DTU Fødevareinstituttet
DTU National Food Institute

EU Reference Laboratory on Cereals & Feeding stuff

Proficiency Test on pesticide residue rye kernels

DANAK
PT Reg.no. 516

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DTU Fødevareinstituttet
DTU National Food Institute

EU Reference Laboratory on Cereals & Feeding stuff
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Pesticide Residues in Rye Kernels

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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 nineteenth 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, EUPT-CF16 on barley kernels, EUPT-CF17 on wheat kernels and EUPT-CF18 wheat straw. 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 rye kernels used for EUPT-CF19 were treated both with formulation in the field and post-harvest in the laboratory. Participation in EUPT-CF19 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.

Lyngby 16 December 2025

Accepted by

Mette Erecius Poulsen
Director of EURL for pesticide residues in Cereal and Feedingstuff

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EUROPEAN COMMISSION EURL PROFICIENCY TEST ON PESTICIDE RESIDUES IN CEREALS EUPT-CF19, 2025

1. INTRODUCTION

On 28 October 2024 the announcement of the 19th European Commission's Proficiency Test on Cereals and Feed (EUPT-CF19) 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 174 individual compulsory compounds and 62 voluntary compounds 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-CF19, were provided via the homepage. Laboratories were able to register online from 27 of November 2024 to 6 of January 2025. In total 151 laboratories from EU and EFTA countries agreed to participate in the test as well as 8 laboratories from EU-Candidate States and Third Countries.
The rye kernels were sprayed in the field with 20 pesticides. The cultivation was performed in 2024 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 ten 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 rye kernels. The Test Items were shipped to participants on 27 January 2025 and the final deadline for submission of results to the Organiser was the 3 March 2025 The deadline for submission of additional information for false negative results was the 12 March 2025. 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 1**). 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 151662:2018): Cold water was added to five grams of 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 5 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 (rye 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 0.005 mg/kg.

1.3 Preparation of the Test Item

The field spraying was performed in 2024 in Denmark and organised by Danish Centre for Food and Agriculture at Aarhus University. Approximately, 30 kg of the harvested rye kernels where were used for this PT.
It was decided to additionally spike in the laboratory with ten pesticides, which were either not included in the field treatments or where residues were too low for the evaluation (**Table 1**). Spiking was performed in the laboratory of the EURL-CF using formulations or pure standards. Seven portions of 1.4 kg of the field -treated rye kernels was spiked and subsequently mixed with 20 kg of field treated rye kernels and homogenised thoroughly. One-hundred-gram portions of the rye 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.
As indicated in Table 1, technical cypermethrin (Cyperb 100) was applied during field treatment; consequently, all isomers, including alpha-cypermethrin, were present in the test material.

Although flonicamid was applied in the field, no parent compound residues were detected. Only its two degradation products, TNFG and TNFA, were identified. Both compounds are associated with single-residue analytical methods and were therefore excluded from the target pesticide list. Although pirimicarb (Pirimor) was applied in the field no residues of this pesticide were detected. Prothioconazole was rapidly transformed into prothioconazole-desthio, and no parent compound residues were present rye kernels. Spirotetramat was metabolized to BY108330-enol; however, the concentration was low and therefore supplemented through overspiking.

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

Pesticides	Application in field	Spike in laboratory	Formulation/standard
Aclonifen	x		Fenix
Azoxystrobin	x	x	Amistar Gold
Bixafen	x		Siltra Xpro
Chlorantraniliprole	x	x	Coragen
Cypermethrin	x		Cyperb 100
Cyprodinil	x		Kayak 300 EC
Difenoconazole	x		Amistar Gold
Dimethomorph	x		Cabrio Duo
Flonicamid	x		Teppeki
Fludioxonil	x	x	BAS 769 00F
Isoprothiolane		x	Analytical Standard
Mefentrifluconazole	x		BAS 769 00F
Metconazole	x	x	Juventus 90
Metribuzin	x	x	Sencor SC 600
Pirimicarb	x		Pirimor
Proquinazid	x		Talius
Prosulfocarb	x		Boxer
Prothioconazole	x		Siltra Xpro
Pyraclostrobin	x		Cabrio Duo
Spirotetramat/metabolite BYI08330-enol	x	x	Movento/Analytical Standard
Tau-Fluvalinate	x		Mavrik
Bifenazate-Diazene*		x	Analytical Standard
Chlordane, cis-*		x	Analytical Standard
HCH, beta-*		x	Analytical Standard

*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 ISO 13528:2022 [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 2**.

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 10 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-CF19.

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	$Ss^2 < c$
Aclonifen	0.314	0.0007	0.0020	Pass
Azoxystrobin	0.061	0.0000	0.0001	Pass
Bixafen	0.030	0.0000	0.0000	Pass
Chlorantraniliprole	0.042	0.0000	0.0000	Pass
Cypermethrin	0.040	0.0000	0.0000	Pass
Cyprodinil	0.616	0.0024	0.0067	Pass
Fludioxonil	0.044	0.0000	0.0000	Pass
Isoprothiolane	0.046	0.0000	0.0000	Pass
Mefentrifluconazole	0.046	0.0000	0.0000	Pass
Metconazole	0.069	0.0000	0.0001	Pass
Metribuzin	0.045	0.0000	0.0000	Pass
Proquinazid	0.052	0.0000	0.0000	Pass
Prosulfocarb	0.071	0.0000	0.0001	Pass
Prothioconazole-destio	0.023	0.0000	0.0000	Pass
Pyraclostrobin	0.028	0.0000	0.0000	Pass
Spirotetramat metabolite BYI08330-enol	0.043	0.0000	0.0000	Pass
Tau-Fluvalinate	0.029	0.0000	0.0001	Pass
Bifenazate-Diazene*	0.025	0.0000	0.0000	Pass
Chlordane, cis-*	0.029	0.0000	0.0000	Pass
HCH, beta*	0.025	0.0000	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 13528:2022, Annex B [4]. 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: 27 January 2025

Day 2: 10 February 2025

Day 3: 3 March 2025

The results of the stability test for storage temperature -18 °C are given in Table 3. All pesticides passed the test at the temperature -18 °C. At room temperature metribuzin and bifenazate-diazene 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 3**.

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
Aclonifen	0.362	0.015	0.022	Pass
Azoxystrobin	0.071	0.000	0.005	Pass
Bixafen	0.035	0.001	0.003	Pass
Chlorantraniliprole	0.047	0.001	0.004	Pass
Cypermethrin	0.043	0.000	0.003	Pass
Cyprodinil	0.668	0.001	0.045	Pass
Difenoconazole	0.036	0.000	0.003	Pass
Dimethomorph	0.030	0.001	0.002	Pass
Fludioxonil	0.048	0.000	0.004	Pass
Isoprothiolane	0.051	0.000	0.004	Pass
Mefentrifluconazole	0.053	0.000	0.004	Pass
Metconazole	0.078	0.003	0.005	Pass
Metribuzin	0.047	0.000	0.003	Pass
Proquinazid	0.056	0.000	0.005	Pass
Prosulfocarb	0.070	0.004	0.005	Pass
Prothioconazole-desthio	0.022	0.001	0.002	Pass
Pyraclostrobin	0.031	0.002	0.002	Pass
Spirotetramat-cis-enol	0.053	0.001	0.005	Pass
Tau-Fluvalinate	0.023	0.001	0.002	Pass
Bifenazate-Diazene*	0.029	0.001	0.002	Pass
Chlordane-cis*	0.032	0.000	0.003	Pass
HCH, beta*	0.028	0.000	0.002	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 27 November 2024. All documents related to this EUPT (Calendar, Target Pesticides List, Specific Protocol, General Protocol) were uploaded to the EURL website (EURL | Pesticides in Cereals and Feedingstuffs | EUPT-CF19 on Rye kernels) 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 14 January 2025, 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 27 January 2025, 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 sent.

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 (3 March 2025). 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 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"9 by the organiser, and/or (ii) "not detected" by the overwhelming majority (e.g. > 95 %) of the participating laboratories that had targeted the specific analytes.
Any results reported lower than the MRRL will not be considered as false positives, even though these results should not have been reported. If these results are additionally lower than the lab's reporting limit, they will be attributed with FR ('False Reporting').

2.1.2 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 organisers to treat the PT item and b) detected by the organisers as well as the majority of the participants that had targeted these specific analytes at or above the respective MRRLs. Such results will also be regarded as "not correctly found" when it comes to categorization in A and B based on scope.
Where for a compound present in the PT item a laboratory reports "not detected" and a RL exceeding the assigned value, the result will still be judged as a false negative, despite this practice being consistent and adequate within a routine working environment. The FN judgement should in this case penalize the laboratory for not being able to achieve sufficient sensitivity for the analyte in question.

2.1.3 False reporting (FR)

A result reported below the laboratories own reporting limit (RL) will be evaluated as a 'False Reporting' (FR). If the analytes concerned are present in the test material, z scores will be calculated as for any other numerical results. Furthermore, these results will be included in the population of results for the determination of the assigned value, unless they are excluded for other reasons (e.g. reported by laboratories of non-EU or EFTA countries, generated using biased methods, etc.).

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 13528:2022 [4], taking into account the results reported by only EU and EFTA countries laboratories. In reports, assigned values will be rounded to 3 significant figures if ≥0.01 mg/kg and to 2 significant figures if <0.01 mg/kg (i.e. 0.0078; 0.123; 1.23; 12.3 mg/kg). For the calculation of z scores, the organisers may opt to use assigned values rounded to more significant figures than those stated above.

2.3 Omission or Exclusion of Results

Results reported by laboratories from non-EU/EFTA member states are excluded from the population used to derive the assigned value. Despite the use of robust statistics, all results 10 times higher than the assigned values will be omitted and the assigned values will be recalculated.

2.4 Uncertainty of the assigned value

The uncertainty of the assigned values u(xpt) is calculated according to ISO 13528:2022 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.

2.5 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 13528:2022; Chapter 7.7 (Consensus value from participant results) following Algorithm A in Annex C [4].

2.6 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 17043:2023 [5]:

$|z| \leq 2$ 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.

2.7 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, 151 EU and EFTA laboratories, from 29 different countries (26 EU member states), agreed to participate in this proficiency test. One lab from the EU participants did not submit results. Additionally, eight participants from non-EU Countries registered for the PT.

An overview of results submitted by laboratories from the EU and EFTA can be seen in **Table 4**. All reported analytical results for the pesticide residues are shown in **Table 10 a, b, c, d, f** and in **Appendix 4**. 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 4.**

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]
Aclonifen	110	40	5	73
Azoxystrobin	143	7	0	95
Bixafen	115	35	3	77
Chlorantraniliprole	135	15	0	90
Cypermethrin (sum)	138	12	5	92
Cyprodinil	143	7	0	95
Difenoconazole	143	7	0	95
Dimethomorph	138	12	0	92
Fludioxonil	138	12	1	92
Isoprothiolane	131	19	2	87
Mefentrifluconazole	84	66	12	56
Metconazole	130	20	0	87
Metribuzin	128	22	1	85
Proquinazid	127	23	1	85
Prosulfocarb	125	25	2	83
Prothioconazole-Desthio	128	22	4	85
Pyraclostrobin	135	15	1	90
Spirotetramat-enol	98	52	12	65
Tau-Fluvalinate	133	17	7	89
Chlordane, cis-*	113	37	5	75
HCH, beta-*	121	29	4	81

* 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 = 150). False negatives are included in reported results.

[2]Results for bifenazate-diazene and alpha-cypermethrin were not evaluated due to the validity of the data therefore they were not included in the overview table.

Azoxystrobin, chlorantraniliprole, cypermethrin (sum), cyprodinil, difenoconazole, dimethomorph, fludioxonil and pyraclostrobin were the most frequently analysed compounds with ≥90 % of the labs submitting results for these compounds. Aclonifen, bixafen, chlordane-cis-, HCH- beta-, isoprothiolane, metconazole, metribuzin, proquinazid, prosulfocarb, prothioconazole-desthio and tau-fluvalinate were analysed and reported by 73-89 % of the participants. Mefentrifluconazole and spirotetramat-enol were only analysed and reported by 56 and 65 % of participants.

Results for bifenazate-diazene and alpha-cypermethrin were not evaluated due to the validity of the data.

Bifenazate-diazene cannot be distinguished from its parent compound, bifenazate, when analyzed by GC, as both share identical retention times and transitions/ions. Of the 46 laboratories that included bifenazate-diazene in their scope, only 31 reported results, yielding an Algorithm A standard deviation of 35%. The remaining 15 laboratories did not report results; however, one laboratory indicated that they used GC and that differentiation between bifenazate and bifenazate-diazene was not possible, and two laboratories reported adding ascorbic acid prior to extraction, which converts bifenazate-diazene to bifenazate.
Additionally, 73 laboratories reported results for bifenazate, which was not present in the test item (see Figure 1). These laboratories either employed GC-based methods or used the EURL-SRM method "Analysis of Bifenazate (sum) by the QuEChERS Method using LC-MS/MS with ascorbic acid addition." This method automatically reports results according to the residue definition for bifenazate, which is "Bifenazate (sum of bifenazate plus bifenazate-diazene expressed as bifenazate)."
However, in this proficiency test, the target list included both bifenazate and bifenazate-diazene as separate compounds. The decision not to evaluate the results also considered that the residue definition was mentioned in the target list for bifenazate.

The rye for the test item was treated in the field with technical cypermethrin. Cypermethrin consists of eight stereoisomers, which elute as four distinct peaks in GC analysis when non-chiral columns are used and if the GC run is adequately long (around 30 min). The third eluting peak corresponds to the two alpha-cypermethrin isomers, see Figure 2. As alpha-cypermethrin was added to the voluntary target list the laboratories was expected to report not only results for the cypermethrin (sum of isomers) but also for alpha-cypermethrin.
Fifty-one laboratories included alpha-cypermethrin in their scope. However, only 15 reported results, with concentrations ranging from 0.004 to 0.046 mg/kg, resulting in an Algorithm A standard deviation of 71%. It appears that many laboratories did not realize that quantification should have been based on the third peak in the cypermethrin chromatogram, as they expected to observe a single peak, as if alpha-cypermethrin had been applied during field treatment.

Following the reporting deadline, laboratories that had not submitted results were given the opportunity to do so. In total, 31 results were received, ranging from 0.004 to 0.050 mg/kg, with an Algorithm A standard deviation of 63%. Due to the poor overall performance and the lack of prior guidance provided to laboratories before the proficiency test, no formal evaluation of these results was conducted.

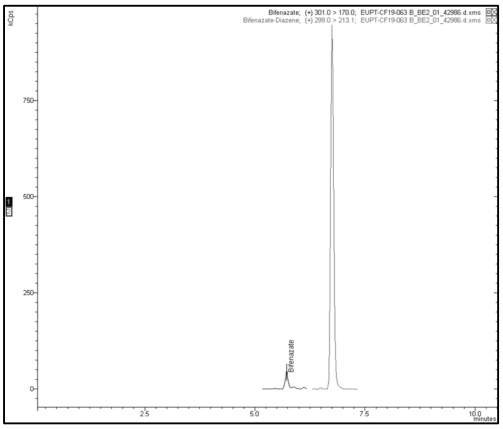


Figure 1. LC-MS/MS chromatogramme of the rye test item showing the presence of bifenazate-diazene.

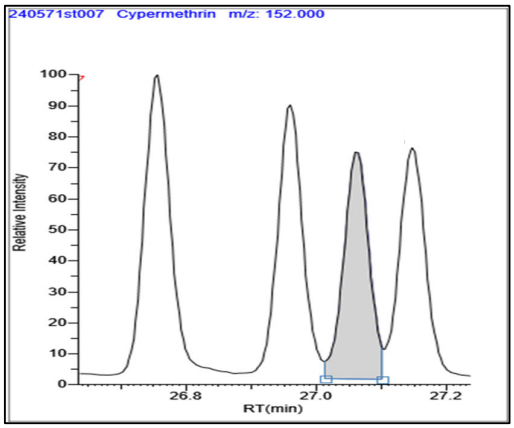


Figure 2. Chromatogram of technical cypermethrin. The third peak corresponds to alpha-cypermethrin.

3.1.1 False positives

Twenty-two participants (all form EU and EFTA) countries reported 25 results for twelve different additional pesticides above the MRRL that had not been used to treat the Test Item (**Table 5**). The pesticides were:
Biphenyl, chlordane-trans, clomazone, dieldrin, endosulfan-alpha, endrin, flonicamid, fluopicolide, HCH-gamma, hexaconazole, spirotetramat and tebuconazole. In all cases the compounds were not detected either by the Organizer, or by the majority of the other participating laboratories. The reported results were therefore considered to be false positives. The likely explanation for the false positive findings for flonicamid is that laboratories reported according to the residue definition for flonicamid. The test item contained only TNFG and TNFA, both degradation products of flonicamid. A similar rationale may apply to the false-positive results for spirotetramat, as only its degradation product BY108330-enol was present in the rye. However, according to the general protocol the individual compounds should be reported not the residue definition.
The false-positive results for chlordane, trans- and HCH, gamma- were presumably due to retention time misidentification, likely confusing these compounds with the correct isomers chlordane, cis and HCH, -beta.

Table 5. False positive results at or above 0.0.05 mg/kg, the concentration detected in mg/kg, the determination technique used, the reporting level and the MRRL in mg/kg.

Lab code	Pesticides	Concentration mg/kg	Determination technique	RL, mg/kg	MRRL, mg/kg
1	Flonicamid	0.166	LC-Q-Orbitrap	0.005	0.005
17	Dieldrin	0.023	GC-MS/MS (QQQ)	0.010	0.005
31	Spirotetramat	0.0551	LC-MS/MS QQQ	0.005	0.005
40	Spirotetramat	0.056	LC-MS/MS QQQ	0.005	0.005
41	Chlordane, trans-	0.027	GC-MS/MS (QQQ)	0.005	0.005
43	Flonicamid	0.148	LC - MS/MS	0.005	0.005
46	HCH, gamma-	0.032	GC-MS/MS (QQQ)	0.010	0.005
49	Chlordane, trans-	0.014	GC-MS/MS (QQQ)	0.005	0.005
55	Chlordane, trans-	0.07	GC-MS/MS (QQQ)	0.005	0.005
65	Endosulfan, alpha-	0.0159	GC-MS/MS (QQQ)	0.005	0.005
70	HCH, gamma-	0.035	GC-MS/MS (QQQ)	0.010	0.005
79	Flonicamid	0.205	LC-MS/MS QQQ	0.005	0.005
79	Spirotetramat	0.14	LC-MS/MS QQQ	0.005	0.005
91	Clomazone	0.017	LC-MS/MS QQQ	0.005	0.005
92	Tebuconazole	0.005	GC-MS/MS (QQQ)	0.005	0.005
93	Flonicamid	0.315	LC-MS	0.010	0.005
93	Fluopicolide	0.45	GC-MS/MS (QQQ)	0.010	0.005
93	Spirotetramat	0.064	LC-MS	0.010	0.005
112	Flonicamid	0.127	LC-MS/MS QQQ	0.005	0.005
121	HCH, gamma-	0.03	GC-MS/MS (QQQ)	0.020	0.005
124	HCH, gamma-	0.019	GC-MS/MS (QQQ)	0.005	0.005
137	Endrin	0.019	GC-MS/MS (QQQ)	0.005	0.005
145	HCH, gamma-	0.0249	GC-MS/MS (QQQ)	0.005	0.005
151	Hexaconazole	13	LC-MS/MS QQQ	0.005	0.005
160	Biphenyl	0.011	GC-MS/MS (QQQ)	0.005	0.005

3.1.2 False reported

Additionally, three participants reported results below their own reporting limits which is evaluated as false reported results, see **Table 6**. Some of the results were for pesticides present in the test material others were not present.

Table 6. False reported results, 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
137	Endosulfan, alpha-	0.0044	GC-MS/MS (QQQ)	0.005	0.005
141	Fluopyram	0.0020	LC-MS/MS QQQ	0.005	0.005
159	Fluopyram	0.0030	LC - MS/MS	0.005	0.005
159	HCH, alpha-	0.0040	GC-MS/MS (QQQ)	0.005	0.005

3.1.3 False negatives

Not reported results for pesticides actually present in the Test Item were judged as false negatives. **Table 7** summarizes the number of reported false negatives for each pesticide. Thirty-five participants submitted 71 false negatives result for 15 different pesticides, which represents 2.4% of the total number of results submitted by EU and EFTA laboratories. 22 % of the EU and EFTA participants (35 laboratories) reported false negative results.

Table 7. False negative results (FN).

Lab code	Aclonifen	Bixafen	Cypermethrin (sum)	Fludioxonil	Isoprothiolane	Mefentrifluconazole	Metribuzin	Proquinazid	Prosulfocarb	Prothioconazole-Desthio	Pyraclostrobin	Spirotetramat-enol	Tau-Fluvalinate	Chlordane, cis-*	HCH, beta-*
22													FN		
36													FN		
41		FN											FN		FN
43							FN								
48					FN										
49		FN	FN				FN		FN				FN		FN
55			FN				FN							FN	FN
57							FN								
59												FN	FN		
63						FN				FN	FN				
64								FN							FN
65							FN								
70															
73														FN	
86				FN			FN								
87													FN		
91		FN	FN				FN				FN		FN		
103													FN		
112													FN		
117						FN									
120														FN	
121				FN											
124							FN				FN				
127															FN
134							FN							FN	
136							FN								
138		FN									FN				
139		FN		FN			FN		FN	FN			FN	FN	FN
141				FN											
143		FN		FN									FN		
145														FN	
149			FN				FN								
152													FN		
154														FN	
163							FN						FN		

* Voluntary pesticides

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 8**. For the evaluated pesticides the assigned values were in the range of 0.020 – 0.51 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 8. Assigned values and their uncertainty in mg/kg, Fit-For-Purpose Relative Standard Deviation (FFP RSD) and Robust Relative Standard Deviation (Alg A RSD) for the pesticides present in the Test Item.

Pesticides	MRRL, mg/kg	Assigned value, mg/kg	Uncertainty, mg/kg	FFP RSD, %	Alg A RSD, %
Aclonifen	0.005	0.249	0.006	25	20
Azoxystrobin	0.005	0.059	0.001	25	17
Bixafen	0.005	0.029	0.001	25	20
Chlorantraniliprole	0.005	0.042	0.001	25	18
Cypermethrin (sum)	0.005	0.039	0.001	25	19
Cyprodinil	0.005	0.511	0.012	25	23
Difenoconazole	0.005	0.030	0.001	25	21
Dimethomorph	0.005	0.026	0.001	25	20
Fludioxonil	0.005	0.044	0.001	25	15
Isoprothiolane	0.005	0.046	0.001	25	15
Mefentrifluconazole	0.005	0.041	0.001	25	24
Metconazole	0.005	0.062	0.001	25	16
Metribuzin	0.005	0.040	0.001	25	20
Metribuzin	0.005	0.040	0.001	25	20
Proquinazid	0.005	0.055	0.001	25	23
Prosulfocarb	0.005	0.058	0.002	25	25
Pyraclostrobin	0.005	0.028	0.001	25	19
Spirotetramat-enol	0.005	0.058	0.002	25	30
Tau-Fluvalinate	0.005	0.028	0.001	25	18
Chlordane, cis-*	0.005	0.031	0.001	25	24
HCH, beta-*	0.005	0.028	0.001	25	21

* 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 15-30 % but on average, the Alg A-RSD was 20%, lower than 25% FFP-RSD used for the z score calculations.

3.3 Assessment of laboratory performance

3.3.1 Z scores

Z scores have been calculated for all the quantified pesticides using the FFP RSD of 25%. **Table 9** shows an overview of the acceptable, questionable, and unacceptable z scores and **Tables 10 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 4**.

Of the reported results for the evaluated pesticides, more than 90% were aclonifen, azoxystrobin, bixafen, chlorantraniliprole, cypermethrin (sum), cyprodinil, difenoconazole, dimethomorph, fludioxonil, HCH- beta, isoprothiolane, metconazole, metribuzin, proquinazid, prosulfocarb, prothioconazole-desthio and pyraclostrobin. For chlordane-cis, mefentrifluconazole, spirotetramat-enol and fluvalinate-tau the range of the acceptable results vary between 77 to 89 %.

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

Pesticides	No. of reported results	Assigned values	Acceptable %	Questionable %	Unacceptable[1] %	False negatives %
Aclonifen	110	0.249	91	2	7	5
Azoxystrobin	143	0.059	96	2	2	0
Bixafen	115	0.029	95	3	3	3
Chlorantraniliprole	135	0.042	94	1	5	0
Cypermethrin (sum)	138	0.039	90	4	6	4
Cyprodinil	143	0.511	92	3	5	0
Difenoconazole	143	0.030	92	4	3	0
Dimethomorph	138	0.03	93	4	4	0
Fludioxonil	138	0.044	96	2	2	1
Isoprothiolane	131	0.046	95	2	2	2
Mefentrifluconazole	84	0.041	79	5	17	14
Metconazole	130	0.062	97	2	1	0
Metribuzin	128	0.040	95	2	4	1
Proquinazid	127	0.055	93	1	6	1
Prosulfocarb	125	0.058	94	3	2	2
Prothioconazole-Desthio	128	0.020	91	4	5	3
Pyraclostrobin	135	0.028	96	3	1	1
Spirotetramat-enol	98	0.058	77	5	18	12
Tau-Fluvalinate	133	0.028	89	2	9	5
Chlordane, cis-*	113	0.031	86	6	8	4
HCH, beta-*	121	0.028	91	3	6	3

* Voluntary pesticides

[1] Unacceptable z scores include 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, 76 %, QuEChERS, Citrate buffered (EN 151662) [3], was used. However, variations in the clean-up procedures were reported by the labs, e.g. some used a freezing out step (18 % of the participants), centrifugation (17 %), some used d-SPE with PSA/MgSO4 (26 %), some used d-SPE with ODS/ $MgSO_4$ (3 %) and other used different combination of ODS, PSA, C18, z-sep (13 %). Liquid-liquid partition was used by 7% 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 10 % and 6 % 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 97 % of the reported results derived from a method where water was added before extraction.

