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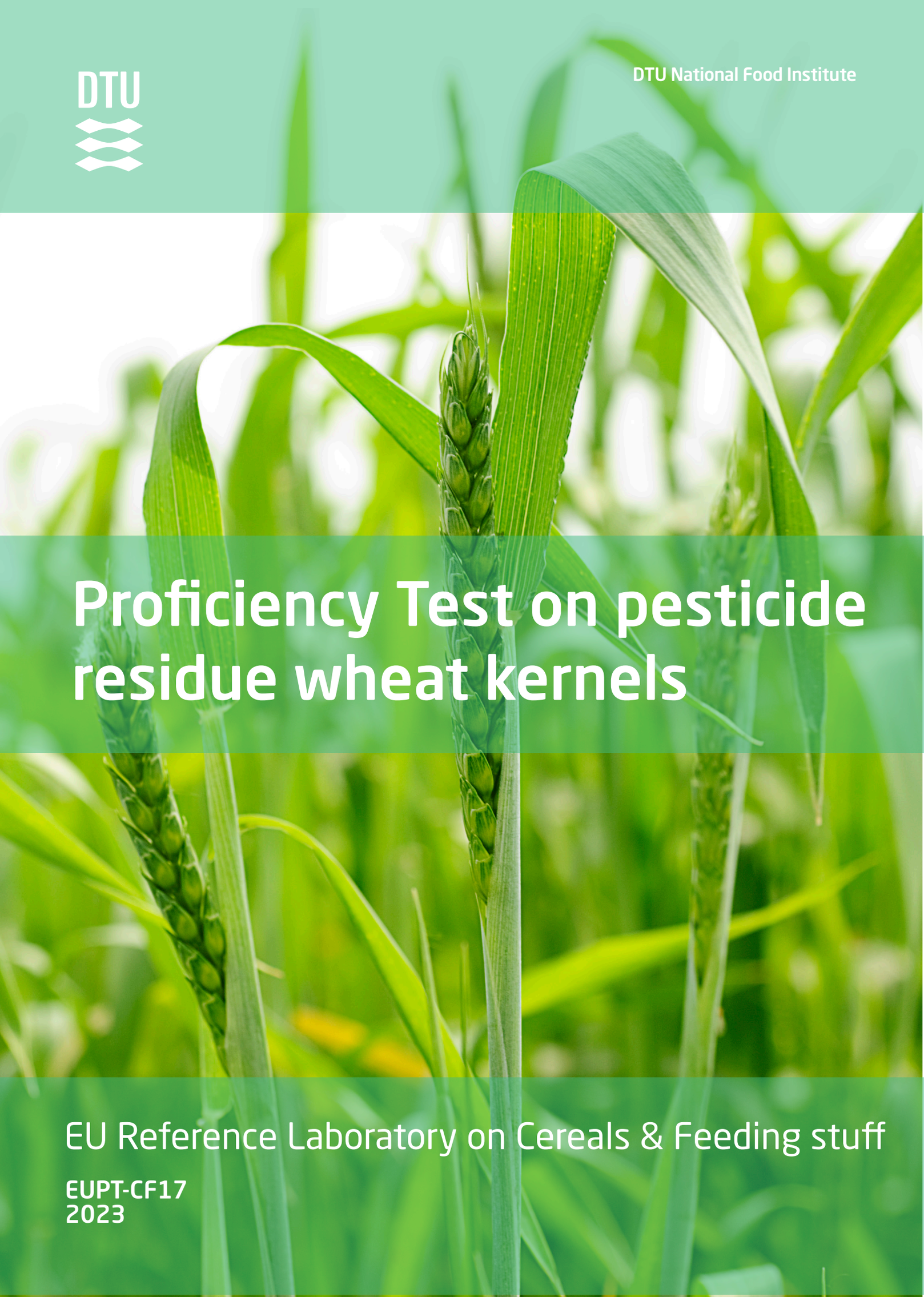

Proficiency Test on pesticide residue wheat kernels

EU Reference Laboratory on Cereals & Feeding stuff

EUPT-CF17
2023

EU PROFICIENCY TESTS
EUPT-CF17, 2023

Pesticide Residues in Wheat Kernels

Final Report

2. edition

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PREFACE

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

The present EUPT was the seventeenth organized within the frame of the EURL activities with cereal or feed matrices as Test Items. The previous PTs were EUPT-C1/SRM2 on wheat, EUPT-C2 on wheat, EUPT-C3/SRM4 on hay, EUPT-C4 on rye, EUPT-C5/SRM6 on rice, EUPT-C6 on barley, EUPT-CF7 on animal feed, EUPT-CF8 on wheat, EUPT-CF9 on maize, EUPT-CF10 on rye flour, EUPT-CF11 on oat flour, EUPT-CF12 on hay flour, EUPT-CF13 on rye kernels, EUPT-CF14 on rice kernels, EUPT-CF15 on rapeseed cake and the EURPT-CF16 on barley kernels. The PTs in 2007, 2009, 2011, 2015 and 2020 were jointly organised by the EURL-CF and EURL-SRM using same cereal and focusing on both MRM and SRM pesticides. The other PTs have only focused on MRM-pesticides. The wheat kernels used for EUPT-CF17 were treated both with formulations in the field and post-harvest in the laboratory.

Participation in EUPT-CF17 was compulsory for all National Reference Laboratories (NRLs) and Official Laboratories (OfLs) within the EU involved in the determination of pesticide residues in cereals for human or animal consumption using multi residue methods for their national programmes. Official laboratories from EFTA countries (Iceland, Norway and Switzerland), as well as official laboratories from EU-candidate states, were invited to take part in this EUPT. Selected laboratories from Third Countries were also allowed to take part in this exercise, but their results, together with the EU-candidate state laboratories, were not used when establishing the Assigned Values for each pesticide.

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

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EUROPEAN COMMISSION EURL PROFICIENCY TEST ON PESTICIDE RESIDUES IN CEREALS EUPT-CF17, 2023

1. INTRODUCTION

On 7 November 2022 the announcement of the 17th European Commission's Proficiency Test on Cereals and Feed (EUPT-CF17) 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 169 individual compulsory compounds and 58 voluntary requiring the use of multi residue methods (MRMs), along with a minimum required reporting level (MRRL) stipulated for each compound. Links to The General Protocol containing information (**Annex 1**) that is common to all EUPTs, the Specific protocol (**Annex 2**), as well as a list of labs that are obliged to take part in the EUPT-CF17, were provided via the homepage. Laboratories were able to register online from December 2022 to 9 of January 2023. In total 149 laboratories from EU and EFTA countries agreed to participate in the test as well as 13 laboratories from EU-Candidate States and Third Countries (**Appendix 1**).

The wheat were sprayed in the field with 16 pesticides. The cultivation was performed in 2022 in Denmark by the Danish Centre for Food and Agriculture at Aarhus University. After analyses of the pesticide residues content, it was decided to additionally spike in the laboratory with six pesticides, which were either not included in the field treatments or where residues were too low for the evaluation.
The pesticides employed for the field treatment were selected by the EURL-CF and the EUPT quality control group. The application rates and harvest intervals chosen were based on previous experience and data from supervised residue trials. The test material was checked for homogeneity before shipping to participants. Furthermore, the stabilities of the pesticides in the Test Item were checked several times during the period of time allowed for laboratories to complete the PT exercise.

The participating laboratories were provided with 100 g portions of the rapeseed cake Test Item. The Test Items were shipped to participants on 6 February 2023 and the deadline for submission of results to the Organiser was the 6 March 2023. The deadline for submission of additional information for false negative results was the 5 March 2023. The participants were asked to analyse the Test Item and report the concentrations of any pesticide residues found that were included in the Target Pesticide List (**Appendix 2**). Submission of results was performed online via the DTU Webtool.

1.1 Analytical methods

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

- QuEChERS - Citrate buffered (EN 151662): 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 1 ml of supernatant was filtrated and transferred in a autosampler vial for the LC/MS/MS analysis. The remaining extract 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.

1.2 Selection of Pesticides for the Target Pesticide List

The pesticides to be included in the target pesticides list were selected by the Organiser and the Quality Control Group, taking into account the present and upcoming scope of the EU multi-annual coordinated control programme, the working document, and pesticides according to their relevance and risk-potential, as well as pesticides relevant to the specific commodity (barley kernels). The overall capacity and capability of the laboratories within the EU, as assessed from previous PTs and surveys, was also taken into account. The minimum required reporting level (MRRL)

for all pesticides in the target list was in general set at 0.01 mg/kg. However, for 20 pesticides the MRRL were set at or below 0.005 mg/kg.

1.3 Preparation of the Test Item

The field spraying was performed in 2022 in Denmark and organised by Danish Centre for Food and Agriculture at Aarhus University. Approximately, 24 kg of the harvested wheat were used for this PT. It was decided to additionally spike in the laboratory with six pesticides, which were either not included in the field treatments or where residues were too low for the evaluation (**Table 1**). Spiking in the laboratory was performed using formulations or pure standards. Seven portions of 1.4 kg of the non-treated wheat was spiked and subsequently mixed with 20 kg of field treated wheat kernels and homogenised thoroughly. One hundred gram portions of the homogenized wheat kernels were then weighed out into screw-capped polyethylene plastic bottles, sealed, numbered, and stored in a freezer at about -20 °C prior to homogeneity testing and distribution to participants.

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

Pesticides	Application in field	Spike in laboratory	Formulation/standard
Azoxystrobin	x		Amistar/Amistar gold
Bixafen	x		Ascra Xpro
Clomazone*	x		Clomate
Cyazofamid	x	x	Ranman Top/Analytical standard
Cyfluthrin		x	Analytical standard
Cyprodinil	x		Kayak
Difenoconazole	x		Amistar gold
Dimethomorph	x		Cabrio Duo
Flonicamid	x		Teppeki
Fluapyram	x		Ascra Xpro
HCH-beta*		x	Analytical standard
Metconazole	x		Juventus
Phenmedipham*		x	Analytical standard
Pirimicarb	x		Pirimor
Pirimicarb-desmethyl		x	Analytical standard
Proquinazid	x		Talius
Prothioconazole	x		Ascra Xpro/Kayak/Madison
Pyraclostrobin	x		Cabrio Duo
Pyriproxyfen	x	x	Admiral/Analytical standard
Trifloxystrobin	x		Madison

*Voluntary pesticides.

1.4 Homogeneity test

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

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

The homogeneity test is to show that the between-bottle variance is not greater than the within-bottle variance. The acceptance criteria to show that the Test Items were sufficiently homogeneous for the proficiency test was that: $S_s^2 < c$ where S_s is the between-bottle sample standard deviation and $c = F_1 \times \sigma_{all}^2 + F_2 \times s_{an}^2$; F1 and F2 being constants with values of 1.83 and 0.93, respectively, from the 11 samples taken, σ_{all}^2 = 0.3 x FFP RSD (25%) x the analytical sampling mean for all pesticides, and s_{an} is the estimate of the analytical standard deviation.

As all pesticides passed the homogeneity test, when the Test Item was stored at -18 °C, the Test Item was considered to be sufficiently homogenous and suitable for the EUPT-CF17.

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	Ss^2	c	$Ss^2 < c$
Azoxystrobin	0.210	0.00021	0.0007	Pass
Bixafen	0.085	0.00000	0.0002	Pass
Clomazone *	0.032	0.00000	0.0000	Pass
Cyazofamid	0.221	0.00009	0.0008	Pass
Cyflutrin	0.069	0.00002	0.0001	Pass
Cyprodinil	0.342	0.00000	0.0027	Pass
Difenoconazole	0.094	0.00000	0.0002	Pass
Dimethomorph	0.165	0.00000	0.0005	Pass
Flonicamid	0.043	0.00000	0.0000	Pass
Fluopyram	0.125	0.00000	0.0003	Pass
HCH-beta *	0.042	0.00001	0.0001	Pass
Metconazole	0.060	0.00000	0.0001	Pass
Phenmedipham *	0.071	0.00002	0.0001	Pass
Pirimicarb	0.037	0.00000	0.0000	Pass
Pirimicarb-desmethyl	0.056	0.00001	0.0000	Pass
Prothioconazole-desthio	0.192	0.00014	0.0006	Pass
Pyraclostrobin	0.109	0.00002	0.0002	Pass
Pyriproxyfen	0.049	0.00001	0.0001	Pass
Trifloxystrobin	0.050	0.00000	0.0000	Pass

*Voluntary pesticides.

1.5 Stability tests

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

The stability test was performed according to ISO 13528, Annex B [5]. Two different storage temperatures were used; room temperature and -18 °C. Six sub-samples (analytical portions) were analysed on each test day. A pesticide is considered to be adequately stable if $| x_1 - y_i | \leq 0.3 \times \sigma$, where x_1 is the mean value of the first stability test, y_i the mean value of the last stability test and σ the standard deviation used for proficiency assessment (25% of the assigned value):

The dates of testing were as follows:

Day 1: 6 February 2023
Day 2: 20 February 2023
Day 3: 6 March 2023

The results of the stability test for storage temperature -18 °C are given in Table 3. All pesticides passed the test at -18 °C. At room temperature only cyazofamid did not pass the test. However, all the laboratories were instructed to store the test item at -18 degree and the stability test was consequently accepted. See the individual stability figures for all pesticides in **Appendix 4**.

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

Pesticides	Mean, mg/kg	| x1 - yi |	0.3×σ	| x1 - yi | ≤ 0.3×σ
Azoxystrobin	0.234	0.008	0.018	Pass
Bixafen	0.096	0.003	0.008	Pass
Clomazone *	0.045	0.000	0.003	Pass
Cyazofamid	0.203	0.002	0.003	Pass
Cyflutrin	0.115	0.007	0.005	Pass
Cyprodinil	0.549	0.004	0.028	Pass
Difenoconazole	0.099	0.003	0.008	Pass
Dimethomorph	0.207	0.014	0.014	Pass
Flonicamid	0.100	0.003	0.004	Pass
Fluopyram	0.146	0.004	0.012	Pass
HCH-beta *	0.051	0.001	0.004	Pass
Metconazole	0.076	0.000	0.006	Pass
Phenmedipham *	0.061	0.001	0.005	Pass
Pirimicarb	0.036	0.003	0.003	Pass
Pirimicarb-desmethyl	0.051	0.001	0.005	Pass
Proquinazid	0.080	0.003	0.007	Pass
Prothioconazole-desthio	0.187	0.006	0.014	Pass
Pyraclostrobin	0.106	0.004	0.085	Pass
Pyriproxyfen	0.063	0.003	0.005	Pass
Trifloxystrobin	0.048	0.002	0.004	Pass

*Voluntary pesticides.

1.6 Organisational details

1.6.1 Access to documents, registration and confidentiality

In the invitation letter, all NRLs and OfLs were requested to register using the online registration link from December 2021. All documents related to this EUPT (Calendar, Target Pesticides List, Specific Protocol, General Protocol) were uploaded to the EURL website and the CIRCA platform. Laboratories that were intending not to participate were given the opportunity to explain the reasons for their non-participation. Participants from Candidate countries and third countries did also have access to another online registration link. On 23 January, the participants received a link to DTU web tool, along with login credentials and were asked to enter the web tool and to select the scope of pesticides they wanted to be evaluated on. This had to be done before the samples were shipped to the participants.

1.6.2 Distribution of the Test Item

On 6 February 2023, the Test Item (100 g) was shipped to all participants in insulated polystyrene boxes containing a freezer block. The laboratories were asked to check the state of the sample on receipt and to enter the web tool to report whether they accept/not accept the Test Item. No blank test material was send.

1.6.3 Submission of results

The participants had to submit their results via a web tool. All participants had access to the result-submission website from a few days after shipment until the result-submission deadline (6 March 2023). Participants were asked not only to report their analytical results, but also to give information regarding accreditation, reporting limits and details regarding the methods they used to analyse the Test Item.

2. EVALUATION OF THE RESULTS

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

2.1 False positives and negatives

2.1.1 False positives

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

2.1.2 False negatives

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

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

In order to minimise the influence of out-lying results on the statistical evaluation, the assigned value xpt (= consensus concentration) will typically be estimated using robust estimate of the participants' mean (x*) as described in ISO 13528:2015, taking into account the results reported by only EU and EFTA countries laboratories. 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(xpt) is calculated according to ISO 13528:2015 as:

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

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 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 rounded to one decimal place. For the calculation of combined z scores (see below) the original z scores will be used and rounded to one decimal place after calculation.

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

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

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

For results considered as false negatives, z scores will be calculated using the MRRL or RL (the laboratory's Reporting Limit) if RL < MRRL. Where, using this approach, the calculated z scores for false negatives are > −3 (still questionable), they will be fixed at −4 to underline that these are unacceptable results. These z-scores will typically appear in the z-score histograms and used in the calculation of combined z-scores.

2.6 Category A and B classification and combined z scores (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, 149 EU and EFTA laboratories, from 29 different countries (26 EU member states), agreed to participate in this proficiency test. Six EU participants did not submit results. Additionally, nine participants from non-EU Countries registered for the PT. The participating laboratories are listed in **Appendix 1**.

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

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

Pesticides	No. of reported results	No. of NA	False negatives	% of labs reporting results [1]
Azoxystrobin	135	8	1	94
Bixafen	115	28	3	80
Clomazone *	93	50	1	65
Cyazofamid	120	23	6	84
Cyfluthrin	129	14	6	90
Cyprodinil	136	7	1	95
Difenoconazole	136	7	2	95
Dimethomorph	129	14	1	90
Flonicamid	120	23	6	84
Fluopyram	131	12	1	92
HCH-beta *	113	30	3	79
Metconazole	123	20	1	86
Phenmedipham *	69	74	0	48
Pirimicarb	136	7	1	95
Pirimicarb-desmethyl	111	32	5	78
Proquinazid	113	30	0	79
Prothioconazole-desthio	120	23	5	84
Pyraclostrobin	130	13	5	91
Pyriproxyfen	129	14	2	90
Trifloxystrobin	134	9	2	94

* Voluntary pesticides

[1] '% results' have been calculated using the number of laboratories that reported results for each particular compound and the total number of EU laboratories that submitted results (n = 143). False negatives are included in reported results.

Azoxystrobin, cyfluthrin, cyprodinil, difenoconazole, dimethomorph, fluopyram, pirimicarb, pyraclostrobin, pyriproxyfen and trifloxystrobin were the most frequently analysed compounds with ≥90 % of the labs submitting results for these compounds. Bixafen, cyazofamid, flonicamid, HCH-beta, metconazole, pirimicarb-desmethyl, proquinazid and prothioconazole-desthio were analysed and reported by 78-86% of the participants. Clomazone and phenmedipham were only analysed and reported by 48-65% of participants.

3.1.1 False positives

Seven participants (all form EU and EFTA) countries reported six results for five different additional pesticides above the MRRL that had not been used to treat the Test Item (**Table 5**). The pesticides were: acephate, biphenyl, lindane and triadimenol. 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. Additionally, one participant reported acetamiprid at 0.006 mg/kg. However, this result were below the MRRL at 0.01 mg/kg and therefore not considered a false positive.

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

Lab code	Pesticides	Concentration mg/kg	Determination technique	RL, mg/kg	MRRL, mg/kg
11	Lindane	0.037	GC-MS/MS (QQQ)	0.01	0.01
24	Lindane	0.011	GC- (μ) ECD	0.01	0.01
38	Acephate	2.447	LC-MS/MS QQQ	0.01	0.01
41	Biphenyl	0.024	GC-MS/MS (QQQ)	0.01	0.01
108	Lindane	0.053	GC-MS/MS (QQQ)	0.01	0.01
109	Triadimenol	0.011	GC-MS/MS (QQQ)	0.01	0.01

3.1.2 False negatives

Not reported results for pesticides actually present in the Test Item were judged as false negatives. **Table 6** summarizes the number of reported false negatives for each pesticide. Twenty nine participants submitted 60 false negatives results for 18 different pesticides, which represents 3.3% of the total number of results submitted by EU and EFTA laboratories. Around 20 % of the EU and EFTA participants (28 laboratories) reported false negative results.

3.2 Assigned values, target standard deviations and Alg A standard deviations

3.2.1 Assigned values

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

All assigned values for the pesticides can be seen in **Table 7**. For the evaluated pesticides the assigned values were in the range of 0.033-0.324 mg/kg.

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 8**)

Table 6. False negative results (FN).

Lab code	Azoxystrobin	Bixafen	Clomazone *	Cyazofamid	Cyfluthrin	Cyprodinil	Difenoconazole	Dimethomorph	Flonicamid	Fluopyram	HCH-beta *	Metconazole	Pirimicarb	Pirimicarb-desmethyl	Prothioconazole-desthio	Pyraclostrobin	Pyriproxyfen	Trifloxystrobin
10					FN													
11				FN							FN							
13		FN																
30															FN			
38				FN			FN		FN									
41					FN													
43									FN									
48					FN										FN			
50														FN				
53			FN															
58									FN									
61															FN			
64	FN		FN		FN		FN					FN			FN		FN	FN
65																FN		
82														FN				
86				FN														
94											FN							
101																		FN
103	FN			FN		FN	FN	FN				FN	FN			FN	FN	FN
104											FN							
107				FN														
109				FN					FN					FN		FN		
116					FN													
120					FN													
129									FN									
135		FN												FN				
150															FN			
154																FN		
170		FN			FN				FN	FN				FN	FN	FN	FN	

* Voluntary pesticides

3.2.2 Target standard deviations and Alg A standard deviations

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 for the evaluated pesticide in the range of 17-27 % but on average, the Alg A-RSD was 22%, lower than 25% FFP-RSD used for the z score calculations.

Table 7. Assigned values and their uncertainty in mg/kg, Fit-For-Purpose Relative Standard Deviation (FFP RSD) and Robust Relative Standard Deviation (Alg A RSD) for the pesticides present in the Test Item.

Pesticides	MRRL, mg/kg	Assigned value, mg/kg	Uncertainty, mg/kg	FFP RSD, %	Alg A RSD, %
Azoxystrobin	0.01	0.207	0.003	25	14
Bixafen	0.01	0.088	0.002	25	19
Clomazone *	0.01	0.035	0.001	25	15
Cyazofamid	0.01	0.038	0.001	25	17
Cyfluthrin	0.01	0.057	0.002	25	24
Cyprodinil	0.01	0.324	0.007	25	20
Difenoconazole	0.01	0.090	0.002	25	19
Dimethomorph	0.01	0.164	0.003	25	16
Flonicamid	0.01	0.046	0.001	25	16
Fluopyram	0.01	0.134	0.003	25	17
HCH-beta *	0.01	0.041	0.001	25	18
Metconazole	0.01	0.069	0.002	25	20
Phenmedipham *	0.01	0.057	0.002	25	25
Pirimicarb	0.01	0.033	0.001	25	19
Pirimicarb-desmethyl	0.01	0.053	0.001	25	14
Proquinazid	0.01	0.085	0.002	25	23
Prothioconazole-desthio	0.01	0.161	0.003	25	17
Pyraclostrobin	0.01	0.097	0.002	25	18
Pyriproxyfen	0.01	0.052	0.001	25	16
Trifloxystrobin	0.01	0.049	0.001	25	19

* Voluntary pesticides

3.3 Assessment of laboratory performance

3.3.1 Z scores

Z scores have been calculated for all the quantified pesticides using the FFP RSD of 25%. **Table 8** shows an overview of the acceptable, questionable, and unacceptable z scores and **Tables 9 a/b/c**- 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**.

Of the reported results for the evaluated pesticides, more than 90% were azoxystrobin, bixafen, clomazone, cyprodinil, difenoconazole, dimethomorph, fluopyram, HCH-beta, metconazole, pirimicarb, pirimicarb-desmethyl, proquinazid, prothioconazole-desthio, pyraclostrobin pyriproxyfen and trifloxystrobin. For cyazofamid, cyfluthrin, flonicamid and phenmediphambetween 87-89% of the results were acceptable.

Table 8. Number of acceptable, questionable, unacceptable z scores, and false negatives.

Pesticides	No. of reported results	Assigned values	Acceptable %	Questionable %	Unacceptable[1] %	False negatives %
Azoxystrobin	135	0.207	96	1	2	1
Bixafen	115	0.088	94	2	4	3
Clomazone *	93	0.035	95	3	2	1
Cyazofamid	120	0.038	88	3	9	5
Cyfluthrin	129	0.057	87	6	7	5
Cyprodinil	136	0.324	94	4	1	1
Difenoconazole	136	0.090	93	4	4	1
Dimethomorph	129	0.164	94	2	5	1
Flonicamid	120	0.046	89	3	8	5
Fluopyram	131	0.134	93	3	4	1
HCH-beta *	113	0.041	90	4	6	3
Metconazole	123	0.069	94	3	2	1
Phenmedipham *	69	0.057	87	7	6	0
Pirimicarb	136	0.033	95	1	4	1
Pirimicarb-desmethyl	111	0.053	95	0	5	5
Proquinazid	113	0.085	93	4	3	0
Prothioconazole-desthio	120	0.161	90	4	6	4
Pyraclostrobin	130	0.097	92	2	5	4
Pyriproxyfen	129	0.052	92	4	4	1
Trifloxystrobin	134	0.049	92	3	5	2

* Voluntary pesticides

[1] Unacceptable z scores includes false negative results.

3.3.2 Analytical methods used

More than five different analytical methods have been used by the laboratories. For the majority of the results, 73%, QuEChERS, Citrate buffered (EN 151662) was used. However, variations in the clean-up procedures were reported by the labs, e.g. some used a freezing out step (24% of the participants), centrifugation (20%), some used d-SPE with PSA/MgSO4 (28%), some used d-SPE with ODS/ $MgSO_4$ (4%) and other used different combination of ODS, PSA, C18, z-sep (11%). Liquid-liquid partition was used by 5% of the participants. Consequently, it was not one specific method.

Other extraction methods have been used; the original QuEChERS version method (J. AOAC 86, 2003) and QuEChERS-Acetate buffered (AOAC Official method 2007.01) were used by 5.5% and 7 % of the laboratory, respectively. The Mini-Luke method and the SweEt method were each used by 3% of the participants. The remaining 3 % of the participants used other methods. More than 93% of the reported results derived from a method where water was added before extraction.

For milling, 53% of the labs used a knife mill and 27% of the labs used centrifugal mill. Moreover, 7% used a disk mill, 1% used an horizontal mill, and 2% used a hammer mill. Furthermore, 9% of the labs did not specify the type of mill used or did not mill at all.

GC instruments was used for 30% of the results, mainly GC-MS/MS (93%), but also GC-MS (1%) and GC- (μ) ECD (2%) was used. GC-NPD was used for 0.1 results, GC-iontrap for 1, GC-TOF, GC-Q-Orbitrap and GC-Orbitrap for 2% result. LC instruments was used for 70% of the reported results, mainly LC-MS/MS (73%) but 7% used high resolution instrument like LC-Orbitrap, LC-Q-Orbitrap or LC-Q-TOF. Finally, 0.1% results were based on LC-MS. No result were analysed using specific detectors such as LC-Iontrap , LC-Fluorescence, LC-UV, or LC-DAD.

Table 9a. Results for the mandatory pesticides azoxystrobin, bixafen, cyazofamid, cyfluthrin, difenoconazole, dimethomorph and flonicamid in mg/kg, the corresponding z scores, MRRLs and the assigned values.

