7					0.036	-1.0						
68	0.809	0.1	9.56	-0.7	2.65	0.3					0.045	-0.3			2.8	-2.1		
69	0.767	-0.1	10.8	-0.3	2.74	0.4					0.040	-0.8						
70	0.796	0.0	10.3	-0.4	3.02	0.9					0.048	-0.1						
71	0.917	0.6	14.3	1.0	2.82	0.6	1.59	0.7	0.872	-0.1			0.723	-0.4	5.91	0.0	0.63	0.1
72	0.777	-0.1	12.3	0.3	2.37	-0.2			0.887	0.0	0.068	1.5			6.25	0.2	0.56	-0.4
73	0.986	1.0	12.8	0.4	2.96	0.8									2.55	-2.3	0.714	0.6
74																		
75	0.641	-0.8	12.27	0.3	2.51	0.1					0.042	-0.6					0.704	0.6
76																		
77	0.553	-1.2	6.28	-1.8							0.033	-1.3			3.45	-1.7	0.504	-0.7
78	0.94	0.7	12.7	0.4	2.72	0.4	1.36	0.0	1	0.5			0.79	-0.1	6.33	0.3	0.781	1.1
79	0.77	-0.1	16.2	1.6	3.1	1.0					0.054	0.4			7.6	1.1	0.62	0.0
80	0.769	-0.1	11.8	0.1	3.09	1.0					0.059	0.8			5.65	-0.2	0.628	0.1
81	0.367	-2.2	10.2	-0.5	1.3	-1.9					0.037	-1.0			4.06	-1.3	0.48	-0.9
82	0.61	-0.9	10.2	-0.5	2.73	0.4	1.74	1.2	1.19	1.3	0.044	-0.4			6.34	0.3	0.595	-0.1

Table 12. Results for azoxystrobin, boscalid, epoxiconazole, fluxapyroxad, metrafenone, pendimethalin, proquinazid, pyraclostrobin and tau-Fluvalinate in mg/kg, and the corresponding z scores, MRRLs and the assigned values.

Laboratory code	Azoxystrobin	Z score (FFP RSD (25%)	Boscalid	Z score (FFP RSD (25%)	Epoxiconazole	Z score (FFP RSD (25%)	Fluxapyroxad	Z score (FFP RSD (25%)	Metrafenone	Z score (FFP RSD (25%)	Pendimethalin [1]	Z score (FFP RSD (25%)	Proquinazid [2]	Z score (FFP RSD (25%)	Pyraclostrobin	Z score (FFP RSD (25%)	Tau-Fluvalinate	Z scores (FFP RSD (25%)
MRRL	**0.05**		**0.05**		**0.05**		**0.05**		**0.05**		**0.05**		**0.05**		**0.05**		**0.05**	
Assigned value	**0.794**		**11.5**		**2.48**		**1.35**		**0.894**		**0.049**		**0.803**		**5.94**		**0.617**	
83	**0.905**	**0.6**	**15.9**	**1.5**	**2.68**	**0.3**			**0.922**	**0.1**	**0.0713**	**1.8**	**0.829**	**0.1**	**6.54**	**0.4**	**0.574**	**-0.3**
84																		
85	0.795	0.0									0.061	0.9			4.27	-1.1		
86	0.77	-0.1	10.8	-0.3	2.05	-0.7					0.053	0.3			9.97	2.7	0.96	2.2
87	0.445	-1.8	6.52	-1.7	1.34	-1.8	0.743	-1.8	0.606	-1.3	0.023	-2.1	FN	-3.8	4.89	-0.7	0.263	-2.3
88	0.671	-0.6	14.4	1.0	2.09	-0.6					0.048	-0.1			5.48	-0.3	0.447	-1.1
89	0.879	0.4	12.7	0.4	2.99	0.8	1.57	0.7	0.939	0.2	0.052	0.2			7.34	0.9	0.581	-0.2
90	0.64	-0.8	9.25	-0.8	1.54	-1.5	FN	-3.9	FN	-3.8					5.26	-0.5	0.747	0.8
91	0.877	0.4	12.61	0.4	2.852	0.6	1.524	0.5	0.939	0.2	0.055	0.4	0.782	-0.1	6.517	0.4	0.648	0.2
92																		
93	0.52	-1.4	10	-0.5	1.529	-1.5	0.693	-1.9	0.349	-2.4	0.05	0.0			1.706	-2.9	0.467	-1.0
94	0.74	-0.3	11.7	0.1	2.65	0.3	1.44	0.3	0.986	0.4	0.0565	0.6			6.69	0.5	0.66	0.3
95	1.04	1.2	10.8	-0.3	2.85	0.6	1.58	0.7	0.979	0.4	0.0675	1.5			6.4	0.3	0.555	-0.4
96	0.442	-1.8	6.023	-1.9	1.394	-1.7									2.228	-2.5	0.291	-2.1
97	0.862	0.3	12.45	0.3	2.81	0.5	1.4	0.2	1.05	0.7					6.19	0.2	0.685	0.4
98	0.71	-0.4	11	-0.2	2.64	0.3	1.41	0.2	0.928	0.2			0.702	-0.5	6.3	0.2	0.73	0.7
99	4.48	>5	2.47	-3.1	1.09	-2.2	0.941	-1.2							0.522	-3.6	0.432	-1.2
100	0.84	0.2	12.5	0.3	2.5	0.0					0.029	-1.7			7.2	0.9	0.27	-2.3
101	0.57	-1.1	8.8	-1.0	2.13	-0.6	1.07	-0.8	0.683	-0.9	0.036	-1.1	0.517	-1.4	4.73	-0.8	0.325	-1.9
102	0.776	-0.1			2.26	-0.4									6.72	0.5	FN	-3.7
103	0.86	0.3	11.7	0.1	2.63	0.2	1.27	-0.2	0.903	0.0			0.813	0.0	6.19	0.2	0.52	-0.6
104	0.873	0.4	11.2	-0.1	2.37	-0.2	1.25	-0.3	0.872	-0.1					6.13	0.1	0.491	-0.8
105	0.868	0.4	11.2	-0.1	2.77	0.5	1.24	-0.3	0.912	0.1			0.816	0.1	6.31	0.3	0.508	-0.7
106	0.882	0.4	10.7	-0.3	2.75	0.4	1.26	-0.3	0.897	0.0			0.781	-0.1	6.21	0.2	0.503	-0.7
107	0.802	0.0	12	0.2	2.52	0.1	1.32	-0.1	0.88	-0.1	0.0534	0.3	0.755	-0.2	6.67	0.5	0.504	-0.7
108	0.89	0.5	12.2	0.2	2.77	0.5	1.53	0.5	0.879	-0.1	0.05	0.0	0.846	0.2	6.38	0.3	0.367	-1.6
109	0.958	0.8	12.1	0.2	2.51	0.1	1.45	0.3	0.786	-0.5	0.0418	-0.6	0.872	0.3	7.03	0.7	0.701	0.5
110	0.664	-0.7	13.332	0.6	2.782	0.5	1.354	0.0	0.923	0.1	0.049	0.0	1.362	2.8	6.448	0.3	0.68	0.4
111	0.836	0.2	15.7	1.4	2.11	-0.6	1.28	-0.2	0.958	0.3	0.052	0.2			10.9	3.3	0.701	0.5
112	0.839	0.2	11.6	0.0	2.52	0.1	1.32	-0.1	0.99	0.4			0.82	0.1	7.06	0.8	0.616	0.0
113	0.85	0.3	13	0.5	1.9	-0.9	1.8	1.3	0.93	0.2	0.056	0.5	0.92	0.6	5.2	-0.5	1.08	3.0
114	0.628	-0.8	9.86	-0.6	1.98	-0.8	1.43	0.2	0.88	-0.1	0.0479	-0.1			6.04	0.1	0.761	0.9
115	0.953	0.8	13.1	0.5	2.91	0.7	1.57	0.7	1.04	0.7	0.058	0.7			6.49	0.4	0.705	0.6
116	0.913	0.6	13.3	0.6	2.45	0.0	1.45	0.3	1.03	0.6	0.054	0.4	0.856	0.3	7.1	0.8	0.742	0.8
117	0.8	0.0	11.88	0.1	3.27	1.3	1.36	0.0	0.935	0.2					6.92	0.7	0.575	-0.3
118	0.7	-0.5	7.7	-1.3	1.6	-1.4	1.04	-0.9	0.65	-1.1	0.04	-0.8			5.96	0.0	0.47	-1.0
119	1.018	1.1	13.027	0.5	2.869	0.6	1.629	0.8	0.99	0.4	0.048	-0.1	0.864	0.3	6.296	0.2	0.732	0.7
120	0.845	0.3	10.4	-0.4	2.8	0.5					0.038	-0.9						
121																		
122	0.516	-1.4	9.5	-0.7	1.79	-1.1	0.94	-1.2	0.686	-0.9	0.0404	-0.7	0.614	-0.9	4.67	-0.9	0.626	0.1
123																		

Table 13. Results for azoxystrobin, boscalid, epoxiconazole, fluxapyroxad, metrafenone, pendimethalin, proquinazid, pyraclostrobin and tau-Fluvalinate in mg/kg, and the corresponding z scores, MRRLs and the assigned values.

Laboratory code	Azoxystrobin	Z score (FFP RSD (25%)	Boscalid	Z score (FFP RSD (25%)	Epoxiconazole	Z score (FFP RSD (25%)	Fluxapyroxad	Z score (FFP RSD (25%)	Metrafenone	Z score (FFP RSD (25%)	Pendimethalin [1]	Z score (FFP RSD (25%)	Proquinazid [2]	Z score (FFP RSD (25%)	Pyraclostrobin	Z score (FFP RSD (25%)	Tau-Fluvalinate	Z scores (FFP RSD (25%)
MRRL	**0.05**		**0.05**		**0.05**		**0.05**		**0.05**		**0.05**		**0.05**		**0.05**		**0.05**	
Assigned value	**0.794**		**11.5**		**2.48**		**1.35**		**0.894**		**0.049**		**0.803**		**5.94**		**0.617**	
125	0.94	0.7	12.7	0.4	2.6	0.2	1.5	0.5	0.99	0.4	0.055	0.4	0.88	0.4	6.5	0.4	0.777	1.0
125	0.94	0.7	12.7	0.4	2.6	0.2	1.5	0.5	0.99	0.4	0.055	0.4	0.88	0.4	6.5	0.4	0.777	1.0
126	0.879	0.4	13.50	0.7	2.51	0.1	1.285	-0.2	1.015	0.5	0.042	-0.6	0.704	-0.5	3.858	-1.4	0.444	-1.1
127	0.855	0.3	14.62	1.1	2.779	0.5	1.43	0.2	0.752	-0.6	0.04	-0.8	0.701	-0.5	6.685	0.5	0.504	-0.7
128	1.23	2.2	17	1.9	3.61	1.8	1.96	1.8	1.29	1.8	0.059	0.8	1.13	1.6	7.86	1.3	0.738	0.8
129	0.768	-0.1	9.91	-0.6	2.45	0.0	1.58	0.7	0.834	-0.3			0.754	-0.2	4.61	-0.9	0.459	-1.0
130	0.581	-1.1	9.52	-0.7	2.23	-0.4	1.38	0.1	0.908	0.1	0.057	0.7			4.9	-0.7	0.48	-0.9

[1] Informative purpose
[2] Voluntary pesticide

3.3.2 Sum of Weighted Z scores (AZ^2) – Category A

To be classified into Category A, the laboratories had to submit quantitative results for at least 90 % of the compulsory pesticides present in the Test Item (≥6 pesticide residues, exclusive of any false negatives results), analyse for more than 90% of the compulsory pesticides on the target list and also report no false positive results. For the 59 EU and EFTA laboratories in Category A (51%), the results were additionally evaluated by calculating the Average of the Squared -Score (AZ^2). Of the 59 participants, 54 participants (92%) obtained AZ^2 values at or below 2 (good) and 2 participants (3.4%) obtained AZ^2 values between 2-3 (satisfactory) and 3 participants (5.1%) obtained AZ^2 values ≥3 (unsatisfactory). Additional five laboratories from Third Countries were evaluated and classified into Category A. The AZ^2 scores achieved by the labs can be seen in **Table 14.**