GC instruments were used for 25% of the results, mainly GC-MS/MS (93%), but also GC-MS (2%) and GC- (μ) ECD (2%) was used. GC-ion trap for2%, GC-TOF, GC-Q-Orbitrap for 1% result. LC instruments were used for 75% of the reported results, mainly LC-MS/MS (66%) but 6% used high resolution instrument like LC-Orbitrap, LC-Q-Orbitrap or LC-Q-TOF. No results were analysed using specific detectors such as GC-NPD, LC-MS, LC-Ion trap, LC-Fluorescence, LC-UV, or LC-DAD.

Table 10a. Results for the mandatory pesticides aclonifen, azoxystrobin, bixafen, chlorantraniliprole, cypermethrin (sum), cyprodinil, difenoconazole, and dimethomorph in mg/kg, the corresponding z scores, MRRLs and the assigned values.

Laboratory code	Aclonifen	Z-scores (FFP RSD (25%)	Azoxystrobin	Z-scores (FFP RSD (25%)	Bixafen	Z-scores (FFP RSD (25%)	Chlorantraniliprole	Z-scores (FFP RSD (25%)	Cypermethrin (sum)	Z-scores (FFP RSD (25%)	Cyprodinil	Z-scores (FFP RSD (25%)	Difenoconazole	Z-scores (FFP RSD (25%)	Dimethomorph	Z-scores (FFP RSD (25%)
MRRL	**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**	
Assigned value	**0.249**		**0.059**		**0.029**		**0.042**		**0.039**		**0.511**		**0.029**		**0.025**	
1	0.283	0.5	0.069	0.7	0.031	0.3	0.044	0.2	0.078	4.1	0.700	1.5	0.050	2.9	0.033	1.2
2	0.231	-0.3	0.057	-0.1	0.024	-0.7	0.034	-0.8	0.029	-1.0	0.430	-0.6	0.029	-0.1	0.023	-0.3
3	0.275	0.4	0.062	0.2	0.026	-0.3	0.047	0.5	0.040	0.1	0.509	0.0	0.026	-0.4	0.030	0.8
4			0.062	0.2	0.020	-1.2	0.040	-0.2	0.034	-0.5	0.620	0.8	0.032	0.4	0.023	-0.3
5	0.211	-0.6	0.059	0.0	0.033	0.6	0.031	-1.1	0.039	0.0	0.500	-0.1	0.026	-0.4	0.025	0.0
6	0.214	-0.6	0.059	0.0	0.022	-0.9	0.039	-0.4	0.035	-0.4	0.471	-0.3	0.021	-1.1	0.022	-0.5
7	0.246	-0.1	0.062	0.2	0.025	-0.5	0.041	-0.1	0.044	0.5	0.522	0.1	0.028	-0.1	0.028	0.4
8	0.300	0.8	0.080	1.4	0.040	1.6	0.059	1.6	0.045	0.6	0.650	1.1	0.038	1.2	0.034	1.4
9	0.271	0.4	0.055	-0.3	0.026	-0.4	0.033	-0.9	0.038	-0.1	0.514	0.0	0.027	-0.3	0.019	-1.0
11			0.056	-0.2	0.023	-0.8	0.039	-0.3	0.034	-0.5	0.525	0.1	0.027	-0.3	0.023	-0.4
12																
13			0.056	-0.2	0.025	-0.6	0.039	-0.3	0.032	-0.7	0.483	-0.2	0.025	-0.6	0.021	-0.6
14	0.295	0.7	0.071	0.8	0.036	1.0	0.044	0.2	0.034	-0.5	0.671	1.2	0.039	1.4	0.031	0.9
15	0.279	0.5	0.047	-0.8	0.021	-1.1	0.033	-0.9	0.039	0.0	0.425	-0.7	0.023	-0.8	0.019	-1.0
16	0.298	0.8	0.048	-0.8	0.022	-0.9	0.040	-0.2	0.041	0.2	0.490	-0.2	0.025	-0.6	0.025	0.0
17	0.260	0.2	0.033	-1.8					0.038	-0.1	0.520	0.1	0.021	-1.1	0.016	-1.5
18	0.280	0.5	0.048	-0.8	0.029	0.0	0.039	-0.3	0.051	1.3	0.540	0.2	0.055	3.6	0.040	2.3
19	0.342	1.5	0.069	0.7	0.033	0.5	0.056	1.3	0.042	0.4	0.660	1.2	0.038	1.3	0.033	1.2
20	0.170	-1.3	0.072	0.9	0.028	-0.1	0.049	0.6	0.034	-0.5	0.424	-0.7	0.033	0.6	0.035	1.6
22	0.227	-0.4	0.057	-0.1	0.022	-0.9	0.043	0.1	0.042	0.3	0.456	-0.4	0.022	-0.9	0.024	-0.1
23	0.344	1.5	0.053	-0.4	0.034	0.7	0.042	-0.1	0.038	-0.1	0.639	1.0	0.036	1.0	0.028	0.5
24			0.065	0.4			0.021	-2.0			0.070	-3.5	0.017	-1.7		
25	0.283	0.5	0.061	0.1	0.025	-0.5	0.060	1.7	0.040	0.1	0.569	0.5	0.034	0.7	0.026	0.1
26	0.253	0.1	0.053	-0.4	0.026	-0.4	0.039	-0.3	0.030	-0.9	0.591	0.6	0.025	-0.6	0.018	-1.1
27			0.059	0.0			0.035	-0.7	0.035	-0.4	0.390	-0.9	0.027	-0.3	0.020	-0.8
28	0.230	-0.3	0.060	0.1	0.030	0.2	0.051	0.8	0.040	0.1	0.576	0.5	0.033	0.6	0.029	0.6
29																
31	0.208	-0.7	0.069	0.7	0.026	-0.4	0.043	0.1	0.056	1.8	0.484	-0.2	0.031	0.3	0.026	0.1
32	310.0	>5	0.077	1.2	0.031	0.3	0.038	-0.4	0.040	0.1	0.550	0.3	0.035	0.8	0.025	0.0
33	0.207	-0.7	0.059	0.0	0.032	0.5	0.042	0.0	0.043	0.5	0.641	1.0	0.033	0.6	0.028	0.4
34																
35			0.068	0.6	0.026	-0.4	0.045	0.3	0.030	-0.9	0.472	-0.3	0.028	-0.2	0.023	-0.4
36	0.184	-1.0	0.054	-0.3	0.021	-1.1	0.041	-0.1	0.036	-0.3	0.338	-1.4	0.026	-0.4	0.025	0.0
37			0.064	0.3	0.030	0.2	0.052	0.9	0.039	0.0	0.797	2.2	0.039	1.4	0.032	1.1
38	0.310	1.0	0.069	0.7			0.046	0.3	0.039	0.0	0.530	0.1	0.038	1.2	0.031	0.9
39	0.230	-0.3	0.100	2.8	0.044	2.1	0.053	1.0	0.041	0.2	0.560	0.4	0.045	2.2	0.042	2.7
40	0.224	-0.4	0.062	0.2	0.036	1.0	0.040	-0.2	0.044	0.5	0.651	1.1	0.031	0.3	0.027	0.3
41	FN	-4.0	0.059	0.0			0.038	-0.4	0.035	-0.4	0.391	-0.9	0.021	-1.1	0.020	-0.8

Laboratory code	Aclonifen	Z-scores (FFP RSD (25%)	Azoxystrobin	Z-scores (FFP RSD (25%)	Bixafen	Z-scores (FFP RSD (25%)	Chlorantraniliprole	Z-scores (FFP RSD (25%)	Cypermethrin (sum)	Z-scores (FFP RSD (25%)	Cyprodinil	Z-scores (FFP RSD (25%)	Difenoconazole	Z-scores (FFP RSD (25%)	Dimethomorph	Z-scores (FFP RSD (25%)
MRRL	**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**	
Assigned value	**0.249**		**0.059**		**0.029**		**0.042**		**0.039**		**0.511**		**0.029**		**0.025**	
42			0.064	0.3			0.037	-0.5	0.047	0.9	0.363	-1.2	0.023	-0.8	0.022	-0.5
43	0.288	0.6	0.063	0.3	0.029	0.0	0.049	0.6	0.043	0.4	0.518	0.1	0.034	0.7	0.029	0.6
44	0.203	-0.7	0.051	-0.6	0.019	-1.3	0.030	-1.2	0.025	-1.4	0.492	-0.2	0.026	-0.4	0.023	-0.3
45	0.222	-0.4	0.053	-0.4	0.026	-0.4	0.042	0.0	0.062	2.4	0.589	0.6	0.025	-0.6	0.023	-0.3
46	0.220	-0.5	0.059	0.0			0.041	-0.1			0.490	-0.2	0.028	-0.1		
47	0.235	-0.2	0.065	0.4	0.027	-0.2	0.030	-1.2	0.039	0.0	0.430	-0.6	0.039	1.4	0.023	-0.3
48			0.066	0.5					0.054	1.6	0.521	0.1	0.035	0.8	0.036	1.7
49	FN	-4.0	0.039	-1.4	FN	-4.0	0.026	-1.5	0.025	-1.4	0.100	-3.2	0.012	-2.3	0.011	-2.3
50	0.268	0.3	0.058	-0.1			0.036	-0.6	0.031	-0.8	0.471	-0.3	0.029	-0.1	0.024	-0.2
51	0.280	0.5	0.056	-0.2	0.029	0.0	0.041	-0.1	0.043	0.4	0.530	0.1	0.030	0.1	0.026	0.1
52	0.185	-1.0	0.055	-0.3	0.026	-0.4	0.036	-0.6	0.037	-0.2	0.521	0.1	0.027	-0.2	0.024	-0.2
53	0.209	-0.6	0.060	0.1	0.024	-0.7	0.040	-0.3	0.034	-0.5	0.438	-0.6	0.023	-0.9	0.023	-0.4
54	0.190	-0.9	0.057	-0.1	0.030	0.2	0.038	-0.4	0.030	-0.9	0.465	-0.4	0.030	0.1	0.026	0.1
55	0.200	-0.8	0.040	-1.3	FN	-4.0	0.042	0.0	0.032	-0.7	0.380	-1.0	0.029	0.0	0.023	-0.3
56	0.323	1.2	0.070	0.7	0.023	-0.8	0.048	0.5	0.045	0.6	0.522	0.1	0.018	-1.5	0.032	1.0
57	0.230	-0.3	0.079	1.3	0.042	1.9	0.030	-1.2	0.037	-0.2	0.310	-1.6	0.034	0.7	0.049	3.8
58	0.307	0.9	0.043	-1.1	0.022	-0.9	0.029	-1.3	0.043	0.5	0.426	-0.7	0.023	-0.8	0.037	1.8
59	0.320	1.1	0.050	-0.6	0.025	-0.5	0.131	8.4	0.029	-1.0	0.445	-0.5	0.031	0.3	0.022	-0.5
60	0.311	1.0	0.062	0.2	0.029	0.0	0.043	0.1	0.059	2.1	0.688	1.4	0.029	0.0	0.029	0.6
61	0.197	-0.8	0.072	0.9	0.031	0.3	0.047	0.4	0.039	0.0	0.486	-0.2	0.030	0.2	0.023	-0.3
62	0.300	0.8	0.050	-0.6	0.030	0.2	0.040	-0.2	0.035	-0.4	0.498	-0.1	0.030	0.1	0.023	-0.3
63			0.059	0.0					0.035	-0.4	0.991	3.8	0.052	3.1	0.058	>5
64			0.061	0.1			0.038	-0.4	0.034	-0.5	0.470	-0.3	0.026	-0.4	0.016	-1.5
65	0.276	0.4	0.055	-0.3	0.032	0.4	0.045	0.3	0.039	0.0	0.599	0.7	0.037	1.0	0.031	0.9
66	0.261	0.2	0.075	1.1	0.035	0.8	0.049	0.6	0.030	-0.9	0.644	1.0	0.035	0.8	0.044	3.0
67	0.245	-0.1	0.040	-1.3	0.036	1.0	0.062	1.9	0.028	-1.1	0.305	-1.6	0.020	-1.2	0.018	-1.1
68			0.019	-2.7					0.012	-2.8	0.037	-3.7	0.013	-2.2	0.018	-1.1
69	0.250	0.0	0.064	0.3	0.027	-0.2	0.049	0.6	0.039	0.0	0.604	0.7	0.029	0.0	0.029	0.6
70	0.199	-0.8	0.063	0.3	0.035	0.9	0.044	0.1	0.034	-0.5	0.443	-0.5	0.026	-0.4	0.020	-0.8
71																
72	0.268	0.3	0.057	-0.1	0.031	0.3	0.041	-0.1	0.038	-0.1	0.510	0.0	0.027	-0.3	0.029	0.6
73			0.036	-1.6							0.089	-3.3	0.013	-2.2		
74			0.049	-0.7					0.037	-0.2	0.480	-0.2	0.029	0.0	0.022	-0.5
75									0.035	-0.4						
76			0.053	-0.4			0.035	-0.7	0.039	0.0	0.173	-2.6	0.025	-0.6	0.025	0.0
77																
78	0.272	0.4	0.051	-0.6	0.030	0.1	0.053	1.0	0.037	-0.1	0.581	0.5	0.030	0.2	0.026	0.1
79	0.237	-0.2	0.058	-0.1	0.028	-0.2	0.104	>5	0.039	0.0	0.389	-1.0	0.024	-0.7	0.022	-0.5
80	0.276	0.4	0.103	3.0	0.040	1.6	0.076	3.2	0.032	-0.7	0.727	1.7	0.047	2.5	0.047	3.5
81	0.230	-0.3	0.065	0.4	0.042	1.9	0.059	1.6	0.030	-0.9	0.310	-1.6	0.030	0.1	0.035	1.6

Laboratory code	Aclonifen	Z-scores (FFP RSD (25%)	Azoxystrobin	Z-scores (FFP RSD (25%)	Bixafen	Z-scores (FFP RSD (25%)	Chlorantraniliprole	Z-scores (FFP RSD (25%)	Cypermethrin (sum)	Z-scores (FFP RSD (25%)	Cyprodinil	Z-scores (FFP RSD (25%)	Difenoconazole	Z-scores (FFP RSD (25%)	Dimethomorph	Z-scores (FFP RSD (25%)
MRRL	**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**	
Assigned value	**0.249**		**0.059**		**0.029**		**0.042**		**0.039**		**0.511**		**0.029**		**0.025**	
82	0.322	1.2	0.065	0.4	0.040	1.6	0.053	1.0	0.049	1.1	0.663	1.2	0.044	2.1	0.034	1.4
83	0.218	-0.5	0.062	0.2			0.035	-0.7	0.037	-0.2	0.511	0.0	0.022	-1.0	0.024	-0.2
84	0.267	0.3	0.049	-0.7	0.027	-0.2	0.042	0.0	0.035	-0.4	0.493	-0.1	0.024	-0.7	0.026	0.1
85	0.218	-0.5	0.061	0.1			0.034	-0.8	0.031	-0.8	0.450	-0.5	0.025	-0.5		
86	0.250	0.0	0.050	-0.6	0.021	-1.1	0.029	-1.3	FN	-4.0	0.500	-0.1	0.021	-1.1	0.035	1.6
87	0.172	-1.2	0.103	3.0	0.036	1.0	0.047	0.4	0.039	0.0	0.425	-0.7	0.040	1.5	0.025	0.0
88			0.069	0.7	0.038	1.3	0.054	1.1	0.050	1.2	0.638	1.0	0.028	-0.1	0.026	0.1
89	0.320	1.1	0.048	-0.8	0.024	-0.7	0.036	-0.6	0.045	0.6	0.579	0.5	0.046	2.3	0.023	-0.3
90																
91	FN	-4.0	0.045	-1.0	FN	-4.0	0.049	0.6	0.080	4.3	0.340	-1.3	0.032	0.4	0.039	2.2
92	0.213	-0.6	0.068	0.6	0.026	-0.4	0.053	1.0	0.050	1.2	0.413	-0.8	0.030	0.1	0.028	0.4
93	0.242	-0.1	0.063	0.3	0.032	0.5	0.041	-0.1	0.036	-0.3	0.728	1.7	0.032	0.4	0.024	-0.2
94																
95									0.040	0.1						
96	0.281	0.5	0.067	0.5	0.031	0.3	0.045	0.3	0.043	0.4	0.551	0.3	0.034	0.7	0.031	0.9
97			0.079	1.3	0.040	1.6					0.624	0.9	0.040	1.5	0.035	1.6
98	0.196	-0.9	0.059	0.0	0.026	-0.4	0.037	-0.5	0.036	-0.3	0.418	-0.7	0.029	0.0	0.019	-1.0
99	0.184	-1.0	0.045	-1.0	0.027	-0.2	0.035	-0.7	0.031	-0.8	0.442	-0.5	0.026	-0.4	0.032	1.1
100	0.255	0.1	0.049	-0.7	0.025	-0.5	0.040	-0.2	0.044	0.6	0.491	-0.2	0.026	-0.4	0.027	0.2
101	0.189	-1.0	0.051	-0.6			0.034	-0.8	0.044	0.5	0.456	-0.4	0.022	-1.0	0.019	-1.0
102			0.068	0.6			0.047	0.4			0.506	0.0	0.029	0.0	0.023	-0.3
103	0.320	1.1	0.061	0.1	0.030	0.2	0.043	0.0	0.046	0.7	0.464	-0.4	0.033	0.6	0.025	0.0
104	0.339	1.4	0.053	-0.4	0.030	0.2	0.039	-0.3	0.043	0.4	0.579	0.5	0.029	0.0	0.026	0.1
105			0.055	-0.3	0.022	-0.9	0.036	-0.6	0.045	0.6	0.500	-0.1	0.028	-0.1	0.026	0.1
106	0.160	-1.4	0.060	0.1	0.033	0.6	0.053	1.0	0.030	-0.9	0.650	1.1	0.052	3.2	0.028	0.4
107	0.185	-1.0	0.044	-1.0	0.022	-0.9	0.033	-0.9	0.035	-0.4	0.420	-0.7	0.024	-0.7	0.025	0.0
108	0.218	-0.5	0.070	0.7	0.035	0.9	0.054	1.1	0.031	-0.8	0.500	-0.1	0.038	1.2	0.030	0.8
109	0.278	0.5	0.062	0.2	0.027	-0.2	0.038	-0.4	0.036	-0.3	0.524	0.1	0.030	0.1	0.024	-0.2
110	0.246	-0.1	0.045	-1.0	0.022	-0.9	0.038	-0.4	0.034	-0.5	0.392	-0.9	0.026	-0.4	0.020	-0.8
111			0.056	-0.2	0.022	-0.9	0.041	-0.2	0.030	-0.9	0.623	0.9	0.031	0.2	0.025	0.0
112	0.204	-0.7	0.062	0.2	0.033	0.6	0.051	0.8	0.031	-0.8	0.581	0.5	0.035	0.8	0.032	1.1
113	0.286	0.6	0.072	0.9	0.037	1.2	0.047	0.4	0.054	1.6	0.681	1.3	0.025	-0.6	0.026	0.1
114	0.180	-1.1	0.067	0.5	0.020	-1.2	0.042	0.0	0.055	1.7	0.500	-0.1	0.027	-0.3	0.022	-0.5
115			0.057	-0.2	0.022	-0.9	0.044	0.2	0.042	0.3	0.359	-1.2	0.020	-1.2	0.020	-0.8
116	0.216	-0.5	0.056	-0.2	0.026	-0.4	0.035	-0.7	0.036	-0.3	0.557	0.4	0.029	0.0	0.026	0.1
117			0.076	1.1	0.027	-0.2	0.054	1.1	0.066	2.8	0.435	-0.6	0.028	-0.1		
118			0.068	0.6			0.040	-0.2	0.036	-0.3	0.433	-0.6	0.031	0.2	0.028	0.4
119			0.056	-0.2	0.026	-0.4	0.040	-0.3	0.039	0.0	0.276	-1.8	0.020	-1.2	0.022	-0.6
120	0.285	0.6	0.063	0.3	0.031	0.3	0.051	0.8	0.039	0.0	0.609	0.8	0.036	1.0	0.029	0.6
121			0.067	0.5	0.045	2.3	0.214	>5	FN	-4.0	0.540	0.2	0.034	0.7	0.024	-0.2

Laboratory code	Aclonifen	Z-scores (FFP RSD (25%)	Azoxystrobin	Z-scores (FFP RSD (25%)	Bixafen	Z-scores (FFP RSD (25%)	Chlorantraniliprole	Z-scores (FFP RSD (25%)	Cypermethrin (sum)	Z-scores (FFP RSD (25%)	Cyprodinil	Z-scores (FFP RSD (25%)	Difenoconazole	Z-scores (FFP RSD (25%)	Dimethomorph	Z-scores (FFP RSD (25%)
MRRL	**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**	
Assigned value	**0.249**		**0.059**		**0.029**		**0.042**		**0.039**		**0.511**		**0.029**		**0.025**	
122			0.065	0.4			0.330	>5			0.383	-1.0	0.029	0.0	0.047	3.5
123	0.292	0.7	0.063	0.3	0.030	0.2	0.044	0.2	0.029	-1.0	0.626	0.9	0.030	0.1	0.025	0.0
124	0.101	-2.4	0.054	-0.3	0.016	-1.8	0.037	-0.5	0.039	0.0	0.385	-1.0	0.036	1.0	0.023	-0.3
125	0.210	-0.6	0.052	-0.5	0.024	-0.7	0.044	0.2	0.051	1.3	0.471	-0.3	0.033	0.6	0.027	0.3
126	0.280	0.5	0.063	0.2			0.044	0.2	0.052	1.4	0.547	0.3	0.028	-0.1	0.024	-0.1
127			0.063	0.3			0.032	-1.0	0.047	0.9	0.216	-2.3	0.027	-0.3	0.018	-1.1
128	0.201	-0.8	0.053	-0.4	0.028	-0.1	0.035	-0.7	0.038	-0.1	0.409	-0.8	0.026	-0.4	0.021	-0.7
129	0.270	0.3	0.048	-0.7	0.023	-0.8	0.035	-0.7	0.040	0.2	0.549	0.3	0.024	-0.7	0.026	0.2
131	0.200	-0.8	0.064	0.3	0.028	-0.1	0.041	-0.1	0.044	0.5	0.570	0.5	0.030	0.1	0.026	0.1
132	0.219	-0.5	0.042	-1.2	0.027	-0.2	0.040	-0.2	0.028	-1.1	0.415	-0.8	0.027	-0.3	0.025	0.0
133	0.281	0.5	0.056	-0.2	0.029	0.0	0.049	0.6	0.041	0.2	0.566	0.4	0.030	0.1	0.023	-0.3
134	0.232	-0.3	0.069	0.7	0.033	0.6	0.044	0.2	0.010	-3.0	0.862	2.7	0.044	2.1	0.025	0.0
135	0.250	0.0	0.057	-0.1	0.027	-0.3	0.042	-0.1	0.049	1.1	0.467	-0.3	0.028	-0.2	0.023	-0.3
136	0.350	1.6	0.058	-0.1	0.034	0.7	0.041	-0.1	0.042	0.3	0.430	-0.6	0.028	-0.1	0.027	0.3
137	0.555	4.9	0.047	-0.8			0.043	0.1	0.049	1.1	0.513	0.0	0.032	0.4	0.014	-1.8
138	FN	-4.0	0.064	0.3			0.043	0.1	0.043	0.4	0.632	0.9	0.027	-0.3	0.027	0.3
139	FN	-4.0	0.089	2.0	0.038	1.3	0.093	4.8	FN	-4.0	0.939	3.3	0.077	>5	0.040	2.3
141	0.293	0.7	0.053	-0.4	0.023	-0.8	0.040	-0.2	FN	-4.0	0.587	0.6	0.027	-0.3	0.025	0.0
142	0.242	-0.1	0.073	0.9	0.033	0.6	0.047	0.5	0.037	-0.2	0.497	-0.1	0.033	0.6	0.030	0.7
143	FN	-4.0	0.062	0.2	0.032	0.5	0.035	-0.7	FN	-4.0	0.750	1.9	0.032	0.4	0.029	0.6
144			0.064	0.3	0.038	1.3	0.036	-0.6	0.035	-0.4	0.520	0.1	0.035	0.8	0.034	1.4
145			0.074	1.0			0.031	-1.1	0.029	-1.0	0.253	-2.0	0.027	-0.3	0.022	-0.5
146	0.284	0.6	0.073	1.0	0.035	0.9	0.051	0.8	0.049	1.0	0.560	0.4	0.037	1.1	0.031	1.0
147	0.274	0.4	0.063	0.3	0.031	0.3	0.046	0.3	0.038	-0.1	0.759	1.9	0.035	0.8	0.033	1.2
148	0.121	-2.1	0.042	-1.2	0.020	-1.2	0.026	-1.6	0.029	-1.0	0.280	-1.8	0.016	-1.8	0.019	-1.0
149	0.190	-0.9	0.046	-0.9	FN	-4.0	0.033	-0.9	0.013	-2.7	0.400	-0.9	0.019	-1.4	0.020	-0.8
150	0.306	0.9	0.047	-0.8	0.023	-0.8	0.035	-0.7	0.042	0.3	0.459	-0.4	0.021	-1.1	0.023	-0.3
151	219.0	>5	45.00	>5			33.00	>5	31.00	>5	383.0	>5	24.00	>5	21.00	>5
152			0.058	-0.1	0.028	-0.1	0.045	0.3	0.030	-0.9	0.437	-0.6	0.026	-0.4	0.024	-0.2
153	0.207	-0.7	0.053	-0.4	0.023	-0.8	0.038	-0.4	0.032	-0.7	0.458	-0.4	0.025	-0.6	0.021	-0.7
154	0.221	-0.5	0.063	0.3	0.034	0.7	0.054	1.1			0.650	1.1	0.034	0.7	0.029	0.6
155			0.028	-2.1	0.032	0.5	0.044	0.2	0.047	0.9	0.675	1.3	0.036	1.0	0.033	1.2
156																
157	0.247	0.0	0.048	-0.8	0.026	-0.4	0.040	-0.2	0.053	1.5	0.502	-0.1	0.030	0.1	0.028	0.4
158	0.288	0.6	0.059	0.0	0.030	0.2	0.038	-0.4	0.036	-0.3	0.596	0.7	0.031	0.3	0.030	0.8
159			0.050	-0.6			0.037	-0.5	0.041	0.2	0.690	1.4	0.030	0.1	0.023	-0.3
160			0.035	-1.6					0.046	0.8	0.046	-3.6	0.014	-2.1	0.012	-2.1
161	0.270	0.3	0.057	-0.1	0.029	0.0	0.036	-0.6	0.039	0.1	0.633	0.9	0.036	1.0	0.025	0.0
162	0.256	0.1	0.059	0.0	0.032	0.5	0.044	0.2	0.060	2.2	0.547	0.3	0.033	0.5	0.031	1.0
163	0.211	-0.6	0.066	0.5	0.032	0.5	0.050	0.7	0.037	-0.2	0.706	1.5	0.038	1.2	0.023	-0.3
165	0.232	-0.3	0.053	-0.4	0.027	-0.2	0.042	0.0	0.046	0.8	0.415	-0.8	0.026	-0.4	0.029	0.6

Table 10b. Assigned values, MRRL, preliminary z scores for the mandatory pesticides fludioxonil, isoprothiolane, mefentrifluconazole, metconazole, metribuzin, proquinazid, prosulfocarb and prothioconazole-desthio.