Laboratory code	Azoxystrobin	Z-scores (FFP RSD (25%)	Bixafen	Z-scores (FFP RSD (25%)	Cyazofamid	Z-scores (FFP RSD (25%)	Cyfluthrin	Z-scores (FFP RSD (25%)	Cyprodinil	Z-scores (FFP RSD (25%)	Difenoconazole	Z-scores (FFP RSD (25%)	Dimethomorph	Z-scores (FFP RSD (25%)	Flonicamid	Z-scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.207**		**0.088**		**0.038**		**0.057**		**0.324**		**0.090**		**0.164**		**0.046**	
3	0.201	-0.1	0.0809	-0.3			0.0626	0.4	0.387	0.8	0.0965	0.3	0.175	0.3		
4	0.176	-0.6							0.316	-0.1	0.091	0.0			0.044	-0.2
5	0.201	-0.1			0.034	-0.4			0.299	-0.3	0.087	-0.2	0.149	-0.4		
6	0.156	-1.0	0.076	-0.5	0.029	-0.9	0.053	-0.3	0.369	0.6	0.067	-1.0	0.127	-0.9	0.049	0.3
7	0.201	-0.1	0.0739	-0.6	0.0412	0.4	0.0535	-0.3	0.27	-0.7	0.0705	-0.9	0.17	0.1	0.0434	-0.2
8	0.222	0.3	0.101	0.6	0.0421	0.5	0.0603	0.2	0.313	-0.1	0.1	0.4	0.193	0.7	0.045	-0.1
9	0.214	0.1	0.1	0.5	0.041	0.3	0.056	-0.1	0.364	0.5	0.1	0.4	0.195	0.8	0.053	0.6
10	0.251	0.9	0.099	0.5	0.0401	0.3	FN	-4.0	0.408	1.0	0.089	-0.1	0.172	0.2	0.049	0.3
11	0.135	-1.4			FN	-4.0	0.042	-1.1	0.347	0.3	0.098	0.3	0.11	-1.3		
12	0.21	0.1	0.07	-0.8	0.03	-0.8	0.043	-1.0	0.28	-0.5	0.083	-0.3	0.14	-0.6	0.048	0.2
13	0.226	0.4	FN	-4.0	0.038	0.0	0.062	0.3	0.316	-0.1	0.099	0.4	0.2	0.9		
14	0.134	-1.4	0.078	-0.5	0.027	-1.1	0.045	-0.8	0.197	-1.6	0.065	-1.1	0.141	-0.6	0.06	1.2
15	0.254	0.9			0.0455	0.8			0.408	1.0	0.092	0.1	0.194	0.7	0.0514	0.5
16	0.185	-0.4					0.185	9.0	0.185	-1.7	0.185	4.2	0.185	0.5	0.185	>5
17	0.194	-0.2	0.0798	-0.4	0.0297	-0.8	0.0456	-0.8	0.272	-0.6	0.0737	-0.7	0.139	-0.6	0.0427	-0.3
18	0.174	-0.6					0.02	-2.6	0.2	-1.5	0.066	-1.1	0.142	-0.5		
19	0.2	-0.1			0.034	-0.4	0.082	1.7	0.31	-0.2	0.086	-0.2	0.17	0.1		
20	0.246	0.8	0.104	0.7	0.0399	0.2	0.0594	0.2	0.394	0.9	0.119	1.3	0.198	0.8	0.0459	0.0
21	0.169	-0.7	0.069	-0.9	0.031	-0.7	0.045	-0.8	0.301	-0.3	0.067	-1.0	0.138	-0.6	0.043	-0.2
22	0.168	-0.8	0.072	-0.7	0.0335	-0.4	0.0468	-0.7	0.2438	-1.0	0.0668	-1.0	0.1178	-1.1	0.0398	-0.5
23	0.219	0.2	0.075	-0.6	0.037	-0.1	0.042	-1.1	0.352	0.4	0.075	-0.7	0.138	-0.6	0.041	-0.4
24	0.172	-0.7	0.07	-0.8	0.036	-0.2	0.067	0.7	0.201	-1.5	0.089	-0.1	0.132	-0.8	0.033	-1.1
25																
26	0.202	-0.1	0.0811	-0.3	0.0384	0.1	0.0714	1.0	0.38	0.7	0.0743	-0.7	0.181	0.4	0.0434	-0.2
27	0.211	0.1	0.0947	0.3	0.038	0.0	0.0545	-0.2	0.352	0.4	0.0962	0.3	0.162	0.0	0.0608	1.3
28	0.24	0.6	0.098	0.5	0.035	-0.3	0.049	-0.6	0.34	0.2	0.11	0.9	0.19	0.6	0.041	-0.4
29	0.221	0.3	0.0729	-0.7	0.0437	0.6	0.0399	-1.2	0.311	-0.2	0.0854	-0.2	0.192	0.7	0.041	-0.4
30	0.191	-0.3			0.038	0.0	0.053	-0.3	0.278	-0.6	0.084	-0.3	0.151	-0.3	0.02	-2.3
31	0.23	0.4	0.096	0.4	0.039	0.1	0.063	0.4	0.39	0.8	0.1	0.4	0.19	0.6	0.048	0.2
33	0.19	-0.3	0.0709	-0.8	0.0353	-0.3	0.0626	0.4	0.268	-0.7	0.0668	-1.0	0.147	-0.4	0.045	-0.1
34	0.16	-0.9	0.095	0.3	0.036	-0.2	0.05	-0.5	0.34	0.2	0.093	0.1	0.16	-0.1	0.044	-0.2
35	0.238	0.6	0.105	0.8	0.0443	0.7	0.0741	1.2	0.389	0.8	0.0984	0.4	0.189	0.6	0.0488	0.3
36																
37	0.18	-0.5	0.074	-0.6	0.031	-0.7	0.059	0.1	0.36	0.4	0.1	0.4	0.14	-0.6	0.055	0.8
38	4.714	>			FN	-4.0	0.032	-1.8	0.093	-2.9	FN	-4.0	0.176	0.3	FN	-4.0
39	0.212	0.1			0.031	-0.7	0.042	-1.1	0.251	-0.9	0.079	-0.5	0.165	0.0	0.042	-0.3
40	0.507	>5	0.447	>5	0.088	>5	0.055	-0.1	0.358	0.4	0.207	>5	0.301	3.3	0.061	1.3
41	0.234	0.5	0.083	-0.2	0.035	-0.3	FN	-4.0	0.263	-0.7	0.089	-0.1	0.185	0.5	0.05	0.4
42	0.208	0.0	0.104	0.7	0.038	0.0	0.079	1.5	0.369	0.6	0.094	0.2	0.165	0.0	0.042	-0.3
43	0.308	2.0	0.095	0.3	0.044	0.7			0.494	2.1	0.115	1.1	0.19	0.6	FN	-4.0

Laboratory code	Azoxystrobin	Z-scores (FFP RSD (25%)	Bixafen	Z-scores (FFP RSD (25%)	Cyazofamid	Z-scores (FFP RSD (25%)	Cyfluthrin	Z-scores (FFP RSD (25%)	Cyprodinil	Z-scores (FFP RSD (25%)	Difenoconazole	Z-scores (FFP RSD (25%)	Dimethomorph	Z-scores (FFP RSD (25%)	Flonicamid	Z-scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.207**		**0.088**		**0.038**		**0.057**		**0.324**		**0.090**		**0.164**		**0.046**	
44	0.227	0.4	0.0818	-0.3			0.0511	-0.4	0.392	0.8	0.0987	0.4				
45	0.183	-0.5	0.0836	-0.2	0.0311	-0.7	0.0594	0.2	0.321	0.0	0.0819	-0.4	0.15	-0.3	0.0453	0.0
46	0.178	-0.6	0.08	-0.4			0.084	1.9	0.273	-0.6	0.084	-0.3	0.186	0.5		
47	0.195	-0.2	0.0783	-0.4	0.055	1.8	0.0561	-0.1	0.488	2.0	0.102	0.5	0.23	1.6	0.051	0.5
48	0.204	-0.1	0.058	-1.4	0.026	-1.2	FN	-4.0	0.323	0.0	0.08	-0.5	0.178	0.3	0.054	0.7
49	0.186	-0.4	0.0814	-0.3	0.0339	-0.4	0.0513	-0.4	0.303	-0.3	0.0855	-0.2	0.157	-0.2	0.0411	-0.4
50	0.216	0.2	0.087	0.0	0.034	-0.4	0.06	0.2	0.337	0.2	0.092	0.1	0.186	0.5	0.042	-0.3
51	0.204	-0.1	0.094	0.3	0.04	0.2	0.061	0.3	0.36	0.4	0.084	-0.3	0.165	0.0	0.048	0.2
52	0.085	-2.4	0.036	-2.4	0.016	-2.3	0.035	-1.5	0.098	-2.8	0.022	-3.0	0.081	-2.0	0.028	-1.6
53									0.272	-0.6	0.075	-0.7	0.169	0.1		
54	0.219	0.2	0.108	0.9	0.0457	0.8	0.0229	-2.4	0.321	0.0	0.104	0.6	0.346	4.4	0.0542	0.7
55	0.228	0.4	0.0996	0.5	0.0391	0.1	0.0917	2.4	0.384	0.7	0.1	0.4	0.168	0.1	0.0474	0.1
56	0.155	-1.0	0.085	-0.1	0.025	-1.3	0.054	-0.2	0.3	-0.3	0.082	-0.4	0.155	-0.2	0.04	-0.5
57																
58	0.213	0.1	0.156	3.1	0.053	1.6	0.134	>5	0.363	0.5	0.111	0.9	0.191	0.7	FN	-4.0
59	0.208	0.0	0.108	0.9	0.07	3.4	0.039	-1.3	0.375	0.6	0.1	0.4	0.168	0.1	0.037	-0.8
60	0.258	1.0	0.105	0.8	0.075	4.0	0.092	2.4	0.483	2.0	0.121	1.4	0.231	1.6	0.054	0.7
61	0.25	0.8	0.11	1.0	0.04	0.2	0.05	-0.5	0.36	0.4	0.09	0.0	0.15	-0.3	0.07	2.1
62	0.218	0.2	0.0811	-0.3	0.0369	-0.1	0.0838	1.9	0.405	1.0	0.106	0.7	0.182	0.4	0.0474	0.1
63	0.226	0.4	0.0919	0.2	0.0566	2.0	0.0344	-1.6	0.496	2.1	0.107	0.7	0.246	2.0	0.059	1.2
64	FN	-4.0					FN	-4.0			FN	-4.0			0.051	0.5
65	0.353	2.8	0.034	-2.5	0.042	0.5	0.056	-0.1	0.279	-0.6	0.094	0.2	0.144	-0.5	0.348	>5
66	0.203	-0.1	0.086	-0.1	0.3362	>5	0.0591	0.1	0.3317	0.1	0.0758	-0.6	0.1377	-0.6	0.0353	-0.9
67	0.222	0.3	0.106	0.8	0.04	0.2	0.055	-0.1	0.407	1.0	0.11	0.9	0.195	0.8	0.046	0.0
68																
69	0.248	0.8	0.11	1.0	0.455	>5	0.0589	0.1	0.191	-1.6	0.135	2.0	0.184	0.5	0.0108	-3.1
71	0.239	0.6	0.0816	-0.3			0.0442	-0.9	0.389	0.8	0.1	0.4				
72	0.221	0.3	0.0867	-0.1	0.0378	0.0	0.0606	0.2	0.29	-0.4	0.0864	-0.2	0.158	-0.1	0.043	-0.2
73	0.216	0.2	0.096	0.4	0.041	0.3	0.052	-0.4	0.252	-0.9	0.103	0.6	0.162	0.0	0.051	0.5
74	0.222	0.3			0.032	-0.6	0.053	-0.3	0.321	0.0	0.087	-0.2	0.137	-0.7	0.051	0.5
75	0.129	-1.5	0.086	-0.1	0.037	-0.1	0.043	-1.0	0.284	-0.5	0.065	-1.1	0.143	-0.5	0.026	-1.7
76	0.214	0.1	0.123	1.6	0.04	0.2	0.078	1.5	0.374	0.6	0.096	0.2	0.163	0.0	0.046	0.0
77	0.213	0.1	0.0977	0.4	0.0391	0.1	0.0859	2.0	0.388	0.8	0.0975	0.3	0.184	0.5	0.0474	0.1
78	0.212	0.1					0.074	1.2	0.288	-0.4	0.101	0.5	0.183	0.5	0.056	0.9
79	0.171	-0.7	0.102	0.6	0.044	0.7	0.051	-0.4	0.358	0.4	0.093	0.1	0.149	-0.4	0.047	0.1
80	0.295	1.7	0.117	1.3	0.0464	0.9	0.0652	0.6	0.327	0.0	0.116	1.1	0.215	1.2	0.062	1.4
81	0.214	0.1	0.083	-0.2	0.033	-0.5	0.069	0.8	0.299	-0.3	0.087	-0.2	0.174	0.2	0.05	0.4
82	0.224	0.3	0.062	-1.2	0.032	-0.6	0.06	0.2	0.301	-0.3	0.109	0.8	0.165	0.0	0.041	-0.4
84	0.206	0.0	0.076	-0.5	0.034	-0.4	0.049	-0.6	0.326	0.0	0.087	-0.2	0.16	-0.1	0.047	0.1
85	0.169	-0.7	0.09	0.1	0.035	-0.3	0.059	0.1	0.28	-0.5	0.085	-0.2	0.148	-0.4	0.047	0.1
86	0.21	0.1	0.113	1.1	FN	-4.0	0.086	2.0	0.306	-0.2	0.102	0.5	0.187	0.6	0.053	0.6
87	0.181	-0.5	0.105	0.8	0.0408	0.3	0.0474	-0.7	0.3	-0.3	0.102	0.5	0.15	-0.3	0.0436	-0.2
88	0.21	0.1	0.112	1.1	0.047	1.0	0.081	1.7	0.291	-0.4	0.098	0.3	0.292	3.1	0.041	-0.4

Laboratory code	Azoxystrobin	Z-scores (FFP RSD (25%)	Bixafen	Z-scores (FFP RSD (25%)	Cyazofamid	Z-scores (FFP RSD (25%)	Cyfluthrin	Z-scores (FFP RSD (25%)	Cyprodinil	Z-scores (FFP RSD (25%)	Difenoconazole	Z-scores (FFP RSD (25%)	Dimethomorph	Z-scores (FFP RSD (25%)	Flonicamid	Z-scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.207**		**0.088**		**0.038**		**0.057**		**0.324**		**0.090**		**0.164**		**0.046**	
89	0.22	0.3	0.11	1.0	0.039	0.1	0.069	0.8	0.393	0.9	0.108	0.8	0.191	0.7	0.053	0.6
90	0.212	0.1	0.0857	-0.1	0.039	0.1	0.0583	0.1	0.369	0.6	0.0925	0.1	0.15	-0.3	0.0465	0.1
91	0.206	0.0	0.052	-1.6	0.032	-0.6	0.061	0.3	0.299	-0.3	0.085	-0.2	0.161	-0.1	0.041	-0.4
92																
93																
94	0.21	0.1	0.06	-1.3	0.03	-0.8	0.042	-1.1	0.23	-1.2	0.083	-0.3	0.14	-0.6	0.042	-0.3
95	0.15	-1.1	0.08	-0.4			0.04	-1.2	0.23	-1.2	0.11	0.9	0.11	-1.3		
96	0.178	-0.6			0.037	-0.1			0.431	1.3	0.083	-0.3	0.161	-0.1	0.05	0.4
97	0.218	0.2	0.089	0.0	0.037	-0.1	0.064	0.5	0.333	0.1	0.091	0.0	0.17	0.1	0.048	0.2
98	0.128	-1.5			0.03	-0.8	0.054	-0.2	0.279	-0.6	0.082	-0.4	0.016	-3.6	0.04	-0.5
99	0.197	-0.2	0.111	1.0	0.041	0.3	0.061	0.3	0.358	0.4	0.097	0.3	0.169	0.1	0.043	-0.2
100																
101	0.177	-0.6	0.0697	-0.8	0.0274	-1.1	0.0513	-0.4	0.35	0.3	0.0681	-1.0	0.126	-0.9	0.035	-0.9
103	FN	-4.0	0.08	-0.4	FN	-4.0	0.063	0.4	FN	-4.0	FN	-4.0	FN	-4.0	0.035	-0.9
104	0.273	1.3					0.091	2.4	0.461	1.7						
105	0.23	0.4	0.075	-0.6	0.044	0.7	0.052	-0.4	0.44	1.4	0.098	0.3	0.169	0.1	0.049	0.3
106	0.215	0.2	0.084	-0.2	0.039	0.1	0.056	-0.1	0.314	-0.1	0.073	-0.8	0.149	-0.4	0.052	0.5
107	0.19	-0.3	0.077	-0.5	FN	-4.0	0.05	-0.5	0.32	0.0	0.041	-2.2	0.131	-0.8	0.059	1.2
108	0.178	-0.6	0.11	1.0	0.065	2.9	0.047	-0.7	0.254	-0.9	0.066	-1.1	0.191	0.7	0.067	1.8
109	0.244	0.7	0.092	0.2	FN	-4.0	0.128	5.0	0.348	0.3	0.084	-0.3	0.069	-2.3	FN	-4.0
110	0.217	0.2			0.0397	0.2	0.0836	1.9	0.0397	-3.5	0.0397	-2.2	0.0397	-3.0	0.0397	-0.5
111	0.232	0.5	0.066	-1.0			0.064	0.5	0.33	0.1	0.096	0.2				
112																
113	0.206	0.0	0.083	-0.2	0.041	0.3	0.07	0.9	0.342	0.2	0.097	0.3	0.191	0.7	0.049	0.3
114	0.195	-0.2	0.069	-0.9	0.041	0.3	0.048	-0.6	0.301	-0.3	0.084	-0.3	0.15	-0.3	0.037	-0.8
115	0.222	0.3	0.0821	-0.3			0.0445	-0.9	0.357	0.4	0.0917	0.1				
116	0.27	1.2	0.086	-0.1	0.031	-0.7	FN	-4.0	0.35	0.3	0.15	2.6	0.25	2.1	0.058	1.1
117	0.213	0.1	0.075	-0.6	0.033	-0.5	0.064	0.5	0.242	-1.0	0.071	-0.9	0.158	-0.1	0.045	-0.1
118																
119	0.1981	-0.2	0.062	-1.2	0.0218	-1.7	0.059	0.1	0.3279	0.1	0.094	0.2	0.1467	-0.4	0.0338	-1.0
120	0.198	-0.2	0.088	0.0	0.041	0.3	FN	-4.0	0.344	0.3	0.082	-0.4	0.161	-0.1	0.09	3.9
121	0.232	0.5					0.0179	-2.7	0.326	0.0	0.0676	-1.0				
122	0.243	0.7	0.0965	0.4			0.0526	-0.3	0.38	0.7	0.109	0.8				
123	0.234	0.5	0.097	0.4	0.033	-0.5	0.048	-0.6	0.303	-0.3	0.093	0.1	0.165	0.0	0.046	0.0
124	0.18	-0.5	0.104	0.7	0.041	0.3	0.064	0.5	0.334	0.1	0.094	0.2	0.176	0.3	0.044	-0.2
125	0.191	-0.3	0.084	-0.2	0.039	0.1	0.063	0.4	0.261	-0.8	0.104	0.6	0.18	0.4	0.047	0.1
126	0.15	-1.1	0.067	-1.0	0.036	-0.2	0.06	0.2	0.23	-1.2	0.072	-0.8	0.14	-0.6	0.045	-0.1
127																
128	0.176	-0.6	0.0739	-0.6	0.0317	-0.6	0.0724	1.1	0.264	-0.7	0.0771	-0.6	0.152	-0.3	0.0494	0.3
129	0.191	-0.3			0.038	0.0	0.071	1.0	0.226	-1.2	0.083	-0.3	0.133	-0.8	FN	-4.0
130	0.196	-0.2	0.077	-0.5	0.037	-0.1	0.058	0.1	0.295	-0.4	0.069	-0.9	0.152	-0.3	0.038	-0.7
131	0.223	0.3	0.102	0.6	0.0413	0.4	0.0606	0.2	0.334	0.1	0.0994	0.4	0.222	1.4	0.05	0.4
132			0.101	0.6							0.115	1.1	0.162	0.0		

Laboratory code	Azoxystrobin	Z-scores (FFP RSD (25%)	Bixafen	Z-scores (FFP RSD (25%)	Cyazofamid	Z-scores (FFP RSD (25%)	Cyfluthrin	Z-scores (FFP RSD (25%)	Cyprodinil	Z-scores (FFP RSD (25%)	Difenoconazole	Z-scores (FFP RSD (25%)	Dimethomorph	Z-scores (FFP RSD (25%)	Flonicamid	Z-scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.207**		**0.088**		**0.038**		**0.057**		**0.324**		**0.090**		**0.164**		**0.046**	
133	0.193	-0.3	0.102	0.6	0.037	-0.1	0.0673	0.7	0.325	0.0	0.0912	0.0	0.172	0.2	0.0449	-0.1
134	0.211	0.1	0.106	0.8	0.052	1.5	0.057	0.0	0.413	1.1	0.115	1.1	0.165	0.0	0.051	0.5
135	0.195	-0.2	FN	-4.0	0.04	0.2	0.064	0.5	0.453	1.6	0.107	0.7	0.145	-0.5	0.046	0.0
136	0.278	1.4	0.1	0.5	0.052	1.5	0.066	0.6	0.409	1.1	0.109	0.8	0.193	0.7	0.04	-0.5
137	0.241	0.7			0.06	2.4			0.362	0.5	0.089	-0.1	0.157	-0.2		
138	0.215	0.2	0.09	0.1	0.0503	1.3	0.0608	0.3	0.3485	0.3	0.0876	-0.1	0.1702	0.2	0.0504	0.4
139							0.061	0.3								
140	0.213	0.1	0.0874	0.0	0.0356	-0.2	0.048	-0.6	0.313	-0.1	0.0925	0.1	0.174	0.2	0.0437	-0.2
141	0.207	0.0	0.0916	0.2	0.0375	0.0	0.0671	0.7	0.348	0.3	0.098	0.3	0.169	0.1	0.0455	0.0
142	0.213	0.1			0.037	-0.1	0.03	-1.9	0.352	0.4	0.092	0.1	0.165	0.0	0.044	-0.2
143	0.186	-0.4	0.069	-0.9	0.029	-0.9	0.057	0.0	0.335	0.1	0.072	-0.8	0.127	-0.9	0.035	-0.9
144	0.209	0.0	0.107	0.9	0.0341	-0.4	0.054	-0.2	0.38	0.7	0.116	1.1	0.141	-0.6	0.0474	0.1
145	0.206	0.0	0.073	-0.7	0.037	-0.1	0.052	-0.4	0.29	-0.4	0.066	-1.1	0.163	0.0	0.036	-0.9
146	0.207	0.0			0.036	-0.2	0.063	0.4	0.362	0.5	0.098	0.3	0.155	-0.2	0.036	-0.9
147																
148	0.101	-2.0					0.031	-1.8	0.14	-2.3	0.042	-2.1	0.082	-2.0		
150	0.196	-0.2	0.086	-0.1					0.278	-0.6	0.08	-0.5			0.022	-2.1
151	0.181	-0.5	0.075	-0.6	0.032	-0.6	0.054	-0.2	0.215	-1.3	0.076	-0.6	0.14	-0.6	0.048	0.2
152	0.17	-0.7	0.096	0.4	0.016	-2.3	0.051	-0.4	0.35	0.3	0.078	-0.5	0.16	-0.1	0.065	1.7
153	0.21	0.1	0.068	-0.9	0.031	-0.7	0.048	-0.6	0.3	-0.3	0.082	-0.4	0.16	-0.1	0.044	-0.2
154	0.268	1.2					0.016	-2.9	0.284	-0.5	0.106	0.7	0.149	-0.4	0.039	-0.6
155	0.177	-0.6	0.09	0.1	0.039	0.1	0.072	1.0	0.299	-0.3	0.084	-0.3	0.177	0.3	0.05	0.4
156	0.226	0.4			0.043	0.6	0.058	0.1	0.231	-1.1	0.077	-0.6	0.122	-1.0	0.042	-0.3
157	0.15	-1.1			0.029	-0.9	0.052	-0.4	0.28	-0.5	0.091	0.0	0.139	-0.6	0.038	-0.7
158	0.205	0.0	0.0819	-0.3	0.0298	-0.8	0.0573	0.0	0.302	-0.3	0.0842	-0.3	0.147	-0.4	0.0437	-0.2
159	0.198	-0.2	0.085	-0.1	0.037	-0.1	0.032	-1.8	0.342	0.2	0.106	0.7	0.183	0.5	0.047	0.1
160	0.184	-0.4	0.094	0.3	0.031	-0.7	0.05	-0.5	0.285	-0.5	0.083	-0.3	0.148	-0.4	0.041	-0.4
161	0.233	0.5	0.108	0.9	0.036	-0.2	0.056	-0.1	0.547	2.8	0.095	0.2	0.184	0.5	0.048	0.2
162	0.242	0.7	0.0703	-0.8	0.0581	2.2	0.0718	1.0	0.299	-0.3	0.15	2.6	0.439	>5	0.0525	0.6
163	0.195	-0.2			0.1292	>5	0.0969	2.8	0.3263	0.0	0.0836	-0.3	0.1652	0.0	0.0501	0.4
164	0.23	0.4	0.084	-0.2	0.037	-0.1	0.018	-2.7	0.295	-0.4	0.099	0.4	0.118	-1.1	0.046	0.0
165																
166	0.177	-0.6	0.07	-0.8	0.018	-2.1	0.077	1.4	0.235	-1.1	0.07	-0.9	0.078	-2.1	0.026	-1.7
167	0.15	-1.1	0.064	-1.1	0.023	-1.6	0.051	-0.4	0.28	-0.5	0.066	-1.1	0.15	-0.3	0.035	-0.9
168	0.217	0.2	0.092	0.2	0.037	-0.1	0.078	1.5	0.38	0.7	0.099	0.4	0.186	0.5	0.045	-0.1
170	0.145	-1.2	FN	-4.0	0.026	-1.2	FN	-4.0	0.16	-2.0	0.04	-2.2	0.18	0.4	FN	-4.0

Table 9b. Results for the mandatory pesticides fluopyram, metconazole, pirimicarb, pirimicarb-desmethyl, proquinazid, prothioconazole-desthio, pyraclostrobin and pyriproxyfen, in mg/kg, the corresponding z scores, MRRLs and the assigned values.