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

Lab code	No. of detected compulsory pesticides	No. of detected voluntary pesticides	AZ^2	No. Of False negatives	Classification	NRL
1	7	1	0.1	0	Good	
3	7	1	0.7	0	Good	
4	7	1	0.8	0	Good	Yes
5	6	1	4.2	1	Unsatisfactory	
6	7	1	0.1	0	Good	
7	7	1	0.4	0	Good	
8	7	1	0.2	0	Good	
10	7	1	0.5	0	Good	Yes
15	6	0	0.1	0	Good	
16	7	1	0.4	0	Good	
20	7	1	0.3	0	Good	
21	7	1	1.6	0	Good	

Lab code	No. of detected compulsory pesticides	No. of detected voluntary pesticides	AZ^2	No. Of False negatives	Classification	NRL
23	6	1	0.1	0	Good	Yes
24	6	1	0.4	0	Good	
25	7	1	0.8	0	Good	
31	7	0	>5	0	Unsatisfactory	Yes
35	7	0	0.5	0	Good	
37	7	1	0.8	0	Good	
39	7	1	0.2	0	Good	
40	7	0	0.7	0	Good	
47	7	1	1.0	0	Good	Yes
48	7	0	1.2	0	Good	Yes
50	7	1	0.3	0	Good	Yes
52	7	0	0.9	0	Good	Yes
54	6	0	0.9	0	Good	
56	7	1	0.7	0	Good	
57	7	1	0.5	0	Good	
59	7	0	1.7	0	Good	Yes
62	7	1	1.8	0	Good	Yes
71	7	1	0.3	0	Good	
72	6	0	0.0	0	Good	
78	7	1	0.3	0	Good	Yes
82	7	0	0.6	0	Good	
87	6	1	2.9	1	Satisfactory	
89	7	0	0.4	0	Good	
91	7	1	0.2	0	Good	
93	7	0	3.3	0	Unsatisfactory	Yes
94	7	0	0.1	0	Good	
98	7	1	0.1	0	Good	
101	7	1	1.2	0	Good	
103	7	1	0.1	0	Good	Yes
104	7	0	0.1	0	Good	
105	7	1	0.1	0	Good	
106	7	1	0.2	0	Good	
107	7	1	0.1	0	Good	Yes
108	7	1	0.5	0	Good	Yes
109	7	1	0.3	0	Good	Yes
110	7	1	0.2	0	Good	Yes
111	7	0	2.0	0	Good	
112	7	1	0.1	0	Good	
113	7	1	1.8	0	Good	

Lab code	No. of detected compulsory pesticides	No. of detected voluntary pesticides	AZ^2	No. Of False negatives	Classification	NRL
114	7	0	0.4	0	Good	Yes
115	7	0	0.4	0	Good	
116	7	1	0.4	0	Good	
117	7	0	0.3	0	Good	Yes
118	7	0	1.0	0	Good	
119	7	1	0.5	0	Good	
122	7	1	1.0	0	Good	
125	7	1	0.3	0	Good	
126	7	1	0.6	0	Good	
127	7	1	0.4	0	Good	
128	7	1	2.9	0	Satisfactory	
129	7	1	0.4	0	Good	
130	7	0	0.4	0	Good	Yes

The laboratories that did not fulfil the requirements described above, were classified in Category B. The number of reported quantitative results, analysed compounds from the Target List and acceptable z scores as well as information on false negative and positive results are shown in **Table 15.** Five participants were moved from Category A to B, due to false positive results (marked with *). One participants fulfilled the criteria of detecting 90 % of the compulsory pesticides in the Test Item but did not fulfil the criteria of analysing for 90 % of the compulsory pesticides on the Target List. The reverse was the case for six participants. Twenty-two participants (19%) analysed and detected less than 70% of the pesticides present in the Test Item.

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

Lab code	No. of compulsory pesticides detected	Compulsory pesticides detected, %	Analysed of compulsory pesticides on Target List	No. Of voluntary pesticides detected	No. of acceptable z scores	No. of false negative	No. of false positive	NRL
2	3	43	42	0	3	0	0	Yes
9	0	0	15	0	0	0	0	
11	5	71	85	0	5	0	0	Yes
12	0	0	6	0	0	0	0	
13	5	71	93	0	5	0	0	Yes
14								
17 *	7	100	95	1	6	0	1	
18	0	0	32	0	0	0	0	Yes
19								
22	4	57	82	0	4	0	0	
26	0	0	99	0	0	0	0	
27	1	14	17	0	0	0	0	Yes
28	5	71	88	0	5	0	1	
29	4	57	41	0	4	0	0	
30	2	29	36	0	2	0	0	

Lab code	No. of compulsory pesticides detected	Compulsory pesticides detected, %	Analysed of compulsory pesticides on Target List	No. Of voluntary pesticides detected	No. of acceptable z scores	No. of false negative	No. of false positive	NRL
32	5	71	43	0	5	0	0	
33	5	71	75	0	5	0	0	
34	1	14	6	0	0	0	0	
36								
38								Yes
41	3	43	37	0	3	0	0	
42	2	29	25	0	0	0	0	
43 *	7	100	100	0	7	0	1	
44 **	7	57	100	0	1	3	0	
45								
46	5	71	57	0	5	0	0	
49	2	29	32	0	0	0	0	
51	6	86	63	0	6	0	0	Yes
53								
55	4	57	41	0	4	0	0	
58	5	71	86	0	2	0	0	
60 *	7	100	100	1	6	0	1	Yes
61	0	0	6	0	0	0	0	
63	6	86	69	0	6	0	0	
64	3	43	45	0	3	0	0	
65	3	43	45	0	3	0	0	
66	3	43	37	0	3	0	0	
67	3	43	38	0	3	0	0	
68	4	57	44	0	3	0	0	
69	3	43	41	0	3	0	0	
70	3	43	45	0	3	0	0	
73	5	71	94	0	4	0	0	
74	0	0	6	0	0	0	0	
75	4	57	61	0	4	0	1	
76								
77	4	57	52	0	4	0	0	
79	5	71	97	0	5	0	0	
80	5	71	87	0	5	0	0	
81	5	71	68	0	4	0	0	
83	6	86	78	1	6	0	0	
84	0	0	23	0	0	0	0	
85	2	29	61	0	2	0	0	
86	5	71	66	0	3	0	0	

Lab code	No. of compulsory pesticides detected	Compulsory pesticides detected, %	Analysed of compulsory pesticides on Target List	No. Of voluntary pesticides detected	No. of acceptable z scores	No. of false negative	No. of false positive	NRL
88	5	71	73	0	5	0	0	
90	7	71	100	0	5	2	1	
92								
95 *	7	100	99	0	7	0	1	Yes
96	5	71	69	0	3	0	0	
97 *	7	100	98	0	7	0	1	
99	6	86	76	0	2	0	0	Yes
100	5	71	61	0	4	0	0	
102	4	43	63	0	3	1	0	
120	3	43	45	0	3	0	0	
121	0	0	6	0	0	0	0	
123	0	0	6	0	0	0	0	
124	0	0	6	0	0	0	0	

* Moved from Category A due to false positive results
** Detected and quantified all pesticide residues in the Test Item but did not fulfil the requirement of analysing 90% or more of the Target List

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. However, it is observed that when the Test Item is a feed commodity, the number of participants decreases (**Table 16**), because many of the laboratories have not feed in their scope. The number of pesticides included in the Target Pesticide List has increased during this 7-year period, from 107 to 155 individual mandatory compounds and 23 voluntary compounds. The number of spiked or incurred pesticides contained in the Test Items has typically been around 18. However, in the EUPT-CF12 only 8 pesticides were included. Still many laboratories have a limited scope and are therefore not able to cover all pesticides in the PT. Among the laboratories submitting results, 19% submitted results for less than 70% of pesticides present in the Test Item.

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

PT and type of test item	EUPT-C6 Barley flour	EUPT-C7 Feed [1)]	EUPT-CF8 Wheat flour	EUPT-CF9 Maize flour [2)]	EUPT-CF10 Rye flour	EUPT-CF11 Oat flour	EUPT-CF12 Hay flour 3[)]
Participants submitting results (EU and EFTA)	127	106	142	143	160	149	111
MRM pesticides in the Target Pesticide List	107	116	111	117	134/7	153/9	155/23
MRM pesticides in the test material	18	23	17	18	16	18	8
No. of results for MRM pesticides	1741	1932	1893	2012	2012	2172	808
Average of 'reported results', %	76	83	78	78	79	83	74
Range of 'reported results', %	50-95	60-97	49-93	61-94	58-90	65-93	40-91
Acceptable z scores, %	90	90	90	89	95	89	93
Questionable z scores, %	5	4	6	2	2	3	3
Unacceptable z scores, %	4	5	4	3	2	8	3
False negatives, %	1	2	3	2	2	4	1
Number of false positives	2	9	4	9	0	19	7
Category A, % of participating laboratories	55	58	50	57	53	45	51
Good SWZ/AZ^2, %	79	82	85	96	93	92	92
Satisfactory SWZ/AZ^2, %	14	8	10	1	5	1.5	3.4
Unsatisfactory SWZ/AZ^2, %	7	10	4	3	2	6.2	5.1

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

The test material in EUPT-CF12 was relatively easy to analyse due to the low number of residues and the high concentration levels in the Test Item (see **Chapter 1.3**). However, the performance was not significantly better. The average % of reported results has been between 41-91%. This was because a lot of participants analysed for less than 50% of the pesticide residues present in the test Item especially the voluntary pesticide, that only 41% of the participant had included in their scope. The number of false negative results has been low (1%) and the number of false positives has been average.

The percentage of Category A laboratories has varied during the years and for the last 7 EUPTs, no improvement can be seen. However, it has increased a bit since EUPT-CF11 where many false positive results were reported. In EUPT-CF12, 51% of the laboratories were evaluated as Category A. For Category A labs, an improvement in AZ^2 was seen in EUPT-CF9 where 96% of the results were good, and in EUPT-CF10 still 93% and EUPT-CF12 92% of the laboratories were evaluated as good. However, it is difficult to assess any improvement/deterioration in laboratory performance between the Proficiency Tests, because the numbers of pesticides in the Test item as well as the number of laboratories participating in the PTs varies from year to year. Furthermore, the matrix varies and thereby the performance can change.

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

The EUPT-CF12 Test Item consisted of hay flour containing incurred pesticides. The hay crop had been sprayed in the field with commercially available pesticide formulations. The final Test Item contained the following pesticides: azoxystrobin, boscalid, epoxiconazole, fluxapyroxad, metrafenone, pendimethalin, proquinazid, pyraclostrobin and tau-Fluvalinate. One hundred nineteen laboratories, representing 29 EU and EFTA countries, submitted results. Eight more laboratories registered, but did not submit any results. A number of NRLs did not participate in the PT as hay was not within their scope. Malta was represented in the PT by the NRL for the UK. An additional 11 laboratories from EU candidate states and Third Countries registered for the PT and 10 of them submitted results. The Target Pesticide List distributed to the laboratories prior to the test contained 155 individual compulsory and 23 voluntary compounds.

The number of false positives and false negatives has varied between the EUPTs. However, the number of false negatives represented in this PT represented only 1% of the total number of results and 7 false positive results were reported. The average Alg A-RSD was at 20%, well below the FFP-RSD of 25%. The range was from 12-26% for the individual compounds.

For metrafenone, epoxiconazole and proquinazid, acceptable z scores were obtained by 96-97% of the laboratories. For boscalid, pendimethalin, azoxystrobin and fluxapyroxad, acceptable z scores were obtained by 93-95% of the laboratories and finally 88-89% of the laboratories obtained acceptable z scores for tau-fluvalinate and pyraclostrobin.

The EUPT-CF13 Test Item will be a cereal commodity, rye kernels, which will be shipped to the laboratories in January 2019. The selection of pesticides will continue to be focused on pesticides included in the scope of the EU multi-annual coordinated control programme, as well as additional pesticides of relevance to feed and/or cereal production in Europe and in other parts of the world from where significant quantities of feed and cereals are imported.

4. ACKNOWLEDGEMENTS

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5. REFERENCES

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

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

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

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

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

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

[7] Guidance document on analytical quality control and validation procedures for pesticide residues analysis in food and feed. SANTE 11945/2015.