Laboratory code	Fludioxonil	Z-scores (FFP RSD (25%)	Isoprothiolane	Z-scores (FFP RSD (25%)	Mefentrifluconazole	Z-scores (FFP RSD (25%)	Metconazole	Z-scores (FFP RSD (25%)	Metribuzin	Z-scores (FFP RSD (25%)	Proquinazid	Z-scores (FFP RSD (25%)	Prosulfocarb	Z-scores (FFP RSD (25%)	Prothioconazole-Desthio	Z-scores (FFP RSD (25%)
MRRL	**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**	
Assigned value	**0.044**		**0.046**		**0.041**		**0.062**		**0.040**		**0.055**		**0.058**		**0.020**	
1	0.046	0.2	0.050	0.3	0.042	0.1			0.049	1.0	0.043	-0.9	0.059	0.0	0.023	0.6
2	0.041	-0.3	0.044	-0.2			0.066	0.2	0.033	-0.7	0.048	-0.5	0.053	-0.4	0.015	-1.0
3	0.045	0.1	0.051	0.4	0.035	-0.6	0.068	0.4	0.045	0.6	0.059	0.3	0.061	0.2	0.021	0.2
4	0.048	0.3	0.049	0.3	0.048	0.7	0.064	0.2	0.040	0.1	0.062	0.5			0.021	0.2
5	0.042	-0.2	0.042	-0.4	0.036	-0.5	0.052	-0.6	0.036	-0.4	0.050	-0.4	0.051	-0.5	0.018	-0.4
6	0.049	0.4	0.042	-0.4	0.029	-1.2	0.054	-0.5	0.035	-0.5	0.058	0.2	0.056	-0.2	0.016	-0.8
7	0.041	-0.3	0.042	-0.4			0.063	0.1			0.047	-0.6	0.054	-0.3	0.021	0.2
8	0.057	1.2	0.065	1.6	0.053	1.2	0.076	0.9	0.048	0.9	0.065	0.7	0.080	1.5	0.027	1.4
9	0.040	-0.4	0.038	-0.7	0.029	-1.2	0.056	-0.4	0.040	0.1	0.056	0.1	0.055	-0.2	0.016	-0.8
11	0.040	-0.4	0.043	-0.3	0.039	-0.2	0.062	0.0	0.038	-0.2	0.059	0.3			0.019	-0.2
12																
13	0.036	-0.7	0.041	-0.4	0.035	-0.6	0.063	0.0	0.038	-0.2	0.052	-0.2			0.018	-0.5
14	0.050	0.6	0.053	0.6	0.053	1.2	0.063	0.1	0.048	0.9	0.060	0.4	0.090	2.2	0.023	0.6
15	0.035	-0.8	0.038	-0.7	0.031	-0.9	0.047	-1.0	0.031	-0.8	0.036	-1.4	0.042	-1.1	0.014	-1.2
16	0.038	-0.5	0.041	-0.5	0.045	0.4	0.049	-0.8	0.029	-1.1	0.051	-0.3	0.058	0.0	0.021	0.2
17	0.046	0.2							0.083	4.4			0.043	-1.0		
18	0.049	0.5	0.040	-0.5			0.057	-0.3	0.031	-0.9	0.065	0.7	0.055	-0.2	0.019	-0.2
19	0.057	1.2	0.055	0.8			0.064	0.2	0.043	0.4	0.065	0.7	0.082	1.6	0.028	1.6
20	0.049	0.5	0.058	1.0			0.044	-1.2	0.047	0.8	0.061	0.4	0.058	0.0	0.011	-1.8
22	0.044	0.0	0.046	-0.1	0.030	-1.1	0.058	-0.2	0.047	0.8	0.053	-0.2	0.052	-0.4	0.020	0.0
23	0.041	-0.3	0.049	0.2	0.050	0.9	0.064	0.1	0.035	-0.4	0.058	0.2	0.076	1.2	0.022	0.4
24							0.033	-1.9								
25	0.047	0.2	0.058	1.0	0.064	2.3	0.059	-0.2	0.047	0.7	0.058	0.2	0.078	1.3	0.026	1.2
26	0.043	-0.1	0.042	-0.4	0.033	-0.8	0.049	-0.8	0.029	-1.1	0.046	-0.7	0.050	-0.6	0.015	-1.0
27	0.044	0.0	0.045	-0.1							0.056	0.1	0.049	-0.6	0.018	-0.4
28	0.043	-0.1	0.043	-0.3	0.039	-0.2	0.066	0.3	0.046	0.7	0.072	1.2	0.065	0.5	0.023	0.6
29																
31	0.047	0.3	0.045	-0.1			0.060	-0.2	0.037	-0.3	0.034	-1.6	0.048	-0.7	0.017	-0.7
32	0.045	0.1	0.043	-0.3	0.042	0.1	0.060	-0.1	0.053	1.4	0.066	0.8	0.068	0.7	0.018	-0.4
33	0.047	0.3	0.050	0.3			0.065	0.2	0.041	0.1	0.061	0.5	0.068	0.6	0.023	0.7
34																
35	0.046	0.2	0.050	0.3			0.068	0.4	0.026	-1.4	0.046	-0.7	0.044	-1.0	0.017	-0.7
36	0.049	0.5	0.046	0.0	0.040	-0.1	0.061	-0.1	0.029	-1.1	0.051	-0.3	0.058	0.0	0.023	0.6
37	0.043	-0.1	0.056	0.8			0.059	-0.2	0.044	0.5	0.068	0.9	0.066	0.5	0.020	0.0
38	0.041	-0.3	0.054	0.7					0.039	-0.1	0.075	1.5	0.065	0.5	0.021	0.2
39	0.053	0.8	0.062	1.4	0.042	0.1	0.079	1.1	0.046	0.7	0.038	-1.2	0.043	-1.0	0.031	2.2
40	0.044	0.0	0.048	0.1	0.043	0.2	0.067	0.3	0.040	0.1	0.057	0.1	0.064	0.4	0.024	0.8
41	0.045	0.1	0.047	0.1	0.031	-1.0	0.052	-0.6			0.043	-0.9	0.036	-1.5	0.017	-0.6

Laboratory code	Fludioxonil	Z-scores (FFP RSD (25%)	Isoprothiolane	Z-scores (FFP RSD (25%)	Mefentrifluconazole	Z-scores (FFP RSD (25%)	Metconazole	Z-scores (FFP RSD (25%)	Metribuzin	Z-scores (FFP RSD (25%)	Proquinazid	Z-scores (FFP RSD (25%)	Prosulfocarb	Z-scores (FFP RSD (25%)	Prothioconazole-Desthio	Z-scores (FFP RSD (25%)
MRRL	**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**	
Assigned value	**0.044**		**0.046**		**0.041**		**0.062**		**0.040**		**0.055**		**0.058**		**0.020**	
42	0.045	0.1	0.041	-0.5			0.063	0.1	0.028	-1.2	0.035	-1.5	0.040	-1.3	0.014	-1.2
43	0.055	1.0	0.052	0.5	FN	-4.0	0.063	0.1	0.034	-0.6	0.102	3.4	0.053	-0.4	0.022	0.4
44	0.030	-1.3	0.039	-0.6	0.032	-0.9	0.050	-0.8	0.030	-1.0	0.045	-0.7	0.049	-0.6	0.017	-0.6
45	0.049	0.5	0.048	0.1	0.058	1.6	0.066	0.2	0.043	0.3	0.052	-0.2	0.072	1.0	0.019	-0.2
46	0.051	0.6							0.046	0.7	0.051	-0.3	0.056	-0.2		
47	0.053	0.8	0.043	-0.3	0.040	-0.1	0.060	-0.1	0.032	-0.8	0.050	-0.4	0.050	-0.6	0.020	0.0
48	FN	-4.0	0.059	1.1												
49	0.030	-1.3	0.033	-1.1	FN	-4.0	0.047	-1.0	0.032	-0.8	FN	-4.0	0.213	>5	0.018	-0.4
50	0.039	-0.5	0.043	-0.3							0.042	-1.0	0.053	-0.3	0.019	-0.3
51	0.045	0.1	0.054	0.7			0.084	1.4			0.052	-0.2	0.058	0.0	0.022	0.4
52	0.038	-0.6	0.041	-0.4	0.037	-0.4	0.053	-0.6	0.036	-0.4	0.057	0.2	0.056	-0.1	0.019	-0.2
53	0.043	-0.1	0.048	0.1	0.035	-0.6	0.058	-0.3	0.042	0.3	0.044	-0.8	0.045	-0.9	0.018	-0.3
54	0.041	-0.3	0.042	-0.4			0.063	0.1			0.041	-1.0	0.047	-0.8	0.019	-0.2
55	0.039	-0.4	0.035	-1.0	FN	-4.0	0.049	-0.8	0.028	-1.2	0.045	-0.7	0.046	-0.8	0.028	1.6
56	0.035	-0.8	0.040	-0.5			0.078	1.0	0.043	0.3	0.060	0.3	0.076	1.2	0.018	-0.5
57	0.043	-0.1	0.044	-0.2	FN	-4.0	0.084	1.4	0.036	-0.4	0.078	1.7	0.028	-2.1	0.031	2.2
58	0.041	-0.3	0.032	-1.3			0.050	-0.8	0.047	0.8	0.043	-0.9	0.048	-0.7	0.010	-2.0
59	0.058	1.3	0.049	0.2	0.039	-0.2	0.058	-0.3	0.039	-0.1	0.053	-0.1	0.052	-0.4	0.019	-0.2
60	0.051	0.6	0.049	0.2	0.032	-0.9	0.069	0.4	0.039	-0.1	0.043	-0.9	0.049	-0.6	0.020	0.0
61	0.050	0.6	0.051	0.4	0.059	1.7	0.062	0.0	0.042	0.3	0.051	-0.3	0.056	-0.1	0.020	0.0
62	0.040	-0.4	0.040	-0.5	0.048	0.7	0.060	-0.1	0.044	0.5	0.050	-0.4	0.068	0.7	0.018	-0.4
63	0.040	-0.4	FN	-4.0			0.064	0.1	0.052	1.2			FN	-4.0	FN	-4.0
64	0.048	0.4	0.046	0.0			0.062	0.0	FN	-4.0	0.047	-0.6	0.043	-1.0		
65	0.044	0.0	0.043	-0.3	FN	-4.0	0.068	0.4	0.043	0.4	0.058	0.2	0.079	1.4	0.018	-0.4
66	0.053	0.8	0.057	0.9			0.077	0.9	0.039	-0.1	0.057	0.2	0.074	1.1	0.021	0.2
67	0.032	-1.1	0.058	1.0	0.032	-0.9	0.077	1.0	0.031	-0.9	0.082	2.0	0.028	-2.1	0.025	1.0
68	0.017	-2.5	0.019	-2.3											0.009	-2.3
69	0.049	0.5	0.045	-0.1	0.047	0.6	0.066	0.3	0.037	-0.3	0.049	-0.4	0.064	0.4	0.022	0.4
70	0.034	-0.9	0.051	0.4			0.062	0.0	0.036	-0.4	0.056	0.1	0.047	-0.8	0.019	-0.3
71																
72	0.034	-0.9	0.052	0.5	0.038	-0.3	0.076	0.9	0.042	0.3	0.067	0.9	0.054	-0.3	0.023	0.6
73	0.049	0.5														
74	0.048	0.4	0.037	-0.8			0.053	-0.6	0.029	-1.1						
75																
76																
77																
78	0.040	-0.4	0.045	-0.1	0.050	0.9	0.064	0.1	0.034	-0.5	0.057	0.1	0.056	-0.1	0.022	0.4
79	0.031	-1.2	0.045	-0.1	0.033	-0.8	0.061	-0.1	0.040	0.1	0.042	-1.0	0.046	-0.9	0.019	-0.3
80	0.067	2.1	0.077	2.7	0.057	1.6	0.081	1.2	0.065	2.6	0.077	1.6	0.068	0.7	0.036	3.2
81	0.031	-1.2	0.047	0.1			0.070	0.5	0.049	1.0	0.052	-0.2	0.035	-1.6		

Laboratory code	Fludioxonil	Z-scores (FFP RSD (25%)	Isoprothiolane	Z-scores (FFP RSD (25%)	Mefentrifluconazole	Z-scores (FFP RSD (25%)	Metconazole	Z-scores (FFP RSD (25%)	Metribuzin	Z-scores (FFP RSD (25%)	Proquinazid	Z-scores (FFP RSD (25%)	Prosulfocarb	Z-scores (FFP RSD (25%)	Prothioconazole-Desthio	Z-scores (FFP RSD (25%)
MRRL	**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**	
Assigned value	**0.044**		**0.046**		**0.041**		**0.062**		**0.040**		**0.055**		**0.058**		**0.020**	
82	0.048	0.4	0.051	0.4	0.044	0.3	0.067	0.3	0.044	0.5	0.065	0.7	0.077	1.3	0.033	2.6
83	0.034	-0.9	0.050	0.3			0.052	-0.6	0.038	-0.2	0.040	-1.1	0.059	0.0	0.017	-0.6
84	0.037	-0.6	0.043	-0.3	0.041	0.0	0.050	-0.8	0.037	-0.3	0.055	0.0	0.063	0.3	0.020	0.0
85	0.040	-0.4	0.048	0.1					0.027	-1.3			0.045	-0.9	0.015	-0.9
86	0.040	-0.4	0.041	-0.5	FN	-4.0	0.054	-0.5	0.030	-1.0	0.044	-0.8	0.063	0.3	0.018	-0.4
87	0.050	0.6	0.053	0.6	0.051	1.0	0.095	2.1	0.043	0.4	0.083	2.0	0.070	0.8	0.023	0.6
88	0.047	0.3	0.045	-0.1	0.040	-0.1	0.051	-0.7	0.049	1.0	0.044	-0.8	0.067	0.6	0.022	0.4
89	0.055	1.0	0.057	0.9			0.057	-0.3	0.025	-1.5	0.026	-2.1	0.088	2.0	0.017	-0.6
90																
91	0.044	0.0	0.046	0.0	FN	-4.0	0.058	-0.3	0.052	1.3	0.038	-1.2	0.049	-0.6	FN	-4.0
92	0.050	0.6	0.060	1.2	0.033	-0.8	0.067	0.3	0.053	1.4	0.047	-0.6	0.040	-1.3	0.020	0.0
93	0.042	-0.2	0.052	0.5			0.074	0.8	0.046	0.7	0.055	0.0	0.056	-0.2	0.020	0.0
94																
95																
96	0.046	0.2	0.054	0.7	0.069	2.7	0.067	0.3	0.046	0.7	0.061	0.4	0.067	0.6	0.025	1.0
97							0.087	1.6	0.048	0.8			0.073	1.0		
98	0.039	-0.4	0.044	-0.2			0.061	-0.1	0.031	-0.9	0.046	-0.6	0.044	-1.0	0.020	-0.1
99	0.031	-1.2	0.039	-0.6	0.072	3.0	0.075	0.8	0.036	-0.4	0.067	0.9	0.050	-0.6	0.020	0.0
100	0.041	-0.3	0.044	-0.2			0.054	-0.5	0.045	0.6	0.056	0.1	0.044	-1.0	0.009	-2.2
101	0.040	-0.4	0.045	-0.1			0.050	-0.8	0.040	0.1	0.049	-0.4	0.057	-0.1	0.023	0.6
102									0.028	-1.2					0.016	-0.8
103	0.048	0.4	0.045	-0.1	0.046	0.4	0.068	0.4	0.043	0.3	0.106	3.7	0.094	2.4	0.020	0.1
104	0.042	-0.2	0.045	-0.1	0.050	0.9	0.061	-0.1	0.037	-0.3	0.067	0.9	0.075	1.1	0.024	0.8
105	0.046	0.2	0.044	-0.2			0.052	-0.6	0.038	-0.2	0.049	-0.4	0.038	-1.4	0.022	0.4
106	0.051	0.6	0.049	0.2	0.048	0.7	0.082	1.3	0.080	4.1	0.065	0.7	0.079	1.4	0.013	-1.4
107	0.032	-1.1	0.042	-0.4	0.035	-0.6	0.053	-0.6	0.033	-0.7	0.050	-0.4	0.054	-0.3	0.020	0.0
108	0.040	-0.4	0.055	0.8	0.052	1.1	0.065	0.2	0.047	0.8	0.072	1.2	0.059	0.0	0.024	0.8
109	0.039	-0.4	0.045	-0.1	0.036	-0.5	0.054	-0.5	0.038	-0.2	0.049	-0.4	0.058	0.0	0.021	0.2
110	0.049	0.5	0.040	-0.5	0.031	-1.0	0.050	-0.8	0.039	-0.1	0.049	-0.4	0.050	-0.6	0.018	-0.4
111	0.043	-0.1	0.041	-0.4	0.037	-0.4	0.066	0.3	0.040	0.1	0.055	0.0			0.018	-0.5
112	0.054	0.9	0.047	0.1			0.069	0.4	0.050	1.1	0.062	0.5	0.072	0.9	0.023	0.6
113	0.047	0.3	0.049	0.2			0.075	0.8	0.043	0.4	0.058	0.2	0.074	1.1	0.023	0.6
114	0.042	-0.2	0.041	-0.5	0.026	-1.5	0.051	-0.7	0.032	-0.8	0.037	-1.3	0.039	-1.3	0.016	-0.8
115	0.044	0.0	0.039	-0.6	0.025	-1.5	0.052	-0.7	0.039	-0.1	0.043	-0.9	0.039	-1.3	0.016	-0.9
116	0.035	-0.8	0.044	-0.2			0.060	-0.1	0.037	-0.2	0.056	0.1	0.063	0.3	0.020	-0.1
117	0.051	0.6	FN	-4.0			0.092	1.9	0.051	1.2						
118	0.045	0.1					0.083	1.4	0.053	1.3					0.027	1.4
119	0.042	-0.2	0.031	-1.3			0.059	-0.2	0.042	0.3	0.038	-1.2	0.034	-1.7	0.016	-0.9
120	0.050	0.6	0.044	-0.2	0.041	0.0	0.065	0.2	0.035	-0.5	0.062	0.5	0.059	0.0	0.018	-0.4
121	0.042	-0.2	0.054	0.7	0.037	-0.4			0.038	-0.2	0.109	3.9	0.085	1.8	0.021	0.2

Laboratory code	Fludioxonil	Z-scores (FFP RSD (25%)	Isoprothiolane	Z-scores (FFP RSD (25%)	Mefentrifluconazole	Z-scores (FFP RSD (25%)	Metconazole	Z-scores (FFP RSD (25%)	Metribuzin	Z-scores (FFP RSD (25%)	Proquinazid	Z-scores (FFP RSD (25%)	Prosulfocarb	Z-scores (FFP RSD (25%)	Prothioconazole-Desthio	Z-scores (FFP RSD (25%)
MRRL	**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**	
Assigned value	**0.044**		**0.046**		**0.041**		**0.062**		**0.040**		**0.055**		**0.058**		**0.020**	
122							0.104	2.7	0.040	0.1	0.049	-0.4				
123	0.045	0.1	0.053	0.6			0.070	0.5	0.044	0.5	0.053	-0.1	0.060	0.1	0.023	0.6
124	0.030	-1.3	0.040	-0.5	FN	-4.0	0.054	-0.5	0.024	-1.6	0.034	-1.5	0.035	-1.6	FN	-4.0
125	0.046	0.2	0.052	0.5	0.042	0.1	0.066	0.3	0.034	-0.6	0.048	-0.5	0.054	-0.3	0.018	-0.4
126	0.047	0.3	0.051	0.4			0.060	-0.1	0.040	0.1	0.053	-0.1	0.061	0.2	0.020	0.0
127	0.035	-0.8	0.035	-1.0			0.059	-0.2								
128	0.041	-0.3	0.042	-0.4	0.039	-0.2	0.061	-0.1	0.036	-0.4	0.058	0.2	0.042	-1.1	0.019	-0.2
129	0.036	-0.8	0.041	-0.5	0.035	-0.6	0.057	-0.3	0.037	-0.3	0.066	0.8	0.060	0.1	0.023	0.6
131	0.041	-0.3	0.051	0.4	0.043	0.2	0.063	0.1	0.043	0.4	0.060	0.4	0.059	0.0	0.020	0.0
132	0.048	0.4	0.040	-0.5			0.053	-0.6	0.038	-0.2	0.101	3.3	0.051	-0.5	0.015	-1.0
133	0.040	-0.4	0.044	-0.2	0.048	0.7	0.066	0.3	0.042	0.3	0.062	0.5	0.068	0.7	0.024	0.8
134	0.070	2.4	0.072	2.2	FN	-4.0	0.070	0.5	0.156	>5	0.067	0.9	0.084	1.8	0.024	0.8
135	0.052	0.8	0.052	0.5	0.038	-0.3	0.063	0.0	0.040	0.1	0.064	0.7	0.049	-0.7	0.018	-0.3
136	0.042	-0.2	0.047	0.1	FN	-4.0	0.064	0.1	0.043	0.4	0.047	-0.6	0.052	-0.4	0.020	0.0
137	0.049	0.5	0.047	0.1			0.042	-1.3	0.053	1.4	0.052	-0.2	0.052	-0.4	0.018	-0.4
138	0.045	0.1					0.056	-0.4	0.040	0.1					FN	-4.0
139	0.094	4.6	0.061	1.3	FN	-4.0	0.084	1.4	0.063	2.4	FN	-4.0	FN	-4.0	0.030	2.0
141	0.042	-0.2	0.044	-0.2			0.057	-0.3	0.054	1.5	0.046	-0.7	0.073	1.0	0.015	-1.0
142	0.061	1.5	0.041	-0.5	0.048	0.7	0.069	0.4	0.034	-0.5	0.057	0.1	0.067	0.6	0.023	0.5
143	0.040	-0.4	0.053	0.6							0.010	-3.3	0.086	1.9	0.021	0.2
144	0.044	0.0	0.049	0.2	0.030	-1.1	0.052	-0.6	0.033	-0.7	0.057	0.1	0.059	0.0	0.021	0.2
145	0.045	0.1					0.070	0.5	0.037	-0.3						
146	0.048	0.4	0.056	0.8			0.068	0.4	0.056	1.6	0.067	0.9	0.065	0.5	0.025	1.1
147	0.055	1.0	0.046	0.0	0.065	2.3	0.086	1.5			0.080	1.8	0.075	1.1	0.018	-0.4
148	0.038	-0.5	0.037	-0.8	0.021	-2.0	0.043	-1.2	0.023	-1.7	0.042	-1.0	0.050	-0.6	0.015	-1.0
149	0.050	0.6	0.040	-0.5	FN	-4.0	0.041	-1.4	0.031	-0.9	0.035	-1.5	0.039	-1.3	0.014	-1.2
150	0.046	0.2	0.033	-1.1	0.033	-0.8	0.045	-1.1	0.030	-1.0	0.058	0.2	0.040	-1.3	0.022	0.4
151	40.00	>5	42.00	>5	29.00	>5	53.00	>5	36.00	>5	44.00	>5	44.00	>5	18.00	>5
152	0.035	-0.8	0.050	0.3			0.058	-0.3			0.056	0.1	0.048	-0.7		
153	0.037	-0.6	0.038	-0.7	0.034	-0.7	0.049	-0.8	0.032	-0.8	0.048	-0.5	0.056	-0.2	0.019	-0.2
154	0.032	-1.1	0.037	-0.8			0.071	0.6	0.038	-0.2	0.063	0.6	0.081	1.6	0.023	0.6
155	0.049	0.5	0.052	0.5			0.072	0.6	0.039	-0.1	0.078	1.7	0.081	1.6	0.025	1.0
156																
157	0.041	-0.3	0.040	-0.5	0.044	0.3	0.069	0.4	0.045	0.6	0.069	1.0	0.063	0.3	0.024	0.8
158	0.040	-0.4	0.045	-0.1	0.050	0.9	0.060	-0.1	0.038	-0.2	0.059	0.3	0.069	0.7	0.020	0.0
159	0.044	0.0	0.045	-0.1			0.055	-0.5	0.040	0.1	0.057	0.1	0.062	0.3	0.019	-0.2
160	0.023	-1.9					0.028	-2.2	0.030	-1.0	0.012	-3.1				
161	0.038	-0.5	0.044	-0.2	0.043	0.2	0.068	0.4	0.029	-1.1	0.081	1.9	0.078	1.4	0.022	0.3
162	0.060	1.5	0.041	-0.5	0.049	0.8	0.062	0.0	0.039	-0.1	0.060	0.3	0.058	0.0	0.023	0.6
163	0.046	0.2	0.055	0.8	FN	-4.0	0.061	-0.1	0.043	0.4	0.068	0.9	0.077	1.3	0.022	0.4
165	0.038	-0.5	0.038	-0.7	0.056	1.5	0.058	-0.3	0.043	0.4	0.036	-1.4	0.052	-0.4	0.018	-0.4

Table 10c. Assigned values, MRRL, preliminary z scores for the mandatory pesticides pyraclostrobin, spirotetramat-enol, and tau-fluvalinate, and the voluntary pesticides chlordane, cis-, and HCH, beta-.