Laboratory code	Fluopyram	Z-scores (FFP RSD (25%)	Metconazole	Z-scores (FFP RSD (25%)	Pirimicarb	Z-scores (FFP RSD (25%)	Pirimicarb-desmethyl	Z-scores (FFP RSD (25%)	Proquinazid	Z-scores (FFP RSD (25%)	Prothioconazole-desthio	Z-scores (FFP RSD (25%)	Pyraclostrobin	Z-scores (FFP RSD (25%)	Pyriproxyfen	Z-scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.134**		**0.069**		**0.033**		**0.053**		**0.085**		**0.161**		**0.097**		**0.052**	
3	0.144	0.3			0.0418	1.1	0.0598	0.5			0.193	0.8	0.0939	-0.1		
4	0.131	-0.1			0.033	0.0	0.051	-0.1	0.08	-0.2			0.072	-1.0	0.052	0.0
5					0.029	-0.5							0.101	0.2	0.049	-0.2
6	0.12	-0.4	0.052	-1.0	0.024	-1.1	0.045	-0.6	0.074	-0.5	0.143	-0.5	0.076	-0.9	0.045	-0.5
7	0.119	-0.5	0.0667	-0.2	0.0338	0.1	0.0495	-0.2	0.0883	0.2	0.172	0.3	0.0828	-0.6	0.0604	0.6
8	0.144	0.3	0.0775	0.5	0.0265	-0.8			0.089	0.2	0.178	0.4	0.106	0.4	0.0443	-0.6
9	0.146	0.4	0.089	1.1	0.041	1.0	0.043	-0.7	0.091	0.3	0.187	0.6	0.121	1.0	0.06	0.6
10	0.163	0.9	0.05	-1.1	0.037	0.5	0.065	0.9	0.101	0.8	0.167	0.1	0.091	-0.3	0.045	-0.5
11	0.107	-0.8	0.041	-1.6	0.03	-0.4	0.057	0.3	0.108	1.1	0.07	-2.3	0.074	-1.0	0.056	0.3
12	0.11	-0.7	0.06	-0.5	0.025	-1.0	0.055	0.2	0.07	-0.7	0.14	-0.5	0.093	-0.2	0.054	0.1
13	0.133	0.0	0.08	0.6	0.03	-0.4	0.055	0.2	0.079	-0.3	0.179	0.4	0.098	0.0	0.055	0.2
14	0.115	-0.6	0.051	-1.1	0.027	-0.7	0.049	-0.3	0.044	-1.9	0.13	-0.8	0.086	-0.5	0.036	-1.2
15	0.133	0.0	0.0683	-0.1	0.0394	0.8			0.0834	-0.1	0.153	-0.2	0.112	0.6	0.053	0.1
16	0.185	1.5			0.185	>5							0.185	3.6	0.185	>5
17	0.122	-0.4	0.0621	-0.4	0.0317	-0.2	0.0557	0.2	0.0753	-0.4	0.145	-0.4	0.0801	-0.7	0.0469	-0.4
18	0.093	-1.2									0.131	-0.7			0.048	-0.3
19					0.034	0.1							0.09	-0.3	0.046	-0.5
20	0.147	0.4	0.0891	1.1	0.0377	0.6	0.0595	0.5	0.102	0.8	0.182	0.5	0.114	0.7	0.0642	0.9
21	0.104	-0.9	0.049	-1.2	0.029	-0.5	0.062	0.7	0.086	0.1	0.132	-0.7	0.079	-0.7	0.047	-0.4
22	0.101	-1.0	0.0485	-1.2	0.0275	-0.7	0.0415	-0.8	0.077	-0.4	0.1372	-0.6	0.0788	-0.8	0.0468	-0.4
23	0.15	0.5	0.068	-0.1	0.037	0.5	0.053	0.0	0.079	-0.3	0.169	0.2	0.098	0.0	0.058	0.5
24	0.095	-1.2	0.057	-0.7	0.026	-0.8	0.043	-0.7	0.047	-1.8	0.132	-0.7	0.103	0.2	0.06	0.6
25																
26	0.148	0.4	0.061	-0.5	0.0325	-0.1	0.046	-0.5	0.116	1.5	0.179	0.4	0.0951	-0.1	0.0612	0.7
27	0.107	-0.8	0.0764	0.4	0.0325	-0.1	0.0507	-0.1	0.082	-0.1	0.181	0.5	0.11	0.5	0.0504	-0.1
28	0.17	1.1	0.08	0.6	0.029	-0.5	0.042	-0.8	0.074	-0.5	0.17	0.2	0.11	0.5	0.048	-0.3
29	0.136	0.1	0.0633	-0.4	0.0326	0.0	0.0477	-0.4	0.0784	-0.3	0.174	0.3	0.0924	-0.2	0.046	-0.5
30	0.128	-0.2	0.067	-0.14	0.032	-0.1	0.053	0.0	0.078	-0.3	FN	-4.0	0.076	-0.9	0.038	-1.1
31	0.15	0.5	0.072	0.1	0.04	0.8	0.052	0.0	0.091	0.3	0.21	1.2	0.11	0.5	0.046	-0.5
33	0.106	-0.8	0.0512	-1.0	0.0316	-0.2	0.0523	0.0	0.0833	-0.1	0.145	-0.4	0.0806	-0.7	0.0508	-0.1
34	0.13	-0.1	0.072	0.1	0.033	0.0	0.064	0.9	0.076	-0.4	0.13	-0.8	0.09	-0.3	0.054	0.1
35	0.162	0.8	0.0718	0.1	0.0362	0.4	0.0632	0.8	0.104	0.9	0.184	0.6	0.114	0.7	0.0615	0.7
36																
37	0.13	-0.1	0.069	0.0	0.03	-0.4	0.048	-0.4	0.074	-0.5	0.14	-0.5	0.082	-0.6	0.045	-0.5
38			0.291	>5	0.058	3.0							0.053	-1.8	0.033	-1.5
39	0.125	-0.3			0.024	-1.1			0.081	-0.2	0.156	-0.1	0.098	0.0	0.048	-0.3
40	0.356	>5	0.27	>5	0.038	0.6	0.057	0.3	0.227	>5	0.325	4.1	0.07	-1.1	0.055	0.2
41	0.135	0.0	0.067	-0.1	0.032	-0.1	0.053	0.0	0.059	-1.2	0.148	-0.3	0.101	0.2	0.049	-0.2
42	0.129	-0.2	0.076	0.4	0.034	0.1	0.061	0.6	0.095	0.5	0.153	-0.2	0.1	0.1	0.056	0.3
43			0.107	2.2	0.076	>5			0.096	0.5			0.119	0.9		

Laboratory code	Fluopyram	Z-scores (FFP RSD (25%)	Metconazole	Z-scores (FFP RSD (25%)	Pirimicarb	Z-scores (FFP RSD (25%)	Pirimicarb-desmethyl	Z-scores (FFP RSD (25%)	Proquinazid	Z-scores (FFP RSD (25%)	Prothioconazole-desthio	Z-scores (FFP RSD (25%)	Pyraclostrobin	Z-scores (FFP RSD (25%)	Pyriproxyfen	Z-scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.134**		**0.069**		**0.033**		**0.053**		**0.085**		**0.161**		**0.097**		**0.052**	
44	0.151	0.5	0.079	0.6	0.0352	0.3					0.165	0.1				
45	0.132	-0.1	0.071	0.1	0.0322	-0.1	0.054	0.1	0.0853	0.0	0.162	0.0	0.0791	-0.7	0.0552	0.2
46	0.107	-0.8	0.089	1.13	0.028	-0.6			0.075	-0.5			0.107	0.4	0.073	1.6
47	0.178	1.3	0.053	-0.9	0.043	1.2	0.063	0.8	0.126	2.0	0.238	1.9	0.139	1.7	0.065	1.0
48	0.128	-0.2	0.06	-0.5	0.028	-0.6	0.061	0.6	0.056	-1.4	FN	-4.0	0.083	-0.6	0.034	-1.4
49	0.119	-0.5	0.0551	-0.8	0.0285	-0.5	0.0417	-0.8	0.0664	-0.9	0.133	-0.7	0.0912	-0.2	0.0477	-0.3
50	0.125	-0.3	0.067	-0.1	0.03	-0.4	FN	-4.0	0.097	0.6	0.189	0.7	0.096	0.0	0.056	0.3
51	0.14	0.2	0.08	0.6	0.032	-0.1	0.052	0.0	0.085	0.0	0.166	0.1	0.1	0.1	0.054	0.1
52	0.04	-2.8	0.027	-2.4	0.021	-1.5	0.028	-1.9	0.034	-2.4	0.06	-2.5	0.042	-2.3	0.031	-1.6
53	0.1	-1.0	0.092	1.3	0.045	1.5							0.097	0.0	0.054	0.1
54	0.153	0.6	0.0876	1.0	0.0362	0.4	0.0623	0.7	0.104	0.9	0.187	0.6	0.107	0.4	0.0623	0.8
55	0.14	0.2	0.0574	-0.7	0.0326	0.0	0.0569	0.3	0.0887	0.2	0.164	0.1	0.111	0.6	0.054	0.1
56	0.123	-0.3	0.066	-0.2	0.03	-0.4	0.05	-0.2	0.085	0.0	0.16	0.0	0.088	-0.4	0.045	-0.5
57																
58	0.193	1.8	0.099	1.7	0.04	0.8	0.051	-0.1			0.213	1.3	0.119	0.9	0.111	4.5
59	0.15	0.5	0.082	0.7	0.04	0.8	0.034	-1.4			0.225	1.6	0.126	1.2	0.055	0.2
60	0.159	0.7	0.098	1.6	0.042	1.1	0.073	1.5	0.122	1.8	0.208	1.2	0.123	1.1	0.071	1.5
61	0.15	0.5	0.07	0.0	0.02	-1.6	0.05	-0.2	0.09	0.3	FN	-4.0	0.09	-0.3	0.06	0.6
62	0.144	0.3	0.0738	0.3	0.0387	0.7	0.0537	0.1	0.129	2.1	0.16	0.0	0.117	0.8	0.0575	0.4
63	0.213	2.4	0.0718	0.1	0.0445	1.4	0.0628	0.8	0.0929	0.4	0.167	0.1	0.1277	1.3	0.0498	-0.2
64			FN	-4.0	0.027	-0.7					FN	-4.0	0.295	>5	FN	-4.0
65	0.314	>5	0.07	0.0	0.034	0.1	0.029	-1.8	0.064	-1.0	0.262	2.5	FN	-4.0	0.062	0.8
66	0.1171	-0.5	0.0666	-0.2	0.0627	3.6	0.052	0.0	0.314	>5	0.1356	-0.6	0.0926	-0.2	0.0501	-0.2
67	0.156	0.7	0.08	0.6	0.041	1.0	0.062	0.7	0.091	0.3	0.194	0.8	0.118	0.9	0.06	0.6
68																
69	0.145	0.3	0.082	0.7	0.035	0.2	0.053	0.0	0.109	1.1			0.14	1.8	0.063	0.8
71	0.16	0.8	0.0794	0.6	0.035	0.2					0.164	0.1				
72	0.119	-0.5	0.0632	-0.4	0.0289	-0.5			0.0723	-0.6	0.159	-0.1	0.0887	-0.3	0.0543	0.2
73	0.146	0.4	0.071	0.1	0.031	-0.2	0.051	-0.1	0.089	0.2	0.172	0.3	0.111	0.6	0.038	-1.1
74	0.15	0.5	0.068	-0.1	0.033	0.0	0.042	-0.8	0.085	0.0	0.17	0.2	0.114	0.7	0.045	-0.5
75	0.109	-0.7	0.058	-0.7	0.025	-1.0	0.043	-0.7	0.074	-0.5	0.129	-0.8	0.058	-1.6	0.037	-1.2
76	0.146	0.4	0.066	-0.2	0.035	0.2	0.045	-0.6	0.097	0.6	0.195	0.8	0.108	0.5	0.06	0.6
77	0.156	0.7	0.0795	0.6	0.036	0.4	0.0528	0.0	0.0981	0.6	0.191	0.7	0.104	0.3	0.0497	-0.2
78	0.145	0.3			0.039	0.7	0.055	0.2			0.176	0.4	0.095	-0.1	0.05	-0.2
79	0.134	0.0	0.076	0.4	0.033	0.0	0.054	0.1	0.105	1.0	0.182	0.5	0.102	0.2	0.06	0.6
80	0.172	1.1	0.107	2.2	0.0449	1.4	0.0709	1.4	0.134	2.3	0.219	1.4	0.139	1.7	0.0603	0.6
81	0.116	-0.5	0.058	-0.7	0.031	-0.2	0.052	0.0	0.085	0.0	0.154	-0.2	0.085	-0.5	0.051	-0.1
82	0.111	-0.7	0.082	0.7	0.035	0.2	FN	-4.0	0.079	-0.3	0.144	-0.4	0.098	0.0	0.048	-0.3
84	0.12	-0.4	0.066	-0.2	0.028	-0.6	0.055	0.2	0.075	-0.5	0.165	0.1	0.095	-0.1	0.051	-0.1
85	0.149	0.4	0.056	-0.8	0.034	0.1	0.049	-0.3	0.088	0.2	0.157	-0.1	0.099	0.1	0.048	-0.3
86	0.151	0.5	0.062	-0.4	0.038	0.6	0.061	0.6	0.099	0.7	0.15	-0.3	0.119	0.9	0.075	1.8
87	0.125	-0.3	0.0728	0.2	0.0319	-0.1	0.0502	-0.2	0.0819	-0.1	0.158	-0.1	0.0945	-0.1	0.0563	0.3
88	0.281	4.4	0.058	-0.7	0.035	0.2	0.07	1.3	0.096	0.5	0.149	-0.3	0.115	0.7	0.045	-0.5

Laboratory code	Fluopyram	Z-scores (FFP RSD (25%)	Metconazole	Z-scores (FFP RSD (25%)	Pirimicarb	Z-scores (FFP RSD (25%)	Pirimicarb-desmethyl	Z-scores (FFP RSD (25%)	Proquinazid	Z-scores (FFP RSD (25%)	Prothioconazole-desthio	Z-scores (FFP RSD (25%)	Pyraclostrobin	Z-scores (FFP RSD (25%)	Pyriproxyfen	Z-scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.134**		**0.069**		**0.033**		**0.053**		**0.085**		**0.161**		**0.097**		**0.052**	
89	0.154	0.6	0.089	1.1	0.038	0.6	0.059	0.5	0.11	1.2	0.166	0.1	0.118	0.9	0.057	0.4
90	0.134	0.0	0.075	0.3	0.0313	-0.2			0.113	1.3	0.152	-0.2	0.102	0.2	0.0583	0.5
91	0.133	0.0	0.058	-0.7	0.03	-0.4	0.045	-0.6	0.053	-1.5	0.146	-0.4	0.079	-0.7	0.052	0.0
92																
93																
94	0.114	-0.6	0.053	-0.9	0.03	-0.4			0.076	-0.4			0.098	0.0	0.038	-1.1
95	0.11	-0.7	0.06	-0.5	0.03	-0.4	0.05	-0.2			0.15	-0.3	0.09	-0.3	0.04	-0.9
96	0.139	0.1			0.036	0.4							0.157	2.5	0.047	-0.4
97	0.14	0.2	0.06	-0.5	0.034	0.1	0.055	0.2	0.076	-0.4	0.168	0.2	0.098	0.0	0.052	0.0
98	0.114	-0.6			0.029	-0.5			0.073	-0.6			0.08	-0.7	0.063	0.8
99	0.142	0.2	0.074	0.3	0.036	0.4	0.052	0.0	0.089	0.2	0.163	0.0	0.094	-0.1	0.048	-0.3
100																
101	0.108	-0.8	0.0579	-0.7	0.0247	-1.0	0.0551	0.2	0.0422	-2.0	0.133	-0.7	0.064	-1.4	0.0464	-0.4
103	0.12	-0.4	FN	-4.0	FN	-4.0			0.089	0.2			FN	-4.0		
104					0.027	-0.7										
105	0.141	0.2	0.067	-0.1	0.033	0.0	0.06	0.6	0.096	0.5	0.186	0.6	0.095	-0.1	0.047	-0.4
106	0.146	0.4	0.069	-0.02	0.03	-0.4	0.054	0.1	0.064	-1.0	0.156	-0.1	0.1	0.1	0.045	-0.5
107	0.12	-0.4	0.069	0.0	0.045	1.5	0.057	0.3	0.055	-1.4	0.141	-0.5	0.099	0.1	0.046	-0.5
108	0.117	-0.5	0.071	0.1	0.024	-1.1	0.049	-0.3	0.052	-1.5	0.148	-0.3	0.106	0.4	0.045	-0.5
109	0.988	>5			0.675	>5	FN	-4.0			0.039	-3.0	FN	-4.0	0.332	>5
110	0.0397	-2.8	0.0397	-1.7	0.0397	0.8	0.217	>5	0.217	>5	0.217	1.4	0.217	4.9	0.0397	-1.0
111			0.07	0.0	0.031	-0.2					0.185	0.6	0.112	0.6	0.046	-0.5
112																
113	0.146	0.4	0.068	-0.1	0.032	-0.1	0.047	-0.4	0.076	-0.4	0.152	-0.2	0.1	0.1	0.049	-0.2
114	0.101	-1.0	0.054	-0.9	0.028	-0.6	0.042	-0.8	0.076	-0.4	0.123	-0.9	0.071	-1.1	0.056	0.3
115	0.14	0.2	0.0808	0.7	0.0306	-0.3					0.15	-0.3				
116	0.17	1.1	0.11	2.3	0.041	1.0	0.049	-0.3	0.13	2.1	0.17	0.2	0.13	1.4	0.058	0.5
117	0.128	-0.2	0.054	-0.9	0.027	-0.7			0.055	-1.4	0.145	-0.4	0.104	0.3	0.045	-0.5
118																
119	0.1479	0.4	0.0643	-0.3	0.0312	-0.2	0.057	0.3	0.0947	0.5	0.1552	-0.1	0.1006	0.1	0.0492	-0.2
120	0.16	0.8	0.064	-0.3	0.031	-0.2	0.055	0.2	0.083	-0.1	0.112	-1.2	0.097	0.0	0.053	0.1
121			0.0812	0.7	0.044	1.3										
122	0.153	0.6	0.0858	0.9	0.0328	0.0					0.16	0.0				
123	0.135	0.0	0.073	0.2	0.035	0.2	0.047	-0.4	0.081	-0.2	0.167	0.1	0.099	0.1	0.047	-0.4
124	0.089	-1.3	0.07	0.0	0.032	-0.1	0.05	-0.2	0.076	-0.4	0.229	1.7	0.077	-0.8	0.053	0.1
125	0.127	-0.2	0.073	0.2	0.031	-0.2	0.05	-0.2	0.074	-0.5	0.163	0.0	0.101	0.2	0.053	0.1
126	0.088	-1.4	0.055	-0.8	0.018	-1.8	0.055	0.2	0.062	-1.1	0.13	-0.8	0.067	-1.2	0.055	0.2
127																
128	0.105	-0.9	0.0601	-0.5	0.024	-1.1	0.0507	-0.1	0.07	-0.7	0.146	-0.4	0.0704	-1.1	0.0526	0.0
129	0.112	-0.7	0.074	0.3	0.023	-1.2	0.045	-0.6	0.064	-1.0	0.141	-0.5	0.086	-0.5	0.04	-0.9
130	0.125	-0.3	0.066	-0.2	0.031	-0.2	0.056	0.3	0.07	-0.7	0.139	-0.6	0.093	-0.2	0.044	-0.6
131	0.149	0.4	0.087	1.0	0.0313	-0.2	0.0516	-0.1	0.102	0.8	0.194	0.8	0.0895	-0.3	0.0499	-0.2
132	0.159	0.7			0.0308	-0.3	0.0486	-0.3					0.102	0.2	0.0547	0.2

Laboratory code	Fluopyram	Z-scores (FFP RSD (25%)	Metconazole	Z-scores (FFP RSD (25%)	Pirimicarb	Z-scores (FFP RSD (25%)	Pirimicarb-desmethyl	Z-scores (FFP RSD (25%)	Proquinazid	Z-scores (FFP RSD (25%)	Prothioconazole-desthio	Z-scores (FFP RSD (25%)	Pyraclostrobin	Z-scores (FFP RSD (25%)	Pyriproxyfen	Z-scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.134**		**0.069**		**0.033**		**0.053**		**0.085**		**0.161**		**0.097**		**0.052**	
133	0.147	0.4	0.0793	0.6	0.03	-0.4	0.0524	0.0	0.103	0.9	0.143	-0.5	0.0931	-0.2	0.062	0.8
134	0.144	0.3	0.095	1.5	0.042	1.1	0.058	0.4	0.092	0.3	0.219	1.4	0.113	0.7	0.059	0.5
135	0.124	-0.3	0.068	-0.1	0.036	0.4	FN	-4.0	0.074	-0.5	0.128	-0.8	0.104	0.3	0.056	0.3
136	0.169	1.0	0.076	0.4	0.039	0.7	0.054	0.1	0.073	-0.6	0.197	0.9	0.106	0.4	0.06	0.6
137	0.102	-1.0	0.094	1.4	0.031	-0.2							0.042	-2.3	0.06	0.6
138	0.1438	0.3	0.07	0.0	0.0334	0.0	0.0569	0.3	0.0817	-0.1	0.1778	0.4	0.0982	0.0	0.0615	0.7
139																
140	0.133	0.0	0.0706	0.1	0.0323	-0.1	0.0534	0.1	0.0967	0.6	0.154	-0.2	0.0982	0.0	0.0404	-0.9
141	0.144	0.3	0.0717	0.1	0.0329	0.0	0.0529	0.0	0.0899	0.2	0.17	0.2	0.107	0.4	0.0552	0.2
142	0.138	0.1	0.069	0.0	0.034	0.1	0.05	-0.2	0.076	-0.4	0.156	-0.1	0.104	0.3	0.052	0.0
143	0.124	-0.3	0.057	-0.7	0.029	-0.5	0.048	-0.4	0.073	-0.6	0.131	-0.7	0.085	-0.5	0.051	-0.1
144	0.145	0.3	0.0757	0.4	0.0367	0.4	0.0519	-0.1	0.0837	0.0	0.201	1.0	0.119	0.9	0.0603	0.6
145	0.126	-0.2	0.069	0.0	0.028	-0.6	0.053	0.0	0.106	1.0	0.139	-0.6	0.119	0.9	0.064	0.9
146	0.148	0.4	0.062	-0.4	0.033	0.0	0.051	-0.1	0.099	0.7	0.157	-0.1	0.104	0.3	0.054	0.1
147																
148	0.049	-2.5	0.035	-2.0	0.013	-2.4	0.026	-2.0			0.059	-2.5	0.049	-2.0	0.02	-2.5
150	0.13	-0.1			0.041	1.0					FN	-4.0	0.08	-0.7	0.045	-0.5
151	0.111	-0.7	0.052	-1.0	0.022	-1.3	0.047	-0.4	0.066	-0.9	0.126	-0.9	0.079	-0.7	0.044	-0.6
152	0.12	-0.4			0.028	-0.6	0.043	-0.7	0.065	-0.9	0.18	0.5	0.12	0.9	0.037	-1.2
153	0.11	-0.7	0.053	-0.9	0.029	-0.5	0.044	-0.7	0.076	-0.4	0.15	-0.3	0.084	-0.5	0.054	0.1
154	0.163	0.9	0.098	1.6	0.018	-1.8							FN	-4.0	0.043	-0.7
155	0.124	-0.3	0.065	-0.3	0.034	0.1	0.065	0.9	0.065	-0.9	0.173	0.3	0.088	-0.4	0.052	0.0
156			0.073	0.2	0.026	-0.8			0.065	-0.9			0.084	-0.5	0.045	-0.5
157	0.108	-0.8	0.073	0.2	0.029	-0.5	0.042	-0.8	0.076	-0.4	0.114	-1.2	0.084	-0.5	0.046	-0.5
158	0.122	-0.4	0.0671	-0.1	0.0251	-1.0	0.0471	-0.4	0.0751	-0.5	0.148	-0.3	0.081	-0.7	0.0435	-0.7
159	0.136	0.1	0.084	0.8	0.036	0.4	0.055	0.2	0.081	-0.2	0.183	0.5	0.103	0.2	0.062	0.8
160	0.124	-0.3	0.068	-0.1	0.03	-0.4	0.042	-0.8	0.097	0.6	0.156	-0.1	0.091	-0.3	0.044	-0.6
161	0.141	0.2	0.091	1.2	0.036	0.4	0.049	-0.3	0.124	1.9	0.194	0.8	0.12	0.9	0.071	1.5
162	0.177	1.3	0.196	>5	0.0221	-1.3	0.0283	-1.9	0.134	2.3	0.319	3.9	0.107	0.4	0.0414	-0.8
163	0.1047	-0.9	0.0639	-0.3	0.0341	0.1							0.0818	-0.6	0.0532	0.1
164	0.124	-0.3	0.061	-0.5	0.043	1.2	0.059	0.5	0.098	0.6	0.076	-2.1	0.095	-0.1	0.052	0.0
165																
166	0.09	-1.3	0.062	-0.4	0.022	-1.3	0.053	0.0	0.081	-0.2	0.12	-1.0	0.071	-1.1	0.061	0.7
167	0.098	-1.1	0.053	-0.9	0.032	-0.1	0.043	-0.7	0.059	-1.2	0.14	-0.5	0.064	-1.4	0.04	-0.9
168	0.155	0.6	0.076	0.4	0.031	-0.2	0.065	0.9	0.103	0.9	0.178	0.4	0.104	0.3	0.065	1.0
170	FN	-4.0	0.034	-2.0	0.017	-1.9	FN	-4.0	0.04	-2.1	FN	-4.0	FN	-4.0	FN	-4.0

Table 9c. Results for trifloxystrobin and for the voluntary compounds, clomazone, HCH-beta and phenmedipham in mg/kg, and the corresponding z scores, MRRLs and the assigned values.

Laboratory code	Trifloxystrobin	Z-scores (FFP RSD (25%)	Clomazone	Z-scores (FFP RSD (25%)	HCH-beta	Z-scores (FFP RSD (25%)	Phenmedipham	Z-scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.049**		**0.035**		**0.041**		**0.057**	
3	0.0491	0.0			0.0457	0.4		
4	0.046	-0.3	0.036	0.2	0.035	-0.6		
5	0.047	-0.2						
6	0.047	-0.2	0.031	-0.4	0.037	-0.4	0.039	-1.3
7	0.0369	-1.0	0.0368	0.2	0.0384	-0.3	0.0607	0.3
8	0.0479	-0.1	0.0332	-0.2	0.0403	-0.1	0.056	-0.1
9	0.056	0.5	0.036	0.2	0.042	0.1	0.038	-1.3
10	0.066	1.3	0.035	0.0	0.0501	0.8		
11	0.033	-1.3	0.025	-1.1	FN	-4.0		
12	0.047	-0.2	0.032	-0.3	0.032	-0.9	0.045	-0.8
13	0.055	0.5	0.038	0.4	0.046	0.4	0.056	-0.1
14	0.033	-1.3						
15	0.0538	0.4	0.0544	2.3				
16	0.185	>5			0.185	>5		
17	0.0494	0.0	0.0319	-0.3	0.0342	-0.7	0.0415	-1.1
18					0.04	-0.1		
19	0.047	-0.2	0.035	0.0	0.043	0.2		
20	0.0655	1.3	0.0314	-0.4	0.0402	-0.1	0.0539	-0.2
21	0.039	-0.8						
22	0.039	-0.8	0.0292	-0.6	0.0392	-0.2		
23	0.05	0.0	0.035	0.0	0.045	0.3		
24	0.043	-0.5	0.026	-1.0	0.126	>5		
25					0.0367	-0.5		
26	0.0445	-0.4	0.0374	0.3	0.046	0.4	0.0506	-0.5
27	0.0626	1.1	0.0336	-0.1	0.0668	2.5	0.0573	0.0
28	0.051	0.1	0.033	-0.2	0.023	-1.8	0.061	0.3
29	0.0469	-0.2	0.0338	-0.1	0.0366	-0.5	0.0469	-0.7
30	0.047	-0.2			0.064	2.2		
31	0.055	0.5	0.042	0.8	0.04	-0.1		
33	0.0415	-0.6	0.0299	-0.5	0.0467	0.5	0.0473	-0.7
34	0.053	0.3			0.031	-1.0		
35	0.0557	0.5	0.0392	0.5	0.0522	1.0	0.0651	0.6
36								
37	0.044	-0.4	0.031	-0.4	0.036	-0.5	0.059	0.1
38	0.023	-2.1			0.035	-0.6	0.023	-2.4
39	0.042	-0.6			0.039	-0.2		
40	0.059	0.8	0.027	-0.9	0.053	1.1	0.065	0.6
41	0.05	0.0	0.036	0.2	0.023	-1.8	0.076	1.3
42	0.058	0.7	0.038	0.4	0.04	-0.1	0.049	-0.6
43	0.076	2.2	0.114	>5	0.046	0.4		

Laboratory code	Trifloxystrobin	Z-scores (FFP RSD (25%)	Clomazone	Z-scores (FFP RSD (25%)	HCH-beta	Z-scores (FFP RSD (25%)	Phenmedipham	Z-scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.049**		**0.035**		**0.041**		**0.057**	
44	0.0542	0.4			0.0388	-0.3		
45	0.046	-0.3	0.0351	0.1	0.0457	0.4	0.0467	-0.7
46	0.067	1.4	0.034	-0.1	0.042	0.1	0.109	3.6
47	0.072	1.8	0.0455	1.3	0.051	0.9		
48	0.042	-0.6						
49	0.0418	-0.6	0.0284	-0.7	0.0402	-0.1		
50	0.05	0.0						
51	0.044	-0.4	0.034	-0.1	0.045	0.3	0.052	-0.4
52	0.021	-2.3	0.013	-2.5	0.024	-1.7	0.021	-2.5
53	0.047	-0.2	FN	-4.0				
54	0.0511	0.1			0.0363	-0.5	0.0906	2.3
55	0.0512	0.1	0.0339	-0.1	0.0512	0.9	0.0541	-0.2
56	0.045	-0.4	0.045	1.2	0.05	0.8		
57					0.0459	0.4		
58	0.052	0.2	0.047	1.4	0.078	3.5		
59	0.07	1.7						
60	0.063	1.1	0.042	0.8	0.063	2.1	0.066	0.6
61	0.04	-0.8	0.03	-0.5	0.04	-0.1	0.24	>5
62	0.0583	0.7	0.0391	0.5	0.0577	1.6	0.0546	-0.2
63	0.0522	0.2	0.0464	1.4	0.058	1.6	0.0648	0.5
64	FN	-4.0	FN	-4.0	0.041	0.0		
65	0.036	-1.1	0.03	-0.5	0.037	-0.4	0.038	-1.3
66	0.043	-0.5	0.0349	0.0	0.0363	-0.5	0.0383	-1.3
67	0.061	0.9	0.043	1.0	0.047	0.5	0.052	-0.4
68								
69	0.061	0.9	0.0184	-1.9				
71	0.0535	0.3			0.0398	-0.2		
72	0.0451	-0.3	0.0332	-0.2	0.035	-0.6	0.0451	-0.8
73	0.041	-0.7	0.04	0.6	0.037	-0.4	0.07	0.9
74	0.048	-0.1	0.033	-0.2	0.037	-0.4	0.061	0.3
75	0.04	-0.8	0.024	-1.2	0.035	-0.6	0.041	-1.1
76	0.057	0.6	0.038	0.4	0.04	-0.1	0.053	-0.3
77	0.0544	0.4	0.038	0.4	0.0458	0.4	0.0615	0.3
78	0.046	-0.3						
79	0.044	-0.4	0.035	0.0	0.035	-0.6	0.046	-0.8
80	0.0709	1.7	0.0538	2.2	0.0704	2.8	0.0726	1.1
81	0.046	-0.3	0.036	0.2	0.045	0.3	0.072	1.0
82	0.051	0.1	0.031	-0.4	0.038	-0.3	0.08	1.6
84	0.053	0.3	0.035	0.0	0.033	-0.8	0.053	-0.3
85	0.039	-0.8						
86	0.088	3.1			0.041	0.0	0.07	0.9
87	0.0586	0.7	0.0299	-0.5	0.0378	-0.3		
88	0.042	-0.6	0.033	-0.2	0.034	-0.7	0.032	-1.8