APPENDICES

Appendix 1 List of laboratories registered to participate in the EUPT-CF12

Participating labs from EU and EFTA member states

Country	Institution	City	NRL-CF	Report data
Austria	Department for Pesticide and Food Analytics (PLMA)	Innsbruck	NRL	Yes
Belgium	FLVV - Tervuren	Tervuren		Yes
Belgium	WIV-ISP	Brussels	NRL	Yes
Belgium	Primoris Belgium	Gent - Zwijnaarde		Yes
Bulgaria	Primoris Bulgaria AD - Pesticide Lab	Plovdiv		Yes
Croatia	Croatiakontrola - Pesticide Lab	Zagreb		Yes
Croatia	Croatian Veterinary Institute - Pesticide Lab	Zagreb	NRL	Yes
Croatia	Inspecto d.o.o.	Osijek		Yes
Croatia	Faculty of Food Technology and Biotechnology, Food Control Center - Croatia, Zagreb	Zagreb		Yes
Croatia	Sample Control d.o.o.	Zagreb		Yes
Cyprus	ANIMAL FEEDS AND FEED ADDITIVES - PESTICIDE LAB, DEPARTMENT OF AGRICULTURE	Nicosia	NRL	No
Czech Republic	CAFIA	Praha	NRL	Yes
Czech Republic	UKZUZ	Brno	NRL	Yes
Denmark	DVFA	Ringsted	NRL	Yes
Estonia	Pesticide Lab (Saku)	Saku	NRL	Yes
Finland	Finnish Food Safety Authority (Evira)	Helsinki	NRL	Yes
France	GIRPA-FREDON Pays de la Loire - Pesticide Lab	Beaucouzé		No
France	INOVALYS Le Mans - Pesticide Lab	Le Mans		Yes
France	CAPINOV	Landerneau		No
France	CERECO (GARONS)	GARONS		Yes
France	Domaine Résidus de pesticides SCL L75	Massy Cedex	NRL	Yes
France	Phytocontrol (Nimes) - Pesticide Lab	Nimes		Yes
Germany	CVUA RRW - Pesticide Lab (Krefeld)	Krefeld		Yes
Germany	Eurofins Dr. Specht Laboratorien GmbH	Hamburg		Yes
Germany	Federal Office of Consumer Protection and Food Safety National Reference Laboratory for Pesticides	Berlin	NRL	Yes
Germany	Landesamt für Landwirtschaft, Lebensmittelsicherheit und Fischerei M-V	Rostock		Yes
Germany	Niedersächsisches Landesamt für Verbraucherschutz und Lebensmittelsicherheit, Lebensmittel- und Veterinärinstitut Oldenburg	Oldenburg		Yes
Germany	LAVES - Pesticide Lab (Stade)	Stade		No
Germany	LLG	Halle/Saale		Yes
Germany	LTZ Augustenberg - Organic Chemistry	Karlsruhe		Yes
Germany	LUFA-ITL GmbH	Kiel		Yes
Germany	Pesticide Lab (Jena)	Jena		Yes
Germany	Landesbetrieb Hessisches Landeslabor Kassel	Kassel		Yes
Germany	Pesticide Lab (Nossen)	Nossen		Yes
Germany	LLBB Potsdam	Potsdam		Yes
Germany	LUFA Speyer	Speyer		Yes

Country	Institution	City	NRL-CF	Report data
Germany	Landesbetrieb Hessisches Landeslabor	Wiesbaden		Yes
Greece	General Chemical State Laboratory	Athens	NRL	Yes
Greece	BPI - Pesticide Lab (Kifissia)	Kifissia	NRL	Yes
Greece	AGROLAB-RDS SA	Thessaloniki - GR 570 22, Sindos		Yes
Hungary	National Food Chain Safety Office, Pesticide Analytical Laboratory, Velence	Velence	NRL	Yes
Hungary	Food Chain Safety Centre Non-profit Ltd. Pesticide Residue Analytical Laboratory, Hódmezovásárhely	Hódmezovásárhely		Yes
Hungary	FCSCN Ltd. Pesticide Residue Analytical Laboratory, Miskolc	Miskolc		Yes
Hungary	Food Chain Safety Centre Non-profit Ltd., Pesticide Residue Analytical Laboratory, Szolnok	Szolnok		Yes
Iceland	Matís - Pesticide Lab	Reykjavík	NRL	Yes
Ireland	The Pesticide Control Laboratory	Co. Kildare	NRL	Yes
Italy	ISS - Pesticide Lab	Roma		No
Italy	IZS LT - Pesticide Lab	Roma		Yes
Italy	IZS LT (sezione Firenze) - Pesticide Lab	San Martino alla Palma Scandicci (FI)		Yes
Italy	IZS PB - Pesticide Lab	Foggia		Yes
Italy	Istituto zooprofilattico Sperimentale Laboratorio Chimico Liguria	Genova	NRL	Yes
Italy	ISTITUTO ZOOPROFILATTICO DELLA SARDEGNA LABORATORIO FARMACO E ANALISI RESIDUI	Sassari		Yes
Italy	IZS Sicilia - Pesticide Lab	Palermo		Yes
Italy	APPA BOLZANO	Bolzano		Yes
Italy	ARPA Puglia - Polo di Specializzazione Alimenti	Bari		Yes
Italy	APPA-Settore Laboratorio-Provincia Autonoma di Trento	Trento		Yes
Italy	ARPA FVG - Pesticide Lab (Udine)	Udine		Yes
Italy	ARPA Lazio (sez. Latina) - Pesticide Lab	Latina		Yes
Italy	ARPA VDA - Pesticide Lab	Saint Christophe		Yes
Italy	ARPA Veneto SO Verona	Verona		No
Italy	ATS Milano - Laboratorio di Prevenzione	Milano		Yes
Italy	IZSAM - Pesticide Lab	Teramo		Yes
Italy	IZSLER - Pesticide Lab	Brescia		Yes
Italy	IZSUM - Pesticide Lab	Perugia		Yes
Italy	IZSVe - Pesticide Lab	Legnaro (Padova)		Yes
Italy	LABORATORIO DI PREVENZIONE - ATS BERGAMO	Bergamo		Yes
Latvia	Institute of Food Safety, Animal Health and Environment “BIOR”	Riga	NRL	Yes
Lithuania	NVFRAI	Vilnius	NRL	Yes
Luxembourg	LNS Food lab	Dudelange	NRL	Yes
Netherlands	Eurofins Lab Zeeuws-Vlaanderen B.V. - Pesticiden	Graauw		Yes
Netherlands	Groen Agro Control - Pesticide Lab	Delfgauw		Yes
Netherlands	Dr. A. Verwey B.V.	Rotterdam		Yes
Netherlands	NVWA - NRL for Pesticide Residues in Food and Feed	Wageningen	NRL	Yes
Netherlands	RIKILT - Pesticide Lab	Wageningen		Yes

Country	Institution	City	NRL-CF	Report data
Norway	NIBIO, Pesticides and Natural Products Chemistry	ÅS	NRL	Yes
Poland	InHort (Skierniewice) - Pesticide Lab	Skierniewice		Yes
Poland	Department of Pesticide Residue Research, Institute of Plant Protection - National Research Institute	Poznan	NRL	Yes
Poland	Institute of Plant Protection - National Research Institute Laboratory of Pesticide Residues	Bialystok		Yes
Poland	Regional Veterinary Laboratory in Bialystok Poland	Bialystok		Yes
Poland	Regional Veterinary Laboratory-Poland, WIW-ZHW Gdansk, Pesticide lab	Gdansk		Yes
Poland	Wojewodzki Inspektorat Weterynarii w Katowicach	Katowice		Yes
Poland	WIW ZHW (Opole) - Pesticide Lab	Opole		Yes
Poland	WIW ZHW (Poznan) - Pesticide Lab	Poznan		Yes
Poland	WIW ZHW (Warsaw) - Pesticide Lab	Warszaw		Yes
Poland	WIW ZHW (Wroclaw) - Pesticide Lab	Wroclaw		Yes
Poland	Pesticide Lab (Sosnicowice)	Sosnicowice		Yes
Poland	Regional Veterinary Laboratory	Szczecin		Yes
Portugal	Laboratório Regional de Veterinária e Segurança Alimentar	Funchal - Madeira Island		Yes
Romania	Institutul de Igiena si Sanatate Publica Veterinara	Bucharest	NRL	Yes
Romania	NATIONAL PHITOSANITARY AUTHORITY-NATIONAL LABORATORY FOR PESTICIDES RESIDUES CONTROL IN PLANTS AND VEGETABLE PRODUCTS - BUCHAREST	Bucharest	NRL	Yes
Romania	LRCRPPPV (Tirgu Mures) - Pesticide Lab	Tirgu Mures		Yes
Romania	Pesticides Residues Laboratory - Bistrita	Bistrita		Yes
Romania	Pesticides Residues Laboratory - Braila	Braila		Yes
Slovakia	Veterinary and Food Institute in Bratislava	Bratislava	NRL	Yes
Spain	Ecosur - Pesticide Lab	Lorquí (Murcia)		Yes
Spain	Laboratorio Agrario y Fitopatologico de Galicia	Abegondo. A Coruña		No
Spain	Analytica Alimentaria GmbH, Sucursal en España	Almeria		Yes
Spain	Servicio de Laboratorio y Control (SeLyC) Consejería de Medio Rural, Pesca y Alimentación.	Santander		Yes
Spain	Lab. Agrario Regional - Junta de Castilla y Leon	Burgos		Yes
Spain	Agencia Salut Pública de Barcelona	Barcelona		No
Spain	Laboratori Agroalimentari	Cabrils		Yes
Spain	Laboratorio Agroalimentario	Valencia		Yes
Spain	Laboratorio Agroalimentario de Extremadura	Cáceres		Yes
Spain	Laboratorio Agroambiental de Zaragoza	Zaragoza		Yes
Spain	Laboratorio Arbitral Agroalimentario	Madrid	NRL	Yes
Spain	Laboratorio de Residuos, Inst. Tecnol. de Canarias	Agüimes, Gran Canaria		Yes
Spain	Laboratorio de Salud Pública de Cuenca	Cuenca		Yes
Spain	Laboratorio de Salud Pública de Galicia	Lugo		Yes
Spain	LABORATORIO KUDAM, S.L.	Pilar de la Horadada (Alicante)		Yes
Spain	Laboratorio Regional de la CCAA de La Rioja	Logroño		Yes
Spain	LARAGA - Pesticide Lab (Toledo)	Toledo		Yes
Spain	Nasertic	Villava		Yes

Country	Institution	City	NRL-CF	Report data
Spain	CNTA	San Adrián (Navarra)		Yes
Spain	Pesticide Lab (Majadahonda)	Majadahonda	NRL	Yes
Sweden	Eurofins Food & Feed Testing Sweden AB	Lidköping		Yes
Sweden	National Food Agency, Science Division, Department of Chemistry	Uppsala	NRL	Yes
Switzerland	Kantonales Labor Zürich	Zürich		Yes
United Kingdom	Fera Science Ltd	York	NRL	Yes
United Kingdom	AFBI - Pesticide Lab	Belfast		Yes

Participating labs from EU candidate states and other non EU countries

Country	Institution	City	Report data
Albania	Pesticides Residue in Products of Animal Origin/Food Safety and Veterinary Institute	Tiranë – Shqipëri	Yes
Argentina	Bolsa de Comercio de Rosario - Lab de ensayos	Rosario	Yes
Argentina	National Univ. of Littoral - Pesticide Lab	Santa Fe	Yes
Australia	Symbio Laboratories Pty Ltd	Eight Mile Plains, QLD	Yes
China	Government Laboratory, Hong Kong	Hong Kong	Yes
India	Export Inspection Agency-Mumbai, Pilot Test House	Mumbai	Yes
Serbia	Gradski zavod za javno zdravlje Beograd	Belgrade	Yes
Serbia	Center for Food Analysis	Belgrade	Yes
Singapore	Agri-Food & Veterinary Authority Veterinary Public Health Laboratory	Singapore	Yes
Sri Lanka	Residue Analysis Laboratory Industrial Technology Institute	Colombo 07	No
Thailand	Central Laboratory (Thailand) Co.,Ltd. Branch Bangkok	Bangkok	Yes