Laboratory code	Prothioconazole-Desthio	Z-scores (FFP RSD (25%)	Pyraclostrobin	Z-scores (FFP RSD (25%)	Spirotetramat-enol	Z-scores (FFP RSD (25%)	Tau-Fluvalinate	Z-scores (FFP RSD (25%)	Chlordane, cis-	Z-scores (FFP RSD (25%)	HCH, beta-	Z-scores (FFP RSD (25%)
MRRL	**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**	
Assigned value	**0.020**		**0.028**		**0.058**		**0.028**		**0.031**		**0.028**	
1	0.023	0.6	0.028	-0.1	0.13	5.0	0.045	2.3	0.024	-0.9	0.014	-2.0
2	0.0152	-1.0	0.0217	-0.9			0.0186	-1.4	0.013	-2.3	0.0135	-2.1
3	0.021	0.2	0.0283	0.0	0.0451	-0.9	0.027	-0.2	0.0421	1.4	0.0417	1.9
4	0.0213	0.2					0.0298	0.2	0.0223	-1.2	0.0237	-0.7
5	0.018	-0.4	0.028	-0.1	0.063	0.4	0.027	-0.2	0.03	-0.2	0.031	0.4
6	0.016	-0.8	0.025	-0.5	0.0235	-2.4	0.028	-0.1	0.032	0.1	0.025	-0.5
7	0.021	0.2	0.032	0.5	0.081	1.6	0.046	2.5	0.036	0.6	0.033	0.6
8	0.027	1.4	0.035	0.9	0.085	1.9	0.025	-0.5	0.033	0.2	0.028	-0.1
9	0.016	-0.8	0.023	-0.8	0.057	-0.1	0.03	0.2	0.035	0.5	0.026	-0.3
11	0.019	-0.2					0.0317	0.5	0.0232	-1.0	0.0227	-0.8
12									0.037	0.7	0.019	-1.3
13	0.0178	-0.5					0.0318	0.5	0.0267	-0.6	0.0235	-0.7
14	0.023	0.6	0.032	0.5			0.028	-0.1	0.03	-0.2	0.026	-0.3
15	0.0142	-1.2	0.0204	-1.1	0.0528	-0.3	0.0251	-0.5	0.0283	-0.4	0.0252	-0.5
16	0.021	0.2	0.02	-1.2	0.048	-0.7	0.03	0.2	0.023	-1.1	0.02	-1.2
17			0.021	-1.0			0.028	-0.1	0.032	0.1	0.024	-0.6
18	0.019	-0.2	0.025	-0.5			0.035	0.9	0.026	-0.7	0.03	0.2
19	0.0282	1.6	0.0389	1.5			0.0286	0.0	0.027	-0.5	0.023	-0.8
20	0.011	-1.8	0.026	-0.3	0.09	2.2	0.03	0.2	0.059	3.5	0.043	2.1
22	0.0201	0.0	0.0237	-0.7	FN	-4.0	0.0306	0.3	0.0871	>5	0.0276	-0.1
23	0.022	0.4	0.029	0.1	0.0545	-0.2	0.03	0.2	0.0271	-0.5	0.0292	0.1
24												
25	0.026	1.2	0.0314	0.4	0.0596	0.1	0.0224	-0.9	0.0317	0.1	0.0317	0.5
26	0.015	-1.0	0.021	-1.0	0.065	0.5	0.016	-1.8	0.028	-0.4	0.025	-0.5
27	0.018	-0.4	0.027	-0.2			0.018	-1.5	0.021	-1.3	0.023	-0.8
28	0.023	0.6	0.03	0.2	0.06	0.2	0.026	-0.3	0.033	0.2	0.027	-0.2
29									0.0327	0.2	0.0275	-0.1
31	0.0165	-0.7	0.0214	-1.0			0.0264	-0.3				
32	0.018	-0.4	0.03	0.2	0.063	0.4	0.031	0.4	0.037	0.7	0.028	-0.1
33	0.0234	0.7	0.0306	0.3	0.0771	1.3	0.0315	0.4	0.0353	0.5	0.0375	1.3
34									0.042	1.4	0.031	0.4
35	0.0168	-0.7	0.029	0.1	0.173	>5	0.0287	0.0			0.0232	-0.7
36	0.023	0.6	0.044	2.2	FN	-4.0	0.031	0.4			0.032	0.5
37	0.02	0.0	0.027	-0.2	0.058	0.0	0.029	0.1			0.032	0.5
38	0.021	0.2	0.035	0.9	0.071	0.9	0.028	-0.1	0.034	0.3	0.032	0.5
39	0.031	2.2	0.044	2.2	0.093	2.4	0.064	5.0	0.04	1.1	0.042	1.9
40	0.024	0.8	0.027	-0.2	0.045	-0.9	0.031	0.4	0.031	0.0	0.033	0.6
41	0.017	-0.6	0.031	0.4	FN	-4.0	0.027	-0.2	FN	-4.0	0.032	0.5

Laboratory code	Prothioconazole-Desthio	Z-scores (FFP RSD (25%)	Pyraclostrobin	Z-scores (FFP RSD (25%)	Spirotetramat-enol	Z-scores (FFP RSD (25%)	Tau-Fluvalinate	Z-scores (FFP RSD (25%)	Chlordane, cis-	Z-scores (FFP RSD (25%)	HCH, beta-	Z-scores (FFP RSD (25%)
MRRL	**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**	
Assigned value	**0.020**		**0.028**		**0.058**		**0.028**		**0.031**		**0.028**	
42	0.014	-1.2	0.023	-0.8	0.033	-1.7	0.016	-1.8	0.032	0.1	0.029	0.1
43	0.022	0.4	0.033	0.7	0.044	-1.0	0.028	-0.1	0.027	-0.5	0.031	0.4
44	0.017	-0.6	0.025	-0.5	0.048	-0.7	0.021	-1.1	0.022	-1.2	0.024	-0.6
45	0.0191	-0.2	0.0253	-0.4	0.0487	-0.6	0.0607	4.5	0.0338	0.3	0.0324	0.6
46			0.024	-0.6					0.029	-0.3	0.032	0.5
47	0.02	0.0	0.03	0.2	0.06	0.2	0.028	-0.1	0.029	-0.3	0.03	0.2
48			0.0341	0.8								
49	0.018	-0.4	0.015	-1.9	FN	-4.0	0.018	-1.5	FN	-4.0	0.021	-1.0
50	0.0188	-0.3	0.0267	-0.2			0.0223	-0.9	0.0239	-0.9	0.0262	-0.3
51	0.022	0.4	0.033	0.7			0.03	0.2	0.029	-0.3	0.029	0.1
52	0.0191	-0.2	0.0275	-0.1	0.0502	-0.5	0.0258	-0.4	0.0311	0.0	0.0251	-0.5
53	0.0184	-0.3	0.0226	-0.8	0.067	0.6	0.0216	-1.0	0.0322	0.1	0.0282	0.0
54	0.019	-0.2	0.022	-0.9			0.03	0.2	0.026	-0.7	0.018	-1.5
55	0.028	1.6	0.024	-0.6	0.035	-1.6	FN	-4.0	FN	-4.0	0.029	0.1
56	0.0176	-0.5	0.0385	1.4	0.0526	-0.4	0.0272	-0.2	0.035	0.5	0.0292	0.1
57	0.031	2.2	0.033	0.7	0.024	-2.3	0.031	0.4	0.025	-0.8	0.019	-1.3
58	0.0103	-2.0	0.0185	-1.4	0.0216	-2.5	0.0148	-1.9	0.0231	-1.0	0.0246	-0.5
59	0.019	-0.2	FN	-4.0	FN	-4.0	0.038	1.3	0.032	0.1	0.027	-0.2
60	0.02	0.0	0.029	0.1	0.043	-1.0	0.189	>5	0.257	>5	0.027	-0.2
61	0.0201	0.0	0.027	-0.2	0.0556	-0.2	0.0239	-0.6	0.0362	0.6	0.0399	1.6
62	0.018	-0.4	0.026	-0.3	0.054	-0.3	0.033	0.6	0.048	2.1	0.035	0.9
63	FN	-4.0	0.0279	-0.1					0.015	-2.1	0.01	-2.6
64			0.018	-1.5			0.015	-1.9	FN	-4.0	FN	-4.0
65	0.018	-0.4	0.028	-0.1	0.058	0.0	0.0226	-0.8			0.0291	0.1
66	0.0211	0.2	0.0324	0.6	0.081	1.6	0.02	-1.2	0.024	-0.9	0.03	0.2
67	0.025	1.0	0.035	0.9	0.074	1.1	0.029	0.1				
68	0.0086	-2.3					0.0333	0.7	0.0124	-2.4	0.0128	-2.2
69	0.022	0.4	0.025	-0.5	0.051	-0.5	0.025	-0.5	0.031	0.0	0.032	0.5
70	0.0186	-0.3	0.0242	-0.6			0.0248	-0.5	0.039	1.0	FN	-4.0
71												
72	0.023	0.6	0.028	-0.1	0.068	0.7	0.029	0.1	0.037	0.7	0.033	0.6
73							FN	-4.0			0.019	-1.3
74			0.024	-0.6			0.022	-0.9				
75									0.027	-0.5	0.022	-0.9
76			0.022	-0.9			0.029	0.1	0.022	-1.2	0.021	-1.0
77									0.04	1.1	0.031	0.4
78	0.0222	0.4	0.0245	-0.5	0.0561	-0.1	0.0253	-0.5	0.0446	1.7	0.0291	0.1
79	0.0188	-0.3	0.0274	-0.1	0.112	3.8	0.0308	0.3	0.0173	-1.8	0.0311	0.4
80	0.036	3.2	0.039	1.5	0.111	3.7	0.028	-0.1				
81			0.022	-0.9			0.029	0.1	0.026	-0.7	0.021	-1.0

Laboratory code	Prothioconazole-Desthio	Z-scores (FFP RSD (25%)	Pyraclostrobin	Z-scores (FFP RSD (25%)	Spirotetramat-enol	Z-scores (FFP RSD (25%)	Tau-Fluvalinate	Z-scores (FFP RSD (25%)	Chlordane, cis-	Z-scores (FFP RSD (25%)	HCH, beta-	Z-scores (FFP RSD (25%)
MRRL	**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**	
Assigned value	**0.020**		**0.028**		**0.058**		**0.028**		**0.031**		**0.028**	
82	0.033	2.6	0.035	0.9	0.063	0.4	0.041	1.8	0.034	0.3	0.034	0.8
83	0.017	-0.6	0.029	0.1			0.021	-1.1	0.023	-1.1	0.03	0.2
84	0.0199	0.0	0.022	-0.9	0.055	-0.2	0.03	0.2	0.0247	-0.8	0.027	-0.2
85	0.0154	-0.9	0.024	-0.6			0.022	-0.9				
86	0.018	-0.4	0.018	-1.5	0.083	1.7	0.025	-0.5	0.028	-0.4	0.023	-0.8
87	0.023	0.6	0.028	-0.1	FN	-4.0	0.035	0.9	0.048	2.1	0.038	1.4
88	0.022	0.4	0.037	1.2	0.05	-0.5	0.028	-0.1	0.029	-0.3	0.091	>5
89	0.017	-0.6	0.022	-0.9	0.061	0.2	0.033	0.6	0.038	0.9	0.035	0.9
90												
91	FN	-4.0	0.029	0.1	FN	-4.0	0.085	>5	0.0275	-0.5	0.036	1.1
92	0.02	0.0	0.029	0.1	0.078	1.4	0.03	0.2	0.041	1.2	0.03	0.2
93	0.02	0.0	0.031	0.4	0.052	-0.4	0.025	-0.5	0.036	0.6	0.03	0.2
94									0.0261	-0.7	0.0208	-1.1
95									0.035	0.5	0.03	0.2
96	0.025	1.0	0.036	1.1	0.069	0.8	0.035	0.9				
97			0.042	1.9			0.0237	-0.7	0.037	0.7	0.0397	1.6
98	0.0197	-0.1	0.0278	-0.1			0.0213	-1.0				
99	0.02	0.0	0.025	-0.5	0.033	-1.7	0.021	-1.1	0.052	2.6	0.027	-0.2
100	0.0089	-2.2	0.0333	0.7	0.0544	-0.2	0.0282	0.0	0.0303	-0.1	0.032	0.5
101	0.023	0.6	0.025	-0.5	0.051	-0.5	0.03	0.2	0.031	0.0	0.035	0.9
102	0.016	-0.8	0.034	0.8					0.027	-0.5	0.021	-1.0
103	0.0204	0.1	0.0348	0.9	FN	-4.0	0.0327	0.6	0.0392	1.0	0.0349	0.9
104	0.024	0.8	0.03	0.2	0.049	-0.6	0.032	0.5	0.031	0.0	0.027	-0.2
105	0.022	0.4	0.025	-0.5			0.026	-0.3				
106	0.013	-1.4	0.042	1.9	0.059	0.1	0.042	1.9	0.024	-0.9	0.031	0.4
107	0.02	0.0	0.025	-0.5	0.044	-1.0	0.025	-0.5	0.03	-0.2	0.028	-0.1
108	0.024	0.8	0.034	0.8	0.015	-3.0	0.042	1.9	0.024	-0.9	0.037	1.2
109	0.021	0.2	0.027	-0.2	0.061	0.2	0.03	0.2				
110	0.018	-0.4	0.023	-0.8	0.064	0.4	0.034	0.8	0.031	0.0	0.028	-0.1
111	0.0176	-0.5					0.03	0.2	0.0248	-0.8	0.022	-0.9
112	0.023	0.6	0.032	0.5	FN	-4.0	0.027	-0.2	0.03	-0.2	0.027	-0.2
113	0.023	0.6	0.034	0.8			0.037	1.2				
114	0.016	-0.8	0.023	-0.8	0.038	-1.4	0.028	-0.1				
115	0.0158	-0.9	0.0288	0.1	0.0483	-0.7	0.0276	-0.1	0.0229	-1.1	0.015	-1.9
116	0.0197	-0.1	0.0269	-0.2			0.0217	-1.0	0.0263	-0.6	0.0268	-0.2
117			0.042	1.9			0.039	1.5	0.04	1.1	0.036	1.1
118	0.027	1.4	0.0305	0.3			0.0388	1.5				
119	0.0155	-0.9	0.026	-0.3			0.027	-0.2				
120	0.018	-0.4	0.03	0.2	0.059	0.1	FN	-4.0				
121	0.021	0.2	0.035	0.9	0.07	0.8	0.09	>5				

Laboratory code	Prothioconazole-Desthio	Z-scores (FFP RSD (25%)	Pyraclostrobin	Z-scores (FFP RSD (25%)	Spirotetramat-enol	Z-scores (FFP RSD (25%)	Tau-Fluvalinate	Z-scores (FFP RSD (25%)	Chlordane, cis-	Z-scores (FFP RSD (25%)	HCH, beta-	Z-scores (FFP RSD (25%)
MRRL	**0.005**		**0.005**		**0.005**		**0.005**		**0.005**		**0.005**	
Assigned value	**0.020**		**0.028**		**0.058**		**0.028**		**0.031**		**0.028**	
122			0.0244	-0.6								
123	0.023	0.6	0.03	0.2	0.058	0.0	0.028	-0.1	0.028	-0.4	0.03	0.2
124	FN	-4.0	0.023	-0.8	0.056	-0.1	0.023	-0.8	0.024	-0.9	FN	-4.0
125	0.018	-0.4	0.027	-0.2	0.12	4.3	0.026	-0.3			0.031	0.4
126	0.02	0.0	0.0291	0.1			0.026	-0.3				
127			0.028	-0.1			0.031	0.4	FN	-4.0	0.018	-1.5
128	0.0193	-0.2	0.0261	-0.3	0.0533	-0.3	0.0284	0.0	0.0263	-0.6	0.0256	-0.4
129	0.0233	0.6	0.022	-0.9	0.0461	-0.8	0.0273	-0.2			0.0251	-0.5
131	0.02	0.0	0.026	-0.3	0.06	0.2	0.031	0.4	0.04	1.1	0.034	0.8
132	0.015	-1.0	0.034	0.8			0.031	0.4	0.047	2.0	0.023	-0.8
133	0.024	0.8	0.027	-0.2	0.084	1.8	0.031	0.4	0.034	0.3	0.03	0.2
134	0.024	0.8	0.03	0.2	0.088	2.1	FN	-4.0	0.048	2.1	0.053	3.5
135	0.0184	-0.3	0.027	-0.2	0.0474	-0.7	0.029	0.1	0.0324	0.1	0.0313	0.4
136	0.02	0.0	0.035	0.9	0.065	0.5	0.033	0.6	0.034	0.3	0.028	-0.1
137	0.018	-0.4	0.032	0.5	0.057	-0.1			0.033	0.2	0.032	0.5
138	FN	-4.0	0.025	-0.5			0.029	0.1				
139	0.03	2.0	0.043	2.1	FN	-4.0	FN	-4.0	FN	-4.0	FN	-4.0
141	0.015	-1.0	0.027	-0.2	0.037	-1.4	0.025	-0.5				
142	0.0228	0.5	0.0332	0.7	0.086	2.0	0.0317	0.5			0.0241	-0.6
143	0.021	0.2	0.028	-0.1	FN	-4.0	0.026	-0.3				
144	0.021	0.2	0.031	0.4			0.026	-0.3				
145			0.0273	-0.2			FN	-4.0	0.0219	-1.2	0.0195	-1.3
146	0.0254	1.1	0.0373	1.3	0.0576	0.0	0.0347	0.9	0.04	1.1	0.0327	0.6
147	0.018	-0.4	0.035	0.9	0.038	-1.4	0.028	-0.1	0.029	-0.3	0.03	0.2
148	0.0151	-1.0	0.0215	-1.0	0.0348	-1.6	0.0247	-0.5	0.0278	-0.4	0.0242	-0.6
149	0.014	-1.2	0.019	-1.3	0.046	-0.8	0.024	-0.6	0.031	0.0	0.016	-1.7
150	0.022	0.4	0.022	-0.9	0.032	-1.8	0.031	0.4	0.027	-0.5	0.026	-0.3
151	18	>5	24	>5	33	>5	28	>5	20	>5	21	>5
152			0.026	-0.3	FN	-4.0						
153	0.019	-0.2	0.029	0.1	0.051	-0.5	0.023	-0.8	0.026	-0.7	0.023	-0.8
154	0.023	0.6	0.03	0.2			FN	-4.0				
155	0.025	1.0	0.026	-0.3	0.048	-0.7			0.042	1.4	0.035	0.9
156												
157	0.024	0.8	0.024	-0.6	0.06	0.2	0.04	1.6	0.036	0.6	0.036	1.1
158	0.02	0.0	0.029	0.1	0.048	-0.7	0.024	-0.6	0.025	-0.8	0.023	-0.8
159	0.019	-0.2	0.025	-0.5			0.034	0.8	0.034	0.3	0.036	1.1
160									0.03	-0.2	0.032	0.5
161	0.0215	0.3	0.03	0.2	0.0406	-1.2	0.0235	-0.7				
162	0.0233	0.6	0.0241	-0.6	0.0752	1.2	0.0247	-0.5				
163	0.022	0.4	0.032	0.5	FN	-4.0	0.027	-0.2				
165	0.018	-0.4	0.028	-0.1	0.046	-0.8	0.041	1.8				

Table 10d. Reported results for bifenazate and bifenazate-Diazene.

Laboratory code	Bifenazate	Bifenazate-Diazene
MRRL	0.005	0.005
1		0.022
2	0.032	
3	0.025	0.025
4		
5		
6		
7		
8		
9		
11		
12		
13		
14		
15	0.03	
16	0.04	
17		
18	0.05	
19		
20	0.028	0.02
22	0.044	
23	0.032	0.019
24		
25		
26	0.01	0.016
27		
28	0.052	0.019
29		
31		
32		
33	0.016	0.017
34		
35		
36		
37	0.031	0.01
38		
39	0.019	
40	0.035	
41		
42	0.011	
43	0.035	
44	0.025	0.027
45		

Laboratory code	Laboratory code	Bifenazate-Diazene
MRRL	MRRL	0.000
46		
47	0.022	0.015
48		
49	0.015	
50		
51	0.02	
52		0.021
53	0.021	
54		
55		0.026
56	0.05	
57		
58	0.01	0.006
59	0.007	
60	0.015	0.007
61	0.04	0.02
62	0.027	
63		
64	0.02	
65		
66	0.033	
67		
68		
69	0.015	0.024
70		
71		
72	0.033	0.031
73		
74		
75		
76		
77		
78		
79	0.013	0.014
80		
81		
82	0.019	0.038
83		
84	0.01	0.02
85		
86		
87	0.421	0.018

Laboratory code	Bifenazate	Bifenazate-Diazene
MRRL	0.000	MRRL
88	0.023	
89	0.03	
90		
91	0.022	
92	0.04	
93	0.014	
94		
95		
96	0.032	0.016
97	0.027	0.023
98		
99	0.053	0.028
100		
101	0.035	
102		
103		
104	0.029	
105		
106		
107	0.03	
108	0.02	
109		
110	0.031	
111		
112	0.025	0.025
113		
114		
115	0.036	0.016
116		
117		
118		
119		
120		
121		
122		
123	0.018	
124	0.012	
125		
126		
127		
128	0.038	
129		

Laboratory code	Bifenazate	Bifenazate-Diazene
MRRL	0.000	0.000
129		
131	0.034	0.011
132	0.022	
133	0.032	
134	0.029	
135	0.035	
136		0.029
137		
138		
139		
141		
142		
143		
144		
145	0.03	
146	0.036	
147		
148	0.025	
149	0.011	0.028
150	0.04	0.023
151		
152	0.049	
153	0.015	0.026
154		
155		
156		
157	0.044	0.029
158		0.014
159	0.014	0.011
160		
161		
162		
163		
165		

Table 10f. Reported results for Alpha-cypermethrin.

Laboratory code	Alpha-cypermethrin
MRRL	0.005
Assigned value	
1	
2	
3	0.008
4	
5*	0.021
6*	0.005
7	
8*	0.045
9*	0.016
11	
12	
13	
14	
15	
16*	0.046
17	
18	
19	
20	
22	
23*	0.014
24	
25	
26*	0.029
27	0.014
28	0.041
29	
31	
32	
33*	0.017
34	
35	0.019

Laboratory code	Alpha-cypermethrin
MRRL	MRRL
Assigned value	
36	
37	
38	
39	
40*	0.008
41*	0.022
42	
43	
44*	0.021
45	
46	
47	
48	
49*	0.016
50	
51	
52	
53	
54	
55	
56	
57	
58	
59	
60*	0.009
61	
62	
63	
64	
65	
66	
67	

Laboratory code	Alpha-cypermethrin
MRRL	0.000
Assigned value	
68*	0.018
69*	0.011
70*	0.007
72	
73	
74	
75	
76	
77	
78*	0.045
79	
80*	0.031
81*	0.025
82	0.010
83	
84*	0.018
85	
86*	0.020
87	
88*	0.047
89	
91	
92	0.010
93	
94	
95	0.017
96	
97	
98	
99	
100*	0.028
101	

Laboratory code	Alpha-cypermethrin
MRRL	0.000
Assigned value	
102	
103	
104	0.016
105	
106	
107	
108*	0.050
109	
110	
111	
112	
113	
114	0.014
115	
116	
117	
118	
119	
120	
121	
122	
123	
124	
125	
126	
127	0.019
128	
129	
131	
132*	0.025
133	
134	

Laboratory code	Alpha-cypermethrin
MRRL	0.005
Assigned value	
135	
136	
137	0.024
138	
139	
141	
142	
143	
144	0.046
145	
146	
147*	0.004
148	
149	
150*	0.013
151	
152	
153	0.005
154	
155*	0.011
157	
158	
159	
160	
161	
162	
163	
165	

* marked participants result were submitted by email after the deadline on request from the organiser.

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 (≥17 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 87 EU and EFTA laboratories in Category A (58%), the results were additionally evaluated by calculating the Average of the Squared Score (AZ^2). Of the 87 participants 75 participants (86%) obtained AZ2 score at or below 2 (good), 7 participants (8%) obtained AZ2 values between 2-3 (satisfactory) and 5 participants (6%) obtained AZ2 values ≥3 (unsatisfactory). An additional three laboratories from Third Countries were evaluated and classified into Category A. The AZ2 scores achieved by the labs can be seen in **Table 11**.