Laboratory code	Trifloxystrobin	Z-scores (FFP RSD (25%)	Clomazone	Z-scores (FFP RSD (25%)	HCH-beta	Z-scores (FFP RSD (25%)	Phenmedipham	Z-scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.049**		**0.035**		**0.041**		**0.057**	
89	0.063	1.1	0.044	1.1	0.044	0.3		
90	0.0523	0.2	0.0357	0.1	0.046	0.4	0.0455	-0.8
91	0.047	-0.2	0.032	-0.3	0.042	0.1	0.079	1.5
92					0.03	-1.1		
93					0.0385	-0.3		
94	0.05	0.0	0.03	-0.5	FN	-4.0	0.05	-0.5
95	0.06	0.9			0.04	-0.1		
96	0.048	-0.1						
97	0.046	-0.3	0.032	-0.3	0.041	0.0	0.061	0.3
98	0.044	-0.4			0.038	-0.3		
99	0.053	0.3	0.031	-0.4	0.036	-0.5	0.065	0.6
100					0.042	0.1		
101	FN	-4.0						
103	FN	-4.0	0.034	-0.1	FN	-4.0		
104					FN	-4.0		
105	0.048	-0.1	0.037	0.3	0.049	0.7	0.055	-0.1
106	0.066	1.3	0.034	-0.1	0.037	-0.4		
107	0.042	-0.6						
108	0.035	-1.2	0.04	0.6	0.054	1.2	0.069	0.8
109	0.24	>5			0.047	0.5		
110	0.0397	-0.8	0.0397	0.6				
111	0.056	0.5	0.033	-0.2	0.044	0.3	0.033	-1.7
112								
113	0.04	-0.8	0.035	0.0	0.041	0.0	0.05	-0.5
114	0.055	0.5	0.03	-0.5	0.038	-0.3		
115	0.0502	0.1			0.0393	-0.2		
116	0.069	1.6						
117	0.041	-0.7	0.027	-0.9			0.048	-0.6
118								
119	0.054	0.4	0.0376	0.3	0.045	0.3	0.197	>5
120	0.053	0.3						
121					0.0363	-0.5		
122	0.058	0.7			0.0391	-0.2		
123	0.053	0.3	0.041	0.7				
124	0.06	0.9	0.03	-0.5	0.028	-1.3	0.059	0.1
125	0.04	-0.8	0.035	0.0	0.041	0.0	0.059	0.1
126	0.051	0.1	0.032	-0.3	0.032	-0.9		
127								
128	0.0439	-0.4						
129	0.042	-0.6			0.033	-0.8	0.088	2.2
130	0.049	0.0	0.032	-0.3	0.034	-0.7	0.052	-0.4
131	0.0501	0.1	0.033	-0.2	0.0407	-0.1	0.0588	0.1
132	0.0476	-0.1						

Laboratory code	Trifloxystrobin	Z-scores (FFP RSD (25%)	Clomazone	Z-scores (FFP RSD (25%)	HCH-beta	Z-scores (FFP RSD (25%)	Phenmedipham	Z-scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.049**		**0.035**		**0.041**		**0.057**	
133	0.0467	-0.2	0.0354	0.1	0.0389	-0.2	0.0522	-0.3
134	0.052	0.2	0.036	0.2			0.052	-0.4
135	0.059	0.8	0.038	0.4	0.05	0.8		
136	0.066	1.3	0.039	0.5			0.047	-0.7
137	0.081	2.6			0.048	0.6	0.17	>5
138	0.0539	0.4	0.0366	0.2	0.0482	0.7	0.057	0.0
139					0.047	0.5		
140	0.0513	0.2			0.0442	0.3		
141	0.0506	0.1	0.0371	0.3	0.0403	-0.1		
142	0.051	0.1	0.035	0.0	0.045	0.3	0.069	0.8
143	0.044	-0.4	0.031	-0.4	0.047	0.5	0.09	2.3
144	0.0563	0.6	0.0404	0.7	0.037	-0.4	0.147	>5
145	0.049	0.0	0.033	-0.2	0.022	-1.9		
146	0.054	0.4	0.035	0.0	0.055	1.3		
147								
148	0.024	-2.1						
150	0.045	-0.4						
151	0.029	-1.7	0.027	-0.9	0.039	-0.2		
152	0.07	1.7						
153	0.04	-0.8	0.032	-0.3	0.041	0.0	0.059	0.1
154	0.045	-0.4			0.032	-0.9		
155	0.05	0.0	0.035	0.0	0.073	3.1	0.069	0.8
156	0.059	0.8	0.03	-0.5	0.041	0.0		
157	0.051	0.1	0.042	0.8	0.052	1.0	0.06	0.2
158	0.0459	-0.3	0.0263	-1.0	0.0361	-0.5	0.0486	-0.6
159	0.049	0.0			0.04	-0.1		
160	0.038	-0.9						
161	0.036	-1.1	0.04	0.6	0.045	0.3	0.051	-0.4
162	0.0522	0.2	0.0444	1.1	0.0447	0.3	0.0625	0.4
163	0.0446	-0.4	0.0335	-0.1	0.0464	0.5		
164	0.049	0.0	0.036	0.2	0.027	-1.4		
165								
166	0.04	-0.8						
167	0.042	-0.6						
168	0.051	0.1						
170	0.022	-2.2						

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

To be classified into Category A, the laboratories had to submit quantitative results for at least 90% of the compulsory pesticides present in the Test Item (≥15 pesticide residues, exclusive of any false negatives results), analyse for more than 90% of the compulsory pesticides on the target list and also report no false positive results. For the 143 EU and EFTA laboratories in Category A (73%), the results were additionally evaluated by calculating the Average of the Squared -Score (AZ^2). Of the 104 participants 92 participants (88%) obtained AZ2 score at or below 2 (good), 6 participants (6%) obtained AZ2 values between 2-3 (satisfactory) and 6 participants (6%) obtained AZ2 values ≥3 (unsatisfactory). An additional four laboratories from Third Countries were evaluated and classified into Category A. The AZ2 scores achieved by the labs can be seen in **Table 10**.

Table 10. Sum of Weighted z scores (AZ^2) for laboratories in Category A, the number of pesticides detected and quantified by the laboratories, the number of false negatives reported and the classification as good, satisfactory and unsatisfactory. The table includes data for both EU and non-EU participants.

Lab code	No. of detected mandatory pesticides	Mandatory pesticides detected %	No. of detected voluntary pesticides	AZ2	False negative	Classification	NRL
6	17	100%	3	0.5	0	Good	
7	17	100%	3	0.2	0	Good	
8	16	94%	3	0.2	0	Good	NRL-CE
9	17	100%	3	0.4	0	Good	
10	16	94%	2	1.4	1	Good	
12	17	100%	3	0.3	0	Good	
13	15	88%	3	1.1	1	Good	
14	17	100%	0	1.2	0	Good	
17	17	100%	3	0.3	0	Good	
20	17	100%	3	0.6	0	Good	NRL-CF
21	17	100%	0	0.5	0	Good	
22	17	100%	2	0.6	0	Good	NRL-CF
23	17	100%	2	0.2	0	Good	
26	17	100%	3	0.4	0	Good	NRL-CE
27	17	100%	3	0.3	0	Good	
28	17	100%	3	0.3	0	Good	
29	17	100%	3	0.2	0	Good	NRL-CF
30	15	88%	1	1.5	1	Good	
31	17	100%	2	0.3	0	Good	
33	17	100%	3	0.3	0	Good	
34	17	100%	1	0.2	0	Good	
35	17	100%	3	0.5	0	Good	
37	17	100%	3	0.2	0	Good	
40	17	100%	3	>5	0	Unsatisfactory	
42	17	100%	3	0.3	0	Good	
45	17	100%	3	0.1	0	Good	

Lab code	No. of detected mandatory pesticides	Mandatory pesticides detected %	No. of detected voluntary pesticides	AZ2	False negative	Classification	NRL
47	17	100%	2	1.8	0	Good	
48	15	88%	0	2.5	2	Satisfactory	
49	17	100%	2	0.3	0	Good	NRL-CF
50	16	94%	0	1.0	1	Good	
51	17	100%	3	0.1	0	Good	
52	17	100%	3	>5	0	Unsatisfactory	
54	17	100%	2	1.9	0	Good	NRL-CE
55	17	100%	3	0.5	0	Good	NRL-CF
56	17	100%	2	0.3	0	Good	
58	15	88%	2	>5	1	Unsatisfactory	NRL-CF
59	16	94%	0	1.6	0	Good	NRL-CF
60	17	100%	3	2.8	0	Satisfactory	
61	16	94%	3	1.6	1	Good	
62	17	100%	3	0.7	0	Good	NRL-CF
63	17	100%	3	1.6	0	Good	NRL-CF
65	16	94%	3	>5	1	Unsatisfactory	NRL-CF
66	17	100%	3	3.9	0	Unsatisfactory	NRL-CF
67	17	100%	3	0.5	0	Good	
69	16	94%	1	3.1	0	Unsatisfactory	
72	16	94%	3	0.1	0	Good	NRL-CE
73	17	100%	3	0.2	0	Good	
74	16	94%	3	0.2	0	Good	
75	17	100%	3	0.9	0	Good	
76	17	100%	3	0.5	0	Good	NRL-CE
77	17	100%	3	0.4	0	Good	
79	17	100%	3	0.2	0	Good	
80	17	100%	3	2.0	0	Good	
81	17	100%	3	0.1	0	Good	NRL-CF
82	16	94%	3	1.2	1	Good	
84	17	100%	3	0.1	0	Good	
85	17	100%	0	0.2	0	Good	NRL-CE
86	16	94%	2	2.2	1	Satisfactory	NRL-CF
87	17	100%	2	0.2	0	Good	
88	17	100%	3	2.2	0	Satisfactory	
89	17	100%	2	0.6	0	Good	
90	16	94%	3	0.2	0	Good	
91	17	100%	3	0.4	0	Good	
94	15	88%	3	0.5	1	Good	

Lab code	No. of detected mandatory pesticides	Mandatory pesticides detected %	No. of detected voluntary pesticides	AZ2	False negative	Classification	NRL
97	17	100%	3	0.1	0	Good	
99	17	100%	3	0.1	0	Good	
101	16	94%	0	1.8	1	Good	
105	17	100%	3	0.3	0	Good	
106	17	100%	2	0.3	0	Good	
107	16	94%	0	1.7	1	Good	
110	16	94%	1	>5	0	Unsatisfactory	
113	17	100%	3	0.2	0	Good	
114	17	100%	2	0.4	0	Good	
116	16	94%	0	2.8	1	Satisfactory	NRL-CF
119	17	100%	3	0.4	0	Good	
120	16	94%	0	2.0	1	Good	
123	17	100%	1	0.1	0	Good	
124	17	100%	3	0.4	0	Good	
125	17	100%	3	0.1	0	Good	NRL-CF
126	17	100%	2	0.8	0	Good	NRL-CF
128	17	100%	0	0.5	0	Good	
129	15	88%	2	1.5	1	Good	
130	17	100%	3	0.2	0	Good	NRL-CF
131	17	100%	3	0.3	0	Good	NRL-CF
133	17	100%	3	0.2	0	Good	
134	17	100%	2	0.7	0	Good	
135	15	88%	2	2.2	2	Satisfactory	
136	17	100%	2	0.7	0	Good	
138	17	100%	3	0.2	0	Good	NRL-CE
140	17	100%	1	0.1	0	Good	
141	17	100%	2	0.1	0	Good	
143	17	100%	3	0.4	0	Good	
144	17	100%	3	0.4	0	Good	
145	17	100%	2	0.4	0	Good	NRL-CF
146	16	94%	2	0.2	0	Good	
151	17	100%	2	0.7	0	Good	
153	17	100%	3	0.3	0	Good	
155	17	100%	3	0.2	0	Good	NRL-CF
157	16	94%	3	0.4	0	Good	
158	17	100%	3	0.2	0	Good	
159	17	100%	1	0.3	0	Good	NRL-CF
160	17	100%	0	0.2	0	Good	

Lab code	No. of detected mandatory pesticides	Mandatory pesticides detected %	No. of detected voluntary pesticides	AZ2	False negative	Classification	NRL
161	17	100%	3	1.1	0	Good	NRL-CF
162	17	100%	3	>5	0	Unsatisfactory	
164	17	100%	2	1.0	0	Good	
166	17	100%	0	1.4	0	Good	
167	17	100%	0	0.9	0	Good	
168	17	100%	0	0.4	0	Good	
6	17	100%	3	0.5	0	Good	

The 54 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 11**. Three laboratories was moved from Category A to B due to false positive results. Two 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. Three participants analysed more than 90% of the pesticides on the Target List but reported <15 pesticides in the Test Item.

Table 11. Number and percentage of compulsory pesticides detected and quantified, number of compulsory compounds analysed from the Target List, number of voluntary pesticides detected and quantified, number of acceptable z scores, false negative and positive results, and NRL status for the laboratories in Category B.

Lab code	No. of compulsory pesticides detected	Compulsory pesticides detected in test item, %	Analysed of compulsory pesticides on Target List, %	No. Of voluntary pesticides detected	No. of acceptable z score	No. of false negative	No. of false positive	NRL
3	12	71	58	1	12	0	0	NRL-CF
4	11	65	67	2	11	0	0	
5	9	53	59	0	9	0	0	
11	14	82	86	2	13	2	1	
15	14	82	89	1	14	0	0	
16	11	65	64	1	4	0	0	
18	8	47	58	1	7	0	0	
19	10	59	59	2	10	0	0	
24 [1]	17	100	93	2	17	0	1	
25	0	0	4	1	0	0	0	
36	0	0	98	0	0	0	0	
38	9	53	76	2	4	3	1	
39	14	82	91	1	14	0	0	
41 [1]	16	94	100	3	16	1	1	
43	11	65	54	2	7	1	0	
44	10	59	46	1	10	0	0	
46	13	76	91	3	13	0	0	
53	9	53	63	1	9	1	0	
57	0	0	4	1	0	0	0	

Lab code	No. of compulsory pesticides detected	Compulsory pesticides detected in test item, %	Analysed of compulsory pesticides on Target List, %	No. Of voluntary pesticides detected	No. of acceptable z score	No. of false negative	No. of false positive	NRL
64	3	18	40	2	2	8	0	
68	0	0	100	0	0	0	0	NRL-CF
71	10	59	47	1	10	0	0	
78	13	76	70	0	13	0	0	
92	0	0	4	1	0	0	0	
93	0	0	4	1	0	0	0	
95	14	82	73	1	14	0	0	NRL-CE
96	11	65	62	0	10	0	0	
98	13	76	79	1	12	0	0	
100	0	0	2	1	0	0	0	NRL-CF
103	5	29	80	2	5	10	0	
104	4	24	34	1	3	1	0	
108 [1]	17	100	100	3	16	0	1	
109	11	65	85	1	4	4	1	NRL-CF
111	11	65	67	3	11	0	0	
112	0	0	57	0	0	0	0	
115	10	59	45	1	10	0	0	
117	16	94	90	2	16	0	0	
118	0	0	100	0	0	0	0	
121	6	35	37	1	5	0	0	
122	10	59	46	1	10	0	0	
127	0	0	95	0	0	0	0	
132	9	53	47	0	9	0	0	
137	11	65	66	2	8	0	0	
139	1	6	9	1	1	0	0	NRL-CF
142	16	94	83	3	16	0	0	
147	0	0	99	0	0	0	0	
148	13	76	72	0	3	0	0	
150	10	59	62	0	9	1	0	NRL-CF
152	16	94	81	0	15	0	0	
154	11	65	76	1	10	1	0	
156	13	76	90	2	13	0	0	
163	13	76	87	2	11	0	0	
165	0	0	38	0	0	0	0	
170	9	53	100	0	4	8	0	

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

3.4 Trends in numbers of participating laboratories and their performance

The number of EU and EFTA laboratories participating in the EUPTs on cereals has increased steadily until EUPT-CF10 where the highest number of laboratories participated. After this, the number has settled at around 150, unless the Test Item is a feed. Then the number of participants drops. The numbers from EUPT-CF10 and forward can be seen in **Table 12**.

Table 12. Overall trends in participation of laboratories, pesticides in the target list and test item, and performance of laboratories in the 7 latest EUPTs cereals (excluding EUPT-CF7 on feed and EUPT-CF12 on hay).

PT and types of test item	EUPT-CF11 Oat flour	EUPT-CF12 Hay flour	EUPT-CF13 Oat Kernels	EUPT-CF14 Rice kernels	EUPT-CF15 Rapeseed cake	EUPT-CF16 Barley Kernels	EUPT-CF17 Wheat Kernels
Participants submitting results (EU+EFTA)	149	111	149	156	129	151	149
MRM pesticides in the Target Pesticide List	153/9	155/23	160/32	164/38	172/41	169/53	169/58
MRM pesticides in the test material	18	8	18	19	22	19	20
No. of results for MRM pesticides	2172	808	2007	2298	1315	2206	2422
Average of 'reported results', %	83	74	75	80	83	78	85
Range of 'reported results', %	65-93	40-91	44-94	26-93	57-93	32-97	48-95
Acceptable z scores, %	89	93	93	91	87	89	92
Questionable z scores, %	3	3	3.1	3	7	4	3
Unacceptable z scores, %	8	3	3.4	6	6	6	5
False negatives, %	4	1	2.3	3.4	2.0	3.3	2.8
Number of false positives	19	7	3	14	9	25	7
Category A, % of participating laboratories	45	51	57	57	57	59	73
Good AZ^2, %	92	92	91	91	67	83	89
Satisfactory AZ^2, %	1.5	3.4	5.7	6.7	12	7	6
Unsatisfactory AZ^2, %	6.2	5.1	3.4	2.2	22	10	6
Alg A RSD%	17	20	18	19	30	22	18

The number of pesticides included in the Target Pesticide List has also increased during this 17-year period, from 43 to 169 compulsory compounds and 58 voluntary compounds. Thus, the demands put on the participating laboratories has increased every year. Many laboratories have a limited scope and are therefore not able to cover all pesticides in the PT. In this EUPT, 19% of the laboratories were not able to analyse and detect more than 70% of pesticides present in the Test Item. Last EUPT the number was 23% and the year before it was also 18%. So no improvement was seen on this issue. The analytical scope was in average 87%.

The overall analytical performance (accuracy of measurement) if looking at the percentage of acceptable, questionable, unacceptable z scores has increased during the last 2 EUPTs, and in EUPT-CF17 92% of the results were acceptable and questionable/unacceptable z scores decreased. The average percent of reported results in the last seven EUPT-CF has been between 74-85%. The false negative results have fluctuated between 1-4%. Also the false positive results has been going up and down, in EUPT-CF17 the number decreased again to 7.

The percentage of Category A laboratories has increased slightly over the years. However, in EUPT-CF17 the highest percentage was seen, were 73% of the participants were evaluated as Category A. For Category A the percentage of participant with AZ^2 was <2 (good) has been >90% for many year. However, for the rapeseed cake EUPT-CF15 this dropped significantly to 67% and in this EUPT it was 89%.

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

The EUPT-CF17 Test Item was wheat kernels containing incurred and spiked pesticides. The wheat kernels have been sprayed in the field with commercially available pesticide formulations and additionally spiked post-harvest in the laboratory. The final Test Item contained the following pesticides: azoxystrobin, bixafen, clomazone, cyazofamid, cyfluthrin, cyprodinil, difenoconazole, dimethomorph, flonicamid, fluopyram, HCH-beta, metconazole, phenmedipham, pirimicarb, pirimicarb-desmethyl, proquinazid, prothioconazole-desthio, pyraclostrobin, pyriproxyfen and trifloxystrobin. One hundred forty-nine EU and EFTA laboratories, from 29 different countries agreed to participate in this proficiency test. Six of them did not report any results due to different reasons. An additional 13 laboratories from EU candidate states and Third Countries registered for the PT and only ne did not submitted results. The Target Pesticide List distributed to the laboratories prior to the test contained 169 individual compulsory and 58 voluntary compounds.

The number of false positives and false negatives has varied between the EUPTs. Twenty-five false positive results were reported and the number of false negatives represented 2% of the total number of results. This is at levels typically seen in EUPT-CFs. The average Alg A-RSD was at 18%, lower than the FFP-RSD of 25%.

Of the reported results for the evaluated pesticides, more than 90% were azoxystrobin, bixafen, clomazone, cyprodinil, difenoconazole, dimethomorph, fluopyram, HCH-beta, metconazole, pirimicarb, pirimicarb-desmethyl, proquinazid, prothioconazole-desthio, pyraclostrobin pyriproxyfen and trifloxystrobin. For cyazofamid, cyfluthrin, flonicamid and phenmediphambetween 87-89% of the results were acceptable.

The Test Item for EUPT-CF18 will be wheat straw, and are planned to be shipped to the laboratories in April 2024. The selection of pesticides will continue to be focused on pesticides included in the scope of the EU multi-annual coordinated control programme, the working document as well as additional pesticides of relevance to feed and/or cereal production in Europe and in other parts of the world from where significant quantities of feed and cereals are imported.

4. ACKNOWLEDGEMENTS

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5. REFERENCES

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

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

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

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

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

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

APPENDICES

Appendix 1 List of Laboratories registered to participate in the EUPT-CF17

Participating labs from EU and EFTA member states

Country	Institution	City	NRL	Results reported
Austria	AGES - Innsbruck	Innsbruck	NRL-CF	
Belgium	Primoris Belgium	Gent - Zwijnaarde		
Belgium	FLVVT	Tervuren		
Belgium	Sciensano	Brussels	NRL-CF	
Bulgaria	Primoris - Bulgaria, Plovdiv	Plovdiv		
Croatia	Croatian Veterinary Institute - Zagreb	Zagreb	NRL-CF	
Croatia	Nastavni Zavod za Javno Zdravstvo PGŽ	Kotar County, Rijeka		
Croatia	Dr. Andrija Štampar - Pesticide Lab	Zagreb	NRL-CE	
Croatia	Sample Control - Pesticide Lab	Lucko		
Croatia	Bioinstitut d.o.o., Cakovec	Cakovec		
Croatia	INSPECTO d.o.o. Laboratorij (Osijek)	Osijek		
Croatia	Eurofins Croatiakontrola	Zagreb		
Croatia	Croatian National Institute of Public Health-HZJZ	Zagreb		
Cyprus	Pesticides Residues Laboratory of SGL of Cyprus	Nicosia	NRL-CE	
Cyprus	Animal Feeds and Feed Additives - Pesticide Lab	Nicosia	NRL-CF	
Czech Republic	Metrology and Testing Laboratory, UCT Prague	Praha		
Czech Republic	UKZUZ - Czech Republic, Brno	Brno	NRL-CF	
Czech Republic	Czech Agriculture and Food Inspection Authority	Praha	NRL-CE	
Denmark	Laboratoriet Ringsted - Pesticide Lab	Ringsted	NRL-CF	
Estonia	National Centre for Laboratory Research and Risk Assessment	Tallinn	NRL-CF	
Finland	Finnish Food Authority, Chemistry Unit	Helsinki	NRL-CF	
Finland	Finnish Customs Laboratory	Espoo	NRL-CE	
France	GIRPA	Beaucouzé		
France	Capinov	Landerneau		
France	CERECO	GARONS		
France	Phytocontrol (Nimes) - Pesticide Lab	Nimes		
France	SCL Montpellier	Montpellier		
France	CAMP Méditerrannée (Perpignan)	PERPIGNAN		
France	Laboratoire SCL de PARIS	Massy Cedex	NRL-CF	
France	INOVALYS	Le Mans		
Germany	LUA Saarland - Pesticide Lab	Saarbrücken		
Germany	LHL - Pesticide Lab (Wiesbaden)	Wiesbaden		
Germany	LAVES, Pesticide Lab (Oldenburg)	Oldenburg		
Germany	TLLLR	Jena		
Germany	LA für Landwirtschaft, Lebensmittelsicherheit und Fischerei	Rostock		
Germany	Landesamt für Verbraucherschutz ST FB3, Dez. 33	Halle/Saale		
Germany	LLG	Halle/Saale		
Germany	AGROLAB LUFA Kiel - Pesticide Lab	Kiel		
Germany	Landesuntersuchungsamt - Institut für Lebensmittelchemie	Speyer		
Germany	BVL NRL for Pesticide Residues	Berlin	NRL-CF	

Country	Institution	City	NRL	Results reported
Germany	BfUL Nossen - FB 42	Nossen		
Germany	Landeslabor Schleswig-Holstein	Neumünster		
Germany	Bavarian Health and Food Safety Authority	Erlangen		
Germany	LAVES - Pesticide Lab (Stade)	Stade		
Germany	Landeslabor Berlin Brandenburg	Berlin		
Germany	LUFA Speyer	Speyer		
Germany	CVUA RRW - Pesticide Lab (Krefeld)	Krefeld		
Germany	LUA Sachsen - Pesticide Lab, Dresden	Dresden		
Germany	Eurofins Dr. Specht Laboratorien GmbH	Hamburg		
Germany	CVUA Stuttgart - Pesticide Lab	Fellbach		
Germany	Eurofins Dr.Specht Express GmbH - Hamburg	Hamburg		
Germany	Labor Friedle GmbH	Tegernheim		
Germany	Hessisches Landeslabor Kassel	Kassel		
Germany	Bundeswehr - Pesticide Lab (Garching-Hochbrück)	Garching-Hochbrück		
Germany	LTZ Augustenberg	Karlsruhe		
Germany	GALAB Laboratories GmbH - Hamburg	Hamburg		
Greece	Laboratory of Pesticide Residues Analysis-Thessaloniki	Thessaloniki		
Greece	GCSL - Pesticide Lab (Athens)	Athens	NRL-CF	
Greece	Benaki Phytopathological Institute, Kifissia	Kifissia	NRL-CF	
Hungary	NFCSO FCSLD PPSCNRL Szolnok	Szolnok	NRL-CF	No
Hungary	NCSFO FCSLD PPSCNRL (Miskolc)	Miskolc		
Hungary	NFCSO PPSCNRL, Site in Hódmezovásárhely	Hódmezovásárhely		No
Hungary	Plant Protection and Soil Conservation NRL Site in Velence	Velence	NRL-CF	
Iceland	Matís - Iceland, Reykjavík	Reykjavík	NRL-CF	
Ireland	The Food Chemistry Laboratories - DAFM	Celbridge	NRL-CF	
Italy	APPA Trento	Trento		
Italy	Azienda Sanitaria Locale di Firenze	Firenze		
Italy	ARPA Puglia Polo Spec. Alimenti	Bari		
Italy	ARPA FVG	Udine		
Italy	ARPA-ER - Pesticide Lab	Ferrara		
Italy	Laboratorio di Prevenzione (Bergamo)	Bergamo		
Italy	IZSVe - Pesticide Lab	Legnaro (Padova)		
Italy	APPA Bolzano	Bolzano		
Italy	Laboratorio Pesticidi, micotossine e tossine vegetali. IZSUM	Perugia		
Italy	IZS LT - Italy, Rome	Roma		
Italy	IZS Sardegna - Pesticide Lab	Sassari		
Italy	Istituto Superiore di Sanità - Roma	Roma		
Italy	ARPAL Sez. di La Spezia	La Spezia		
Italy	IZS PLVA - U.O. Chimico , sez. Genova	Genova	NRL-CF	
Italy	IZS PB - Foggia	Foggia		
Italy	ATS Milano - Laboratorio di Prevenzione	Milano		
Italy	ARPA Lazio (sez. Latina) - Pesticide Lab	Latina		No
Italy	IZS Lombardia Emilia Romagna	Brescia		
Italy	IZSAM - Pesticide Lab	Teramo		
Italy	ARPAM	Macerata		No