Appendix 2

Target Pesticide List

Pesticides	MRRL (mg/kg)
Compulsory Compounds (will be considered in Category A/B classification)	
2-phenylphenol	0.05
Acephate	0.05
Acetamiprid	0.05
Acrinathrin	0.05
Aldrin	0.05
Azinphos-methyl	0.05
Azoxystrobin	0.05
Bifenthrin	0.05
Biphenyl	0.05
Bixafen	0.05
Boscalid	0.05
Bromuconazole	0.05
Buprofezin	0.05
Cadusafos	0.05
Carbaryl	0.05
Carbendazim	0.05
Carbofuran	0.05
Carbofuran, 3-hydroxy-	0.05
Carboxin	0.05
Chlorantraniliprole	0.05
Chlorfenapyr	0.05
Chlorfenvinphos	0.05
Chlorpropham (parent compound only)	0.05
Chlorpyrifos	0.05
Chlorpyrifos-methyl	0.05
Clothianidin	0.05
Cyfluthrin (sum of isomers)	0.05
Cymoxanil	0.05
Cypermethrin (sum of isomers)	0.05
Cyproconazole	0.05
Cyprodinil	0.05
DDD, p,p'-	0.05
DDE, p,p'-	0.05
DDT, o,p'-	0.05
DDT, p,p'-	0.05
Deltamethrin, cis-	0.05
Demeton-S-methylsulfone	0.05
Diazinon	0.05
Dichlorvos	0.05
Dieldrin	0.05
Difenoconazole	0.05
Diflubenzuron	0.05
Dimethoate	0.05
Dimethomorph	0.05
Diniconazole	0.05
Endosulfan, alpha-	0.05

Pesticides	MRRL (mg/kg)
Endosulfan, beta-	0.05
Endosulfan-sulfate	0.05
Epoxiconazole	0.05
Ethion	0.05
Ethirimol	0.05
Ethoprophos	0.05
Famoxadone	0.05
Fenbuconazole	0.05
Fenhexamid	0.05
Fenitrothion	0.05
Fenpropathrin	0.05
Fenpropidin	0.05
Fenpropimorph	0.05
Fenpyroximate	0.05
Fenthion	0.05
Fenthion-oxon	0.05
Fenthion-oxon-sulfone	0.05
Fenthion-oxon-sulfoxide	0.05
Fenthion-sulfone	0.05
Fenthion-sulfoxide	0.05
Fenvalerate and Esfenvalerate (Sum of RR/SS and RS/SR isomers)	0.05
Fipronil (parent compound only)	0.05
Flonicamid	0.05
Flubendiamide	0.05
Fludioxonil	0.05
Flufenoxuron	0.05
Fluopicolide	0.05
Fluopyram	0.05
Fluquinconazole	0.05
Flusilazole	0.05
Flutolanil	0.05
Flutriafol	0.05
Fluxapyroxad	0.05
Formetanate	0.05
Hexaconazole	0.05
Imazalil	0.05
Imidacloprid	0.05
Indoxacarb	0.05
Iprodione	0.05
Isocarbophos	0.05
Isoprothiolane	0.05
Isoproturon	0.05
Kresoxim-methyl	0.05
Lambda-cyhalothrin	0.05
Lindane	0.05
Linuron	0.05
Malaoxon	0.05
Malathion	0.05
Mandipropamid	0.05
Metalaxyl	0.05
Metconazole	0.05

Pesticides	MRRL (mg/kg)
Methacrifos	0.05
Methamidophos	0.05
Methomyl	0.05
Metolachlor	0.05
Metrafenone	0.05
Metribuzin	0.05
Omethoate	0.05
Oxydemeton-methyl	0.05
Paclobutrazol	0.05
Parathion	0.05
Penconazole	0.05
Pencycuron	0.05
Pendimethalin	0.05
Permethrin (sum of isomers)	0.05
Phosphamidon	0.05
Pirimicarb	0.05
Pirimicarb-desmethyl	0.05
Pirimiphos-methyl	0.05
Prochloraz (parent compound only)	0.05
Procymidone	0.05
Profenofos	0.05
Propamocarb	0.05
Propiconazole	0.05
Prosulfocarb	0.05
Prothioconazole-desthio	0.05
Prothiofos	0.05
Pyraclostrobin	0.05
Pyridaben	0.05
Pyrimethanil	0.05
Pyriproxyfen	0.05
Quinoxyfen	0.05
Spirodiclofen	0.05
Spiromesifen	0.05
Spiroxamine	0.05
Tau-Fluvalinate	0.05
Tebuconazole	0.05
Tebufenozide	0.05
Teflubenzuron	0.05
Tefluthrin	0.05
Terbuthylazine	0.05
Tetraconazole	0.05
Tetradifon	0.05
Tetramethrin	0.05
Thiabendazole	0,05
Thiacloprid	0.05
Thiamethoxam	0.05
Thiodicarb	0.05
Thiophanate-methyl	0.05
Tolclofos-methyl	0.05
Triadimefon	0.05
Triadimenol	0.05

Pesticides	MRRL (mg/kg)
Triazophos	0.05
Tricyclazole	0.05
Trifloxystrobin	0.05
Trifluralin	0.05
Triticonazole	0.05
Vinclozolin (parent compound only)	0.05
Zoxamide	0.05
Voluntary Compounds	
(will not be considered in Category A/B classification)	
Ametoctradin	0.05
Benalaxyl including other mixtures of constituent isomers including benalaxyl-M (sum of isomers)	0.05
Benzovindiflupyr	0.05
Chlorfluazurone	0.05
Clomazone	0.05
Cyazofamid	0.05
Cyflufenamid	0.05
Etoxazole	0.05
Fenpyrazamine	0.05
Isopyrazam	0.05
Novaluron	0.05
Penflufen	0.05
Penthiopyrad	0.05
Proquinazid	0.05
Pyridalil	0.05
Pyriofenone	0.05
Spinetoram	0.05
Spirotetramat	0.05
Spirotetramat metabolite BYI08330-enol	0.05
Spirotetramat metabolite BYI08330-ketohydroxy	0.05
Spirotetramat metabolite BYI08330-monohydroxy	0.05
Spirotetramat metabolite BYI08330 enol-glucoside	0.05
Sulfoxaflor	0.05

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		Boscalid, mg/kg		Epoxiconazole, mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
007	0.992	0.986	14.156	10.299	2.807	2.840
028	1.066	1.041	11.678	13.081	3.267	3.040
055	1.025	1.008	11.980	13.357	3.051	3.034
085	0.979	0.922	11.538	11.917	2.660	2.591
096	0.882	0.934	11.760	11.936	2.546	2.665
126	1.027	1.025	11.135	10.531	2.999	3.048
156	1.090	1.013	11.844	12.100	3.293	3.088
187	1.066	0.911	11.283	11.901	3.167	2.530
207	0.907	0.915	12.091	11.942	2.590	2.630
210	1.053	0.950	12.198	10.884	3.150	2.917
226	0.947	1.064	11.130	12.011	2.707	3.112

	Metrafenone, mg/kg		Fluxapyroxad, mg/kg		Proquinazad, mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
007	1.010	1.016	1.618	1.465	0.865	0.840
028	1.134	1.087	1.476	1.489	0.965	0.884
055	1.091	1.081	1.493	1.549	0.870	0.874
085	0.957	0.919	1.307	1.463	0.854	0.824
096	0.916	0.942	1.414	1.535	0.804	0.812
126	1.037	1.069	1.530	1.549	0.879	0.912
156	1.168	1.104	1.489	1.505	0.967	0.915
187	1.100	0.902	1.499	1.455	0.944	0.832
207	0.939	0.977	1.653	1.481	0.840	0.826
210	1.113	1.020	1.458	1.392	0.890	0.862
226	0.984	1.101	1.485	1.325	0.802	0.904

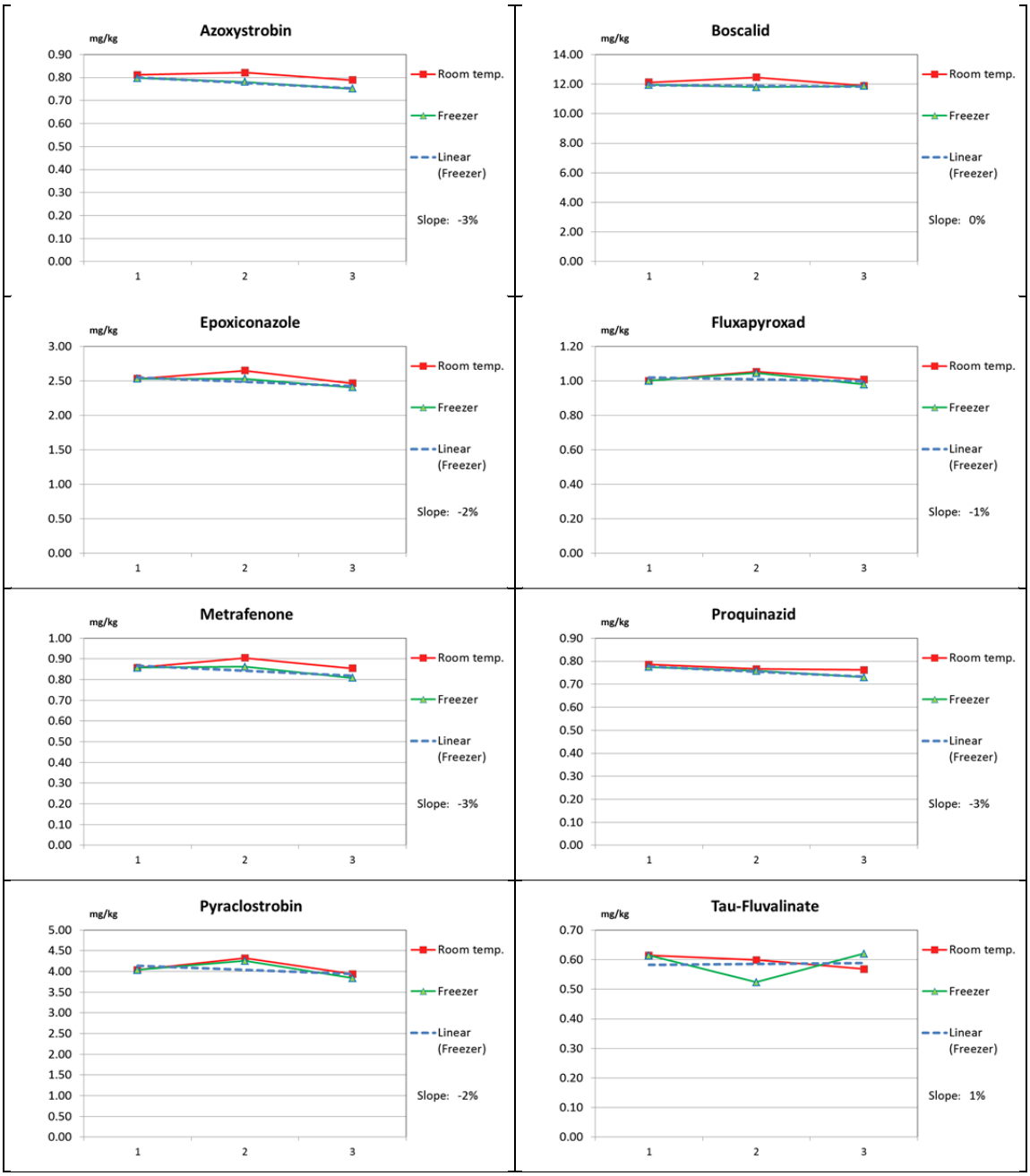
	Pyraclostrobin, mg/kg		Tau-fluvalinate, mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2
007	7.055	6.054	0.7189	0.7012
028	6.582	5.489	0.8925	0.8671
055	5.938	6.406	0.8028	0.8693
085	6.339	5.837	0.8786	0.8227
096	5.636	6.426	0.7881	0.7511
126	5.547	5.615	0.7530	0.7782
156	6.756	6.319	0.9609	0.7939
187	6.839	6.488	0.8749	0.8540
207	6.868	6.411	0.7745	0.7467
210	6.388	5.879	0.8539	0.8040
226	6.029	6.591	0.7904	0.7894