Table 11. 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	Analysed from mandatory target list, %	No. of detected voluntary pesticides	AZ^2	False negative	Classification	NRL
2	17	91	2	0.4	0	Good	
3	**19**	**100**	**3**	**0.2**	**0**	**Good**	
5	**19**	**100**	**3**	**0.2**	**1**	**Good**	
6	19	99	3	0.6	1	Good	NRL-CF
7	17	95	2	0.6	0	Good	
8	19	98	3	1.5	1	Good	
9	19	100	3	0.3	1	Good	
14	18	92	2	0.8	0	Good	
15	19	100	2	0.8	0	Good	NRL-CF
16	19	98	3	0.4	0	Good	
18	17	95	2	1.4	0	Good	
19	17	94	2	1.1	0	Good	
20	18	95	2	1.0	0	Good	
22	18	99	3	1.1	1	Good	
23	19	100	3	0.4	1	Good	
25	19	98	2	0.8	0	Good	NRL-CF
26	19	100	3	0.6	1	Good	
28	19	100	3	0.2	0	Good	
31	17	97	0	0.5	0	Good	
33	18	99	3	0.3	1	Good	
35	17	96	2	1.8	0	Good	
36	18	99	2	1.4	2	Good	NRL-CF
37	17	91	1	0.7	0	Good	NRL-FE
39	19	100	2	3.8	0	Unsatisfactory	
44	19	100	3	0.7	1	Good	
45	19	97	2	1.7	0	Good	
47	19	100	2	0.3	0	Good	
52	19	100	3	0.2	1	Good	
53	19	99	2	0.4	0	Good	
56	18	98	2	0.7	0	Good	NRL-CE
57	18	100	2	3.2	1	Unsatisfactory	

Lab code	No. of detected mandatory pesticides	Analysed from mandatory target list, %	No. of detected voluntary pesticides	AZ[2]	False negative	Classification	NRL
58	18	97	2	1.6	0	Good	
59	17	100	2	3.4	2	Unsatisfactory	NRL-CF
60	19	100	3	1.9	1	Good	NRL-CF
61	19	100	2	0.3	0	Good	
62	19	100	3	0.2	1	Good	
66	18	97	2	1.2	0	Good	
67	19	100	0	1.5	0	Good	NRL-CE
69	19	99	3	0.2	1	Good	
72	19	100	2	0.2	0	Good	
78	19	97	3	0.2	1	Good	
80	19	98	1	4.9	1	Unsatisfactory	
82	19	97	3	1.4	0	Good	NRL-CF
84	19	97	3	0.2	1	Good	NRL-FE
86	17	98	3	2.5	3	Satisfactory	NRL-CF
87	18	100	2	2.2	1	Satisfactory	
88	18	97	3	0.6	1	Good	
89	18	97	2	1.3	0	Good	
96	19	97	0	0.8	0	Good	
98	17	95	0	0.4	0	Good	
99	19	100	2	1.2	0	Good	
100	18	97	3	0.5	1	Good	NRL-CF
101	17	95	2	0.4	0	Good	
103	18	99	2	2.1	1	Satisfactory	NRL-CF
104	19	100	3	0.4	0	Good	NRL-CF
106	19	100	2	2.5	0	Satisfactory	
107	19	100	2	0.5	0	Good	
108	19	100	3	1.2	1	Good	
109	19	100	0	0.1	0	Good	NRL-FE
110	19	99	2	0.4	0	Good	
113	17	97	0	0.7	0	Good	
114	19	98	1	0.9	0	Good	
115	18	98	2	0.6	0	Good	NRL-CF
116	17	94	2	0.2	0	Good	
120	18	99	0	1.1	1	Good	
123	18	93	2	0.2	0	Good	
124	17	100	2	2.8	3	Satisfactory	
125	19	100	1	1.2	0	Good	
128	19	96	2	0.2	0	Good	
129	19	100	1	0.3	0	Good	NRL-CE
131	19	99	3	0.1	1	Good	
132	17	94	3	1.0	1	Good	NRL-FE

Lab code	No. of detected mandatory pesticides	Analysed from mandatory target list, %	No. of detected voluntary pesticides	AZ[2]	False negative	Classification	NRL
133	19	100	3	0.3	1	Good	
134	17	100	2	>5	2	Unsatisfactory	
135	19	98	2	0.2	0	Good	
136	18	98	2	1.2	1	Good	
142	19	99	1	0.6	0	Good	NRL-CE
144	17	90	1	0.4	0	Good	
146	18	98	2	0.8	0	Good	NRL-CE
147	18	97	3	1.3	1	Good	
148	19	100	2	1.8	0	Good	
149	17	100	2	3.0	2	Satisfactory	
150	19	100	3	0.7	1	Good	
153	19	100	3	0.3	0	Good	
157	19	100	2	0.5	0	Good	
158	19	100	2	0.2	0	Good	
161	19	100	0	0.6	0	Good	
162	19	100	0	0.6	0	Good	
163	17	100	0	2.2	2	Satisfactory	
165	19	100	0	0.6	0	Good	

The 64 laboratories from EU and EFTA countries 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 12**. Six laboratories were moved from Category A to B due to false positive results. Two participants fulfilled the criteria of detecting at least 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. Fifteen participants analysed more than 90% of the pesticides on the Target List but reported <17 pesticides in the Test Item.

Table 12. 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 mandatory pesticides detected	Mandatory pesticides detected in test item, %	Analysed of mandatory pesticides on Target List, %	No. Of voluntary pesticides detected	No. of acceptable z score	No. of false negative	No. of false positive	NRL
1	18	95	87	2	14	0	1	
4	15	79	53	2	15	0	0	
11	15	79	49	2	15	0	0	
12	0	0	3	2	0	0	0	
13	15	79	53	2	15	0	0	
17	11	58	59	2	10	0	1	
24	5	26	18	0	3	0	0	
27	13	68	80	3	13	0	0	
29	0	0	3	2	0	0	0	
32[1]	19	100	100	2	18	0	1	NRL-CE
34	0	0	2	2	0	0	0	NRL-FE

Lab code	No. of mandatory pesticides detected	Mandatory pesticides detected in test item, %	Analysed of mandatory pesticides on Target List, %	No. Of voluntary pesticides detected	No. of acceptable z score	No. of false negative	No. of false positive	NRL
38	16	84	91	2	16	0	0	
40[1]	19	100	99	3	19	1	1	NRL-CF
41	15	79	92	3	15	4	0	
42	16	84	91	2	16	0	0	
43[1]	18	95	100	3	17	2	1	
46	10	53	63	2	10	0	0	
48	7	37	57	0	7	1	0	
49	14	74	99	3	10	7	0	
50	14	74	86	2	14	0	0	
51	16	84	81	2	16	0	0	
54	16	84	80	2	16	0	0	
55	16	84	100	2	16	4	0	
63	9	47	76	2	6	3	0	
64	13	68	69	2	13	3	0	
65[1]	18	95	100	1	18	1	1	NRL-FE
68	9	47	57	3	2	1	0	
70	17	89	90	3	17	2	0	NRL-CF
71	0	0	7	0	0	0	0	
73	4	21	56	1	2	1	0	
74	11	58	55	0	11	0	0	
75	1	5	21	2	1	0	0	NRL-CF
76	8	42	61	2	7	0	0	
77	0	0	3	2	0	0	0	
79[1]	19	100	99	3	17	0	2	
81	16	84	88	3	16	0	0	
83	16	84	93	2	16	0	0	
85	13	68	63	0	13	0	0	
90	0	0	32	0	0	0	0	
91	14	74	99	3	11	6	1	NRL-CF
92[1]	19	100	100	3	19	0	1	
93	18	95	98	2	18	0	3	NRL-CF
94	0	0	3	2	0	0	0	
95	1	5	9	3	1	0	0	NRL-FE
97	10	53	56	2	10	0	0	
102	8	42	49	2	8	0	0	
105	16	84	90	0	16	0	0	NRL-CF
111	15	79	49	2	15	0	0	
112	17	89	99	2	17	1	1	
117	11	58	79	2	10	1	0	NRL-CF
118	12	63	75	0	12	0	0	
119	16	84	91	0	16	0	0	
121	16	84	86	0	12	1	0	
122	9	47	35	0	6	0	0	
126	16	84	91	0	16	0	0	
127	11	58	74	3	10	1	0	

Lab code	No. of mandatory pesticides detected	Mandatory pesticides detected in test item, %	Analysed of mandatory pesticides on Target List, %	No. Of voluntary pesticides detected	No. of acceptable z score	No. of false negative	No. of false positive	NRL
137	16	84	89	3	15	0	0	
138	11	58	90	0	11	2	0	
139	12	63	100	3	4	10	0	NRL-CE
141	17	89	88	0	17	1	0	
143	13	68	90	1	12	4	0	NRL-CF
145	10	53	68	2	9	1	0	
151	18	95	99	2	0	0	1	
152	13	68	76	0	13	1	0	
154	15	79	90	0	15	1	0	NRL-CE
155	16	84	94	3	15	1	0	
156	0	0	70	0	0	0	0	
159	15	79	88	3	15	1	0	
160	9	47	56	2	4	0	1	

[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-CF13 and forward can be seen in Table 13.

Table 13. Overall trends in participation of laboratories, pesticides in the target list and test item, and performance of laboratories in the 7 latest EUPTs cereals.

PT and types of test item	EUPT-CF13 Oat kernels	EUPT-CF14 Rice kernels	EUPT-CF15 Rapeseed cake	EUPT-CF16 Barley Kernels	EUPT-CF17 Wheat Kernels	EUPT-CF18 Wheat Straw	EUPT-CF19 Rye Kernels
Participants submitting results (EU+EFTA)	149	156	129	151	149	90	151
MRM pesticides in the Target Pesticide List	160/32	164/38	172/41	169/53	169/58	170/63	174/62
MRM pesticides in the test material	18	19	22	19	20	12	21
No. of results for MRM pesticides	2007	2298	1315	2091	2422	925	2656
Average of 'reported results', %	75	80	83	78	85	85	84
Range of 'reported results', %	44-94	26-93	57-93	32-97	48-95	48-95	56-95
Acceptable z scores, %	93	91	87	89	92	92	91
Questionable z scores, %	3.1	3	7	4	3	4	3
Unacceptable z scores, %	3.4	6	6	6	5	4	6
False negatives, %	2.3	3.4	1.9	2.8	2.8	0.6	1.7
Number of false positives	3	14	9.0	25	7	10	19
Category A, % of participating laboratories	57	57	57	59	73	71	58
Good AZ^2, %	91	91	67	83	89	87	86
Satisfactory AZ^2, %	5.7	6.7	12	7	6	6	8
Unsatisfactory AZ^2, %	3.4	2.2	22.0	10	6	7	6
Alg A RSD%	18	19	30	22	18	23	20

The number of pesticides included in the Target Pesticide List has also increased during this 18-year period, from 43 34 to 174 compulsory compounds and 62 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, 21% of the laboratories were not able to analyse and detect more than 70% of pesticides present in the Test Item. The analytical scope was in average 84%.

The overall analytical performance (accuracy of measurement) if looking at the percentage of acceptable, questionable, unacceptable z scores has increased during the last 3 EUPTs, and in EUPT-CF19 91% 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 75-85%. The false negative results have fluctuated between 1-4%. Also, the false positive results have been going up and down, in EUPT-CF19 the number increased to 12.

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. In EUPT-CF19 it decreases to 58%. For Category A the percentage of participant with AZ2 was <2 (good) has been >90% for many years. However, for the rapeseed cake EUPT-CF15 this dropped significantly to 67% and in this EUPT it was 86%.

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

The EUPT-CF19 Test Item was rye kernels containing incurred and spiked pesticides. The rye 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: aclonifen, azoxystrobin, bixafen, chlorantraniliprole, cypermethrin (sum), cyprodinil, difenoconazole, dimethomorph, fludioxonil, isoprothiolane, mefentrifluconazole, metconazole, metribuzin, proquinazid, and prosulfocarb, prothioconazole-desthio, pyraclostrobin, spirotetramat-enol, and tau-fluvalinate, and the voluntary pesticides chlordane-cis-, and HCH-beta, alpha-cypermethrin and bifenazate-diazene. One hundred fifty-one EU and EFTA laboratories, from 29 different countries agreed to participate in this proficiency test. One of them did not report any results. An additional 8 laboratories from EU candidate states and Third Countries registered for the PT and only one did not submitted results. The Target Pesticide List distributed to the laboratories prior to the test contained 174 individual compulsory and 62 voluntary compounds.

The number of false positives and false negatives has varied between the EUPTs. Nineteen false positive results were reported and the number of false negatives represented 22% of the total number of results. This is at levels typically seen in EUPT-CFs. The average Alg A-RSD was at 20%, lower than the FFP-RSD of 25%.

Of the reported results for the evaluated pesticides, more than 90% were aclonifen, azoxystrobin, bixafen, chlorantraniliprole, cypermethrin (sum), cyprodinil, difenoconazole, dimethomorph, fludioxonil, HCH- bet, isoprothiolane, metconazole, metribuzin, proquinazid, prosulfocarb, prothioconazole-desthio and pyraclostrobin. For chlordane-cis, mefentrifluconazole, spirotetramat-enol and fluvalinate-tau the range of the acceptable results vary between 77 to 89 %.

Results reported for two voluntary compounds, bifenazate-diazene and alpha-cypermethrin, were not evaluated due to the validity of the data. Many laboratories used a method that converted bifenazate-diazene back to bifenazate all though the laboratories are asked to analyse for the specific compounds on the target list. For alpha-cypermethrin only 14 laboratories gave results before the deadline.

The Test Item for EUPT-CF20 will be oat kernels and are planned to be shipped to the laboratories in February 2026. 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

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

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 15662:2018 - Foods of plant origin - Multimethod for the determination of pesticide residues using GC- and LC-based analysis following acetonitrile extraction/partitioning and clean-up by dispersive SPE - Modular QuEChERS-method.

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

[5] ISO 17043:2023– Conformity assessment — General requirements for the competence of proficiency testing providers

APPENDICES

Appendix 1: Target Pesticide List

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

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

Pesticides	MRRL (mg/kg)
Indoxacarb	0.005
Iprodione	0.005
Isocarbophos	0.005
Isofetamid*	0.005
Isoprothiolane	0.005
Isoproturon	0.005
Kresoxim-methyl	0.005
Lambda-cyhalothrin	0.005
Lindane	0.005
Linuron	0.005
Malaoxon	0.005
Malathion	0.005
Mandipropamid	0.005
Mefentrifluconazole*	0.005
Metaflumizone	0.005
Metalaxyl	0.005
Metamitron*	0.005
Metconazole	0.005
Methacrifos	0.005
Methamidophos	0.005
Methomyl	0.005
Metolachlor	0.005
Metrafenone	0.005
Metribuzin	0.005
Omethoate	0.003
Oxydemeton-methyl	0.005
Paclobutrazol	0.005
Parathion	0.005
Penconazole	0.005
Pencycuron	0.005
Pendimethalin	0.005
Permethrin	0.005
Phosphamidon	0.005
Pirimicarb	0.005
Pirimiphos-methyl	0.005
Prochloraz	0.005
Procymidone	0.005
Profenofos	0.005
Propamocarb	0.005
Propiconazole	0.005
Proquinazid	0.005
Prosulfocarb	0.005
Prothioconazole-desthio	0.005
Prothiofos	0.005
Pymetrozine	0.005
Pyraclostrobin	0.005
Pyridaben	0.005

Pesticides	MRRL (mg/kg)
Pyridalyl	0.005
Pyrimethanil	0.005
Pyriproxyfen	0.005
Quinoxyfen	0.005
Spinetoram	0.005
Spirodiclofen	0.005
Spiromesifen	0.005
Spirotetramat	0.005
Spirotetramat metabolite BYI08330-enol	0.005
Spiroxamine	0.005
Sulfoxaflor	0.005
Tau-Fluvalinate	0.005
Tebuconazole	0.005
Tebufenozide	0.005
Teflubenzuron	0.005
Tefluthrin	0.005
Terbuthylazine	0.005
Tetraconazole	0.005
Tetradifon	0.005
Tetramethrin	0.005
Thiabendazole	0.005
Thiacloprid	0.005
Thiamethoxam	0.005
Thiodicarb	0.005
Thiophanate-methyl	0.005
Tolclofos-methyl	0.005
Triadimefon	0.005
Triadimenol	0.005
Triazophos	0.005
Tricyclazole	0.005
Trifloxystrobin	0.005
Triflumizole	0.005
Triflumizole metabolite (FM-6-1)	0.005
Trifluralin	0.005
Triticonazole	0.005
Vinclozolin	0.005
Zoxamide	0.005
Voluntary Compounds (will not be considered in Category A/B classification)	
Alpha-cypermethrin*	0.005
1,4-Dimethylnaphthalene	0.005
Bifenazate *	0.005
Bifenazate-Diazene*	0.005
Benalaxyl (sum)	0.005
Benzovindiflupyr	0.005
Chlordane-cis	0.005
Chlordane-oxy	0.005

Pesticides	MRRL (mg/kg)
Chlordane-trans	0.005
Chlorfluazuron	0.005
Clomazone	0.005
Cyflufenamid	0.005
Cyhalofop-butyl	0.005
DDD-pp	0.005
DDE-pp	0.005
DDT-op	0.005
DDT-pp	0.005
Dinotefuran	0.005
Diuron	0.005
Endrin	0.005
Endrin-ketone	0.005
Fenobucarb	0.005
Fenpicoxamid	0.005
Florpyrauxyfen-benzyl	0.005
Fluazinam	0.005
Fluensulfone	0.005
Flufenacet	0.005
Flutianil	0.005
Forchlorfenuron	0.005
HCH-alpha	0.005
HCH-beta	0.005
Heptachlor	0.005
Heptachlorepoxid-cis	0.005
Heptachlorepoxid-trans	0.005
Isopyrazam	0.005
Isoxaflutole	0.005
Metaldehyde	0.005
Metazachlor	0.005
Metobromuron	0.005
Molinate	0.005
Novaluron	0.005
Oxadiargyl	0.005
Oxathiapiprolin	0.005
Oxyfluorfen	0.005
Penflufen	0.005
Pentachloro-aniline	0.005
Penthiopyrad	0.005
Phenmedipham	0.005
Picolinafen	0.005
Propaquizafop	0.005
Pyrethrins	0.005
Pyridate	0.005
Pyriofenone	0.005
Quinalphos	0.005
Quinoclamine	0.005

Pesticides	MRRL (mg/kg)
Quintozene	0.005
Rotenone	0.005
Tolfenpyrad	0.005
Tri-allate	0.005
Triclopyr	0.005
Trinexapac (free acid)*	0.005
Tritosulfuron	0.005

*New compounds

Appendix 2 Homogeneity data

	Aclonifen mg/kg		Azoxystrobin mg/kg		Bixafen mg/kg		Chlorantraniliprole mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
013	0.327	0.348	0.066	0.070	0.031	0.033	0.047	0.048
025	0.273	0.281	0.064	0.062	0.030	0.028	0.045	0.043
063	0.260	0.273	0.061	0.057	0.030	0.026	0.042	0.039
092	0.310	0.311	0.060	0.056	0.030	0.029	0.042	0.039
139	0.354	0.388	0.060	0.055	0.030	0.028	0.042	0.040
159	0.293	0.277	0.063	0.061	0.031	0.030	0.043	0.042
197	0.319	0.342	0.059	0.064	0.029	0.030	0.041	0.045
215	0.339	0.279	0.066	0.062	0.031	0.028	0.045	0.042
229	0.285	0.346	0.062	0.055	0.030	0.027	0.043	0.038
240	0.362	0.314	0.060	0.054	0.032	0.029	0.042	0.038

	Cypermethrin mg/kg		Cyprodinil mg/kg		Fludioxonil mg/kg		Isoprothiolane mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
013	0.039	0.046	0.664	0.671	0.047	0.049	0.048	0.054
025	0.034	0.035	0.567	0.563	0.044	0.046	0.049	0.049
063	0.032	0.032	0.530	0.518	0.037	0.036	0.047	0.044
092	0.045	0.038	0.645	0.604	0.046	0.043	0.045	0.042
139	0.050	0.048	0.719	0.681	0.049	0.049	0.044	0.042
159	0.038	0.039	0.581	0.562	0.040	0.040	0.047	0.046
197	0.038	0.042	0.597	0.641	0.043	0.045	0.045	0.049
215	0.044	0.038	0.630	0.559	0.050	0.038	0.052	0.048
229	0.032	0.043	0.580	0.642	0.038	0.046	0.047	0.042
240	0.048	0.046	0.735	0.627	0.047	0.043	0.044	0.039

	Mefentrifluconazole mg/kg		Metconazole mg/kg		Metribuzin mg/kg		Proquinazid mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
013	0.049	0.053	0.073	0.080	0.049	0.052	0.055	0.057
025	0.045	0.040	0.069	0.064	0.045	0.046	0.047	0.047
063	0.048	0.043	0.071	0.065	0.040	0.039	0.042	0.044
092	0.048	0.046	0.070	0.068	0.045	0.044	0.058	0.051
139	0.044	0.046	0.063	0.067	0.050	0.049	0.062	0.061
159	0.048	0.048	0.076	0.071	0.044	0.041	0.051	0.049
197	0.046	0.053	0.067	0.078	0.044	0.048	0.050	0.054
215	0.042	0.040	0.064	0.064	0.049	0.039	0.054	0.048
229	0.050	0.045	0.074	0.064	0.040	0.046	0.049	0.055
240	0.051	0.042	0.075	0.057	0.051	0.044	0.060	0.054

	Prosulfocarb mg/kg		Prothioconazole-destio mg/kg		Pyraclostrobin mg/kg		Spirotetramat-enol mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 1	Portion 2	Portion 1	Portion 1	Portion 2
013	0.078	0.085	0.024	0.078	0.085	0.024	0.049	0.049
025	0.068	0.068	0.022	0.068	0.068	0.022	0.045	0.044
063	0.071	0.066	0.022	0.071	0.066	0.022	0.043	0.038
092	0.072	0.068	0.023	0.072	0.068	0.023	0.043	0.038
139	0.070	0.070	0.023	0.070	0.070	0.023	0.042	0.038
159	0.078	0.072	0.024	0.078	0.072	0.024	0.044	0.045
197	0.077	0.082	0.023	0.077	0.082	0.023	0.043	0.043
215	0.067	0.068	0.023	0.067	0.068	0.023	0.049	0.045
229	0.069	0.066	0.023	0.069	0.066	0.023	0.045	0.039
240	0.073	0.063	0.026	0.073	0.063	0.026	0.045	0.038

	Tau-Fluvalinate mg/kg		Bifenazate-Diazene mg/kg		Chlordane, cis- mg/kg		HCH, beta mg/kg	
Sample no.	Portion 1	Portion 1	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
013	0.021	0.028	0.031	0.034	0.031	0.034	0.028	0.030
025	0.017	0.026	0.029	0.031	0.029	0.031	0.026	0.027
063	0.019	0.026	0.026	0.026	0.026	0.026	0.026	0.025
092	0.039	0.022	0.027	0.028	0.027	0.028	0.022	0.026
139	0.027	0.022	0.030	0.027	0.030	0.027	0.022	0.025
159	0.024	0.027	0.029	0.028	0.029	0.028	0.027	0.024
197	0.033	0.026	0.028	0.032	0.028	0.032	0.026	0.024
215	0.026	0.029	0.032	0.027	0.032	0.027	0.029	0.024
229	0.019	0.025	0.026	0.031	0.026	0.031	0.025	0.021
240	0.046	0.023	0.033	0.029	0.033	0.029	0.023	0.020

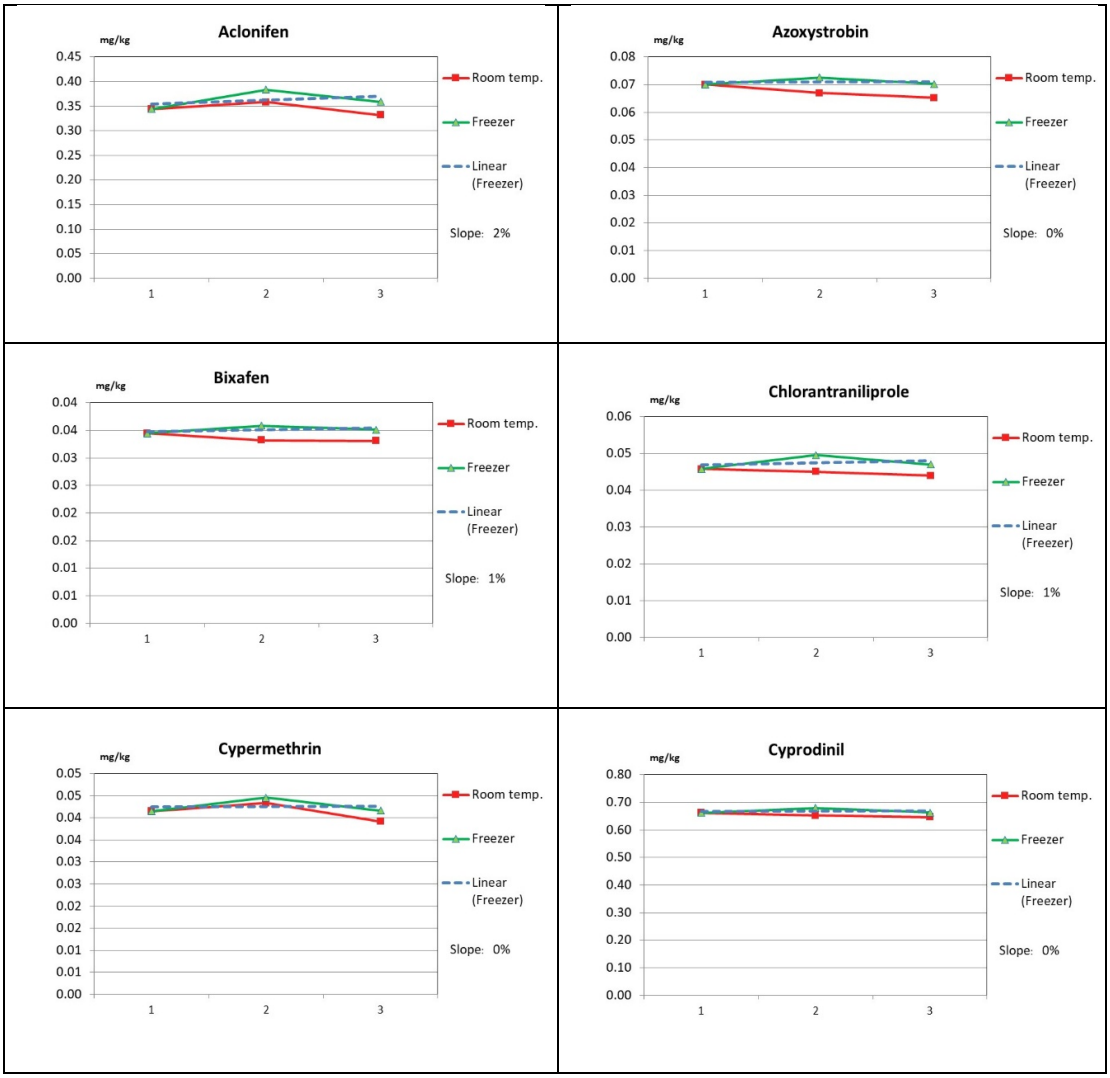
Appendix 4 Stability figures

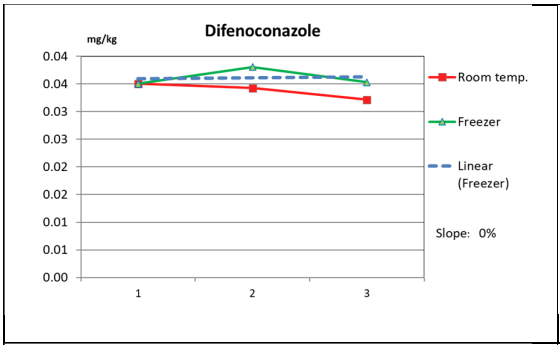
The stability test was performed according to ISO 13528 [5]. Two different storage temperatures were used; room temperature and -18 °C.