Country	Institution	City	NRL	Results reported
Italy	IZSM - Pesticide Lab	Portici (NA)		
Latvia	BIOR (Riga) - Pesticide Lab	Riga	NRL-CF	
Lithuania	NATIONAL FOOD AND VETERINARY RISK ASSESSMENT INSTITUTE	Vilnius	NRL-CF	
Luxembourg	LNS Food lab	Dudelange	NRL-CE	
Netherlands	Wageningen Food Safety Research	Wageningen	NRL-CF	
Netherlands	Groen Agro Control - Netherlands	Delfgauw		
Netherlands	Eurofins Lab Zeeuws-Vlaanderen	Graauw		
Netherlands	AGROLAB Dr. Verwey B.V.	Barendrecht		
Norway	NIBIO Pesticides & Natural Products Chemistry	ÅS	NRL-CF	
Norway	Institute of Marine Research	Bergen	NRL-CF	
Poland	JARS S.A. - Legionowo	Legionowo		
Poland	WIW ZHW Poznan Pesticide Lab	Poznan		
Poland	WIW ZHW (Szczecin) - Pesticide Lab	Szczecin		
Poland	VSES Opole - Pesticide Lab	Opole		
Poland	Laboratory of Food & Feed Safety in Bialystok	Bialystok		
Poland	WIW ZHW (Katowice) - Pesticide Lab	Katowice		
Poland	IPP-NRI - Pesticide Lab (Sosnicowice)	Sosnicowice		
Poland	WIW ZHW (Bialystok) - Pesticide Lab	Bialystok		
Poland	Department of Pesticide Residue Research, IPP-NRI	Poznan	NRL-CF	
Poland	VSES Rzeszow - Pesticide Lab	Rzeszow		
Poland	VSES Warszawa - Pesticide Lab	Warszaw	NRL-CE	
Poland	InHort (Skierniewice) - Pesticide Lab	Skierniewice		
Poland	Hamilton UO-Technologia, Grójec	Grójec		
Portugal	Laboratório Regional de Veterinária e Segurança Alimentar	Funchal - Madeira Island		
Portugal	INIAV Pesticide Lab (Vegetable & Animal Products)	Vairão - Vila do Conde	NRL-CE	
Romania	LRCRPPPV MURES	Tirgu Mures		
Romania	Sanitary Veterinary and Food Safety Directorate Cluj	Cluj Napoca		
Romania	National Phytosanitary Authority- LCRPPPV	Bucharest	NRL-CF	
Romania	DSVSA Bucuresti - Pesticide Lab	Bucharest		
Slovakia	UKSUP SLAPe	Bratislava		
Slovakia	State Veterinary and Food Institute (Bratislava)	Bratislava	NRL-CF	
Slovakia	Pesticide Lab of PHA SR - Bratislava	Bratislava		
Slovenia	Pesticide Lab - Maribor	Maribor	NRL-CE	
Slovenia	Kmetijski institut Slovenije	Ljubljana	NRL-CF	
Spain	Laboratorio Regional de la CCAA de La Rioja	Logroño		
Spain	LABORATORIO KUDAM, S.L.	Pilar de la Horadada (Alicante)		
Spain	LAC - Generalitat de Catalunya	Cabrils		
Spain	LABORATORIO DE SALUD PUBLICA DE CUENCA-SPAIN	Cuenca		
Spain	Nasertic - Spain, Villava	Villava		
Spain	Laboratorio Agroalimentario - Spain, Valencia	Burjassot, Valencia		
Spain	Laboratorio Químico-Microbiológico, S.L.	Mairena del Aljarafe, Sevilla		
Spain	Labs & Technological Services AGQ - Burguillos	Burguillos		
Spain	Ainia (Valencia)	Valencia		

Country	Institution	City	NRL	Results reported
Spain	Agrolab Ibérica S.L.U.	Burgos		
Spain	Analytica Alimentaria GmbH - Almeria, Spain	Almeria		
Spain	EURL-FV University of Almería	Almeria		
Spain	Laboratorio Analítico Bioclínico - Spain, Almeria	Almeria		
Spain	Laboratorio Agroambiental (Gobierno de Aragón)	Zaragoza		
Spain	Please fill-in	San Ginés (Murcia)		
Spain	LABORATORIOA AGRARIO REGIONAL de CASTILLA Y LEÓN	Burgos		
Spain	Laboratorio Agrario y Fitopatologico de Galicia	Abegondo. A Coruña		
Spain	Laboratorio de Residuos, Inst. Tecnol. de Canarias	Agüimes, Gran Canaria		No
Spain	Laboratorio Agroalimentario de Extremadura	Cáceres		
Spain	Laboratorio Arbitral Agroalimentario	Madrid	NRL-CF	
Spain	Laboratorio de Salud Pública (Madrid Salud). Ayto. Madrid	Madrid		
Spain	Laboratorio de Salud Pública de Galicia, Lugo	Lugo		
Spain	EUROFINS ECOSUR - Pesticide Lab	LORQUI - MURCIA		No
Spain	Agència de Salut Pública de Barcelona	Barcelona		
Spain	National Center For Technology and Food Safety (CNTA)	San Adrián (Navarra)		
Spain	LARAGA - Pesticide Lab (Toledo)	Toledo		
Sweden	Eurofins Food and Feed Testing Sweden	Lidköping		
Sweden	Swedish Food Agency	Uppsala	NRL-CF	
Switzerland	Kantonales Laboratorium Zürich	Zürich		
Switzerland	Kantonales Laboratorium Bern	Bern		

Participating labs from EU candidate states and other non-EU countries

Country	Institution	City	Report data
Argentina	Bolsa de Comercio de Rosario	Rosario	
Australia	Symbio Laboratories - Australia, Eight Mile Plains	Eight Mile Plains, QLD	
Egypt	Royal International Inspection Laboratories	Suez	
India	Assurance Laboratory, L T Foods Limited	Sonipat	
Peru	Unidad Centro de Control d Insumos y Residuos Tóxicos SENASA	Lima	
Peru	Inspectorate Services Perú S.A.C	LIMA - CALLAO	
Serbia	A BIO TECH LAB - Serbia, Sremska Kamenica	Sremska Kamenica	
Serbia	Gradski zavod za javno zdravlje Beograd	Belgrade	
Serbia	SP Laboratorija - Pesticide Lab	BECEJ	
Serbia	Directorate for national reference laboratories	Belgrade	
Serbia	Field Test - Serbia, Belgrade	Belgrade	
Thailand	Central Laboratory - Pesticide Lab (Bangkok)	Bangkok	No
United Kingdom	FERA - Pesticide Lab	York	

Appendix 2 Target Pesticide List

Pesticides	MRRL (mg/kg)
Compulsory Compounds (will be considered in Category A/B classification)	
2-phenylphenol	0.01
Acephate	0.01
Aclonifen	0.01
Acetamiprid	0.01
Acrinathrin	0.01
Aldrin	0.005
Ametoctradin	0.01
Azinphos-methyl	0.005
Azoxystrobin	0.01
Bifenthrin	0.01
Biphenyl	0.01
Bitertanol	0.01
Bixafen	0.01
Boscalid	0.01
Bromuconazole	0.01
Buprofezin	0.01
Cadusafos	0.005
Carbaryl	0.005
Carbendazim	0.01
Carbofuran	0.005
Carbofuran-3-hydroxy	0.005
Carboxin	0.01
Chlorantraniliprole	0.01
Chlorfenapyr	0.01
Chlorfenvinphos	0.01
Chlorpropham	0.01
Chlorpyrifos	0.005
Chlorpyrifos-methyl	0.01
Clothianidin	0.01
Cyantraniliprole	0.01
Cyazofamid	0.01
Cyfluthrin	0.01
Cymoxanil	0.01
Cypermethrin	0.01
Cyproconazole	0.01
Cyprodinil	0.01
Deltamethrin	0.01
Demeton-S-methylsulfone	0.005
Diazinon	0.005
Dichlorvos	0.005
Dieldrin	0.005
Difenoconazole	0.01
Diflubenzuron	0.01
Dimethoate	0.003
Dimethomorph	0.01

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

Pesticides	MRRL (mg/kg)
Kresoxim-methyl	0.01
Lambda-cyhalothrin	0.01
Lindane	0.01
Linuron	0.01
Malaoxon	0.01
Malathion	0.01
Mandipropamid	0.01
Metaflumizone	0.01
Metalaxyl	0.01
Metconazole	0.01
Methacrifos	0.01
Methamidophos	0.01
Methomyl	0.01
Metolachlor	0.01
Metrafenone	0.01
Metribuzin	0.01
Omethoate	0.003
Oxydemeton-methyl	0.005
Paclobutrazol	0.01
Parathion	0.01
Penconazole	0.01
Pencycuron	0.01
Pendimethalin	0.01
Permethrin	0.01
Phosphamidon	0.01
Pirimicarb	0.01
Pirimicarb-desmethyl	0.01
Pirimiphos-methyl	0.01
Prochloraz	0.01
Procymidone	0.01
Profenofos	0.01
Propamocarb	0.01
Propiconazole	0.01
Proquinazid	0.01
Prosulfocarb	0.01
Prothioconazole-desthio	0.01
Prothiofos	0.01
Pymetrozine	0.01
Pyraclostrobin	0.01
Pyridaben	0.01
Pyridalyl	0.01
Pyrimethanil	0.01
Pyriproxyfen	0.01
Quinoxyfen	0.01
Spinetoram	0.01
Spirodiclofen	0.01
Spiromesifen	0.01

Pesticides	MRRL (mg/kg)
Spirotetramat	0.01
Spirotetramat metabolite BYI08330-enol	0.01
Spiroxamine	0.01
Sulfoxaflor	0.01
Tau-Fluvalinate	0.01
Tebuconazole	0.01
Tebufenozide	0.01
Teflubenzuron	0.01
Tefluthrin	0.01
Terbuthylazine	0.01
Tetraconazole	0.01
Tetradifon	0.01
Tetramethrin	0.01
Thiabendazole	0.01
Thiacloprid	0.01
Thiamethoxam	0.01
Thiodicarb	0.01
Thiophanate-methyl	0.01
Tolclofos-methyl	0.01
Triadimefon	0.01
Triadimenol	0.01
Triflumizole	0.01
Triflumizole metabolite (FM-6-1)	0.01
Triazophos	0.005
Tricyclazole	0.01
Trifloxystrobin	0.01
Trifluralin	0.01
Triticonazole	0.01
Vinclozolin	0.01
Zoxamide	0.01
Voluntary Compounds (will not be considered in Category A/B classification)	
Azadirachtin	0.01
Benalaxyl (sum)	0.01
Benzovindiflupyr	0.01
Chlordane-cis	0.01
Chlordane-oxy	0.01
Chlordane-trans	0.01
Chlorfluazuron	0.01
Clomazone	0.01
Cyflufenamid	0.01
Cyflumetofen	0.01
Cyhalofop-butyl	0.01
DDD-pp	0.01
DDE-pp	0.01
DDT-op	0.01
DDT-pp	0.01

Pesticides	MRRL (mg/kg)
Dinotefuran	0.01
Diuron	0.01
Endrin	0.01
Endrin-ketone	0.01
Fenobucarb	0.01
Fenpicoxamid	0.01
Florpyrauxyfen-benzyl	0.01
Fluensulfone	0.01
Flufenacet	0.01
Flupyradifurone	0.01
Flutianil	0.01
Forchlorfenuron	0.01
HCH-alpha	0.01
HCH-beta	0.01
Heptachlor	0.01
Heptachlorepoxid-cis	0.01
Heptachlorepoxid-trans	0.01
Isofetamid	0.01
Isopyrazam	0.01
Isoxaflutole	0.01
Mefentrifluconazole	0.01
Metaldehyde	0.01
Molinate	0.01
Novaluron	0.01
Oxadiargyl	0.01
Oxathiapiprolin	0.01
Oxyfluorfen	0.01
Penflufen	0.01
Pentachloro-aniline	0.01
Penthiopyrad	0.01
Phenmedipham	0.01
Picolinafen	0.01
Propaquizafop	0.01
Pyrethrins	0.01
Pyridate	0.01
Pyriofenone	0.01
Quinalphos	0.01
Quinoclamine	0.01
Quintozene	0.01
Rotenone	0.01
Tolfenpyrad	0.01
Tri-allate	0.01
Tritosulfuron	0.01

Appendix 3 Homogeneity data

	Azoxystrobin mg/kg		Bixafen mg/kg		Clomazone mg/kg		Cyazofamid mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
013	0.177	0.176	0.107	0.105			0.233	0.241
024	0.186	0.177	0.111	0.109	0.032	0.031	0.228	0.232
034	0.168	0.164	0.116	0.106	0.029	0.030	0.233	0.211
062	0.187	0.149	0.104	0.100	0.033	0.027	0.200	0.194
094	0.197	0.201	0.113	0.122	0.036	0.034	0.230	0.234
124	0.168	0.166	0.092	0.100	0.029	0.030	0.192	0.216
158	0.168	0.187	0.128	0.120	0.029	0.032	0.250	0.218
186	0.203	0.162	0.113	0.102	0.034	0.029	0.240	0.213
201	0.181	0.177	0.108	0.097	0.031	0.031	0.211	0.211
212	0.191	0.184	0.108	0.108	0.034	0.033	0.219	0.212
230	0.191	0.209	0.101	0.110	0.032	0.037	0.218	0.235

	Cyflutrin mg/kg		Cyprodinil mg/kg		Difenoconazole mg/kg		Dimethomorph mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
013	0.087	0.068	0.309	0.339	0.102	0.108	0.165	0.157
024	0.071	0.072	0.373	0.318	0.117	0.105	0.169	0.154
034	0.057	0.067	0.318	0.319	0.119	0.103	0.155	0.150
062	0.070	0.056	0.365	0.292	0.097	0.104	0.175	0.143
094	0.077	0.065	0.394	0.375	0.115	0.125	0.174	0.185
124	0.067	0.061	0.317	0.317	0.095	0.102	0.156	0.160
158	0.062	0.069	0.320	0.372	0.132	0.112	0.155	0.173
186	0.063	0.068	0.406	0.297	0.113	0.103	0.183	0.146
201	0.069	0.068	0.348	0.347	0.115	0.105	0.171	0.157
212	0.072	0.074	0.348	0.335	0.119	0.114	0.183	0.169
230	0.079	0.083	0.349	0.368	0.104	0.114	0.175	0.185

	Phenmedipham mg/kg		Pirimicarb mg/kg		Pirimicarb-desmethyl mg/kg		Prothioconazole-desthio mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
013	0.076	0.080	0.036	0.036	0.066	0.049	0.183	0.184
024	0.072	0.075	0.041	0.037	0.051	0.047	0.205	0.196
034	0.074	0.070	0.041	0.038	0.040	0.044	0.220	0.191
062	0.060	0.060	0.035	0.035	0.051	0.039	0.176	0.170
094	0.070	0.074	0.040	0.044	0.049	0.048	0.218	0.217
124	0.062	0.069	0.034	0.037	0.045	0.046	0.177	0.188
158	0.074	0.069	0.041	0.039	0.043	0.047	0.223	0.200
186	0.079	0.070	0.036	0.034	0.050	0.046	0.198	0.178
201	0.067	0.067	0.037	0.037	0.047	0.048	0.184	0.181
212	0.073	0.068	0.040	0.035	0.049	0.047	0.193	0.179
230	0.069	0.073	0.035	0.037	0.051	0.055	0.170	0.193

	Pyraclostrobin mg/kg		Pyriproxyfen mg/kg		Trifloxystrobin mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Sample no.	Portion 1	Portion 2
013	0.099	0.108	0.071	013	0.099	0.108
024	0.114	0.106	0.050	024	0.114	0.106
034	0.119	0.105	0.042	034	0.119	0.105
062	0.103	0.104	0.048	062	0.103	0.104
094	0.111	0.121	0.052	094	0.111	0.121
124	0.097	0.100	0.045	124	0.097	0.100
158	0.126	0.112	0.046	158	0.126	0.112
186	0.110	0.099	0.050	186	0.110	0.099
201	0.113	0.104	0.050	201	0.113	0.104
212	0.120	0.111	0.051	212	0.120	0.111
230	0.103	0.112	0.056	230	0.103	0.112

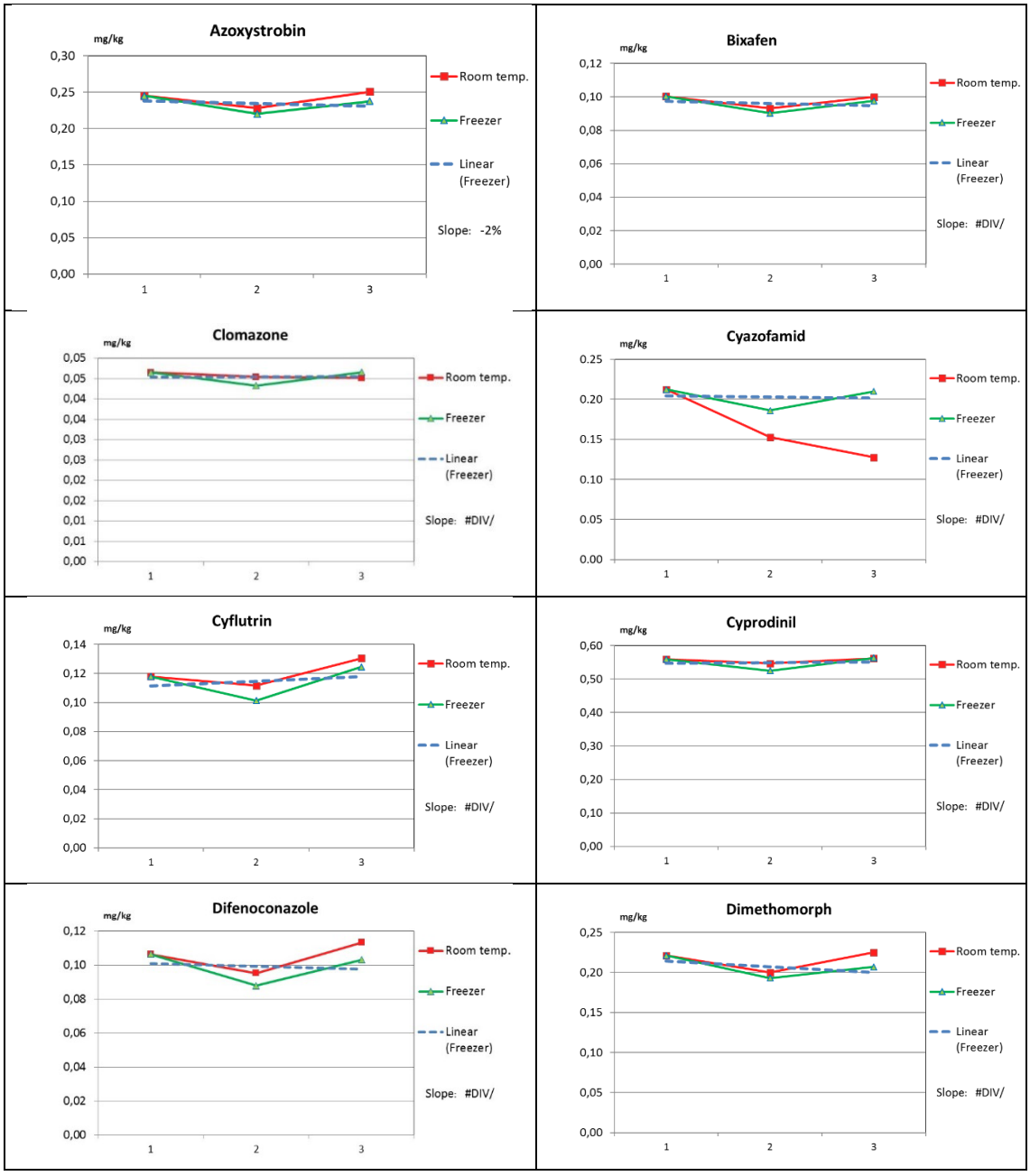
Appendix 4 Stability figures

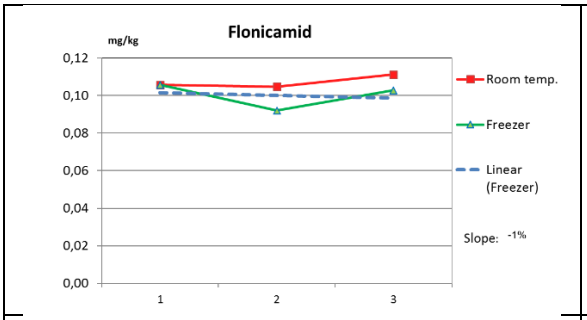
The stability test was performed according to ISO 13528 Annex B [5]. Two different storage temperatures were used; room temperature and -18 °C.

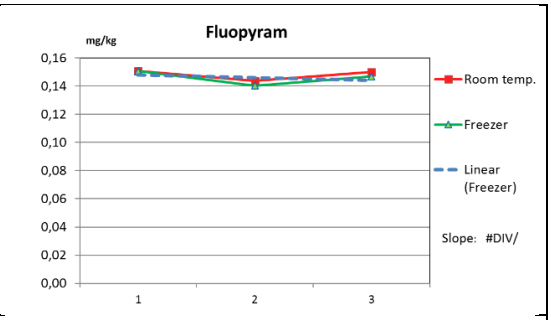
The dates of testing were as follows:

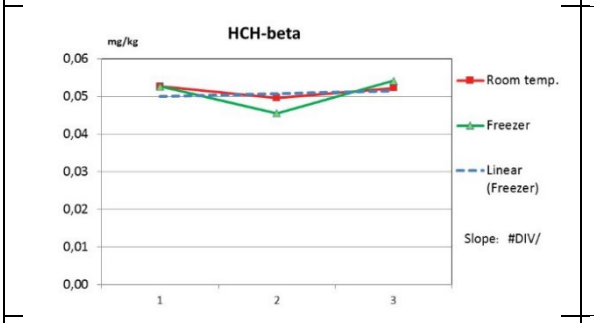
Day 1: 6 February 2023
Day 2: 20 February 2023
Day 3: 6 March 2023

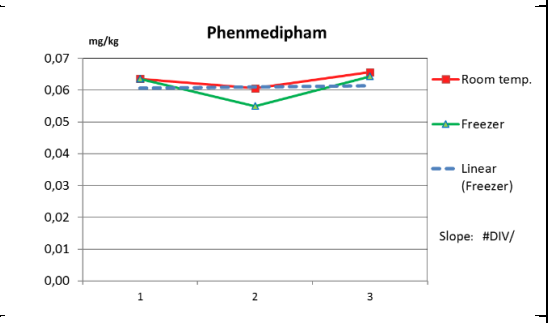
All pesticides passed the test at -18 °C see **1.6 Stability test**. At room temperature indoxacarb and pirimicarb did not pass the test when stored for 11 weeks.

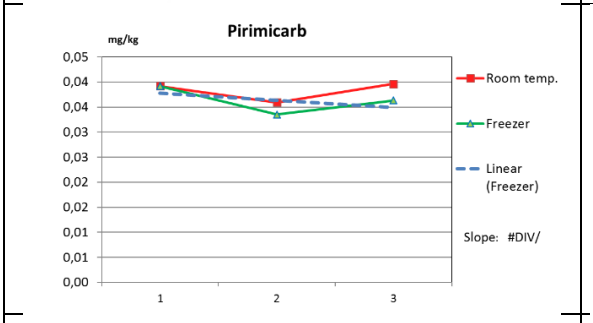


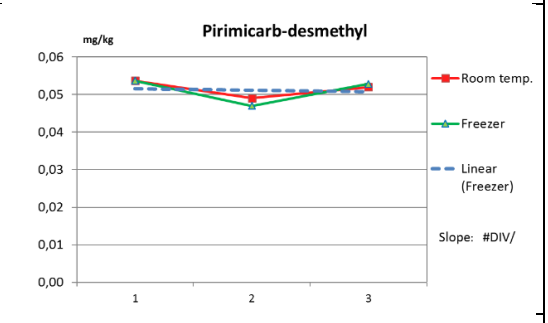

Flonicamid
mg/kg
0,12
0,10
0,08
0,06
0,04
0,02
0,00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: -1%

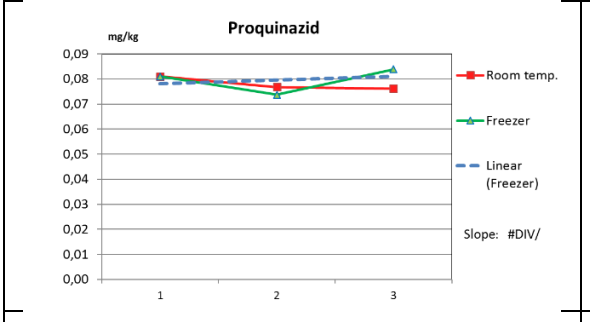

Fluopyram
mg/kg
0,16
0,14
0,12
0,10
0,08
0,06
0,04
0,02
0,00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: #DIV/

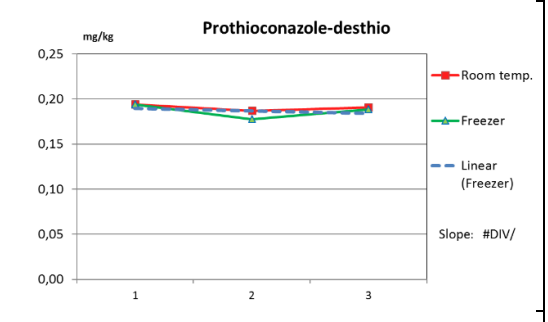

HCH-beta
mg/kg
0,06
0,05
0,04
0,03
0,02
0,01
0,00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: #DIV/

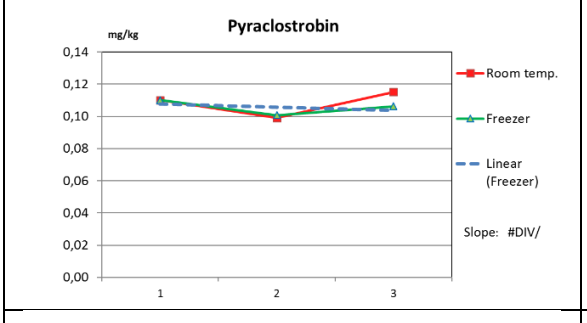

Phenmedipham
mg/kg
0,07
0,06
0,05
0,04
0,03
0,02
0,01
0,00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: #DIV/

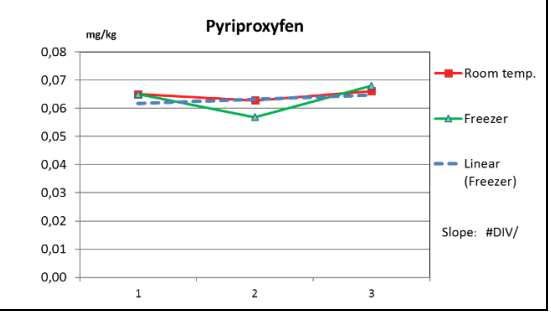

Pirimicarb
mg/kg
0,05
0,04
0,04
0,03
0,03
0,02
0,02
0,01
0,01
0,00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: #DIV/

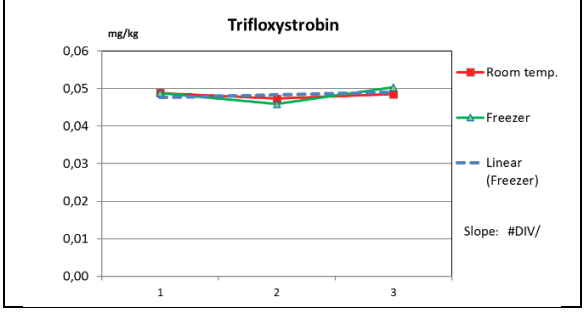

Pirimicarb-desmethyl
mg/kg
0,06
0,05
0,04
0,03
0,02
0,01
0,00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: #DIV/


Proquinazid
mg/kg
0,09
0,08
0,07
0,06
0,05
0,04
0,03
0,02
0,01
0,00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: #DIV/