Appendix 4 Stability figures

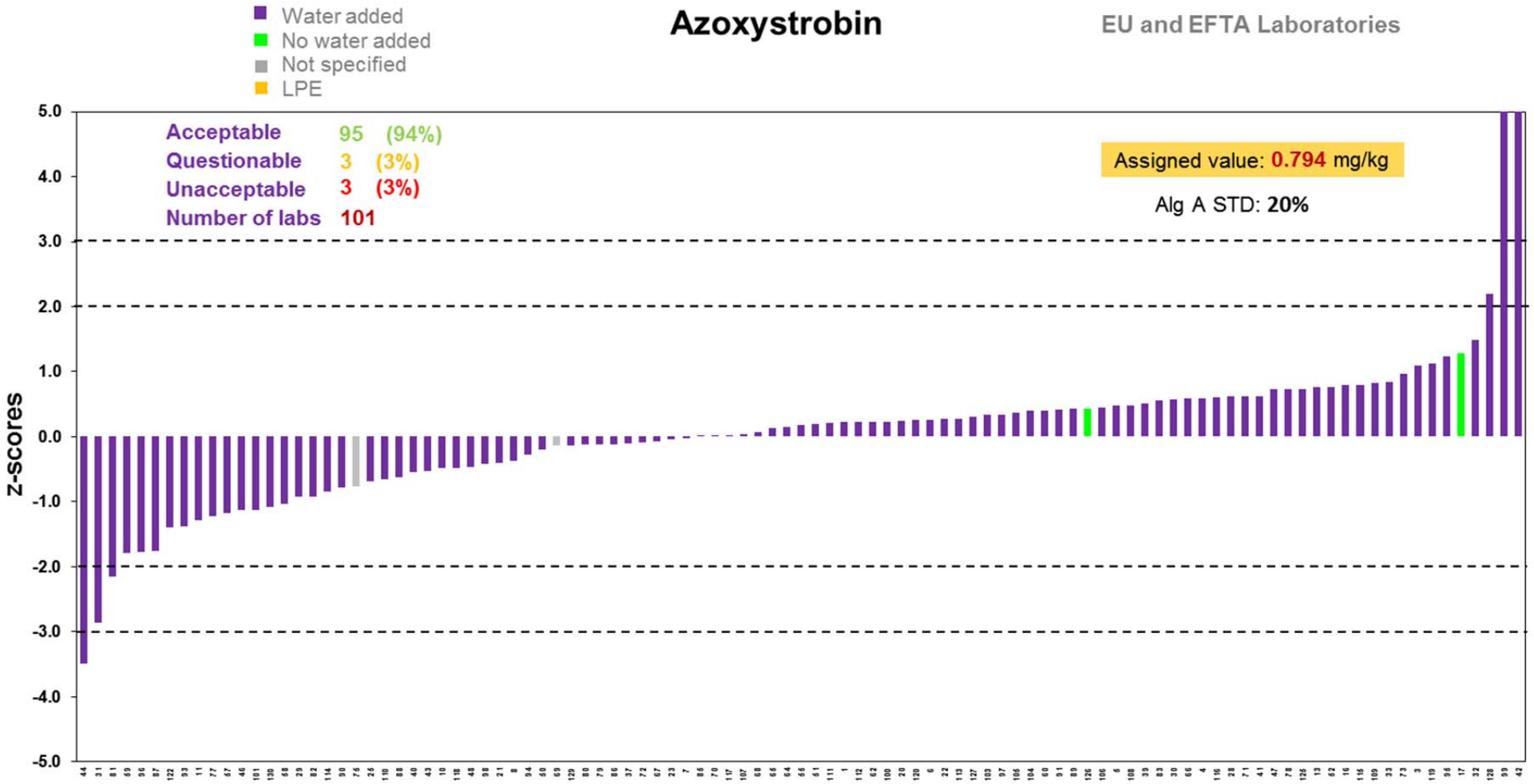
The stability test was performed according to ISO 13528, Annex B [5]. Two different storage temperatures were used; room temperature and -18 °C.

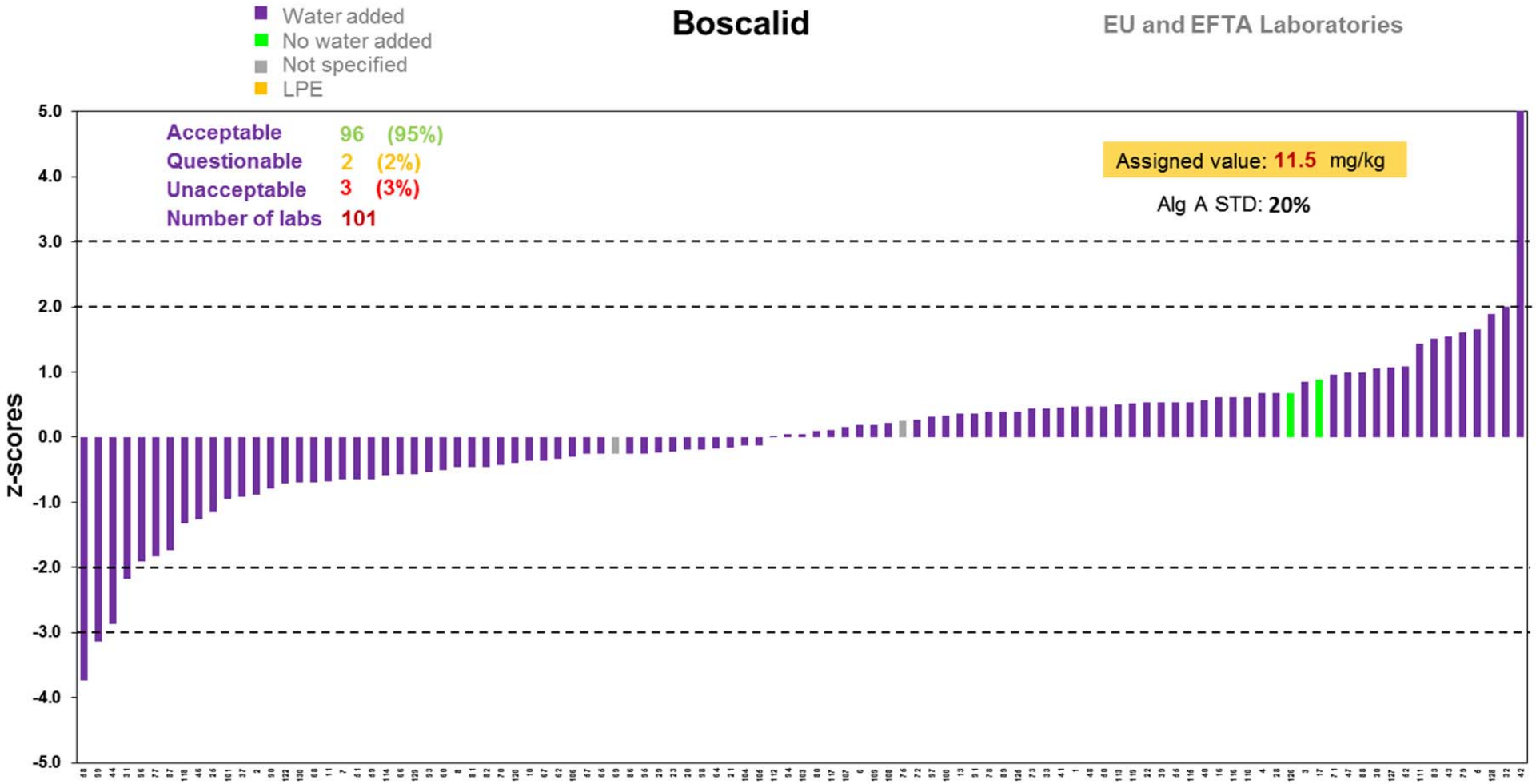
The dates of testing were as follows:
Day 1: 30 January 2018
Day 2: 16 February 2018
Day 3: 5 March 2018

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



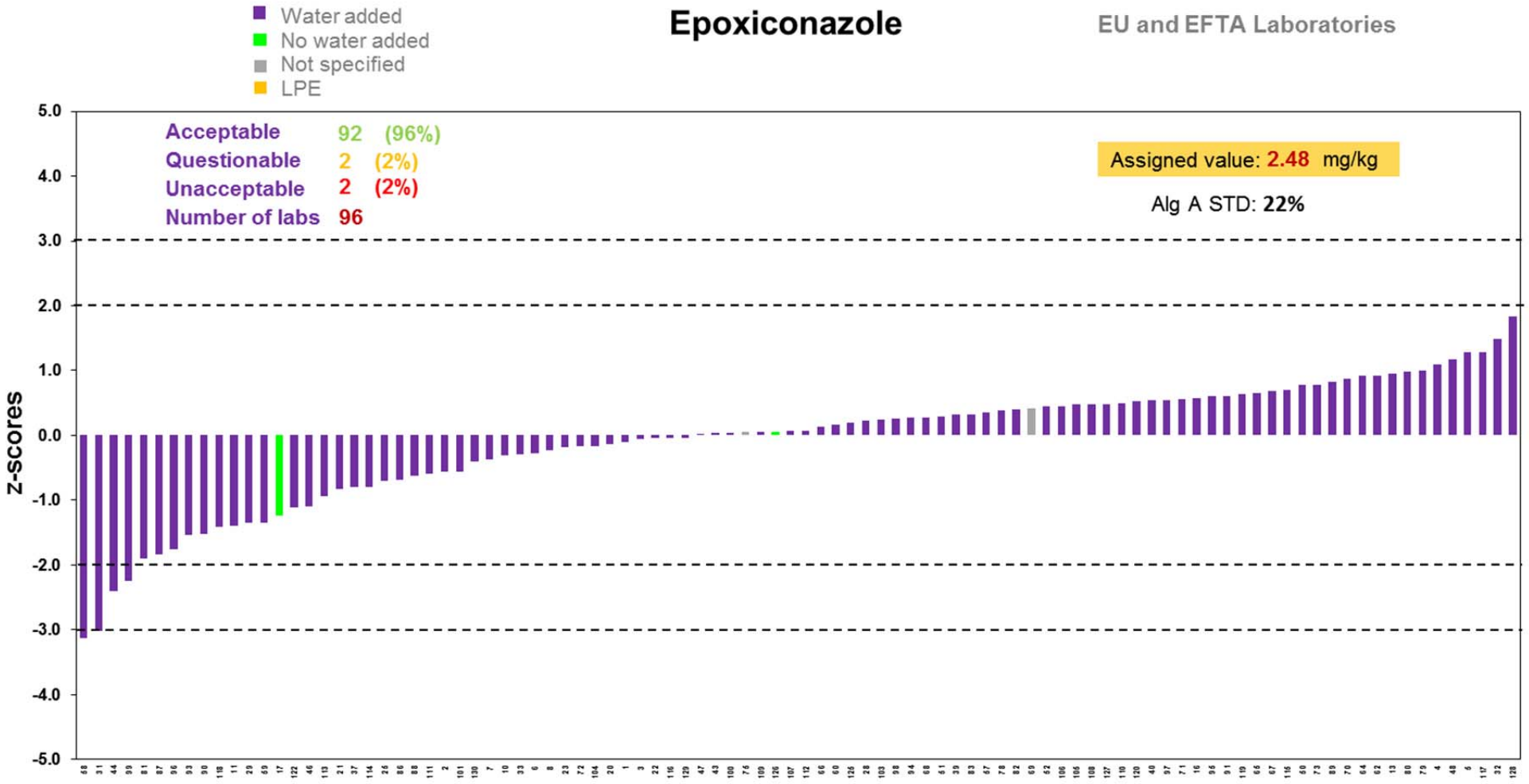
Appendix 5 Graphical presentation of z-scores