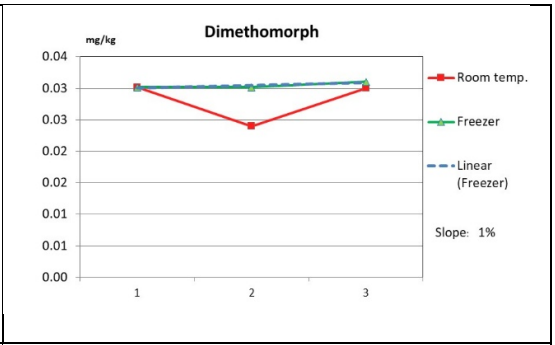
The dates of testing were as follows:

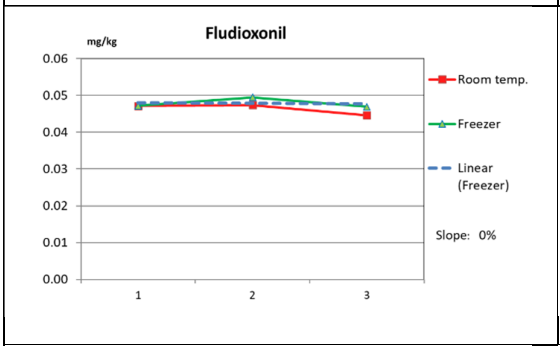
Day 1: 27 January 2025
Day 2: 10 February 2025
Day 3: 3 March 2025

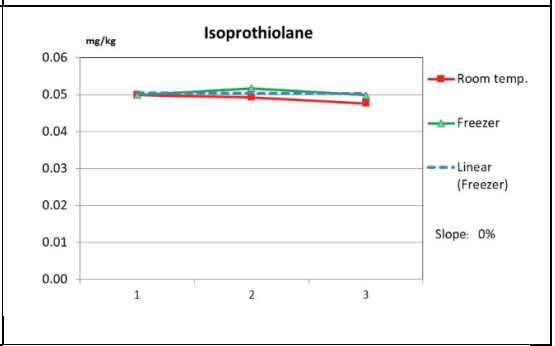
All pesticides passed the test at -18 °C see **1.6 Stability test**. At room temperature metribuzin and bifenazate-Diazene did not pass the test when stored for 11 weeks.

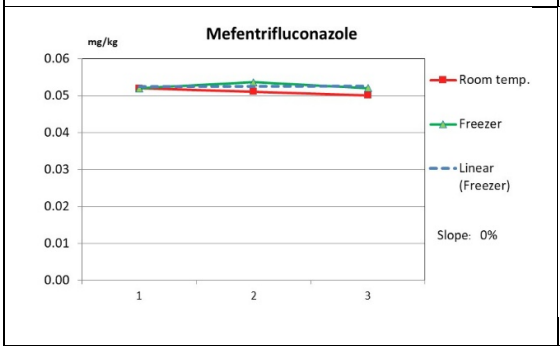


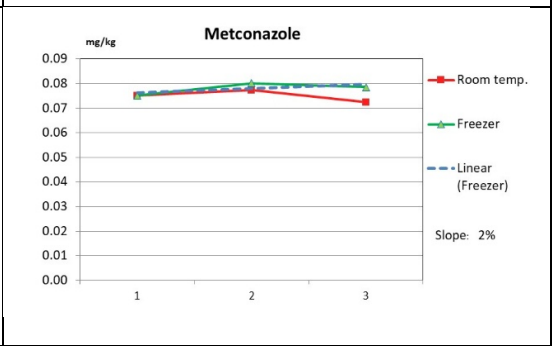

Difenoconazole
mg/kg
0.04
0.04
0.03
0.03
0.02
0.02
0.01
0.01
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: 0%

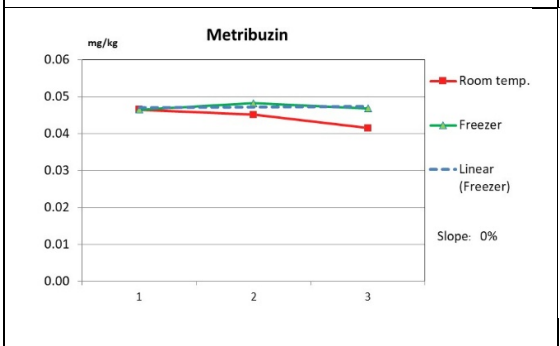

Dimethomorph
mg/kg
0.04
0.03
0.03
0.02
0.02
0.01
0.01
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: 1%

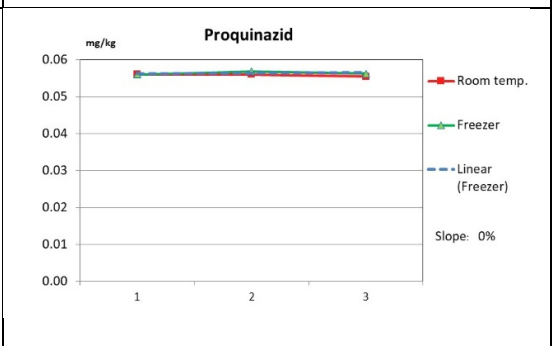

Fludioxonil
mg/kg
0.06
0.05
0.04
0.03
0.02
0.01
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: 0%

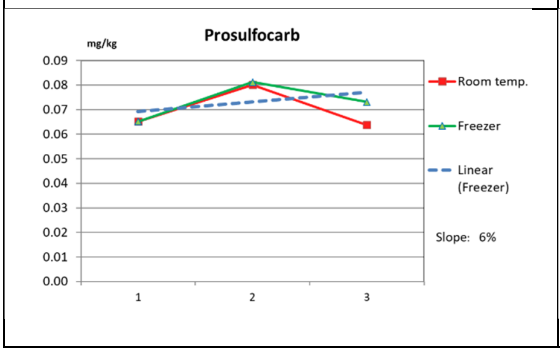

Isoprothiolane
mg/kg
0.06
0.05
0.04
0.03
0.02
0.01
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: 0%

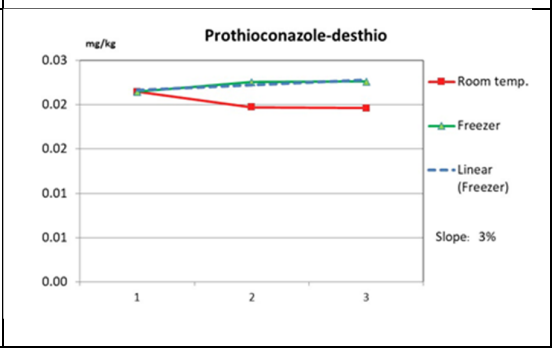

Mefentrifluconazole
mg/kg
0.06
0.05
0.04
0.03
0.02
0.01
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: 0%

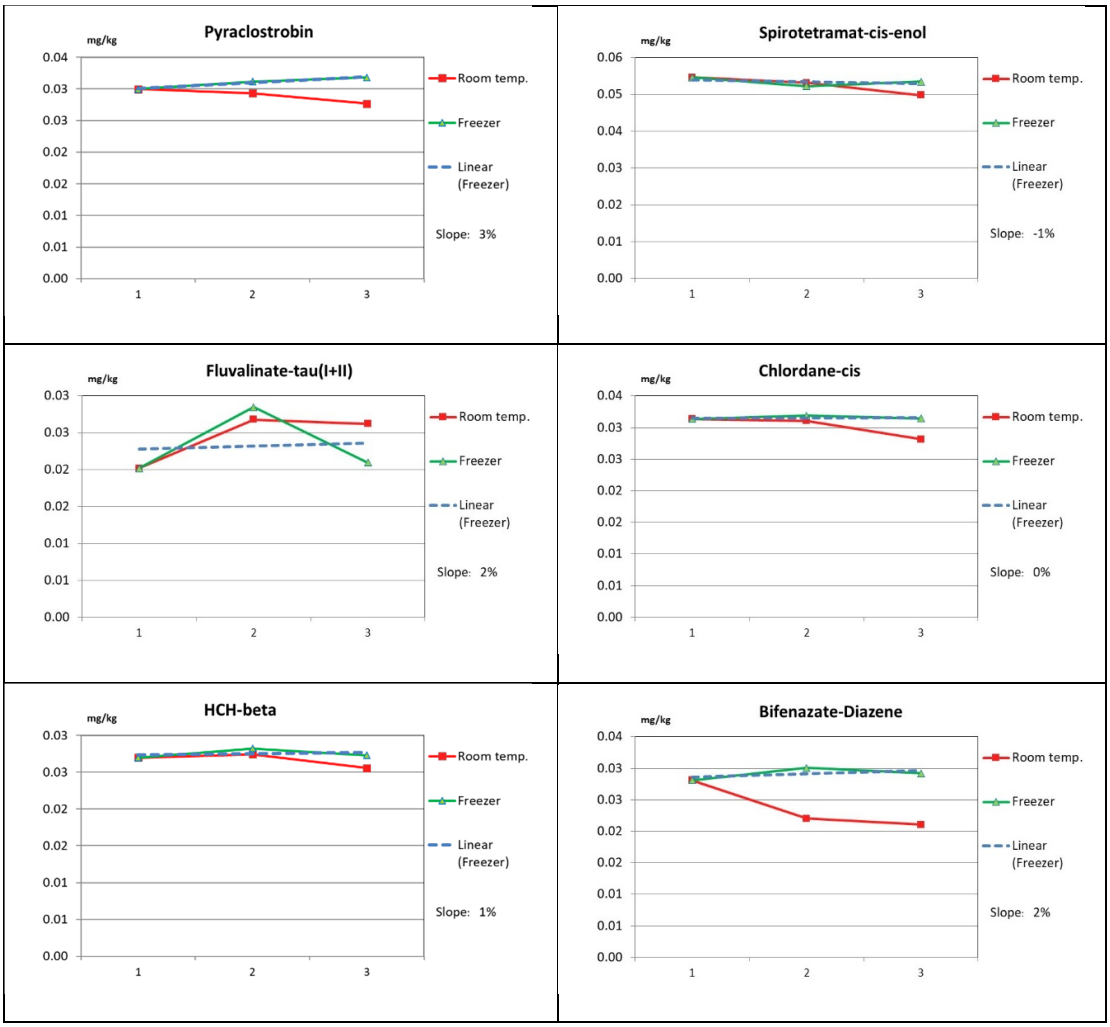

Metconazole
mg/kg
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: 2%


Metribuzin
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: 0%

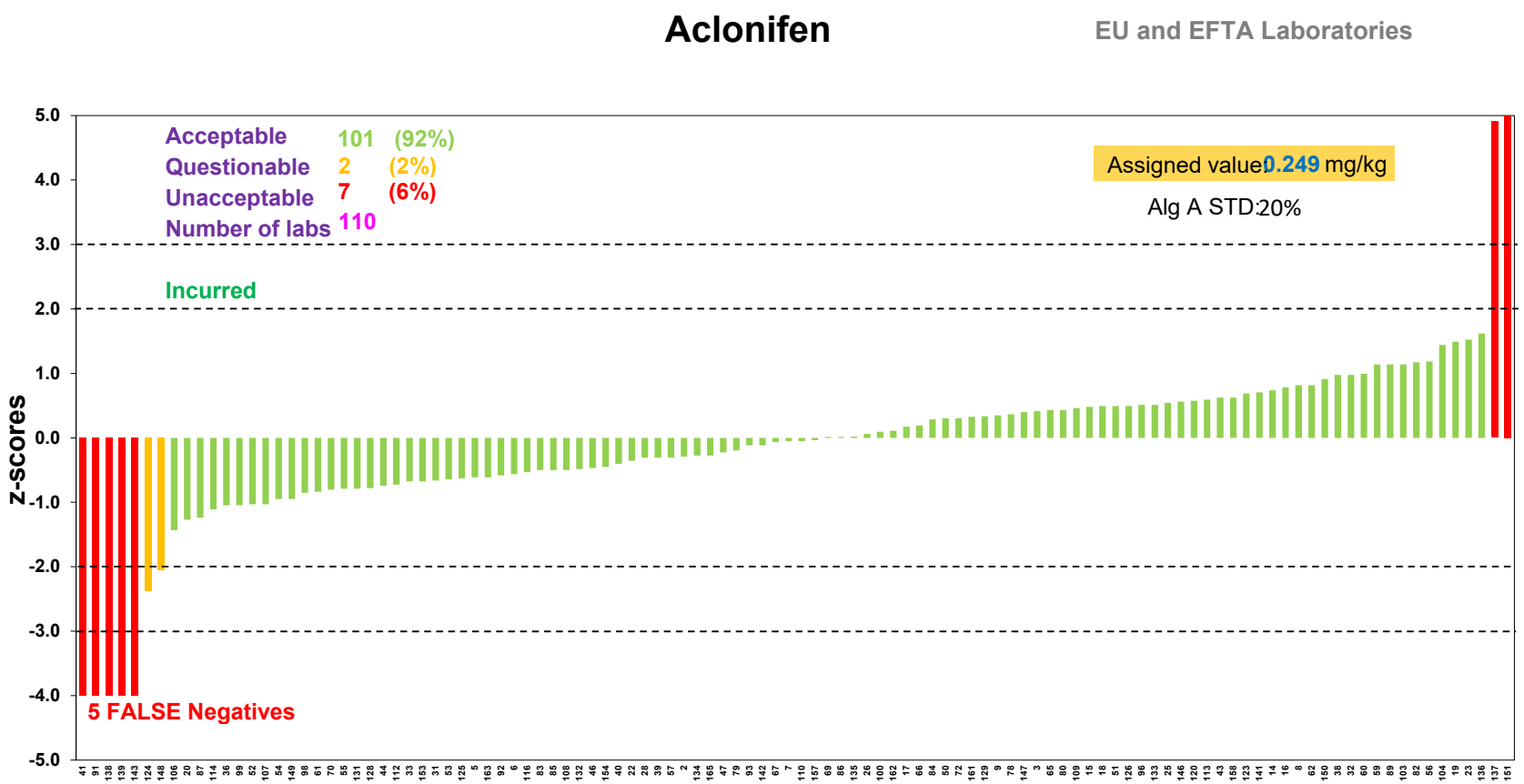

Proquinazid
mg/kg
0.06
0.05
0.04
0.03
0.02
0.01
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: 0%


Prosulfocarb
mg/kg
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: 6%


Prothioconazole-desthio
mg/kg
0.03
0.02
0.02
0.01
0.01
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: 3%

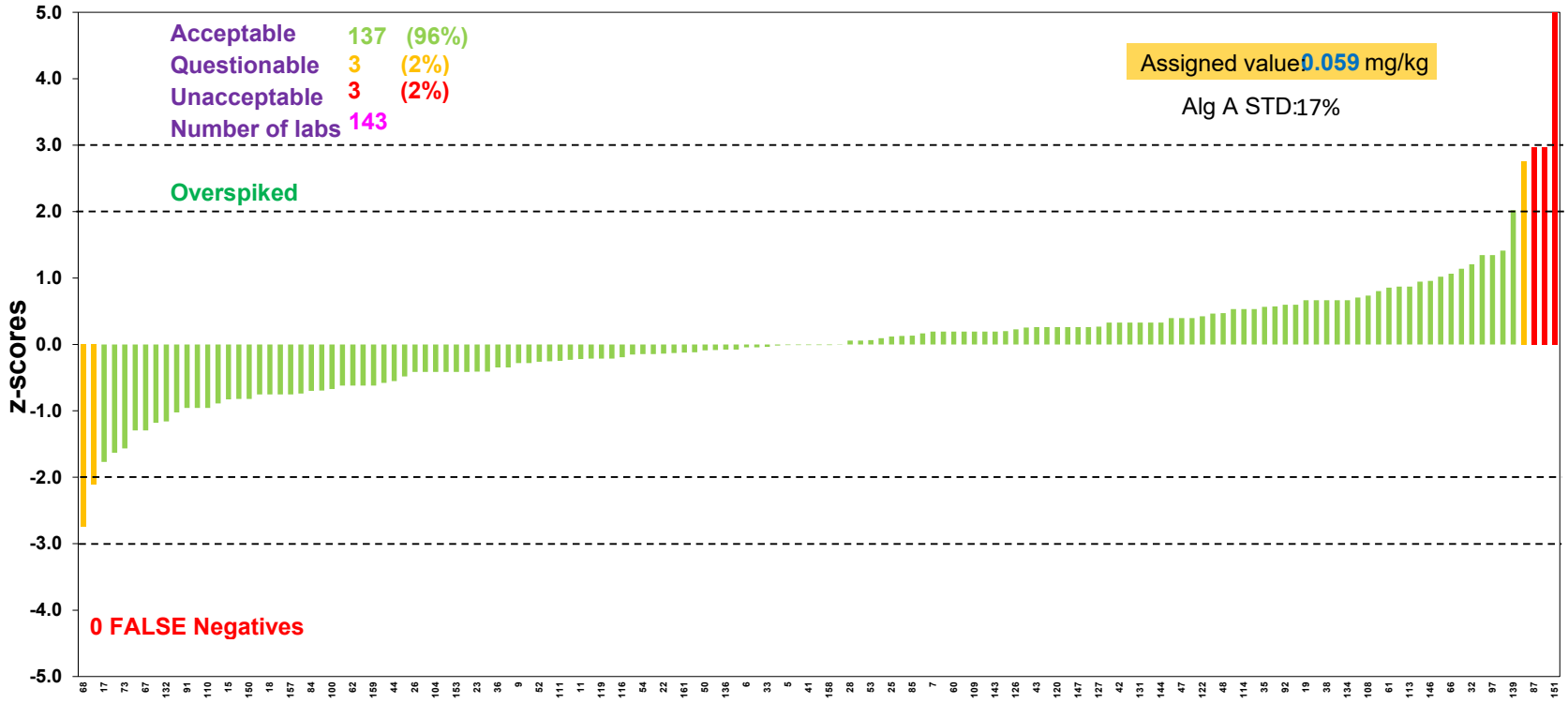

Pyraclostrobin
mg/kg
0.04
0.03
0.03
0.02
0.02
0.01
0.01
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: 3%
Spirotetramat-cis-enol
mg/kg
0.06
0.05
0.04
0.03
0.02
0.01
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: -1%
Fluvalinate-tau(I+II)
mg/kg
0.03
0.03
0.02
0.02
0.01
0.01
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: 2%
Chlordane-cis
mg/kg
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: 0%
HCH-beta
mg/kg
0.03
0.03
0.02
0.02
0.01
0.01
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: 1%
Bifenazate-Diazene
mg/kg
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: 2%