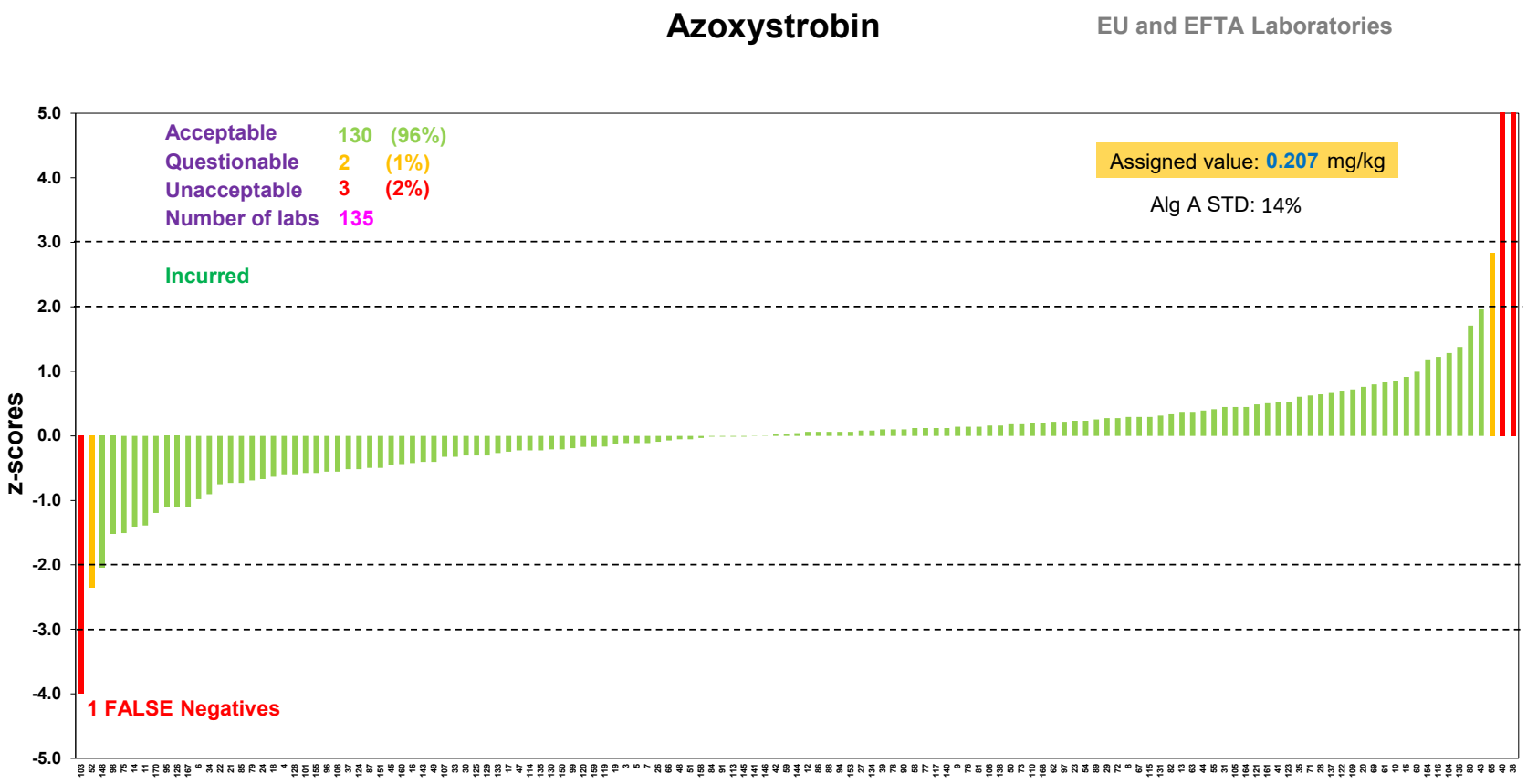

Prothioconazole-desthio
mg/kg
0,25
0,20
0,15
0,10
0,05
0,00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: #DIV/


Pyraclostrobin
mg/kg
0,14
0,12
0,10
0,08
0,06
0,04
0,02
0,00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: #DIV/


Pyriproxyfen
mg/kg
0,08
0,07
0,06
0,05
0,04
0,03
0,02
0,01
0,00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: #DIV/

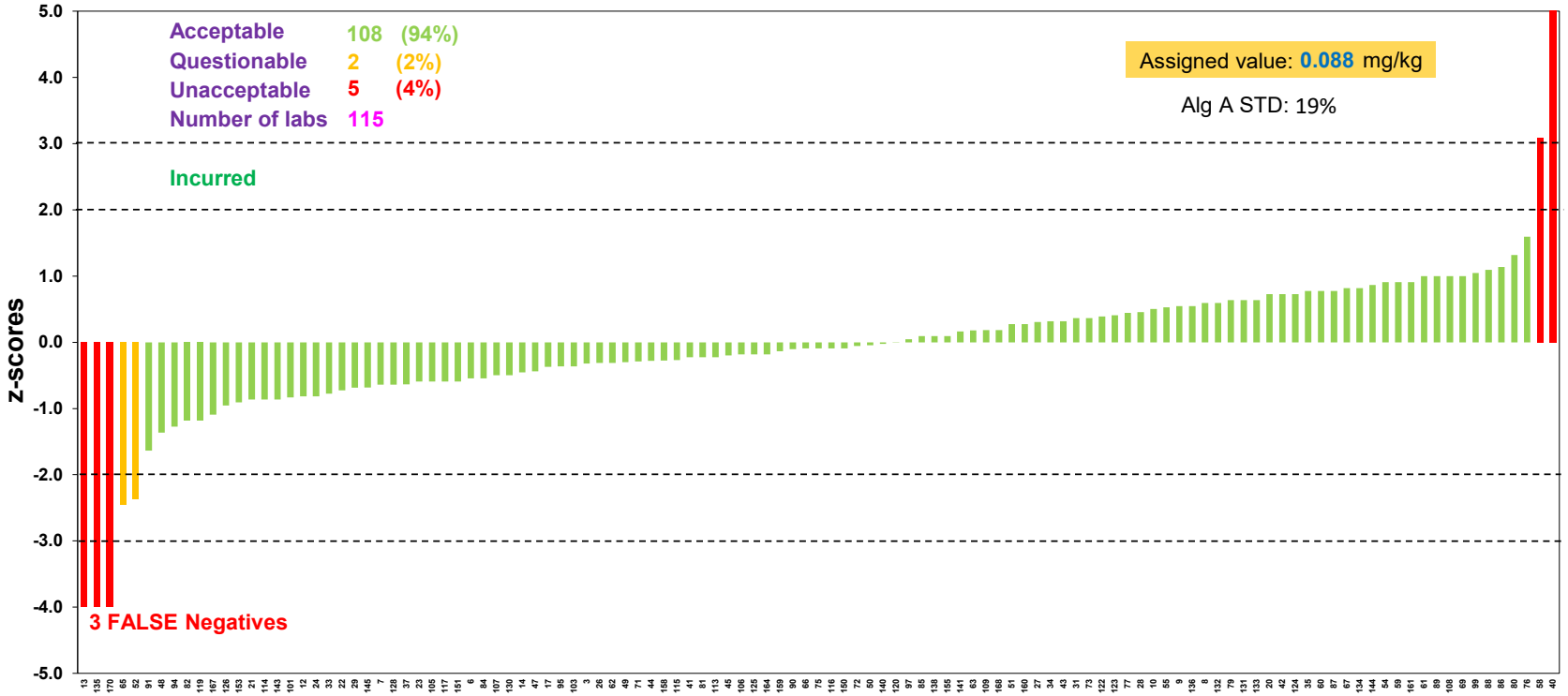

Trifloxystrobin
mg/kg
0,06
0,05
0,04
0,03
0,02
0,01
0,00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: #DIV/