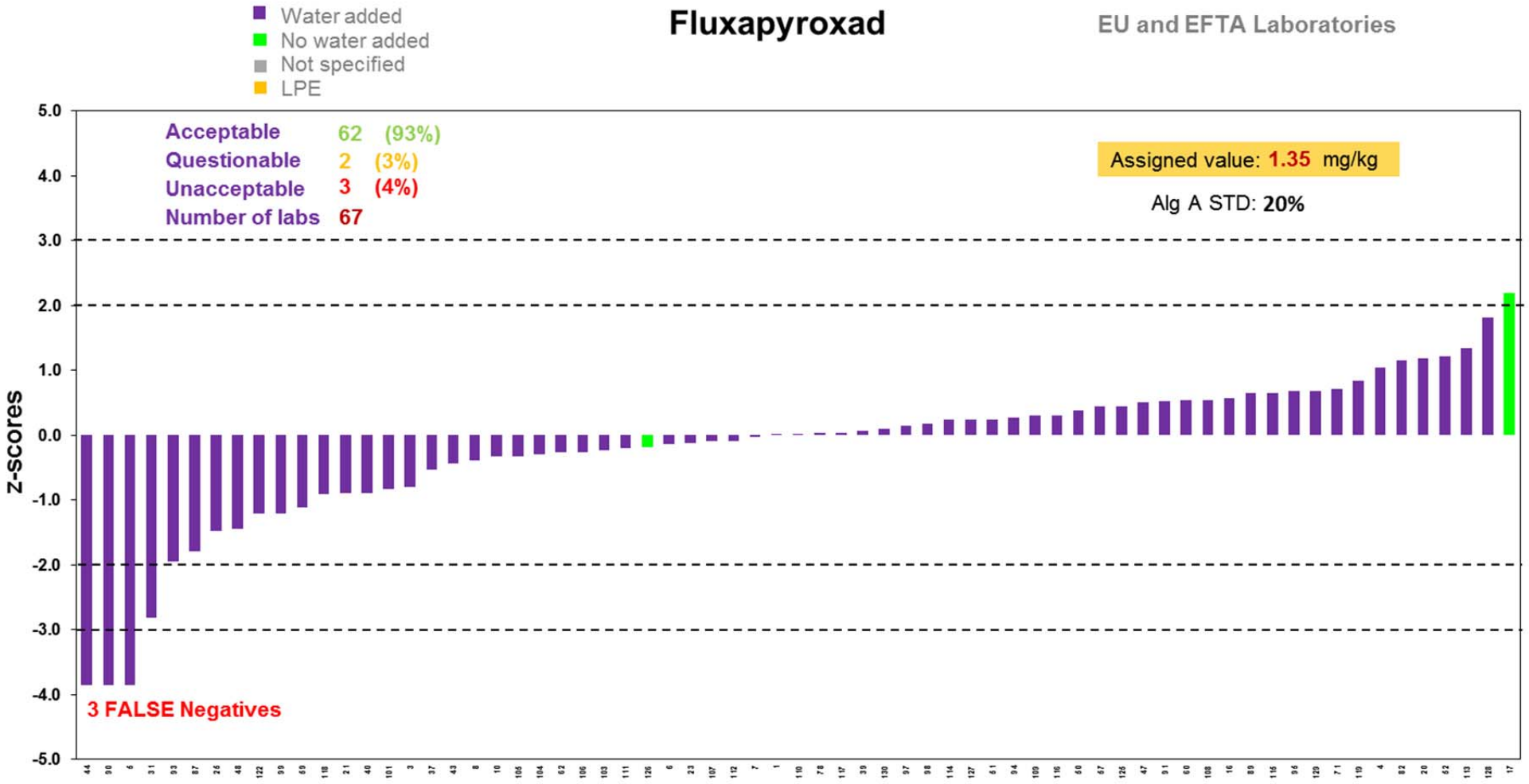


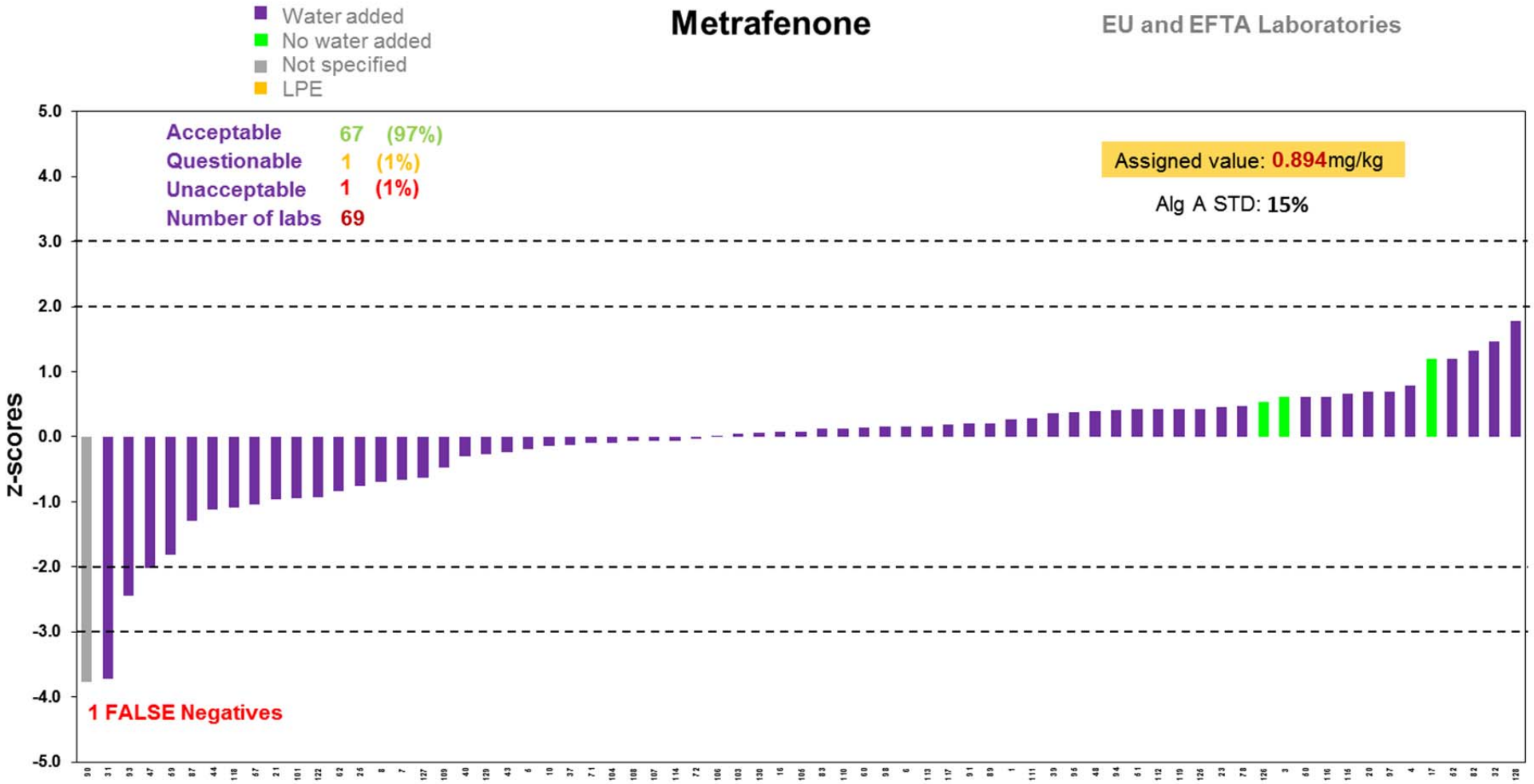

Water added
No water added
Not specified
LPE
Boscalid
EU and EFTA Laboratories
Acceptable 96 (95%)
Questionable 2 (2%)
Unacceptable 3 (3%)
Number of labs 101
Assigned value: 11.5 mg/kg
Alg A STD: 20%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0