Appendix 4 Graphical presentation of z-scores



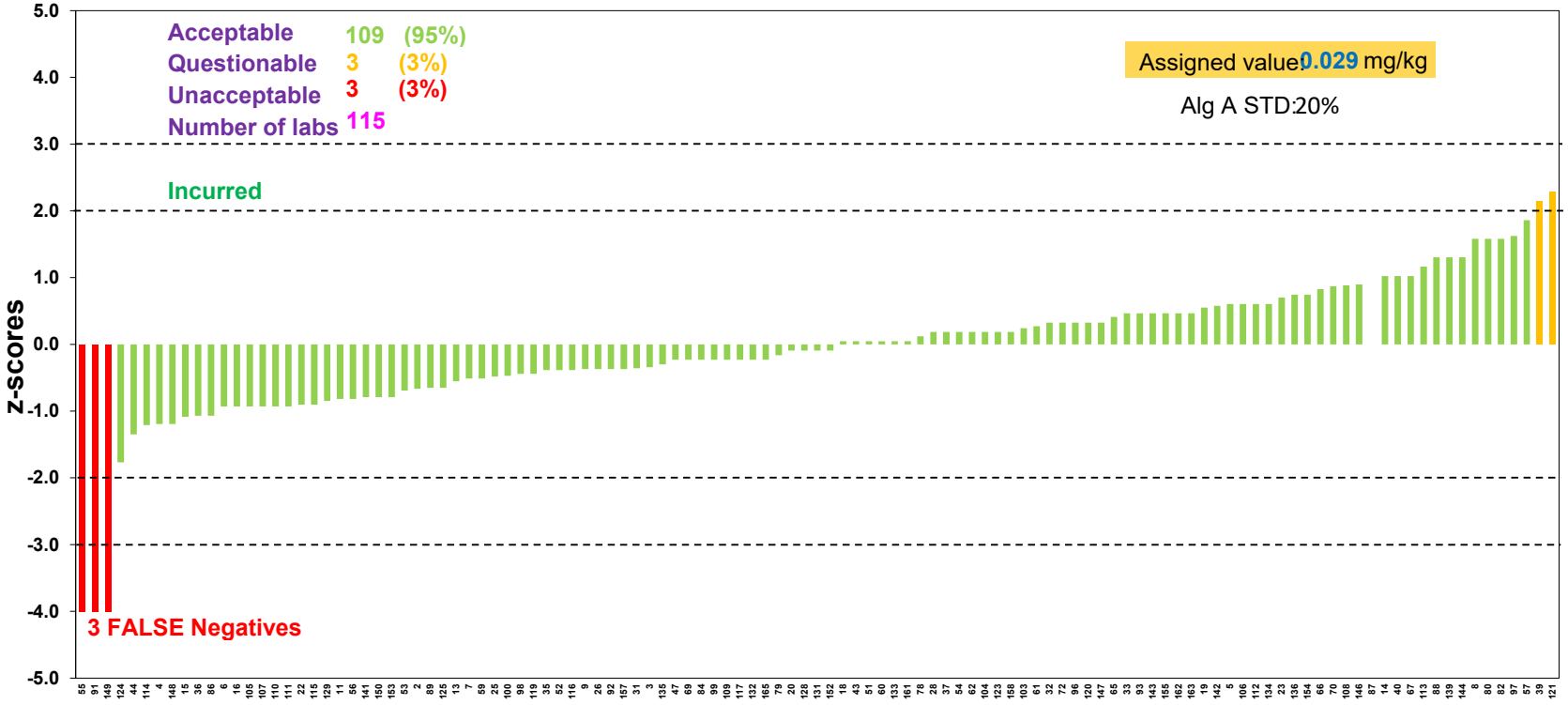
Azoxystrobin

EU and EFTA Laboratories



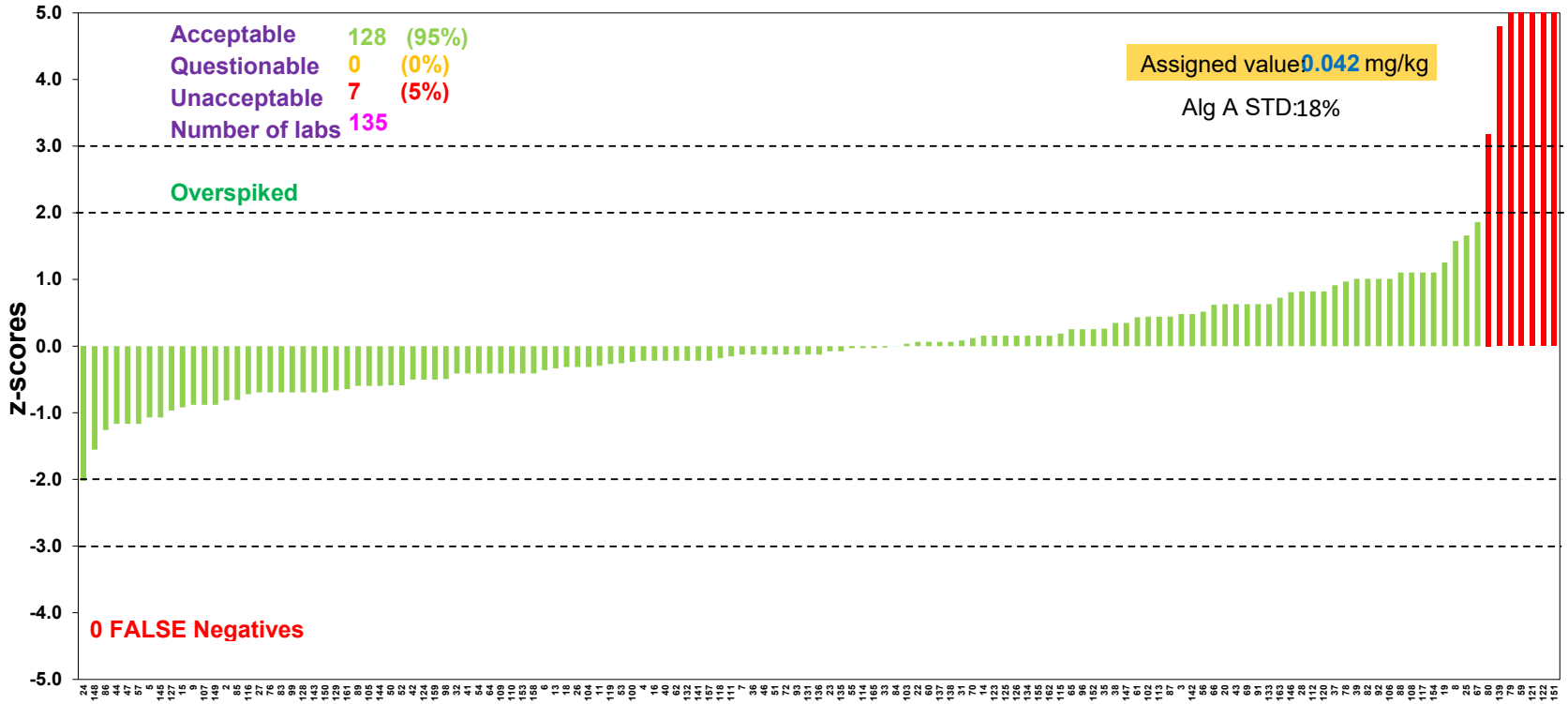
Bixafen

EU and EFTA Laboratories



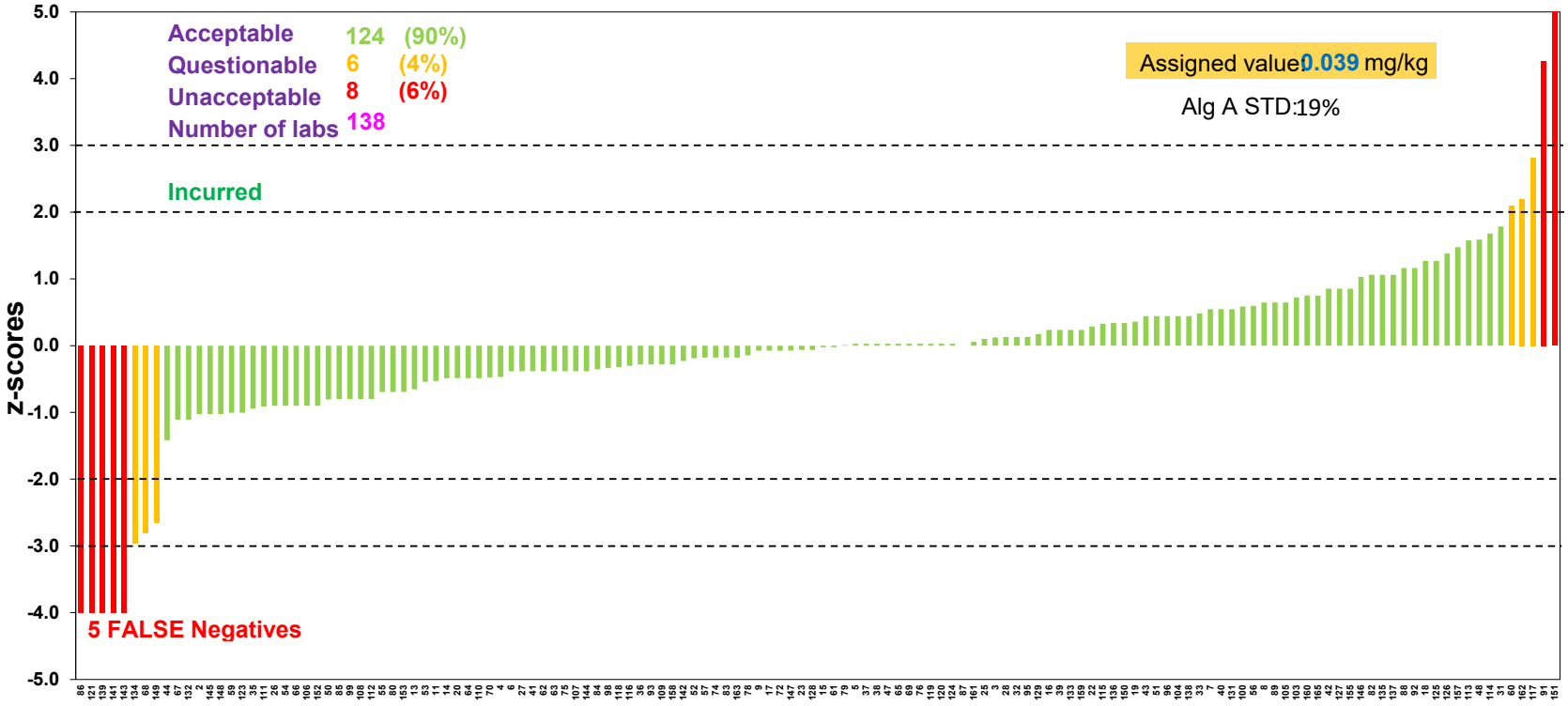
Chlorantraniliprole

EU and EFTA Laboratories



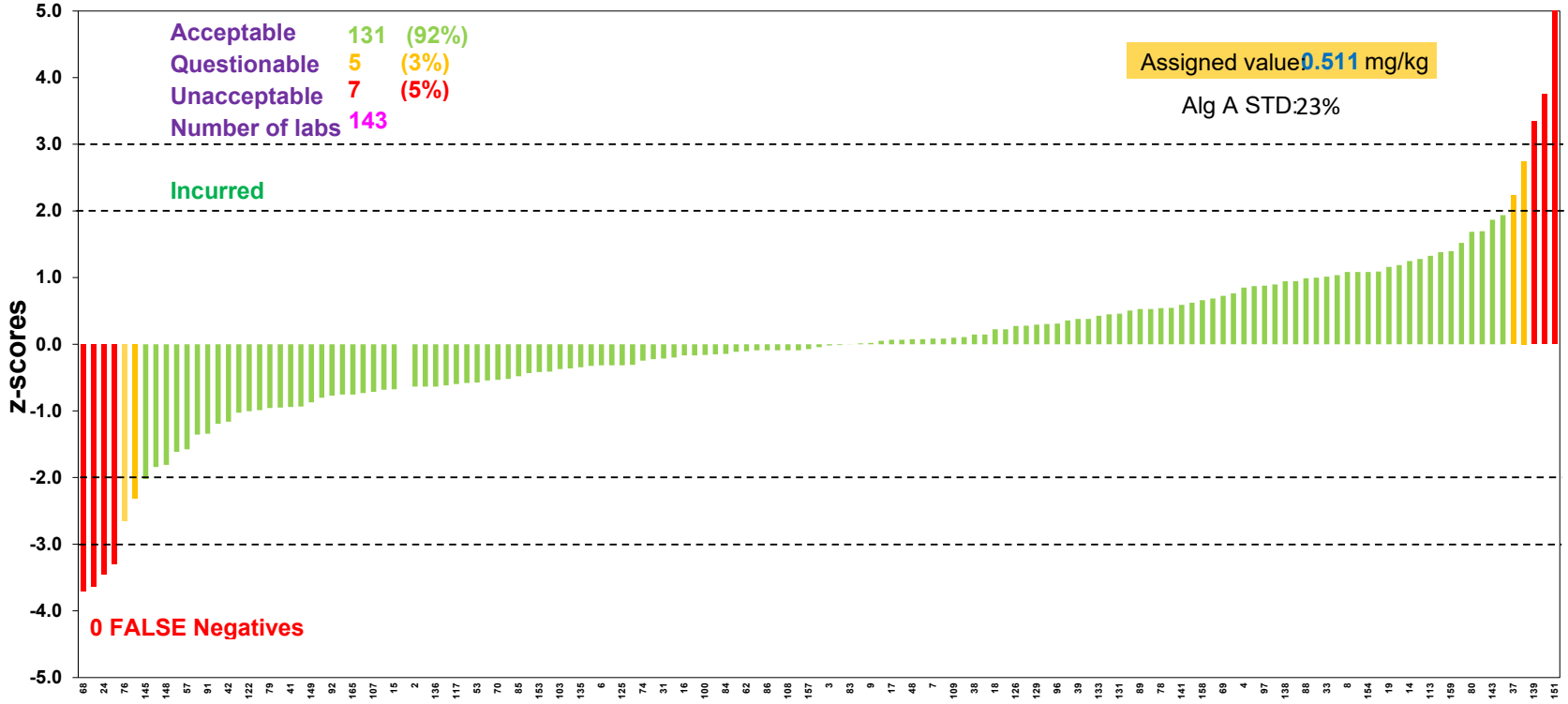
Cypermethrin (sum)