Appendix 5 Graphical presentation of z-scores



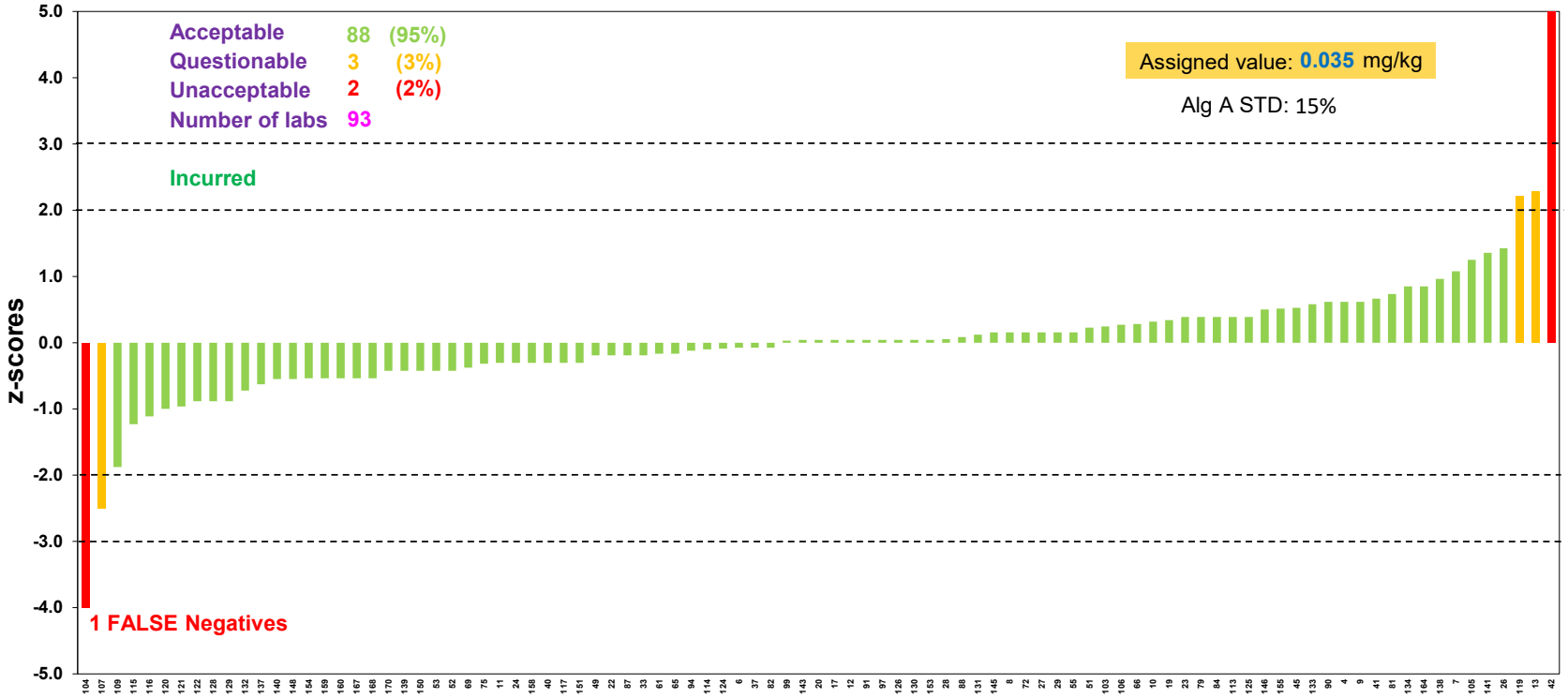
Bixafen

EU and EFTA Laboratories



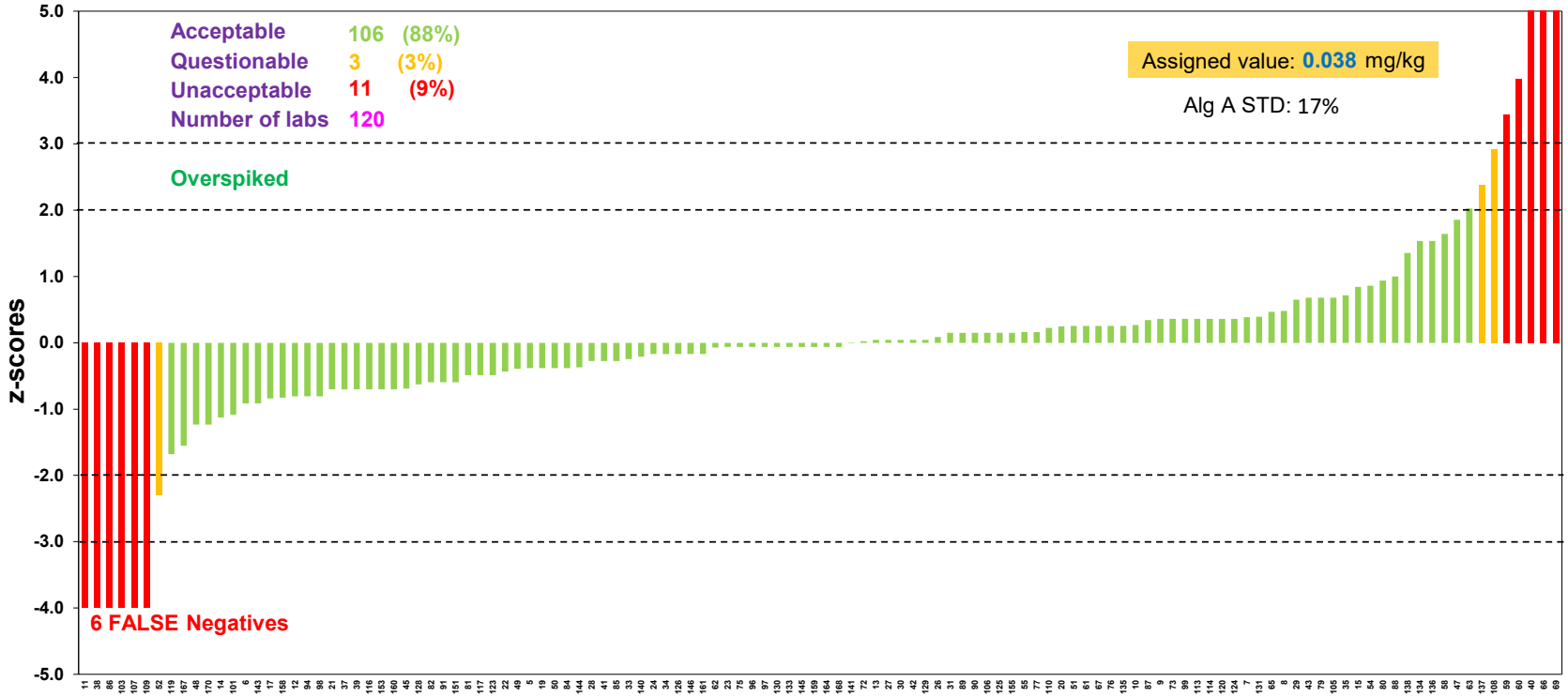
Clomazone

EU and EFTA Laboratories



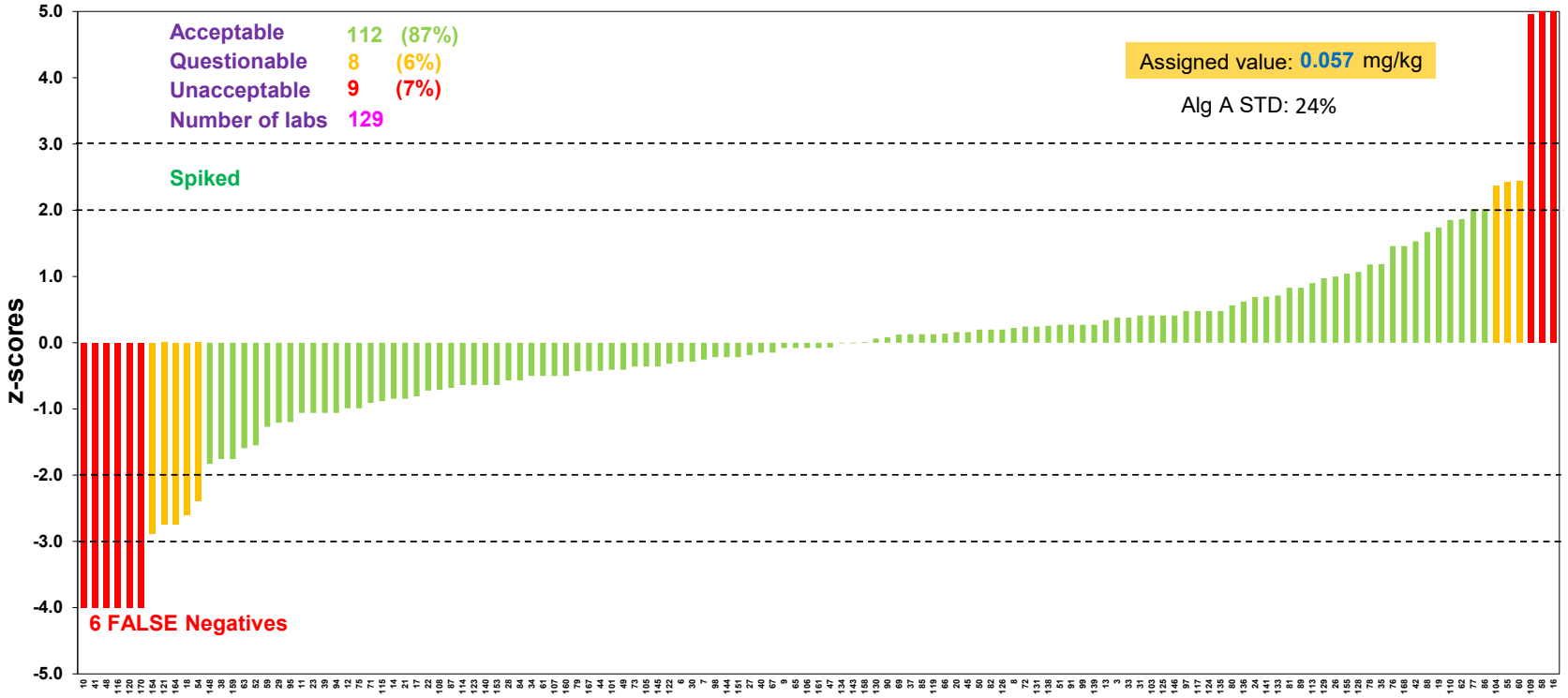
Cyazofamid

EU and EFTA Laboratories



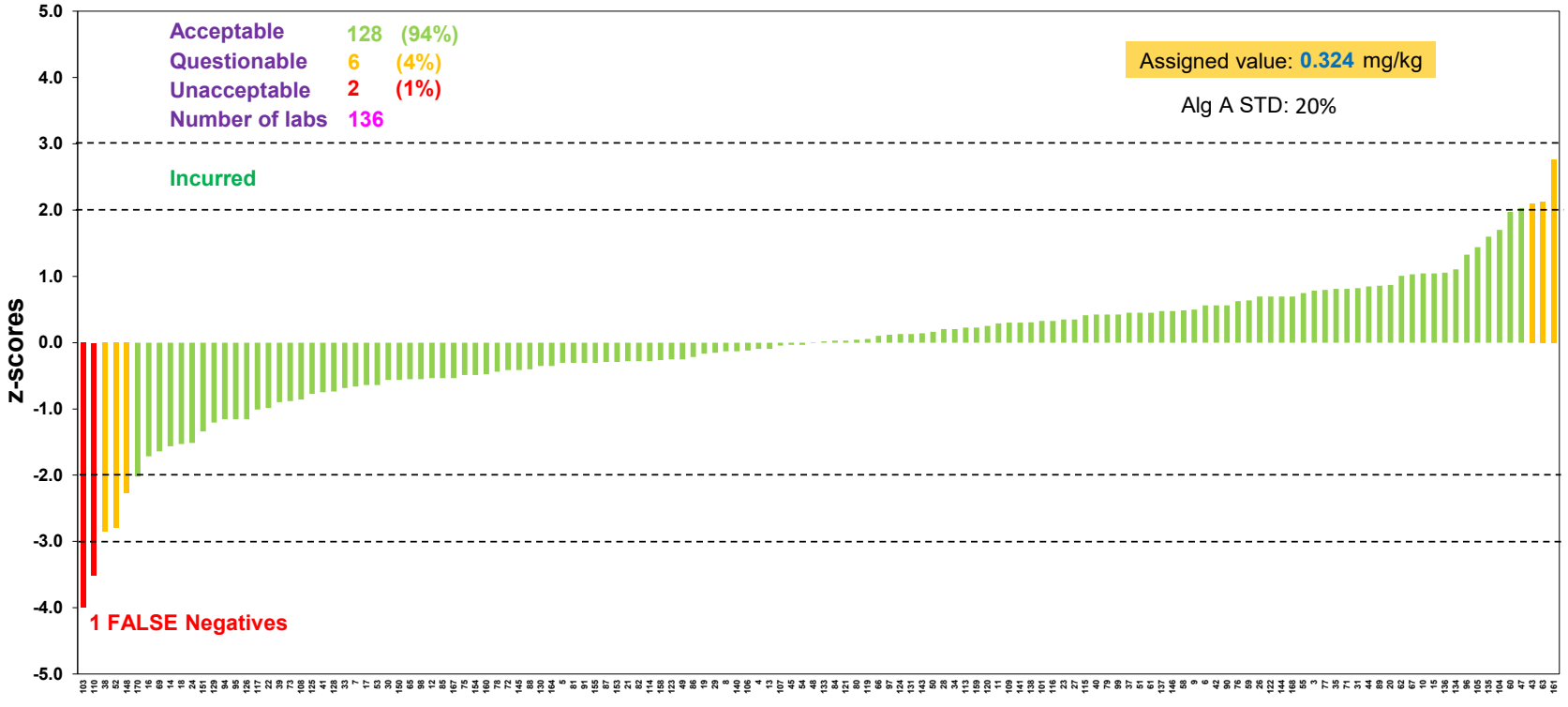
Cyfluthrin

EU and EFTA Laboratories



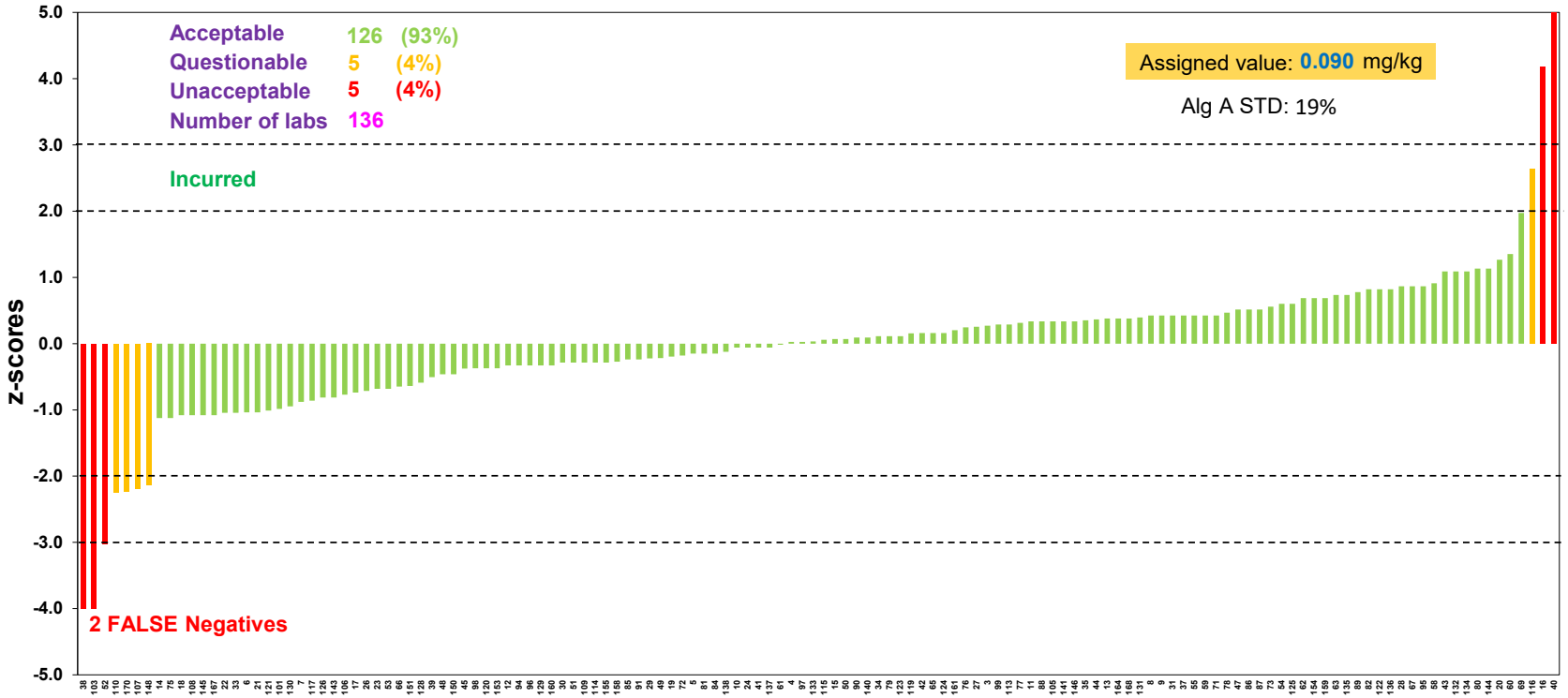
Cyprodinil

EU and EFTA Laboratories



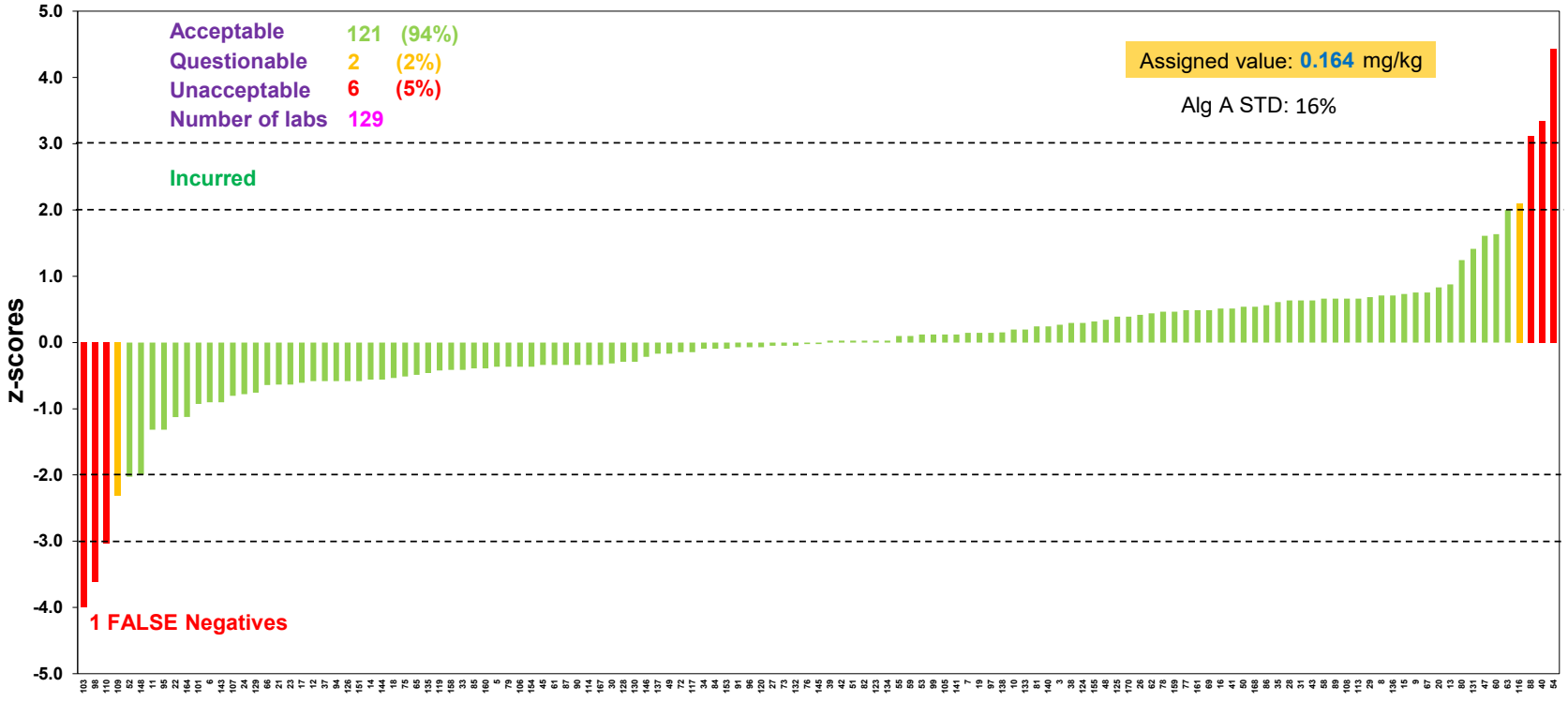
Difenoconazole

EU and EFTA Laboratories



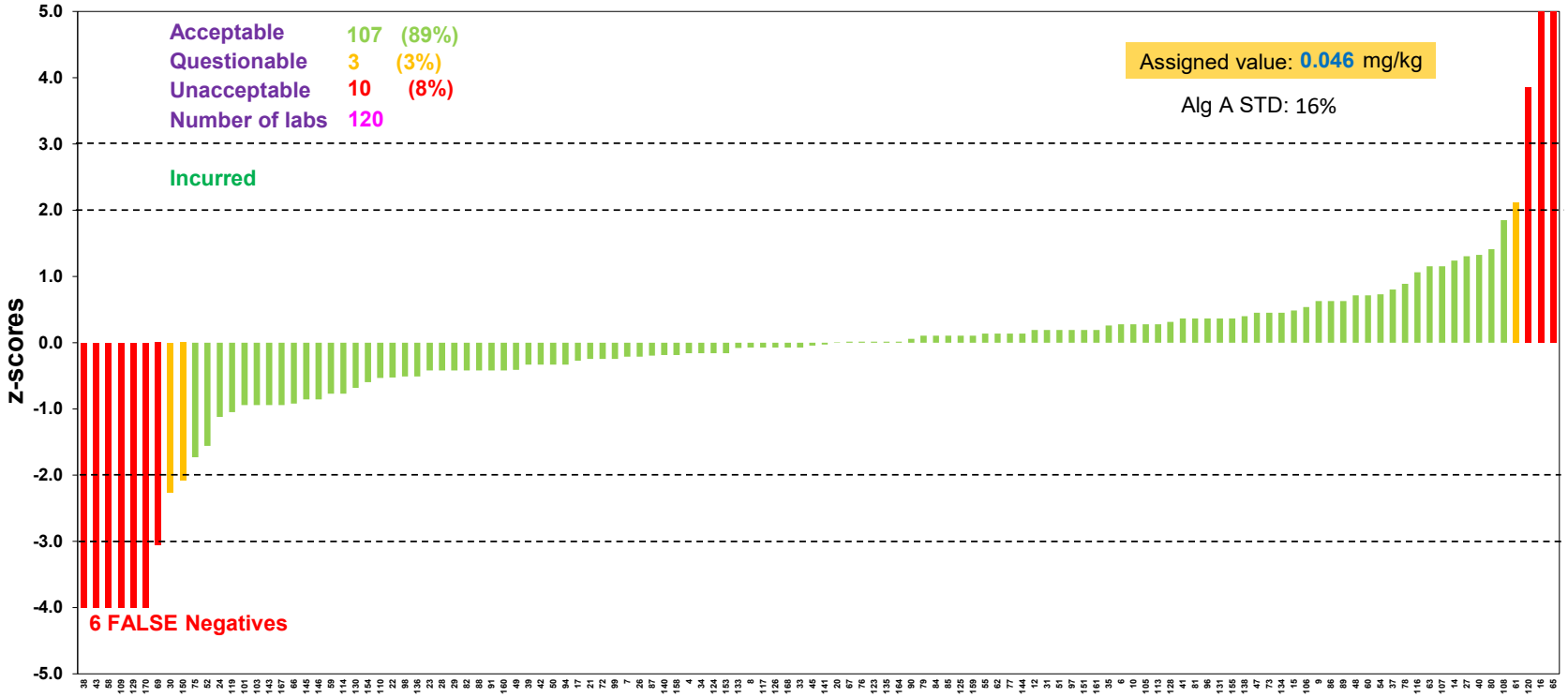
Dimethomorph

EU and EFTA Laboratories



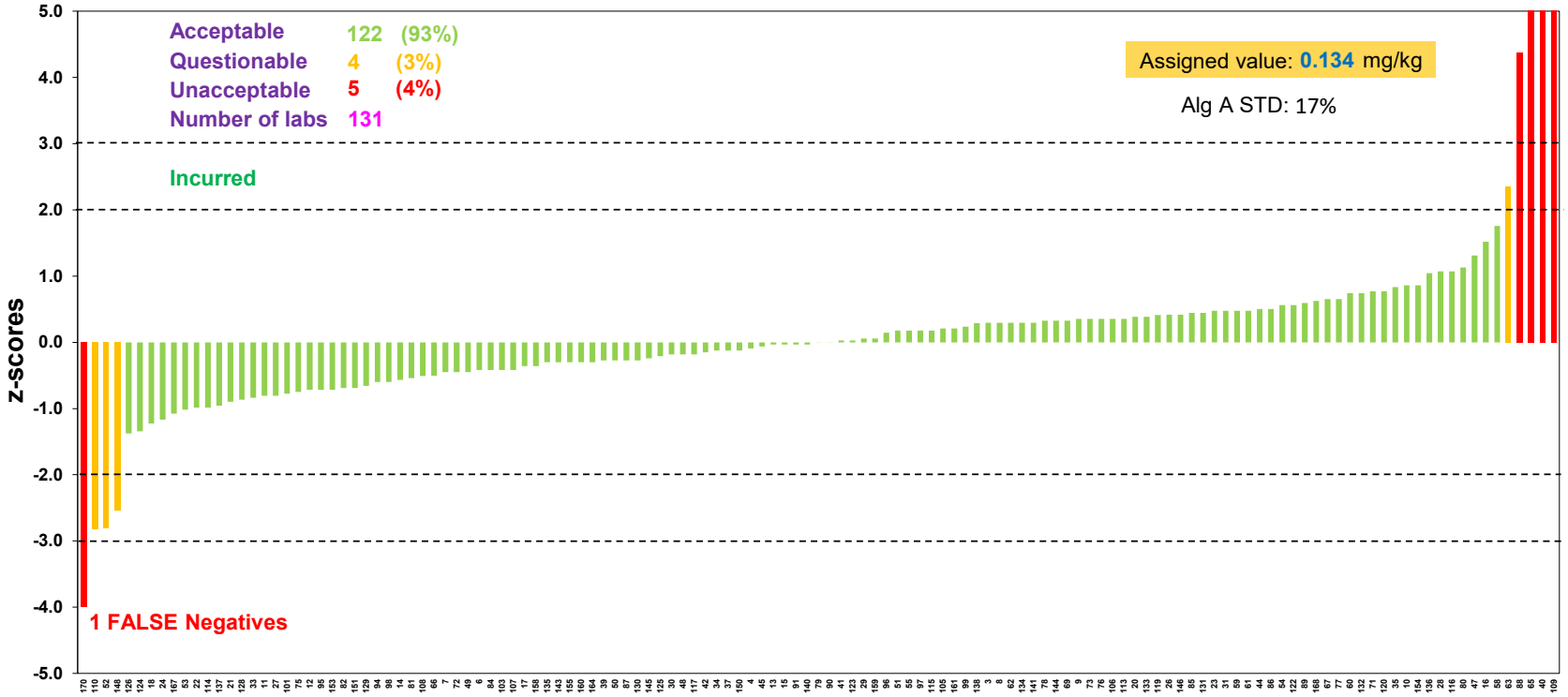
Flonicamid

EU and EFTA Laboratories



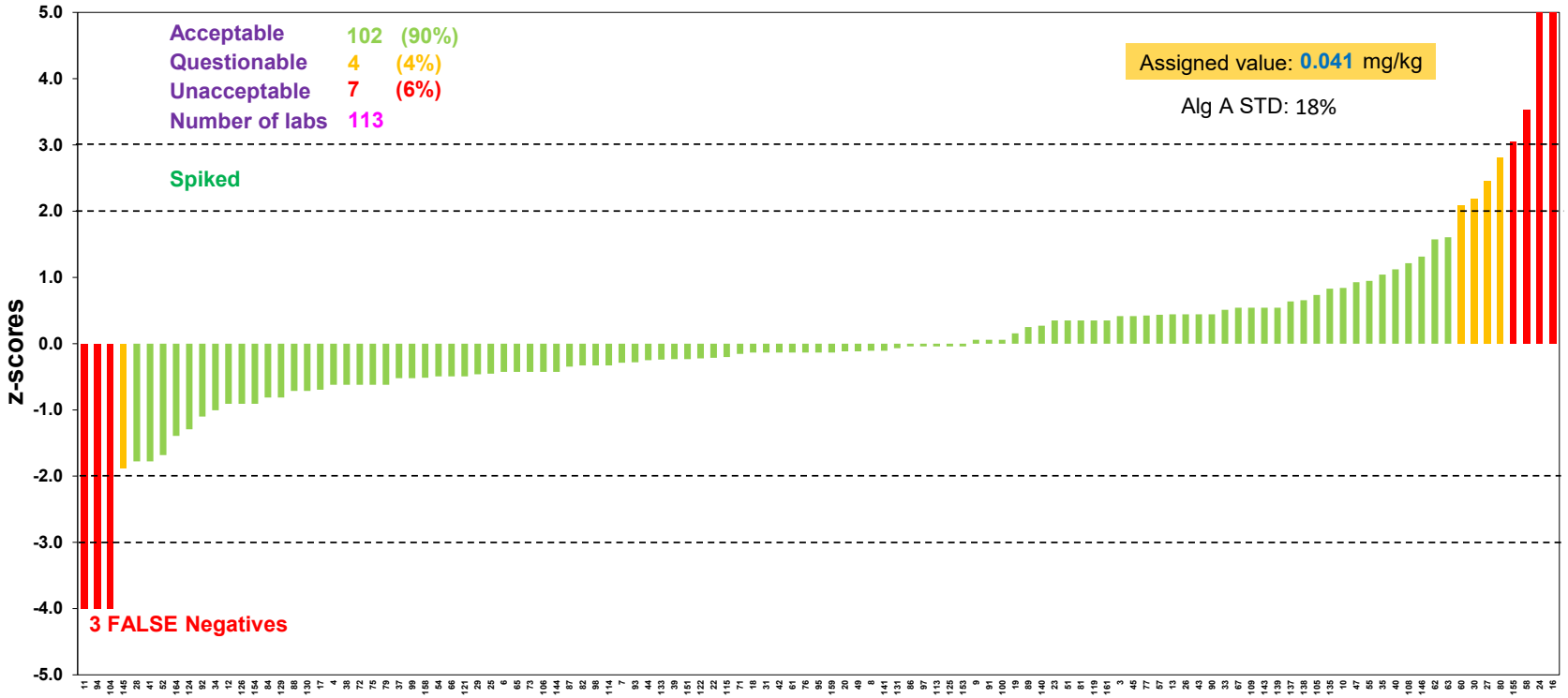
Fluopyram

EU and EFTA Laboratories



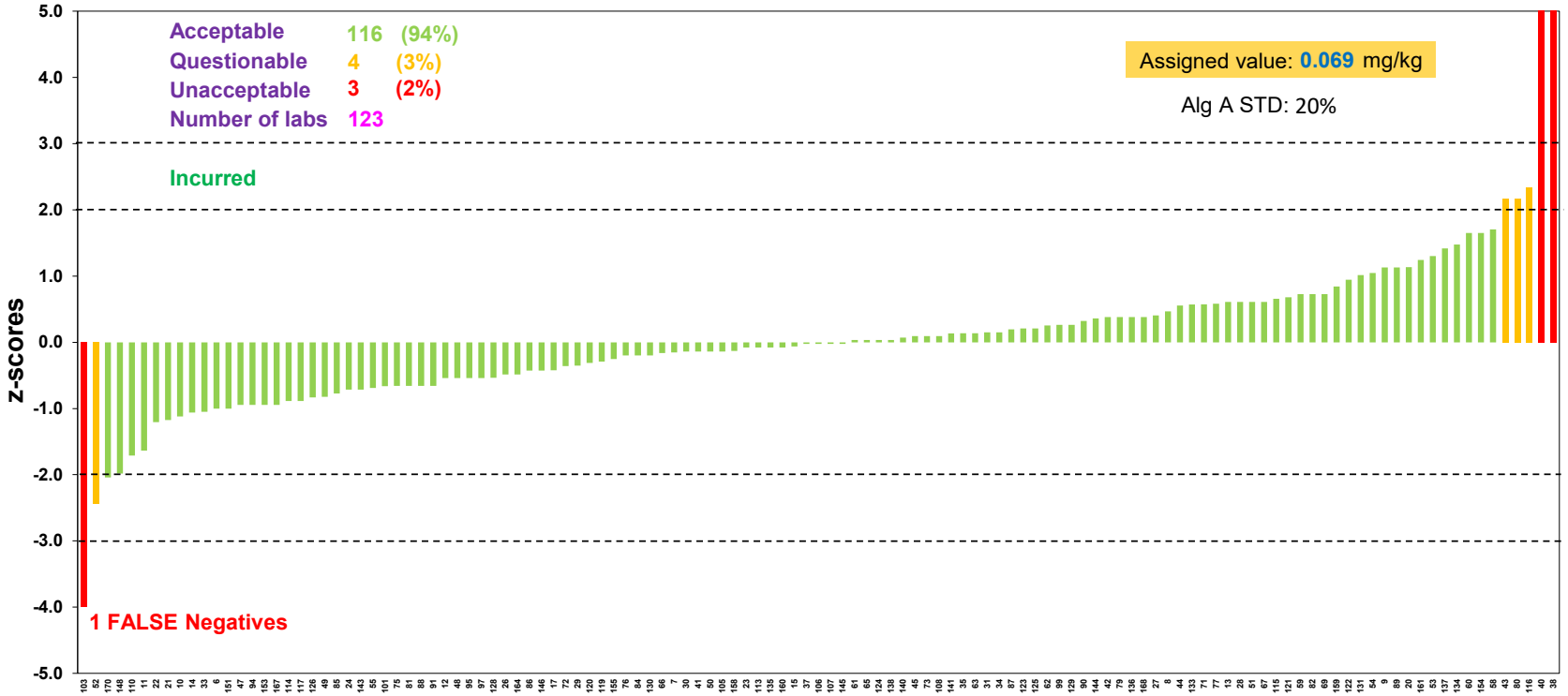
HCH-beta

EU and EFTA Laboratories



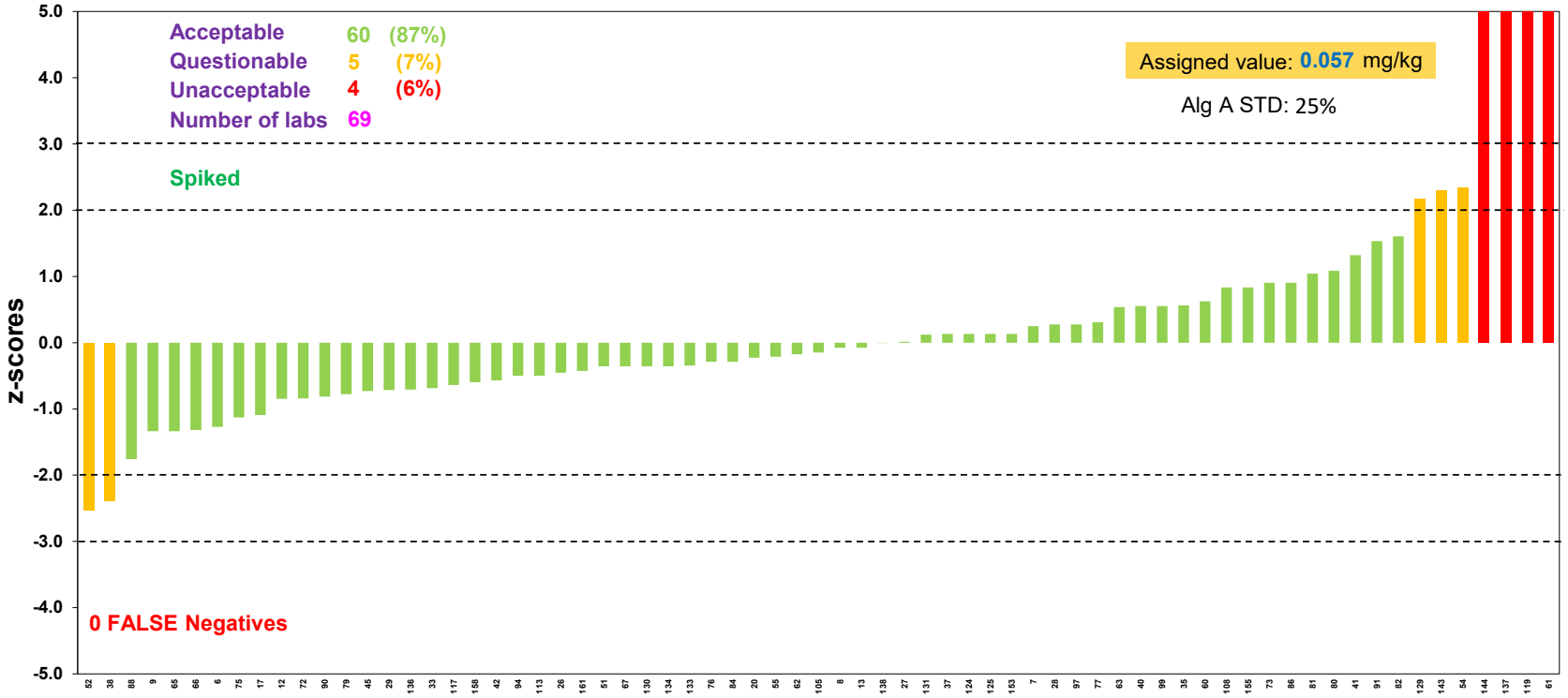
Metconazole

EU and EFTA Laboratories



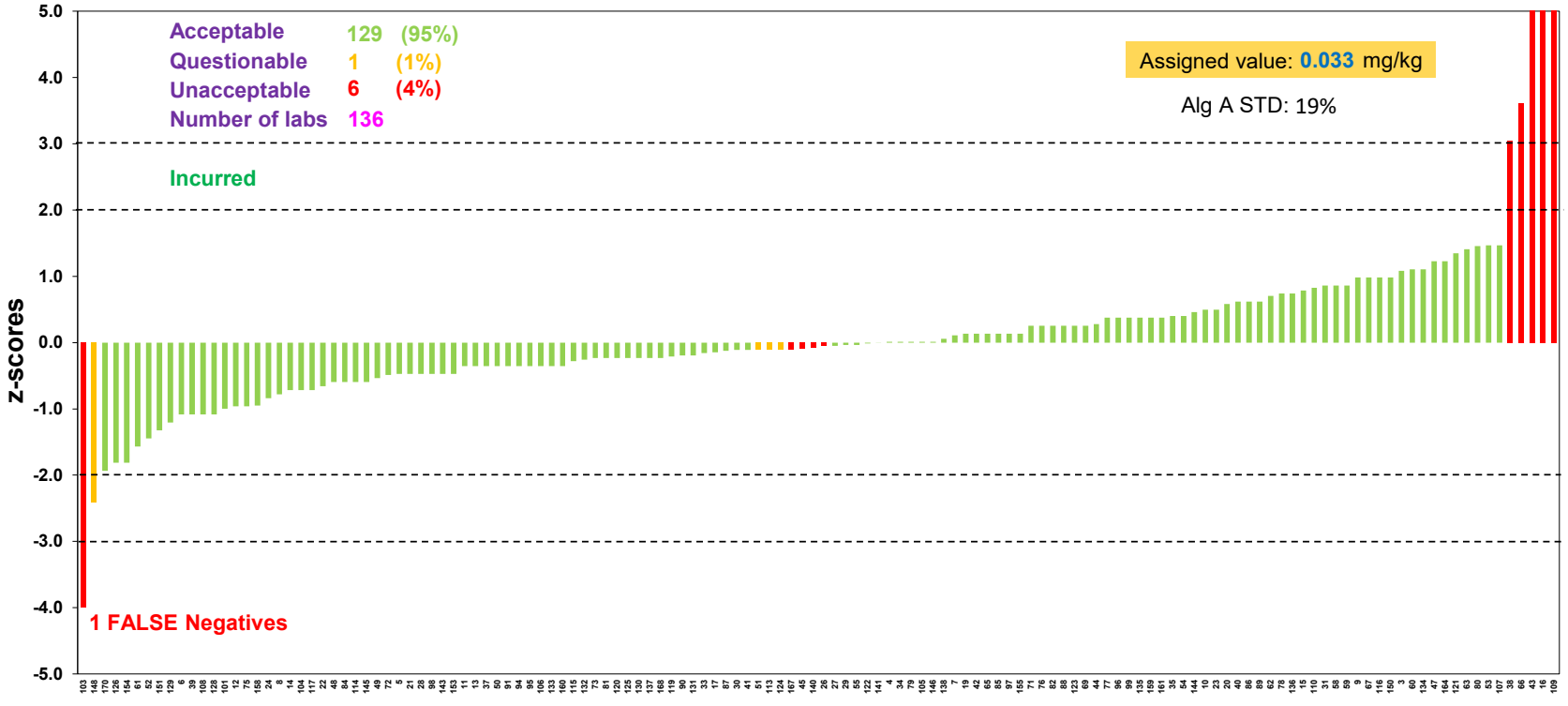
Phenmedipham

EU and EFTA Laboratories



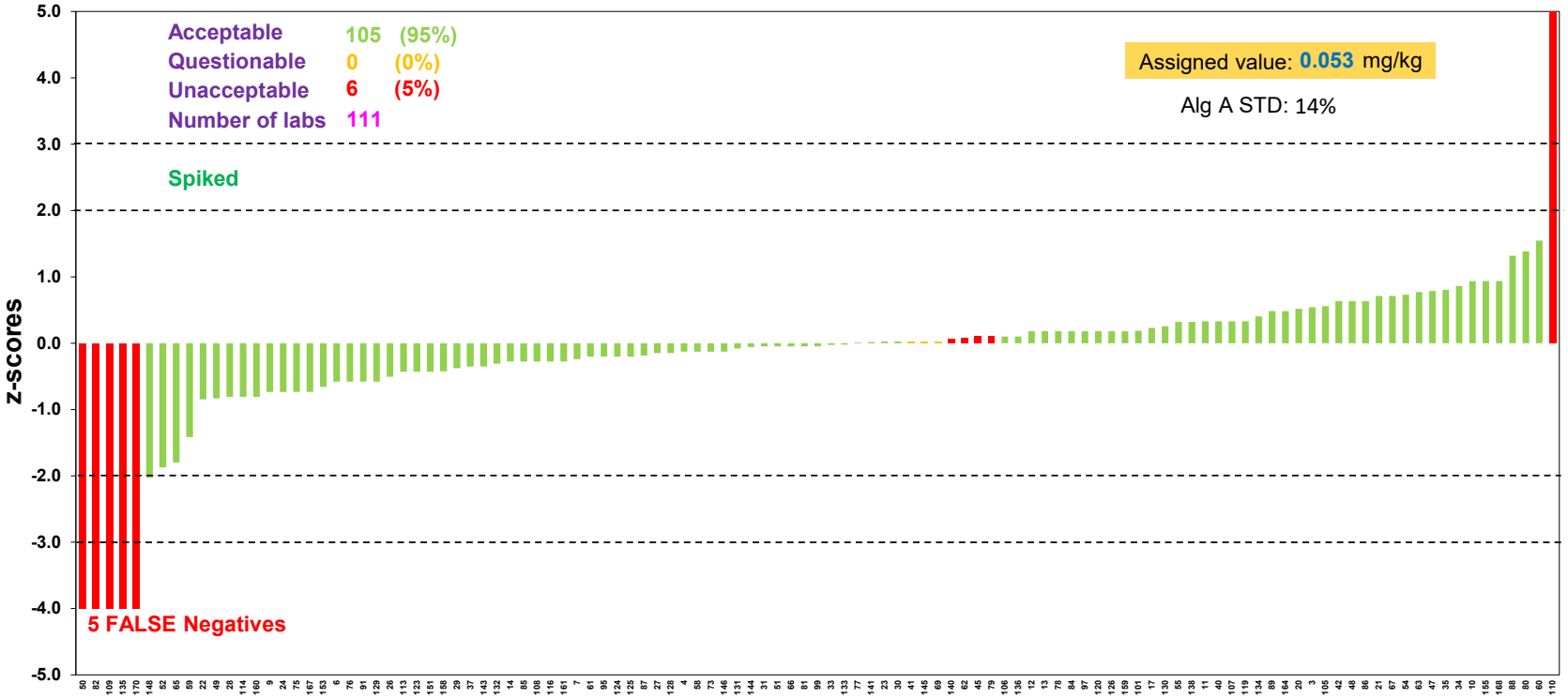
Pirimicarb

EU and EFTA Laboratories



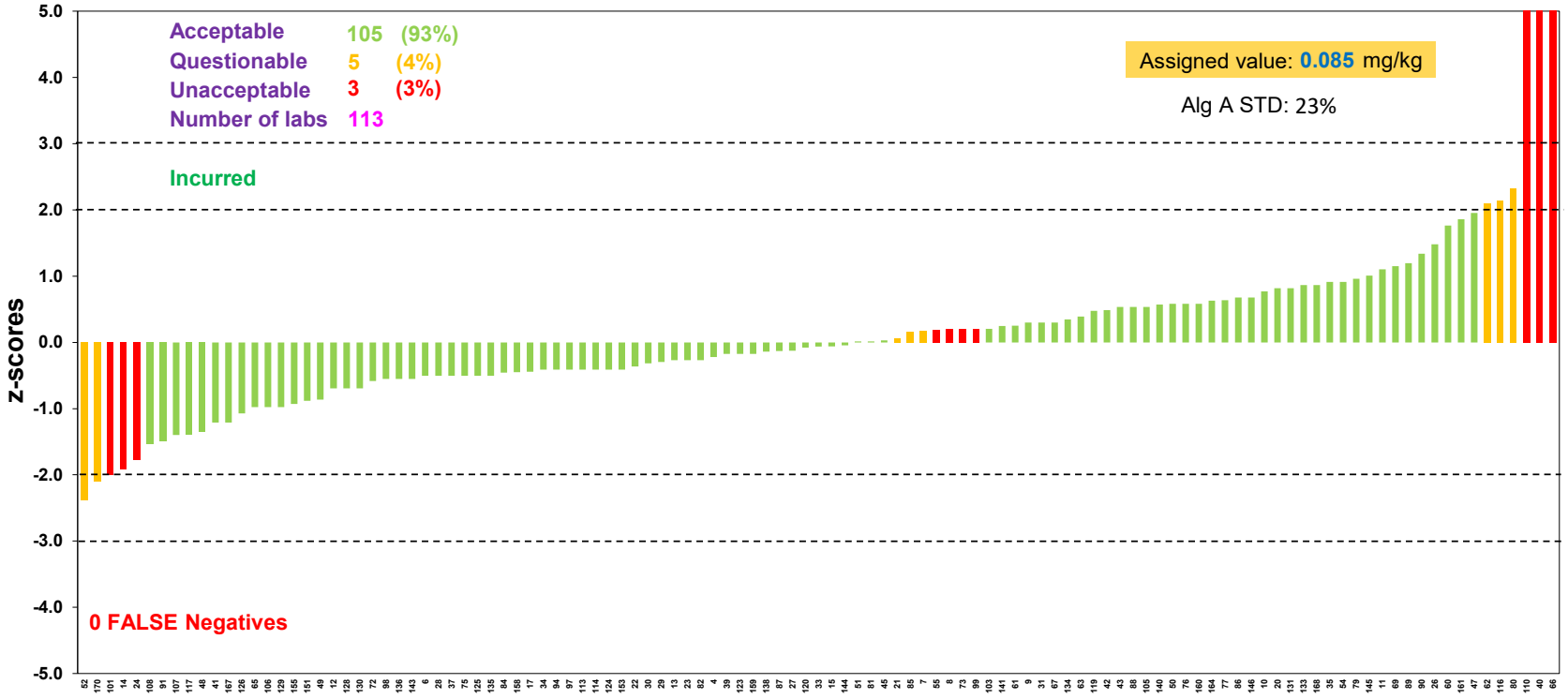
Pirimicarb-desmethyl

EU and EFTA Laboratories

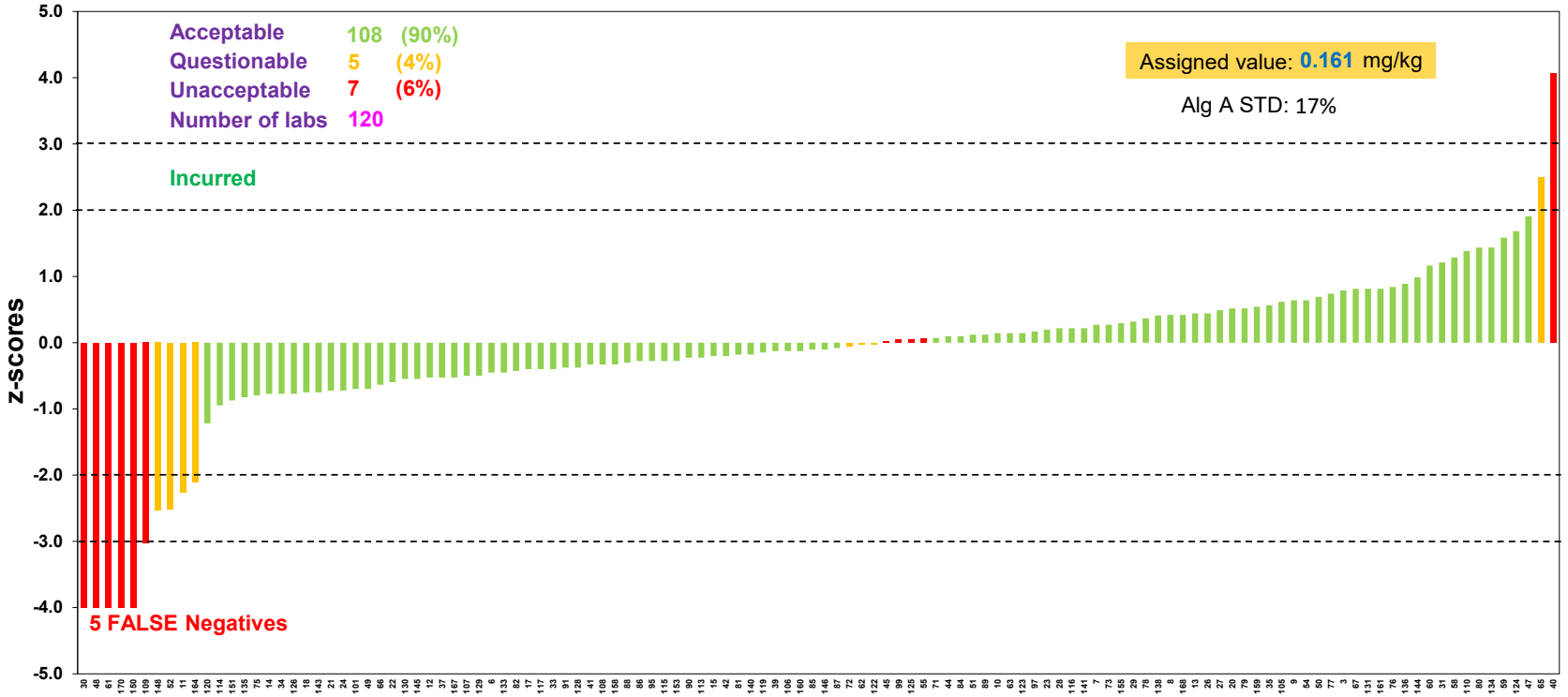


Proquinazid

EU and EFTA Laboratories

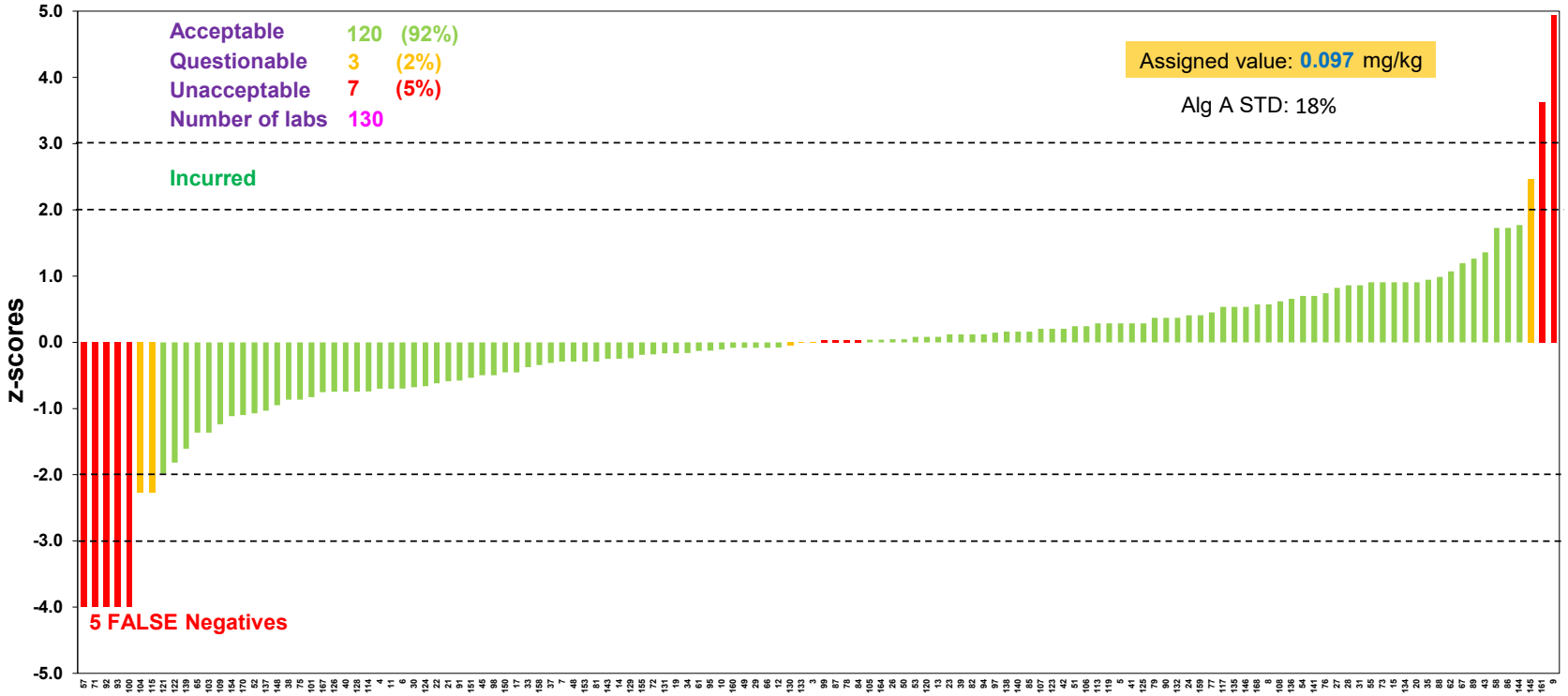


Prothioconazole-desthio EU and EFTA Laboratories



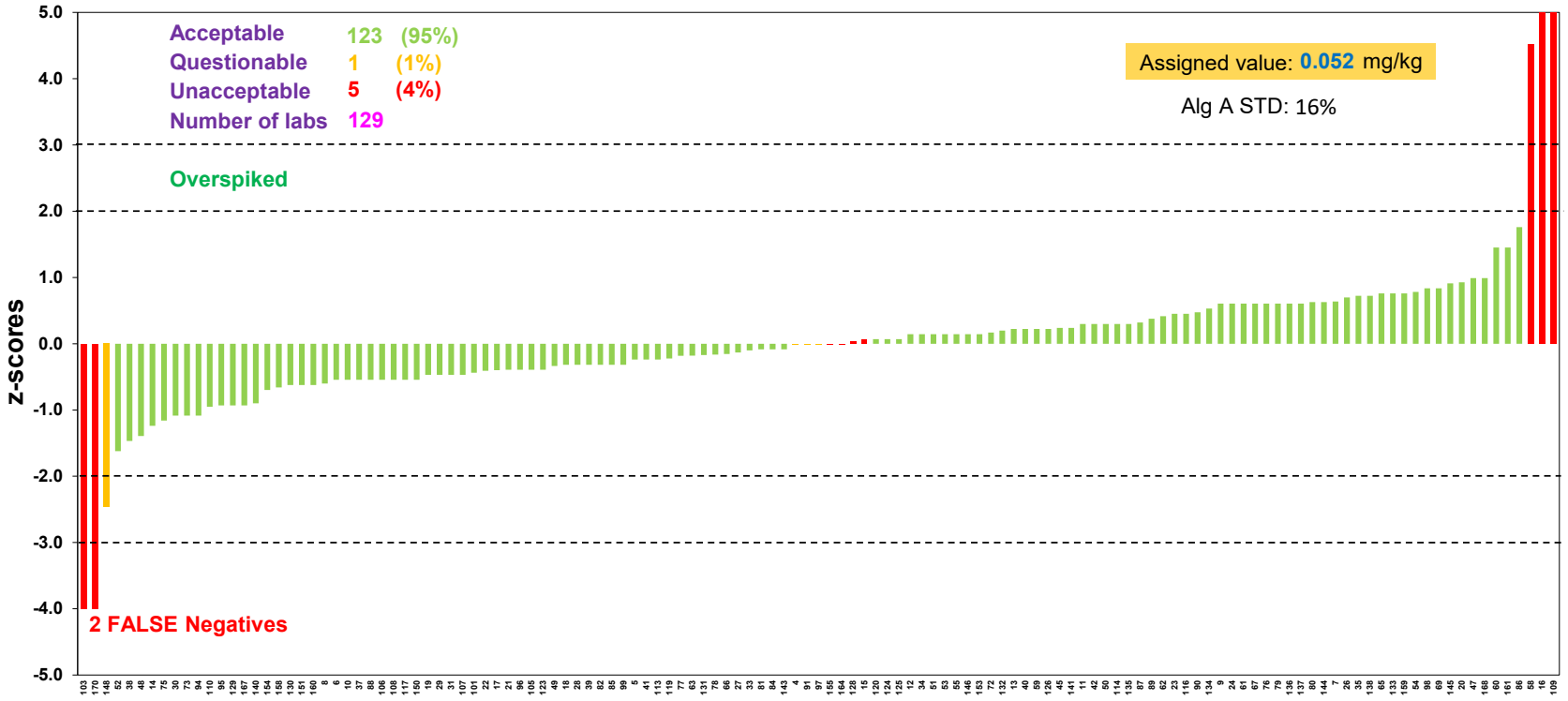
Pyraclostrobin

EU and EFTA Laboratories



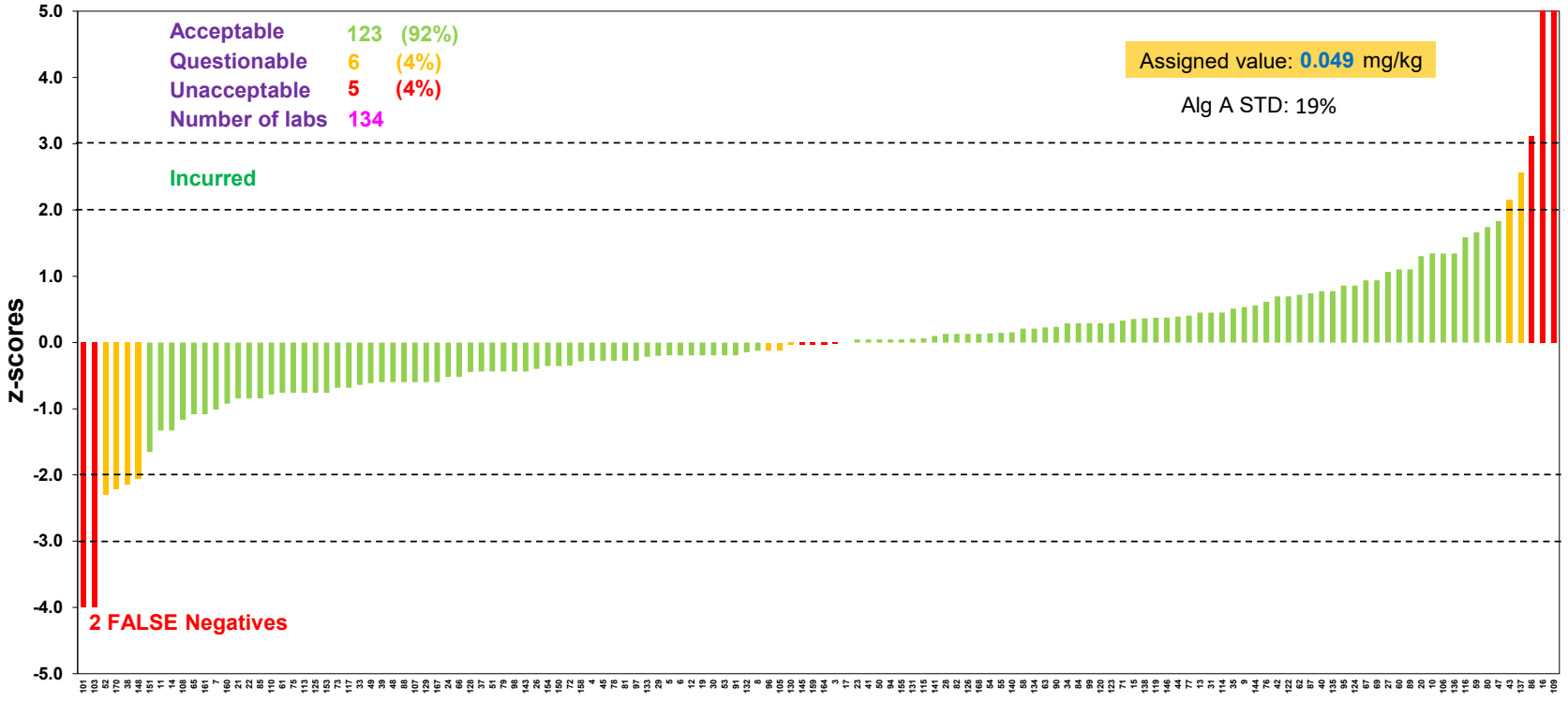
Pyriproxyfen

EU and EFTA Laboratories



Trifloxystrobin

EU and EFTA Laboratories



GENERAL PROTOCOL

for EU Proficiency Tests on Pesticide Residues in Food and Feed

Introduction

This protocol contains general procedures valid for all European Union Proficiency Tests (EUPTs) organised on behalf of the European Commission, DG-SANTE[1] by the four European Union Reference Laboratories (EURLs) responsible for pesticide residues in food and feed. These EUPTs are organised for 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 the official controls Regulation (EU) No. 2017/625[3]:

- EURL for Fruits and Vegetables (EURL-FV),
- EURL for Cereals and Feedingstuff (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 they can use to demonstrate their analytical performance and compare themselves with other participating laboratories.

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[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.

EUPT-organisers and Scientific Committee

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

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

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

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

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

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

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 analytes to be present in the Test Item and the approximate concentrations at which they should be present.

The EUPT-SC typically meets once a year, after all EUPTs of the season have been conducted and preliminarily evaluated by the four pesticide EURLs. The aim of these meetings is to discuss the EUPT-results, especially where case-by-case decisions are needed. PT plans for the next EUPT season and, if needed, possible changes in the EUPT-General Protocol are also discussed during these meetings. The main topics and decisions on these meetings are documented.

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

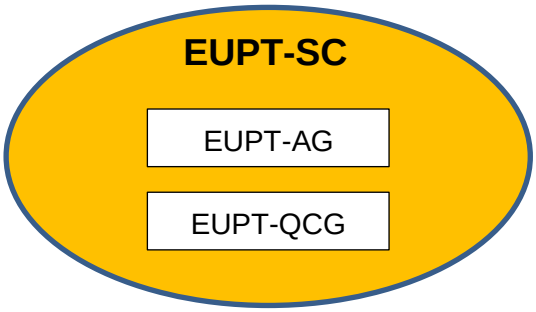

Figure 1: Composition of EUPT-Scientific Committee

The present EUPT General Protocol (EUPT-GP) was drafted by the EURLs and reviewed by the EUPT-SC. Follow the link to access a website giving an *overview of the GP-versions*.

EUPT Participants

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

- Art 38 (2) of Regulation (EU) No. 2017/625[3] Art. 28 (3) of Reg. (EC) No. 2005/396 (for all OfLs analysing for pesticide residues within the framework of official controls of food or feed[6])
- Art. 101 (1)(a) of Regulation (EU) No. 2017/625[3] (for all NRLs)

Every year, shortly before launching the registration period of the first of the four EUPTs in a given EUPT-Season, all OfLs and NRLs are asked to update their routine scope of commodities as well their contact information within the EURL-DataPool. Based on this information the OfLs are classified into those that are obliged and those that are eligible participate in each of the EUPTs to be conducted within a given year..

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

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

[6] Official controls in the sense of Regulation (EU) 2017/625. This includes labs involved in controls within the framework of national and/or EU programs, as well as labs involved in import controls according to Regulation (EU) 2019/1793 (which repealed Regulation (EC) No. 2009/669).

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

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

Confidentiality and Communication

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

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

As laid down in Regulation (EU) No. 2017/625[3], NRLs are responsible for evaluating and improving their own OfL-Network. On request from the NRLs, the EURLs will provide them with the PT-codes of the participating OfLs belonging to their OfL-Network. This will allow NRLs to follow the participation and performance of the laboratories within their network.

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

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

The official language used in all EUPTs is English.

Announcement / Invitation Letter

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

Target Pesticide List

This list contains all analytes (pesticides and metabolites) to be sought for, along with the Minimum Required Reporting Levels (MRRLs) valid for the specific EUPT. The MRRLs are typically based upon the lowest MRLs found either in Regulation (EC) No. 2005/396 and Regulation (EU) No. 2016/128 (Baby Food Directive). Labs must express their results as stated in the Target Pesticides List.

Specific Protocol

For each EUPT the organising 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 analysis of two replicate analytical portions, taken from at least ten randomly chosen units of treated Test Item. Measurements should be conducted in random order. The homogeneity test data are statistically evaluated according to ISO 13528:2022, Annex B[7] or to the International Harmonized Protocols jointly published by ISO, AOAC and IUPAC[8]. 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 analytes spiked at the same time, the overall distribution of the participants' results (CV*), the analytical difficulties faced during the test, knowledge of the analytical behaviour of the compound in question), may decide to overrule the test. The reasons of this overruling have to be transparently explained in the Final EUPT-Report. For certain analytes with comparable properties, an equivalent distribution within the sample can be expected if they were spiked/used at simultaneously. The homogeneity test, of one or more of these analytes, may thus be skipped or simplified. If, however, the distribution of participants' results for an analyte that was not or not fully tested for homogeneity, is found to be atypically broad, compared to the tested analytes, the EUPT-SC may decide that a homogeneity test should be performed a posteriori.

[7] ISO 13528:2022: 'Statistical methods for use in proficiency testing by interlaboratory comparisons", International Organization for Standardization.

[8] Thompson M., Ellison S.L.R., Wood R., "The International Harmonized Protocol for the proficiency testing of analytical chemistry laboratories" (IUPAC Technical Report). Pure Appl. Chem. 2006, 78, 145 – 196

Stability of the analytes contained in the Test Item

The Test Items will also be tested for stability - according to ISO 13528:2022, Annex B[7]. 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 analytes 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 EUPT-Report, considering all relevant aspects such as the distribution of the participant's results (CV*).

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

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

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

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

taken by the EUPT-SC considering all relevant aspects including the duration and conditions of the shipment to the laboratory as well as the feedback by the laboratory.

Methodologies to be used by the participants

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

General procedures for reporting results

Participating laboratories are responsible for reporting their own quantitative results to the organiser within the stipulated deadline. Any analyte 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 analytes detected should be expressed in 'mg/kg' unless indicated otherwise in the specific protocol. Laboratories should not report results below their reporting limits.

Correction of results for bias

According to the DG-SANTE Guidelines, the result of an analyte needs to be adjusted for method bias if the bias exceeds 20%. Unless a method is used that inherently accounts for method bias (see cases a-c below), laboratories are required to report the recovery (in percent), and whether their results were corrected mathematically using a recovery factor reflecting the reported recovery.

The EUPT-Panel will examine whether results, for which no correction for recovery was undertaken, should be omitted from the population used for calculating the assigned value.

When the laboratory uses any of the following approaches inherently accounting for method bias, this needs to be indicated in the appropriate fields within the Web-Tool. In such cases, reporting of the recovery rate is not mandatory.

a) use of stable isotope labelled analogues of the target analytes as Internal Standard (ILISs), added to the analytical portion at an early stage of the procedure

b) 'procedural calibration' approach

c) 'standard addition' approach with additions of analyte(s) to the analytical portions before extraction.

Methodology information

All laboratories are requested to provide information on the analytical method(s) they have used. The Web-Tool, which also serves for submitting analytical results, is typically used for collecting method information.

The collection of method information is considered very important by the EUPT-SC, as it facilitates the interpretation of results and the identification of analytical patterns associated with systematically biased results. A compilation of the methodology information submitted by all participants may be presented in an Annex of the Final EUPT-Report or in a separate report. Where the initial method information provided by the participating laboratories is not sufficient for evaluating methodology-related errors, or where additional information critical for results evaluation is needed, the EURLs and/or the EUPT-Panel may decide to conduct specific follow-up surveys among the concerned laboratories. If no sufficient information on the methodology used is provided, the organisers reserve the right not to accept the analytical results reported by the participants concerned or even refuse participation in the following PT.

Where necessary the methods are evaluated and discussed within the EUPT-SC, especially in those cases where the result distribution is not unimodal or very broad (e.g. CV^* > 35 %).

Results evaluation

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

- ***False Positive (FP) results***

These are results of analytes 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 analytes. In certain instances, case-by-case decisions by the EUPT-SC 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 (FN) results***

These are results for analytes 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 analytes 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-SC 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-SC may decide to take case-by-case decisions in this respect after considering all relevant factors such as the result distribution and the RLs of the affected labs.

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