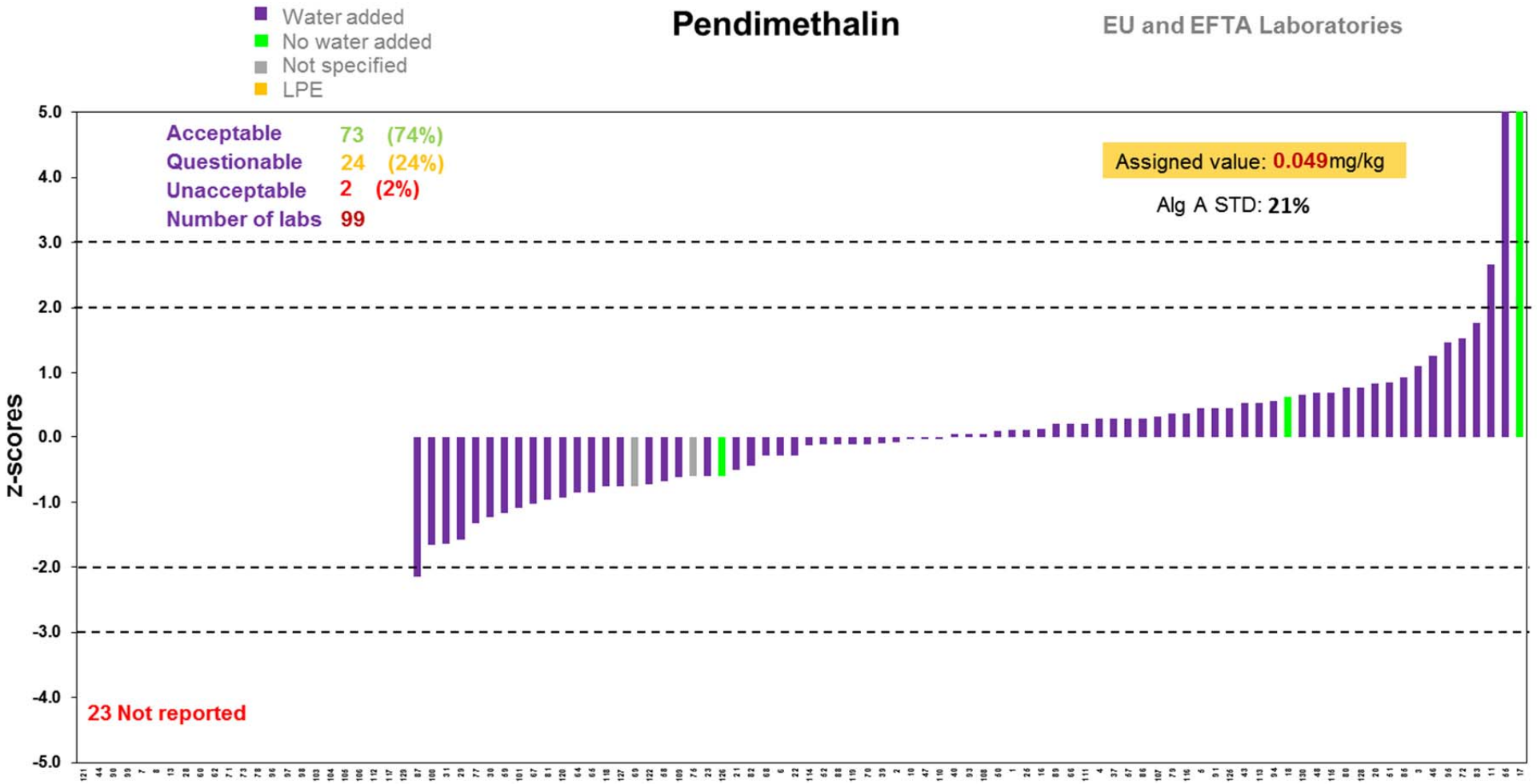
Epoxiconazole

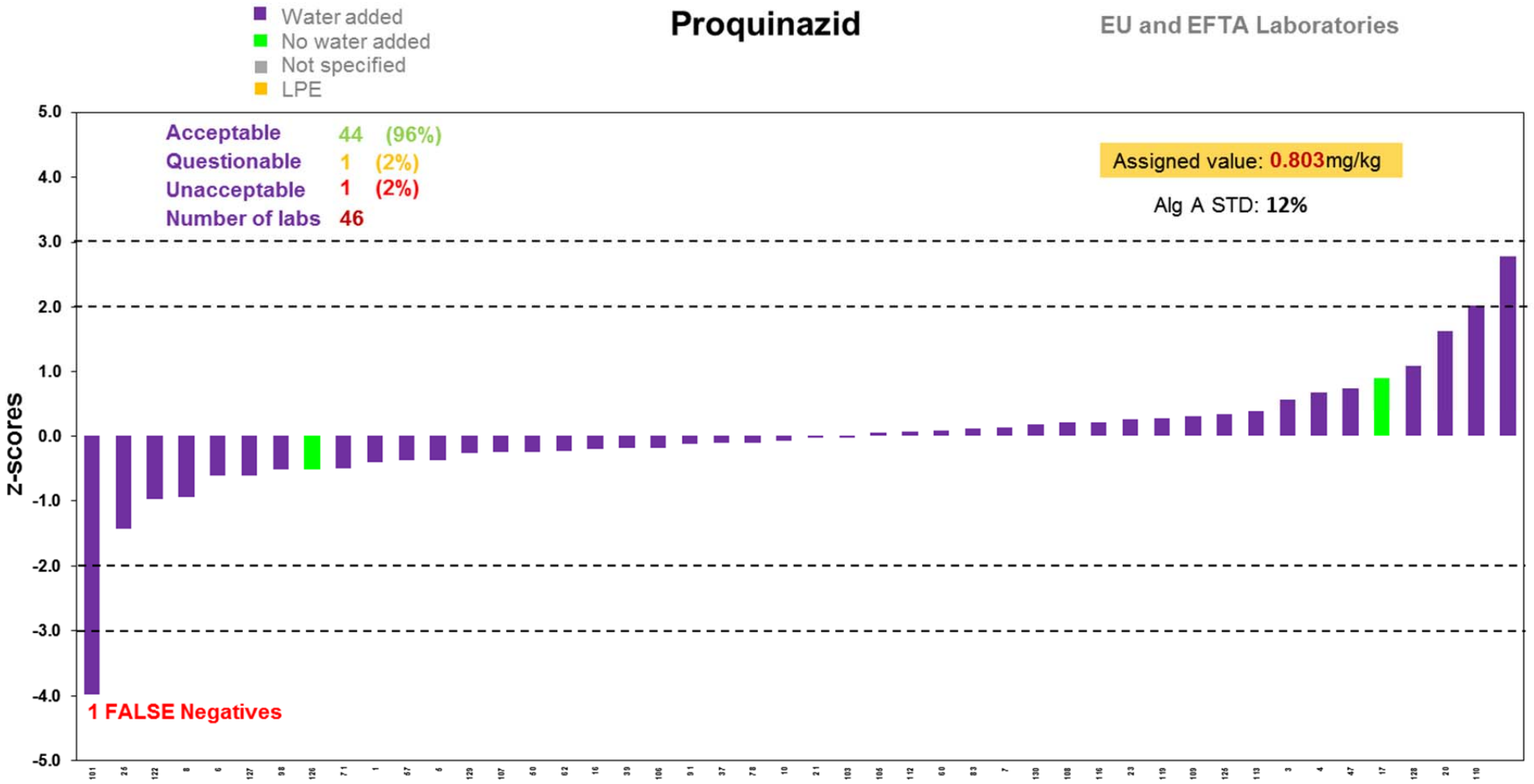
EU and EFTA Laboratories

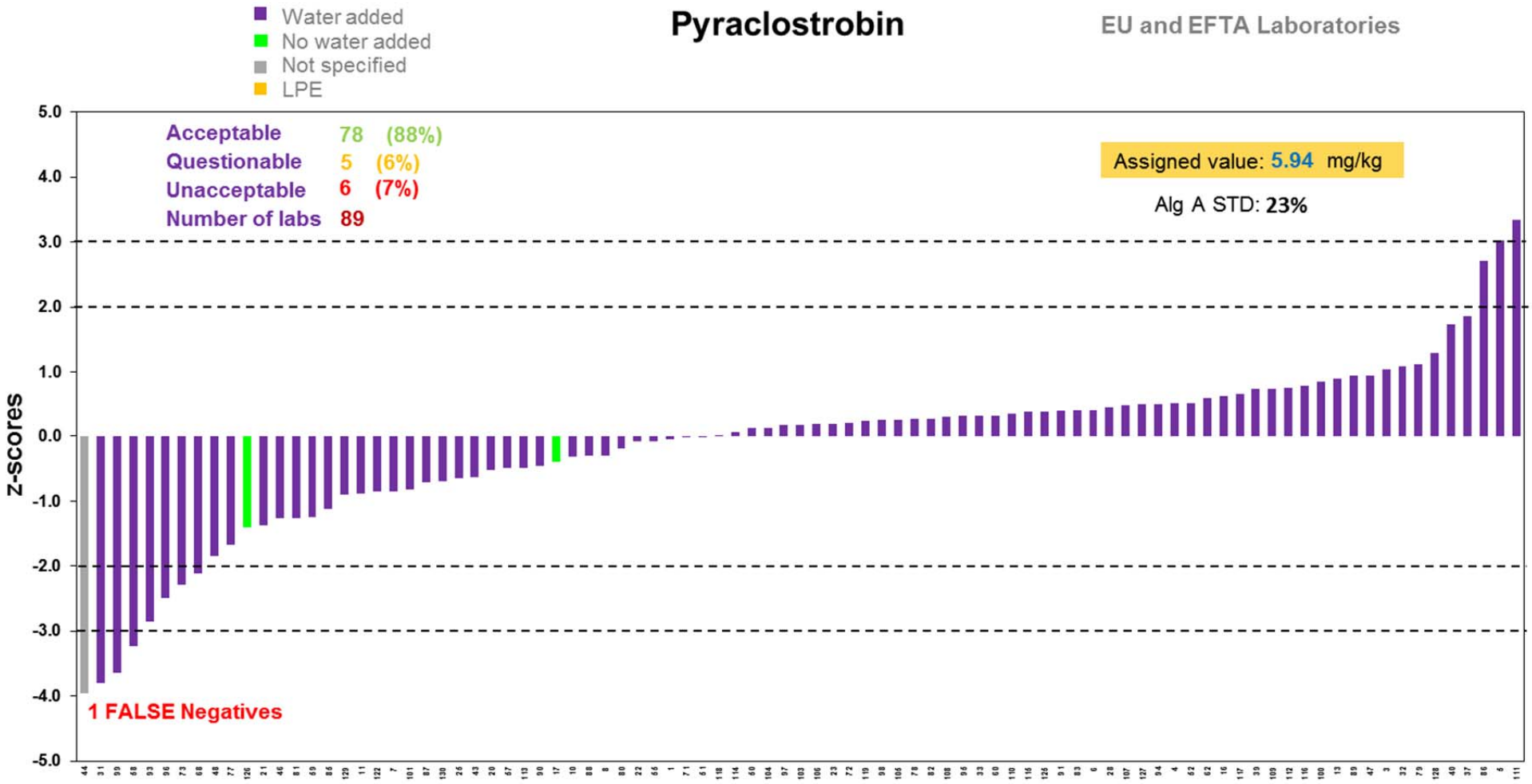


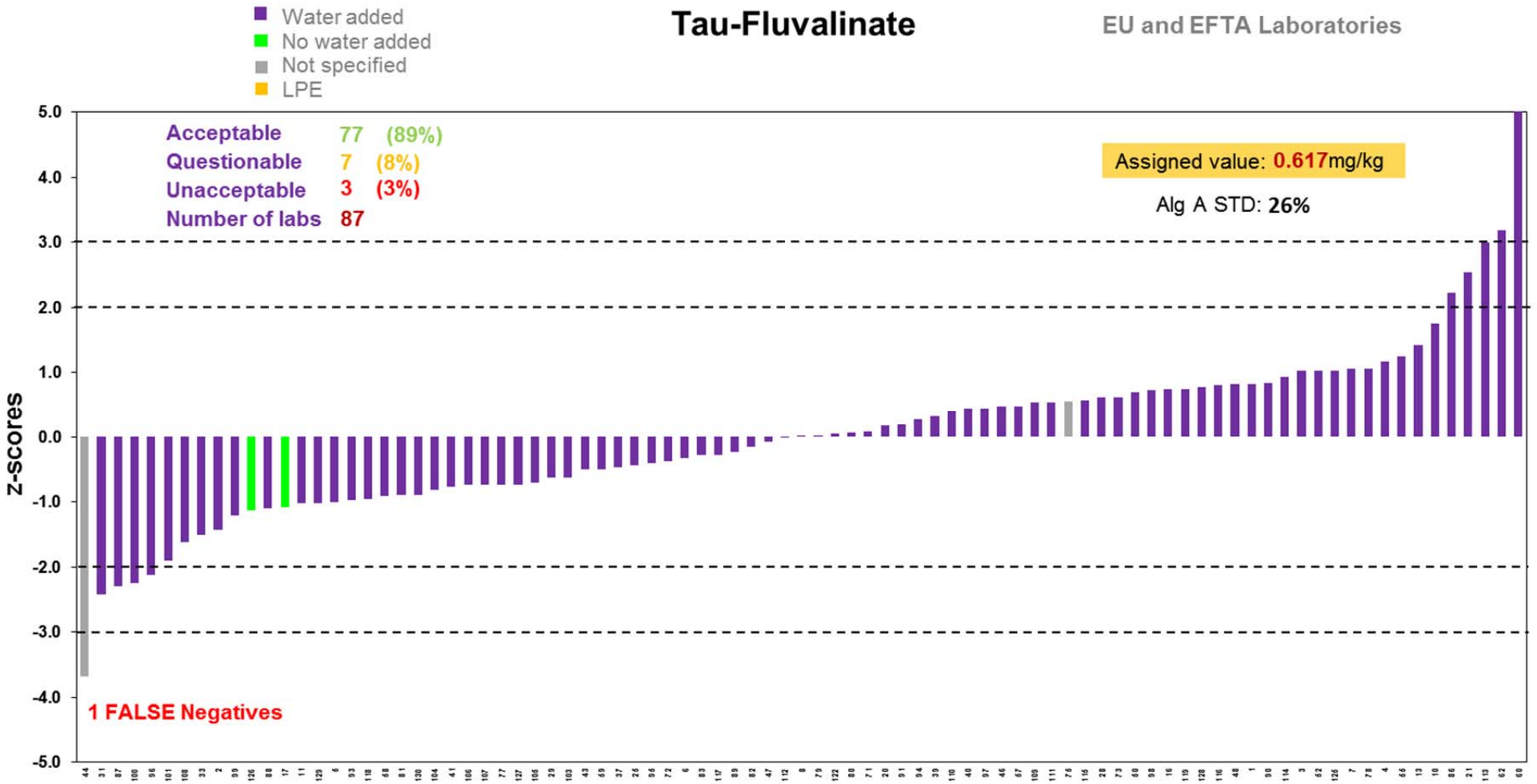

Water added
No water added
Not specified
LPE
Fluxapyroxad
EU and EFTA Laboratories
Acceptable 62 (93%)
Questionable 2 (3%)
Unacceptable 3 (4%)
Number of labs 67
Assigned value: 1.35 mg/kg
Alg A STD: 20%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
3 FALSE Negatives


Water added
No water added
Not specified
LPE
Metrafenone
EU and EFTA Laboratories
Acceptable 67 (97%)
Questionable 1 (1%)
Unacceptable 1 (1%)
Number of labs 69
Assigned value: 0.894mg/kg
Alg A STD: 15%
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
1 FALSE Negatives


Water added
No water added
Not specified
LPE
Pendimethalin
EU and EFTA Laboratories
Acceptable 73 (74%)
Questionable 24 (24%)
Unacceptable 2 (2%)
Number of labs 99
Assigned value: 0.049mg/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
23 Not reported


Water added
No water added
Not specified
LPE
Proquinazid
EU and EFTA Laboratories
Acceptable 44 (96%)
Questionable 1 (2%)
Unacceptable 1 (2%)
Number of labs 46
Assigned value: 0.803mg/kg
Alg A STD: 12%
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
Pyraclostrobin
EU and EFTA Laboratories
Acceptable 78 (88%)
Questionable 5 (6%)
Unacceptable 6 (7%)
Number of labs 89
Assigned value: 5.94 mg/kg
Alg A STD: 23%
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
Tau-Fluvalinate
EU and EFTA Laboratories
Acceptable 77 (89%)
Questionable 7 (8%)
Unacceptable 3 (3%)
Number of labs 87
Assigned value: 0.617mg/kg
Alg A STD: 26%
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

Appendix 6

Reported results in mg/kg at or below the MRRL at 0.05 mg/kg.