To minimise the influence of out-lying results on the statistical evaluation, the assigned value x_{pt} (= consensus concentration) will typically be estimated using the robust estimate of the participant's mean (x^*) as described in ISO 13528:2022[9], taking into account the results reported by EU and EFTA countries laboratories only. In special justifiable cases, the EUPT-Panel may decide including results submitted by laboratories not belonging to the EU-/EFTA-OfLs network or to even to only use the results of a subgroup of ('expert') laboratories that have previously repeatedly demonstrated good performance for the specific or similar compounds.

Furthermore, the EUPT-Panel may decide to eliminate certain results traceably associated with bias or gross errors for establishing the assigned value (see 'Omission or Exclusion of results' below).

Since the assigned values of the EUPT analytes are typically generated using robust mean concentrations of participant results (x_{pt}), which are generated by a variety of analytical standards and methods, the assigned values of EUPTs are typically metrologically not traceable.

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

Before estimating the assigned value, results associated with obvious mistakes have to be examined to decide whether they should be removed from the population. Such gross errors may include incorrect recording (e.g. due to transcription errors by the participant, decimal point faults or transposed digits, incorrect unit), calculation errors (e.g. missing factors), analysis of a wrong sample/extract (e.g. a spiked blank), use of wrong concentrations of standard solutions, incorrect data processing (e.g. integration of wrong peak), inappropriate storage or transport conditions (in case of susceptible compounds), and the use of inappropriate analytical steps or procedures that demonstrably lead to significantly biased results (e.g. employing inappropriate internal standards or analytical steps or conditions leading to considerable losses, due to degradations, adsorptions, incomplete extractions, partitioning etc.). Where the organisers (e.g. after the publication of the preliminary report) receive information of such gross errors, having a significant impact on a

[9] ISO 13528:2022 '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).

generated result, the affected results will be examined on a case-by-case basis to decide whether, or not, they should be excluded from the population used for robust statistics. Results may also be omitted e.g. if an inappropriate method has been used even if they are not outliers. All decisions to omit/exclude results will be discussed with the EUPT-SC and the reasoning for the omission of each result clearly stated in the Final EUPT-Report. However, z scores will be calculated for all results irrespective of the fact that they were omitted from the calculation of the assigned value.

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

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

- ***Uncertainty of the assigned value***

The uncertainty of the assigned values $u(x_{pt})$ is calculated according to ISO 13528:2022 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-SC 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 13528:2022 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).

Based on experience from previous EUPTs[10], a percentage FFP-RSD of 25 % is currently used for all analyte-matrix combination, with the target standard deviation being calculated as follows:

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

[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. DOI:10.1021/jf104060h

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

For informative purposes the robust relative standard deviation (CV*) of the participants results is calculated according to ISO 13528:2022; Chapter 7.7 following Algorithm A in Annex C (so called "consensus approach").

- ***z scores***

This parameter is calculated using the following formula:

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

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

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

Following ISO 17043:2010[11], z scores will be classified as follows::

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

All false negatives will be assigned a z score of -4. These z scores will typically appear in the z score histograms and will be used in the calculation of combined z scores.

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

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

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

- ***Category classification***

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

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

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

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

For evaluation of the overall performance of laboratories within Category A, the Average of the Squared z score (AZ^2)[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 AZ^2, z scores > 5 will be set as 5. Based on the AZ^2 achieved, the laboratories are classified as follows:

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

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

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

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

Publication of results

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

[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.

The Final EUPT-Report will be published after the EUPT-SC has discussed the results. Taking into account that the EUPT-SC meets normally only once a year (typically in late summer or autumn) to discuss the results of all EUPTs organised by the EURLs earlier in the year, the Final EUPT-Report may be published up to 12 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 EUPT organiser will deliver a Certificate of Participation to each participating laboratory showing the z scores achieved for each individual analyte, the classification into Categories, and if deemed necessary also combined z scores. The certificates of participation will be uploaded onto the EURL-DataPool where they can be accessed by the concerned laboratories only.

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-SC will decide whether a corrigendum will be issued and how this should look like. The online version of the Final EUPT report will be replaced by the new one and all affected labs will be contacted.

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

Follow-up activities

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

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

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

NRLs will be considered as **underperforming in relation to scope** if in at least two of the last four EUPTs falling within their responsibility area they: a) haven't participated, or b) targeted less than 90% of the compulsory analytes in the target lists (80% for SRM-compounds), or c) detected less than 90% of the compulsory compounds present in the test items (80% for SRM-compounds). Additionally, NRLs that obtained AZ^2 higher than 3 (AAZ higher than 1.3 for SRM-compounds) in two consecutive EUPTs of the last four EUPTs, will be considered as **underperforming in accuracy**. As soon as underperformance of an NRL is detected, a two-step protocol established by DG-SANTE will be applied[14]:

Phase 1:

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

Phase 2:

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

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

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

Disclaimer

The EUPT-SC 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-CF17 (2023)

(last updated: 20 January 2023)

Introduction

This protocol is complementary to the General Protocol for EU Proficiency Tests for Pesticide Residues in Food and Feed (9th Edition). The current proficiency test covers pesticides that are determined by Multi Residue Methods. This EUPT is to be performed by all National Reference Laboratories for Cereals and/or Feeding stuffs (NRL-CFs) as well as by all official EU laboratories (OfLs) responsible for official pesticide residue controls on feeding stuff, as far as their scope overlaps with that of the EUPT-CF17.

Test Item (Test Material)

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

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

Analytical Parameters

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

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

Amount of Test Item

The participants will receive:

- approximately 100 g of wheat kernels Test Item with incurred and spiked pesticides

Blank material will not be distributed to the participants.

Shipment of Test Items

The Test Items are planned to be shipped on 6 February 2023.

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

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

Instructions on Test Item Handling

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

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

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

EUPT Webtool and Deadlines

To select pesticide scope and report results and method information, the participants should log in to the **EUPT Webtool** using the username send by email, the password can be retrieved via https://guest.dtu.dk/Sites/GuestLogin/RetrievePassword.aspx using your email address or your username. Please, update the password every year.

Selection/deselection of scope: The analytical scope must be selected prior to the shipment of the samples. This is done via the **EUPT Webtool**. The scope selection subpage will be open from 22 January to 6 March 2023. As default all mandatory pesticides are preselected.

Results and method submission: The **EUPT Webtool** will be accessible from 7 February 2023 for sample receipt acknowledgement and submission results and method information.

The deadline for submission is 15 March 2023 at 23.00 CET.

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

Test Item Receipt and Acceptance: Once the laboratory has received the Test Items it must report to the organiser, via the **EUPT Webtool**, the date of receipt, and its acceptance. If the laboratory does not respond by 10 February 2023 at 12.00 CET, the Organiser will assume that the Test Items have been received and accepted.

If participants have not received the Test Items by **the 10 February 2023 at noon**, they must inform the Organiser immediately by e-mail to eurl-cf@food.dtu.dk.

Reporting Quantitative Results:

Results should not be reported where a pesticide

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

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

Reporting of supplementary information in case of false negative results

In case of false negative results, the affected laboratories will be asked to provide details on the methodology used after the deadline for result submission. This has also to be done by accessing EUPT Webtool. Deadline for this is 11 May 2022.

Follow-up actions

In accordance with Art. 32 1b of Regulation (EC) No 2017/625, underperformance of any NRL-CF in comparative testing will be followed by EURL-CF.

Documents

All documents related to EUPT-CF17 can be found on EUPT-CF15 website.

https://www.eurl-pesticides.eu/docs/public/tmplt_article.asp?LabID=400&CntID=1231&Theme_ID=1&Pdf=False&Lang=EN

Calendar

Activity	Dates
Announcement Calendar Target Pesticide List	November 2022
EUPT-Registration Website open	December 2022
Deadline for registration	9 January 2023
Specific Protocol published	23 January 2023
Website for selecting pesticide scope open	23 January 2023
Website for selecting pesticide scope closed	6 February 2023
Distribution of Test items	6 February 2023
Deadline for receipt and acceptance of Test Materials	within 24 hr on receipt
Deadline for Result Submission	6 March 2023 at 23.00 CET
Deadline for submission of additional method information for false negative results	15 March 2023 at 24.00 CET
Preliminary Report (only compilation of results) published	15 May 2023
Final Report published	December 2023

Participation Fees

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

- 250 €

The participation fees for laboratories from third countries will be:

- 400 €

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.

If the participant ask DTU to issue a new invoice because additional/new information are needed on the invoice, or just want a copy of the original invoice, that may add additional cost due to the administrative workload.

Any questions concerning invoices must be directed to Mikkel Lau Mikkelsen, mlami@dtu.dk at the financial department of DTU.

Contact information:

DTU Food
National Food Institute

PT Reg.nr. 516

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