Lab code	Pesticide	Concentration, mg/kg	Reporting Limit, mg/kg
1	Pirimicarb	0.0073	0.005
20	Pirimicarb	0.0072	0.005
33	Pirimicarb	0.0099	0.005
35	Pirimicarb	0.01	0.01
55	Pirimicarb	0.01	0.05
119	Pirimicarb	0.011	0.02
122	Pirimicarb	0.0058	0.01
1	Pirimicarb-desmethyl	0.0061	0.005
6	Pirimicarb-desmethyl	0.01	0,01
10	Pirimicarb-desmethyl	0.0123	0.01
16	Pirimicarb-desmethyl	0.011	0.05
20	Pirimicarb-desmethyl	0.0133	0.005
31	Pirimicarb-desmethyl	0.0058	0.005
35	Pirimicarb-desmethyl	0.0133	0.01
108	Pirimicarb-desmethyl	0.0117	0.01
119	Pirimicarb-desmethyl	0.013	0.02
122	Pirimicarb-desmethyl	0.0112	0.01
125	Pirimicarb-desmethyl	0.014	0.01
1	Prosulfocarb	0.015	0.01
4	Prosulfocarb	0.018	0.05
6	Prosulfocarb	0.019	0,01
16	Prosulfocarb	0.025	0.05
17	Prosulfocarb	0.0141	
20	Prosulfocarb	0.0221	0.005
37	Prosulfocarb	0.017	0.01
39	Prosulfocarb	0.0176	0.01
52	Prosulfocarb	0.017	0.01
80	Prosulfocarb	0.028	0.01
81	Prosulfocarb	0.011	0.02
88	Prosulfocarb	0.022	0.05
98	Prosulfocarb	0.023	0.01
100	Prosulfocarb	0.019	0.01
108	Prosulfocarb	0.0185	0.01
109	Prosulfocarb	0.0161	0.008
119	Prosulfocarb	0.017	0.02
122	Prosulfocarb	0.0161	0.01
125	Prosulfocarb	0.026	0.02
127	Prosulfocarb	0.02	0.01

Lab code	Pesticide	Concentration, mg/kg	Reporting Limit, mg/kg
128	Prosulfocarb	0.023	0.05
1	Prothioconazole-desthio	0.0095	0.005
6	Prothioconazole-desthio	0.013	0,01
10	Prothioconazole-desthio	0.0215	0.01
16	Prothioconazole-desthio	0.018	0.05
17	Prothioconazole-desthio	0.018	
20	Prothioconazole-desthio	0.0141	0.005
31	Prothioconazole-desthio	0.0081	0.005
35	Prothioconazole-desthio	0.0189	0.01
37	Prothioconazole-desthio	0.011	0.01
98	Prothioconazole-desthio	0.019	0.01
108	Prothioconazole-desthio	0.0146	0.01
110	Prothioconazole-desthio	0.012	0.01
122	Prothioconazole-desthio	0.0131	0.01
125	Prothioconazole-desthio	0.016	0.01
1	Tebuconazole	0.0065	0.005
9	Tebuconazole	0.012	0.01
20	Tebuconazole	0.0079	0.005
55	Tebuconazole	0.01	0.05
122	Tebuconazole	0.0054	0.01

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GENERAL PROTOCOL

for EU Proficiency Tests on Pesticide Residues in Food and Feed

Introduction

This protocol contains general procedures valid for all European Union Proficiency Tests (EUPTs) organised on behalf of the European Commission, DG-SANTE[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-SANTE based on regulation (EC) 625/2017[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-SANTE = European Commission, Health and Food Safety Directorate-General

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

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

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

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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-SANTE. The EUPT-SC consists of expert scientists with many years of experience in PTs and/or pesticide residue analysis. The actual composition of the EUPT-SC, the affiliation of each member is shown on the EURL-Website. The members of the EUPT-SC will also be listed in the Specific Protocol and the Final Report of each EUPT.

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

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

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

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

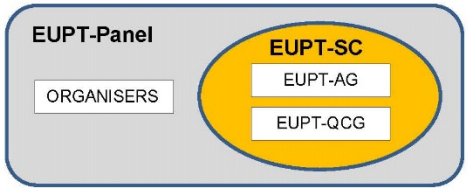
The EUPT-QCG has the additional function of supervising the quality of EUPTs and of assisting the EURLs in confidential aspects such as the choice of the pesticides to be present in the Test Item and the concentrations at which they should be present.

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

www.eurl-pesticides.eu

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

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



The decisions of the EUPT-Panel will be documented.

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

EUPT Participants

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

- Art. 28 of Reg. 396/2005/EC[6] (for all OfLs analysing for pesticide residues within the framework of official controls[7] of food or feed)
- Art. 101 (1)(a) of Reg. (EC) 625/2017 (for all NRLs)

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

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

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

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

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

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

Based on Reg. (EC) 625/2017, OfLs not paying the EUPT sample delivery fee will be initially warned that their participation in subsequent EUPTs could be denied. In case of a repetitive non-payment, the EUPT organisers will inform the competent authority to take action.

Confidentiality and Communication

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

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

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

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

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

The official language used in all EUPTs is English.

Announcement / Invitation Letter

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

Target Pesticide List

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

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

Specific Protocol

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

Homogeneity of the Test Item

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

The homogeneity test data are statistically evaluated according to ISO 13528, Annex B or to the International Harmonized Protocols jointly published by ISO, AOAC and IUPAC. The results of all homogeneity tests are presented to the EUPT-SC. In special cases, where the above homogeneity test criteria are not met, the EUPT-SC considering all relevant aspects (e.g. the homogeneity results of other pesticides spiked at the same time, the overall distribution of the participants'

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

Stability of the analytes contained in the Test Item

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

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

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

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

Considering knowledge about the expected susceptibility of pesticides in the Test Item to possible losses, the Organisers will choose the shipment conditions to be such that pesticide losses are minimised (e.g. shipment of frozen samples, addition of dry ice). As shipment time can differ

between labs/countries it is recommended that the Organisers conduct additional stability tests at conditions simulating shipment. Should critical losses be detected for certain pesticides the EUPT-SC will be informed (or the EUPT-QCG before or during the test). Case-by-case decisions may be taken considering all relevant aspects including the shipment time of the samples to each laboratory.

Methodologies to be used by the participants

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

General procedures for reporting results

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

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

Correction of results for recovery

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

[8] Document N° SANTE/11813/2017; Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed

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of the target analytes as Internal Standards (ILISs), the 'procedural calibration' approach as well as the approach of 'standard addition' with additions of analyte(s) being made to analytical portions. Results may be corrected for recovery only in cases where this correction is applied in routine practice (including cases of MRL-violations). Laboratories are required to report whether their results were adjusted for recovery and, if a recovery factor was used, the recovery rate (in percentage) must also be reported. No recovery data are required where correction for recovery is automatic by adding amounts of analytes to the test portion for using the 'standard addition' approach, or isotopically-labelled internal standards (in both cases with spiking into the Test Item at the beginning of the extraction procedures) or procedural calibration. In these cases, the laboratories should report the actual approach that was followed.

Methodology information

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

Results evaluation

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

- ***False Positive results***

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

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

– ***False Negative results***

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

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

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

In order to minimise the influence of out-lying results on the statistical evaluation, the assigned value x_{pt} (= consensus concentration) will typically be estimated using robust estimate of the participant's mean (x^*) as described in ISO 13528:2015[9], taking into account the results reported by EU and EFTA countries laboratories only. In special justifiable cases, the EUPT-Panel may decide to eliminate certain results traceably associated with gross errors (see "Omission or Exclusion of results" below) or to use only the results of a subgroup consisting of laboratories that have repeatedly demonstrated good performance for the specific compound in the past.

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

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

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

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

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

Uncertainty of the assigned value

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

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

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

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

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

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

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

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

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

– ***z scores***

This parameter is calculated using the following formula:

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

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

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

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

$\lvert z \rvert \leq 2.0$	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.

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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Publication of results

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

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

Certificates of participation

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

Feedback

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

Correction of errors

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

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

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

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

Follow-up activities

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

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

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

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

Phase 1:

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

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

EURL EU REFERENCE LABORATORIES FOR RESIDUES OF PESTICIDES

8th Edition: Revised 23th January, 2018

- Actions: On the spot visits and training if necessary and repetition of the comparative test if feasible and close the assessment of results by the EURL.

Phase 2:

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

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

Disclaimer

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

SPECIFIC PROTOCOL

for the EU Proficiency Test for Pesticide Residues in Cereals/Feeding stuff using Multi Residue Methods, EUPT-CF12 (2018)

(last updated: 22 January 2018)

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-CF12.

Test Item (Test Material)

This proficiency test concerns the analysis of pesticide residues in hay flour. The hay has been grown in Denmark and pesticides were applied in the field.

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 Organiser will check the Test Items for sufficient homogeneity and for stability at conditions reproducing sample shipment and storage during the duration of the test. 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 20 g of hay flour Test Item with incurred pesticides and
- approximately 50 g of blank hay flour Test Item.

Shipment of Test Items

The Test Items are planned to be shipped on 29 January, 2018.

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

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

Instructions on Test Item Handling

Once received, the Test Items should be stored deep-frozen (at -18°C or below) before analysis to avoid any possible deterioration/spoilage and to minimize pesticide losses. The Test 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. Because the hay matrix will swell up when water is added, only 1g samples should be used for extraction. The MRRL are adjusted to this.

The homogeneity test is conducted using 1 g of Test Item in all cases. As sub-sampling variability increases with decreasing analytical portion size, sufficient homogeneity can only be guaranteed where participants employ sample portions that are equal to or larger than the ones stated above.

Results Submission Website and Deadlines

The analytical scope should be selected prior to the shipment of the samples. This can be done via the **EUPT-CF12 Result Submission Website**. The scope selection subpage will be open from 16-26 January 2018

Sample receipt acknowledgement, analytical results and method information are to be submitted via the **EUPT-CF12 Result Submission Website** .

Relevant links and documents can be found on the EURL-CF webpage: **EUPT–CF12 Website** .

The Result Submission Website will be accessible from 30 January 2018. 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) send to the laboratory via email.

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 1 March 2018 at 24.00 CET.

Test Item Receipt and Acceptance - Subpage 0

Once the laboratory has received the Test Items it must report to the organiser, via the **EUPT-CF12 Result Submission Website**, the date of receipt, the condition of the Test Item, and its acceptance. The deadline for acceptance is the 2 February 2018. If the laboratory does not respond by this deadline, the Organiser will assume that the Test Items have been received and accepted.

If participants have not received the Test Items by the 2 February 2018 at noon, they must inform the Organiser immediately by e-mail to eurl-cf@food.dtu.dk.

Reporting Qualitative and Quantitative Results - Subpages 1 and 2

To report their results, laboratories must access the **EUPT-CF12 Result Submission Website**.

Deadline: All results must be reported on the online result submission website by 1 March 2018 at 24.00 CET. The website will NOT be accessible for result submission after this date and time, and any results reported after the deadline will not be included in the statistical treatment, or in the final report.

Results should **not** be reported where a pesticide

a) was not detected,

b) was detected below the RL (Reporting Limit) of the laboratory, or

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

Significant Figures:

Residue levels <0.010 mg/kg;

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

Residue levels ≥ 0.010 mg/kg;

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

Reporting Information on Analytical Methodology - Subpage 3

All laboratories are requested to provide information on the analytical method(s) they have used via the **EUPT-CF12 Result Submission Website**. The laboratories are asked to thoroughly fill in this important information in order to minimize the administrative burden of collecting this information at a later stage.

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

In case of false negative results the affected laboratories will be asked to provide details on the methodology used after the deadline for result submission. This can be done by accessing subpage 4 on the **EUPT-CF12 Result Submission Website**. This subpage will be accessible from 5 March 2018.

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

Follow-up actions

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

Documents

All documents related to EUPT-CF12 can be found on **EUPT–CF12 Website**.

Calendar

Activity	Dates
Announcement Calendar Target Pesticide List	November 2017
EUPT-Registration Website open	5 December 2017
Deadline for registration	12 January 2018
Specific Protocol published	15 January 2018
Website for selecting pesticide scope open	15 January 2018
Website for selecting pesticide scope closed	26 January 2018
Distribution of Test items	29 January 2018
Deadline for receipt and acceptance of Test Materials	within 24 hr on receipt
Deadline for Result Submission	1 March 2018 at 24.00 CET
Deadline for submission of additional method information for false negative results	5 March 2018 at 24.00 CET
Preliminary Report (only compilation of results) published	30 April 2018
Final Report published	December 2018

Participation Fees

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

- 200 €

The participation fees for laboratories from third countries will be:

- 350 €

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

Delays in Payment

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

Any questions concerning invoices must be directed to Miche Micereme Esati, mmes@adm.dtu.dk at the financial department of DTU.

Contact information:

DTU Food
National Food Institute

DANAK
PT Reg.nr. 518

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