EU and EFTA Laboratories



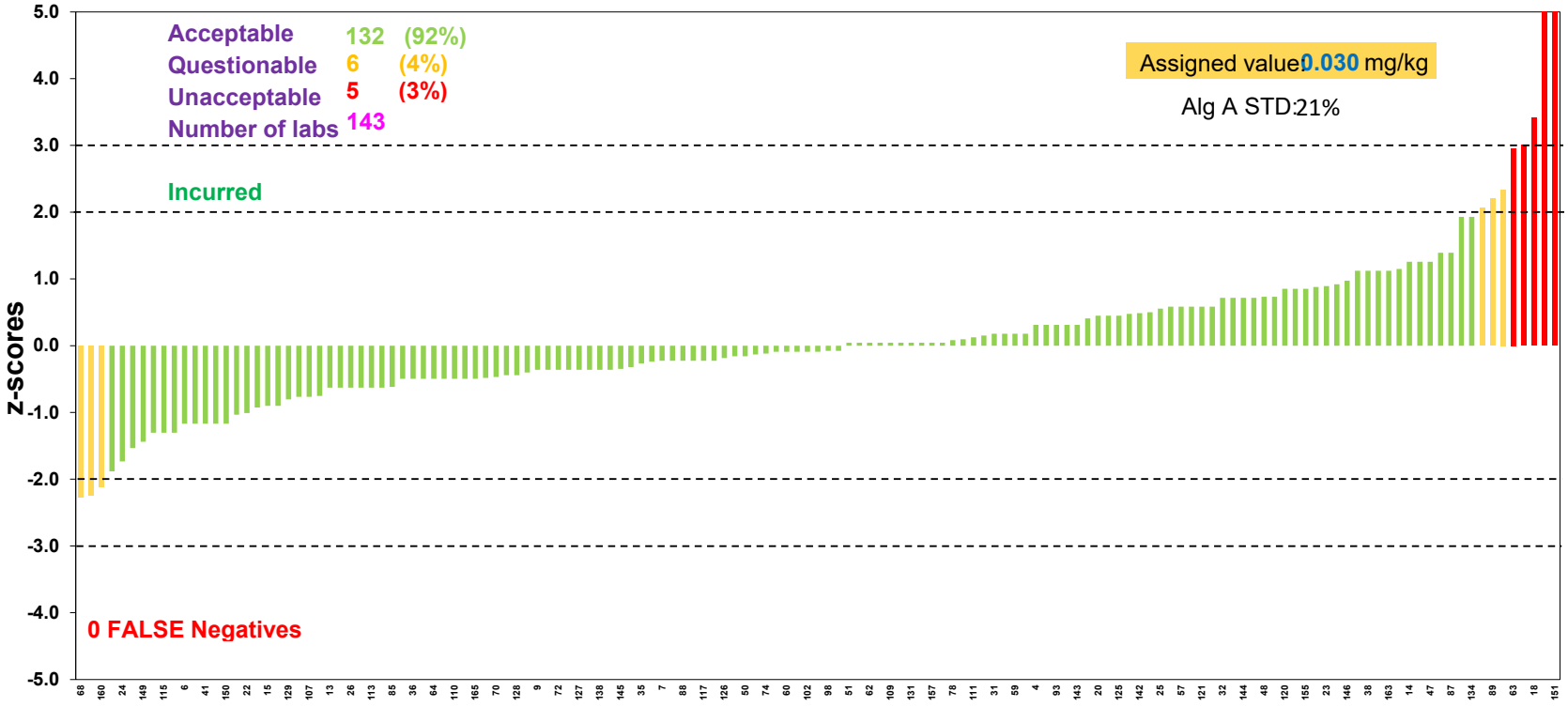
Cyprodinil

EU and EFTA Laboratories



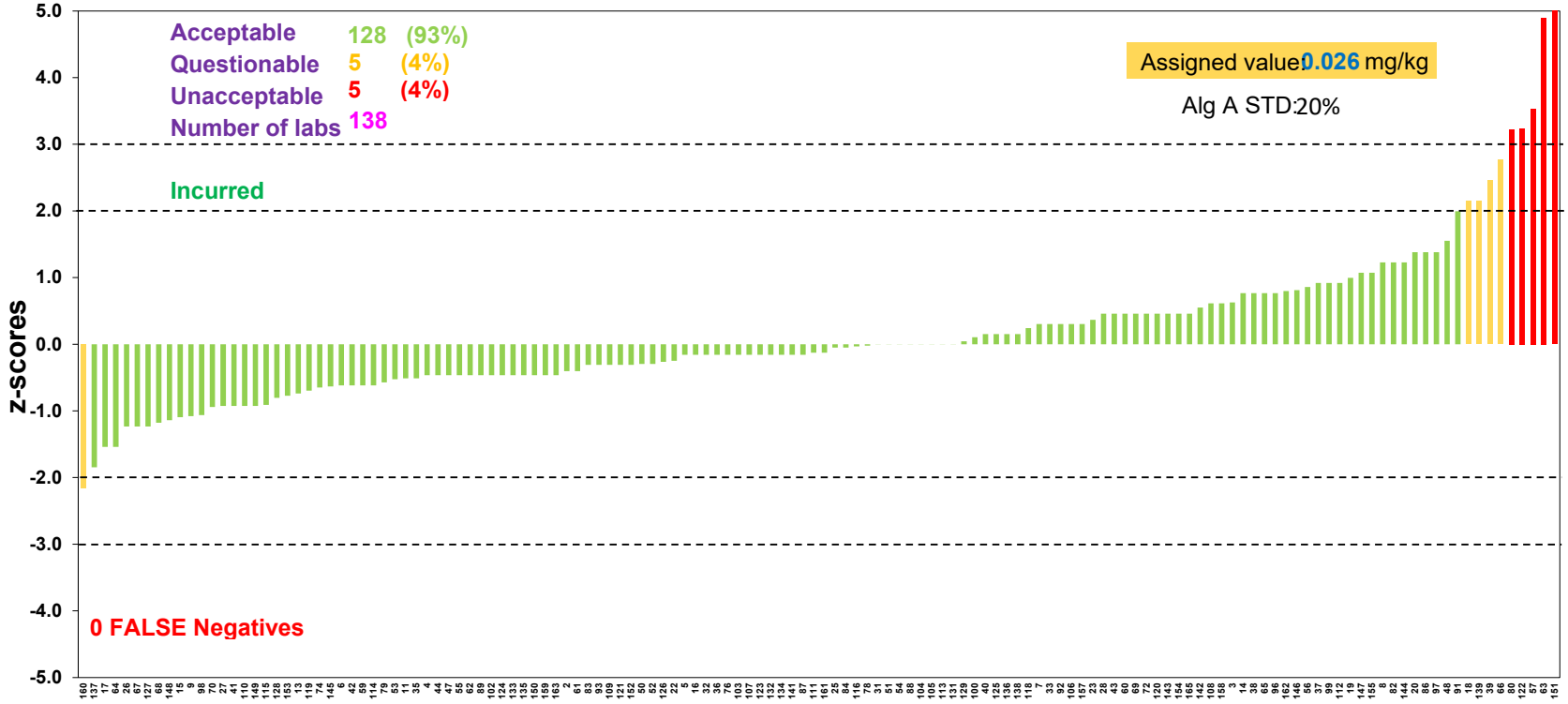
Difenoconazole

EU and EFTA Laboratories



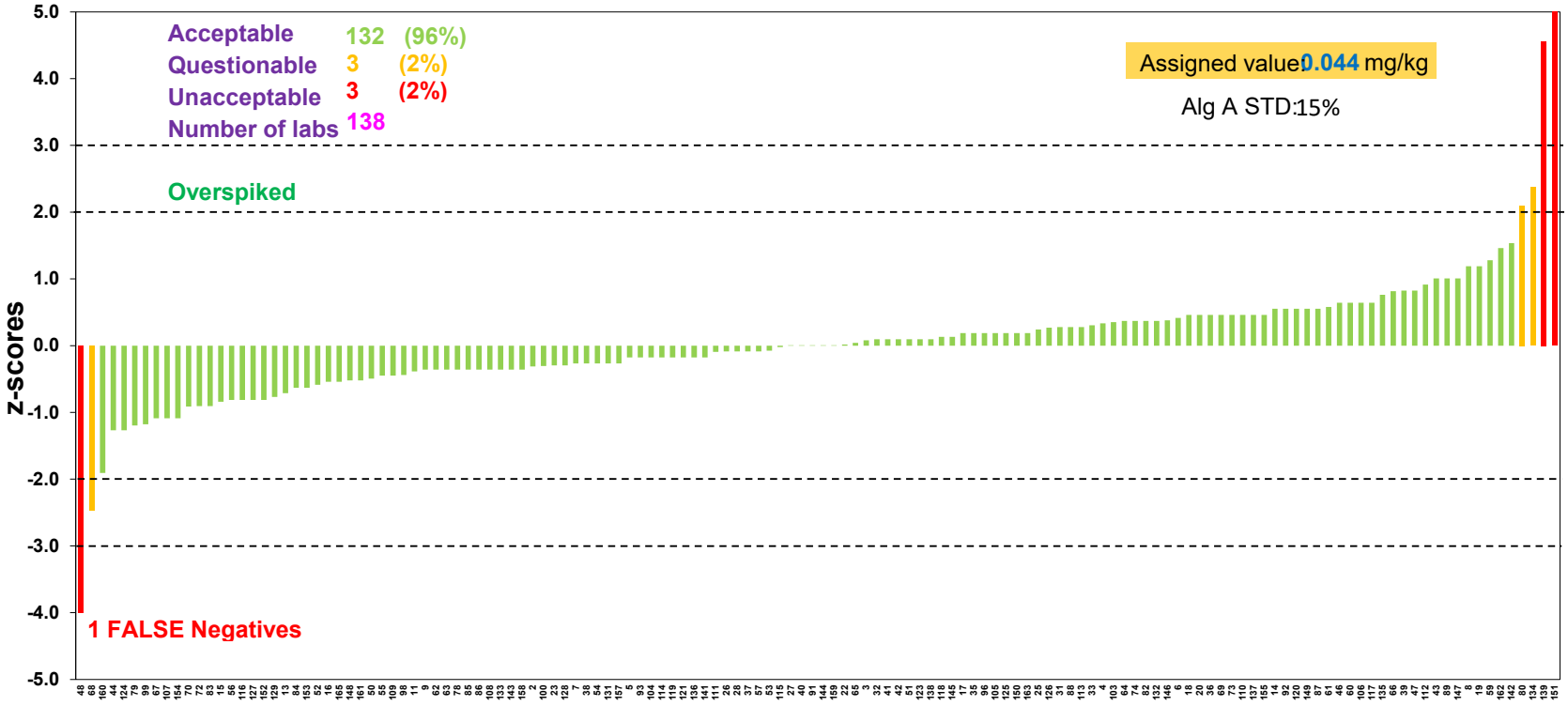
Dimethomorph

EU and EFTA Laboratories



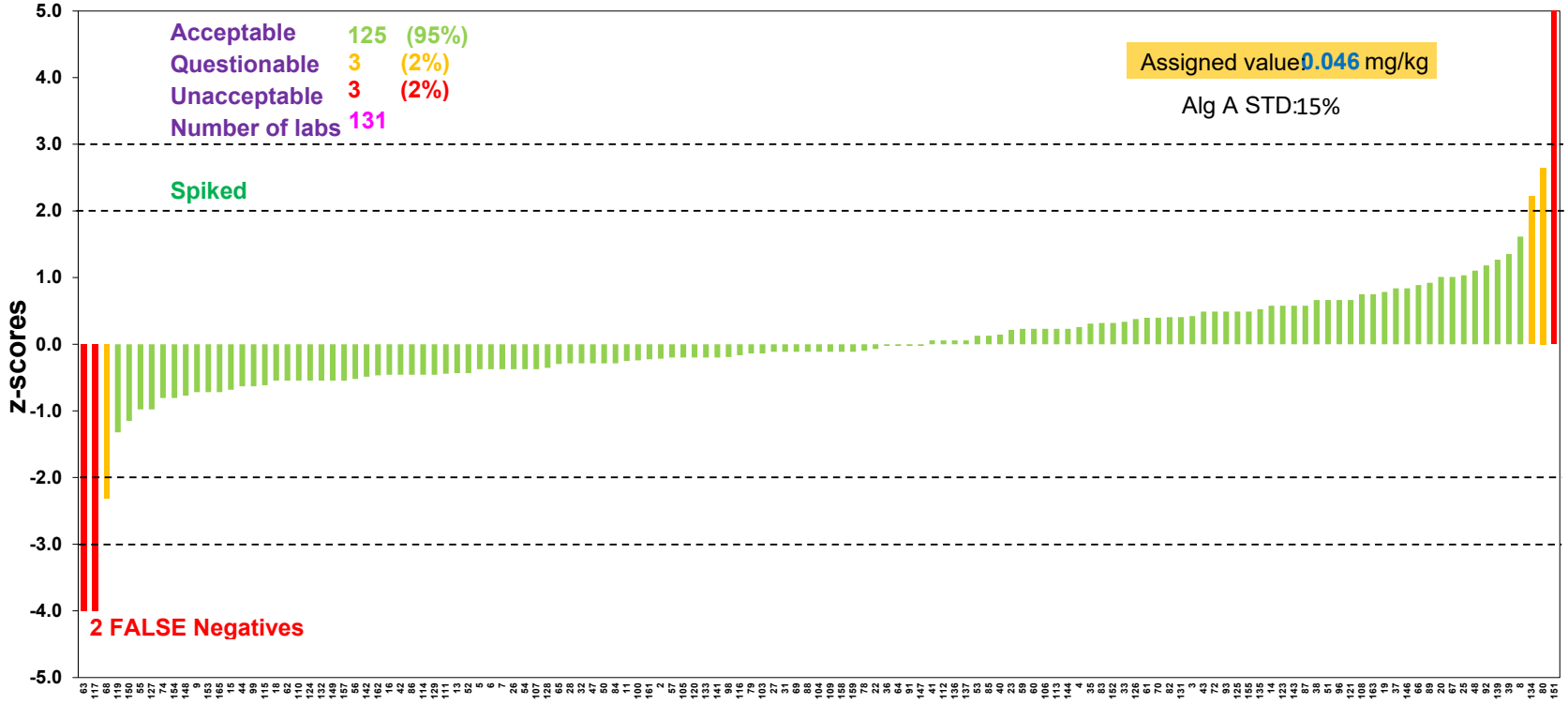
Fludioxonil

EU and EFTA Laboratories



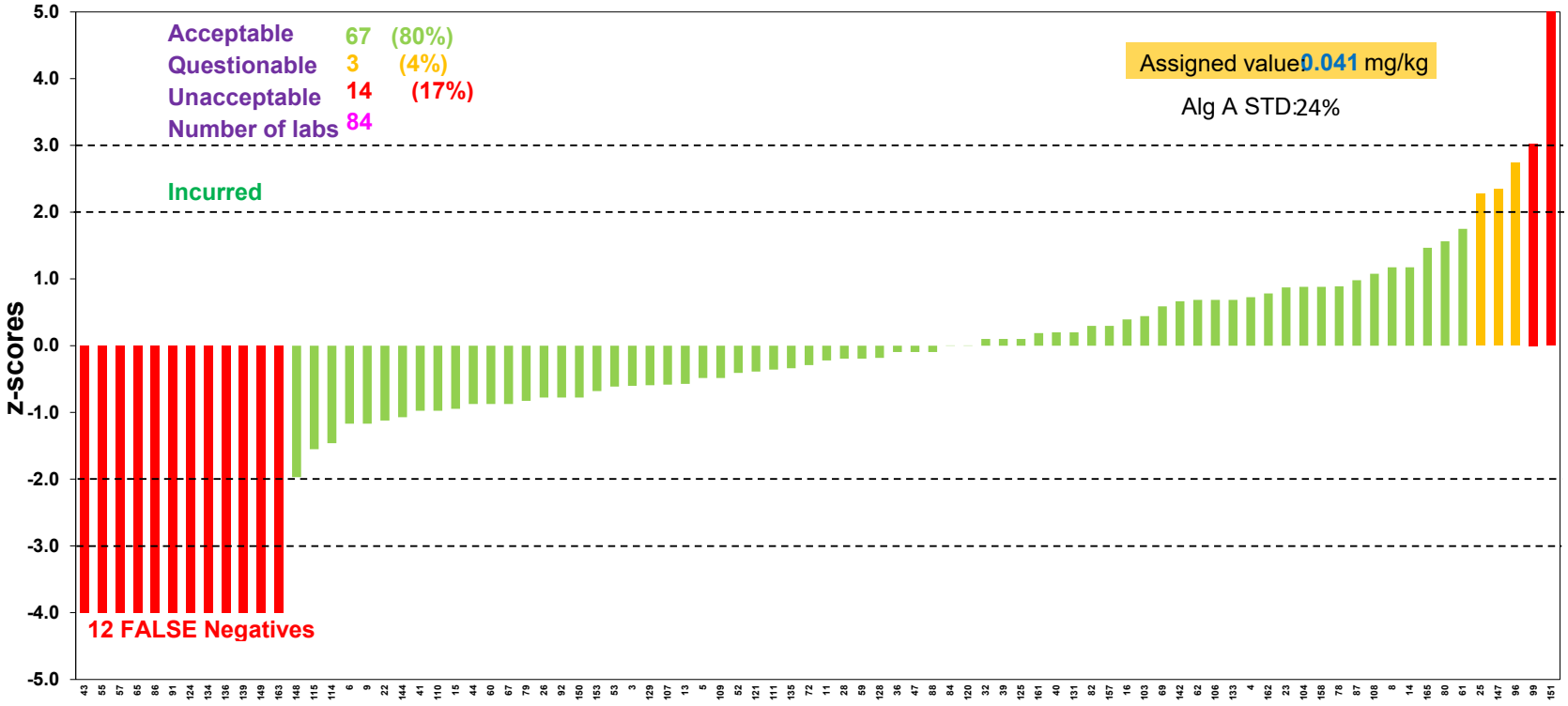
Isoprothiolane

EU and EFTA Laboratories



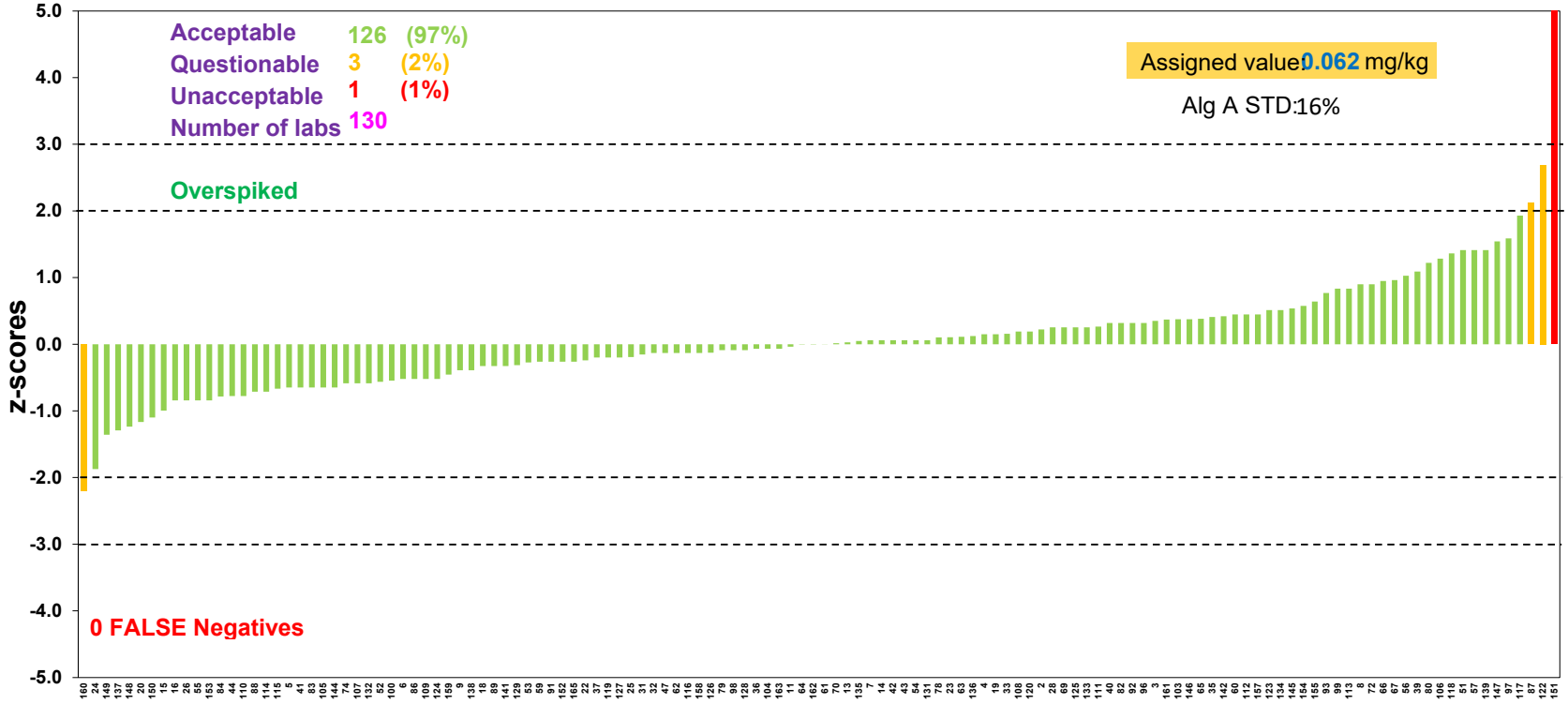
Mefentrifluconazole

EU and EFTA Laboratories



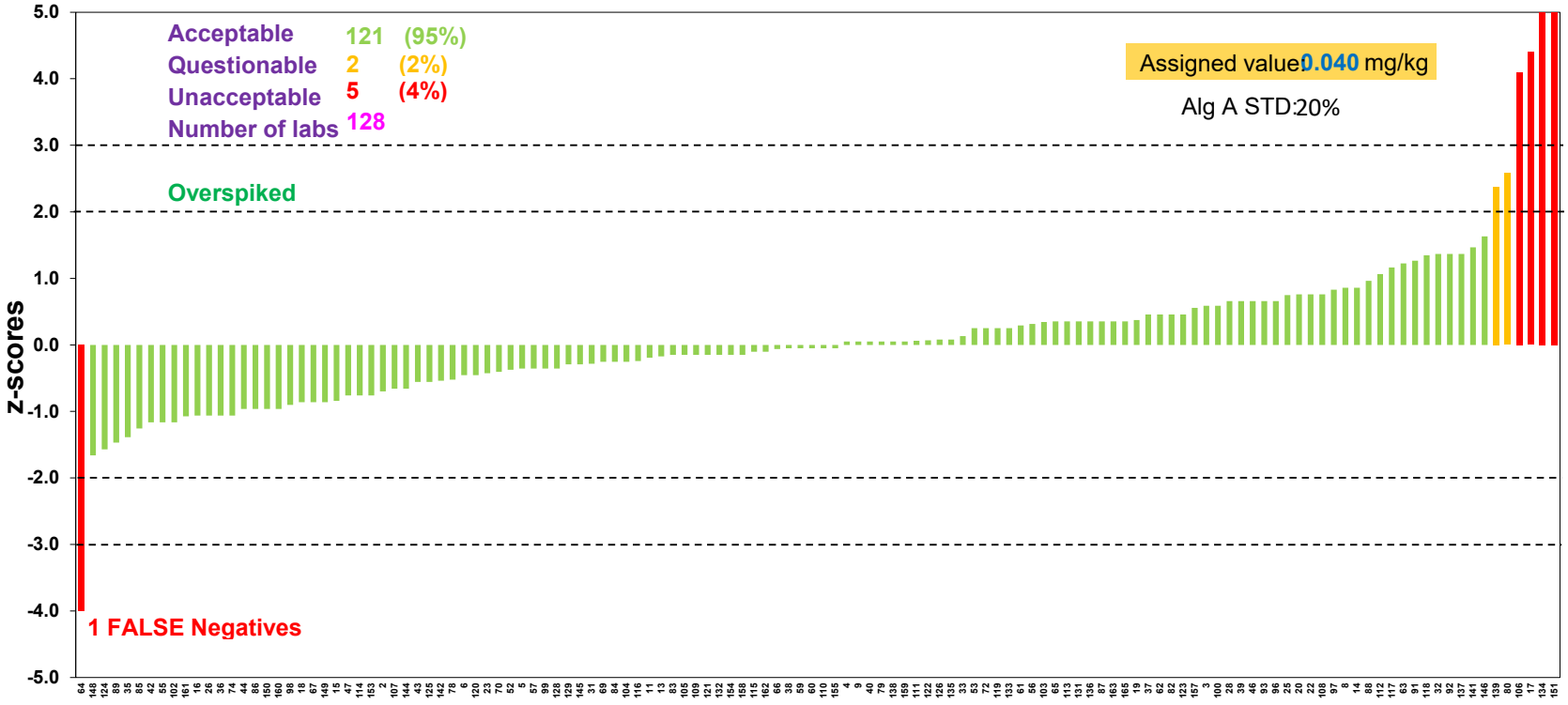
Metconazole

EU and EFTA Laboratories



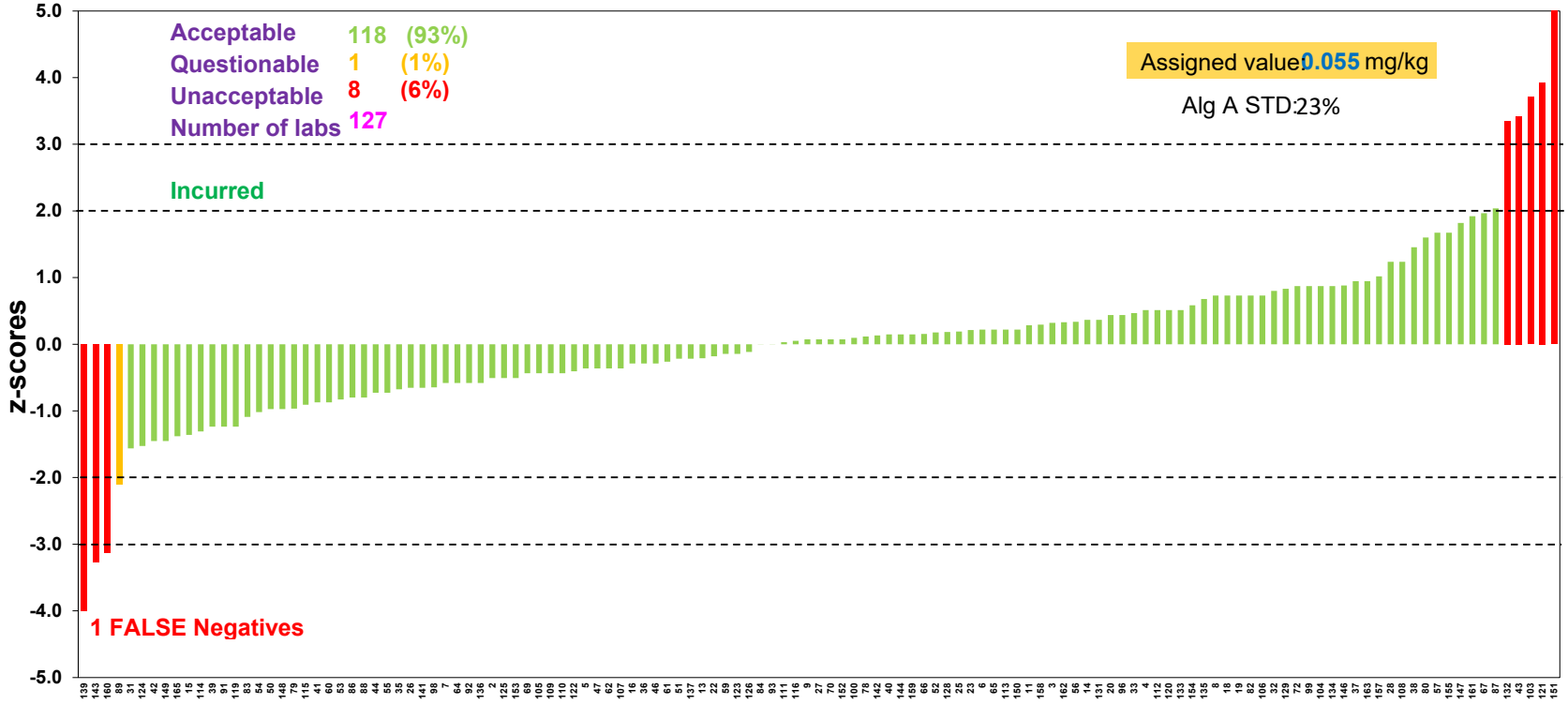
Metribuzin

EU and EFTA Laboratories



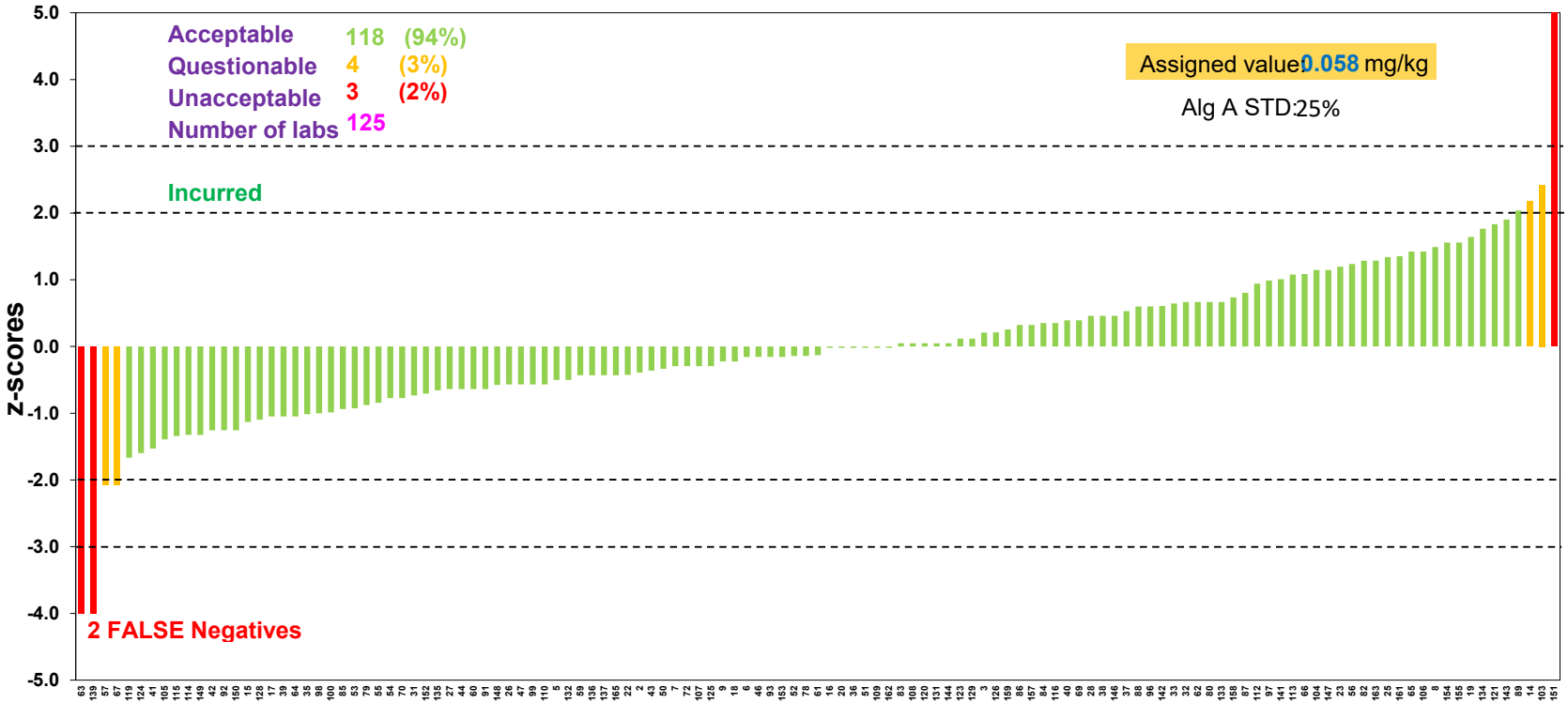
Proquinazid

EU and EFTA Laboratories

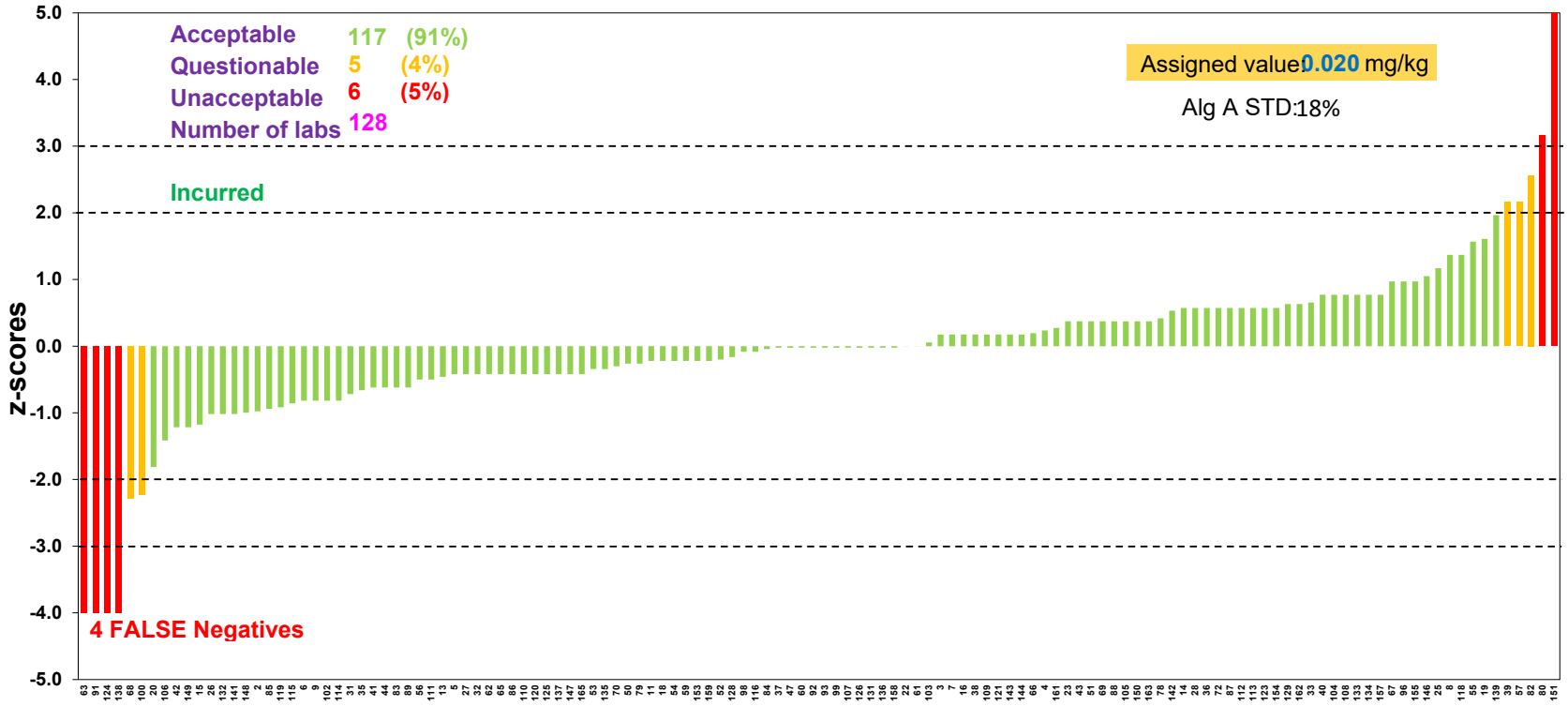


Prosulfocarb

EU and EFTA Laboratories

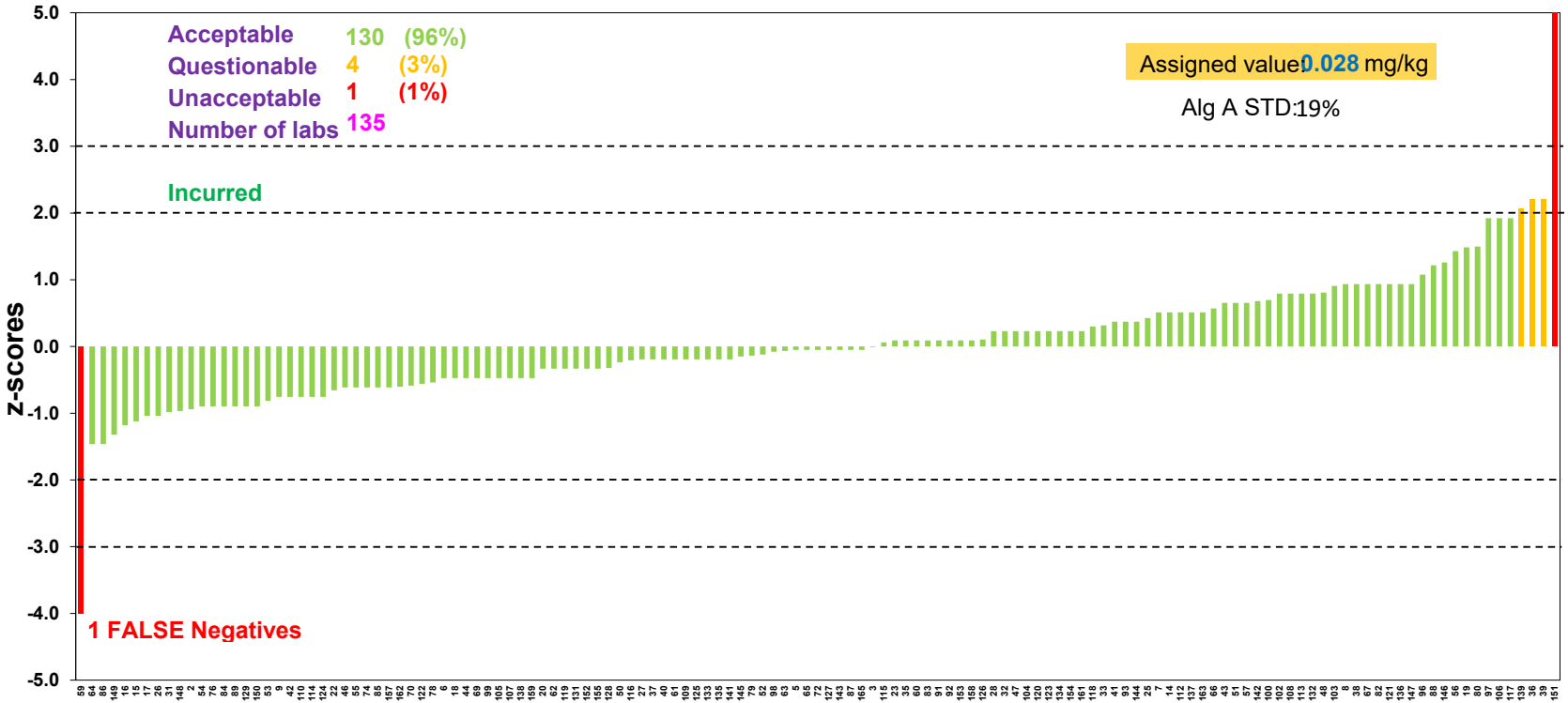


Prothioconazole-Desthio EU and EFTA Laboratories



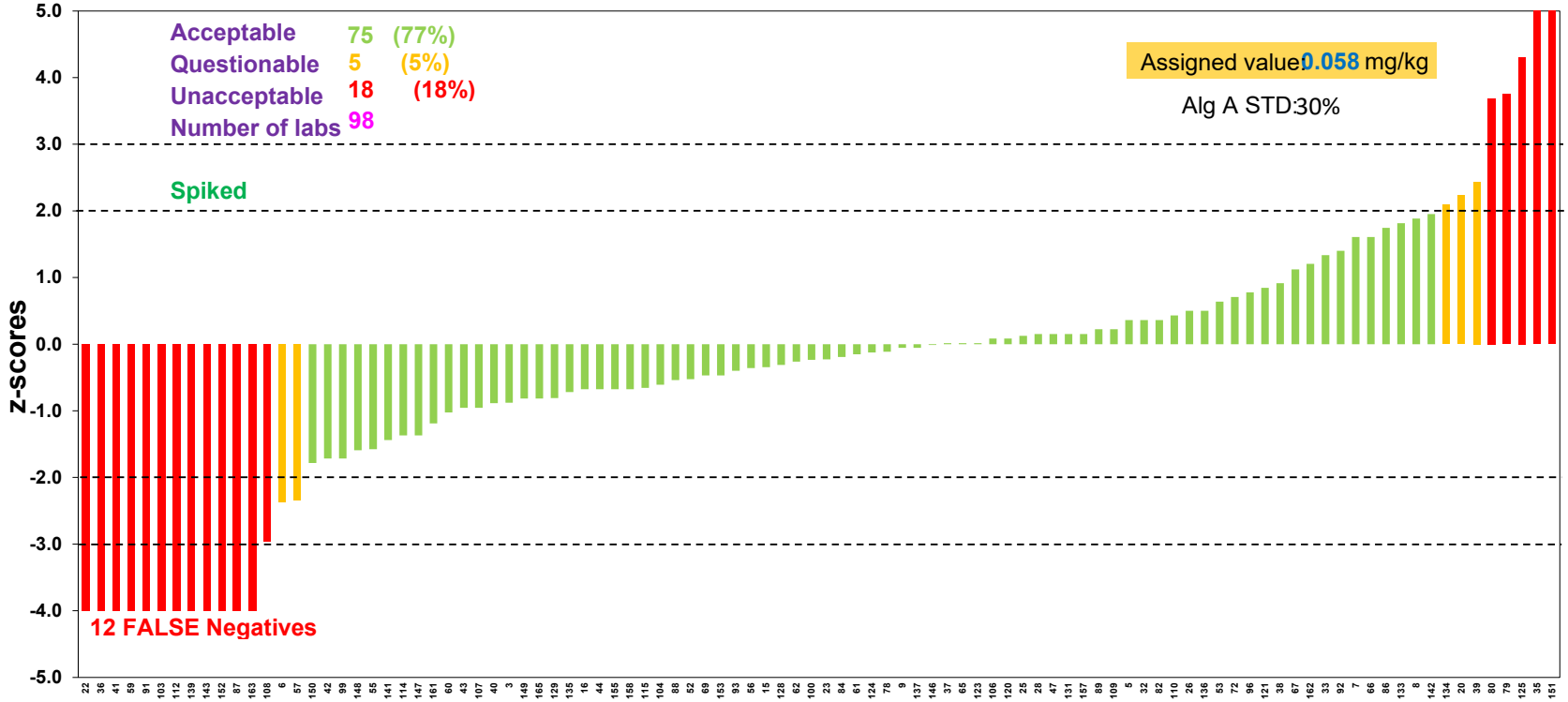
Pyraclostrobin

EU and EFTA Laboratories



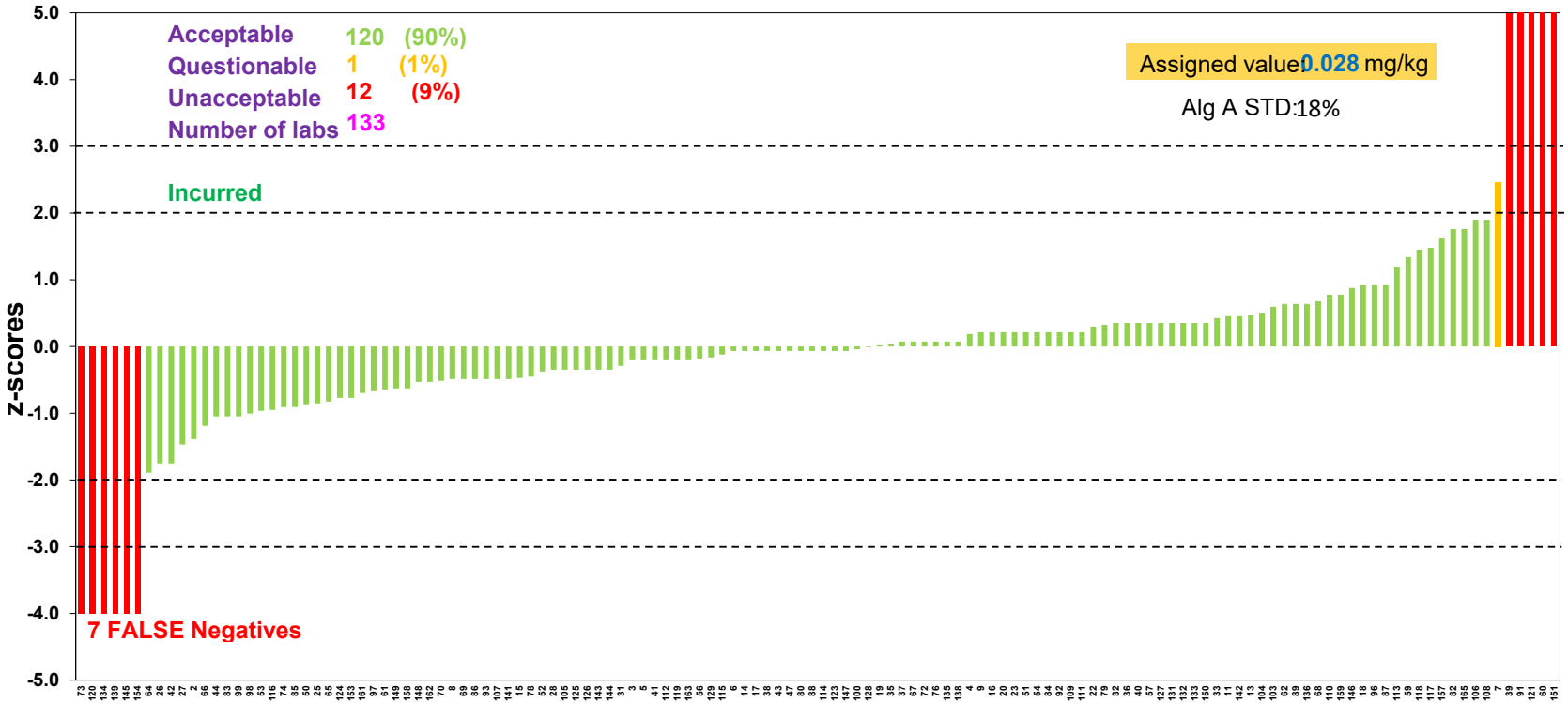
Spirotetramat-enol

EU and EFTA Laboratories



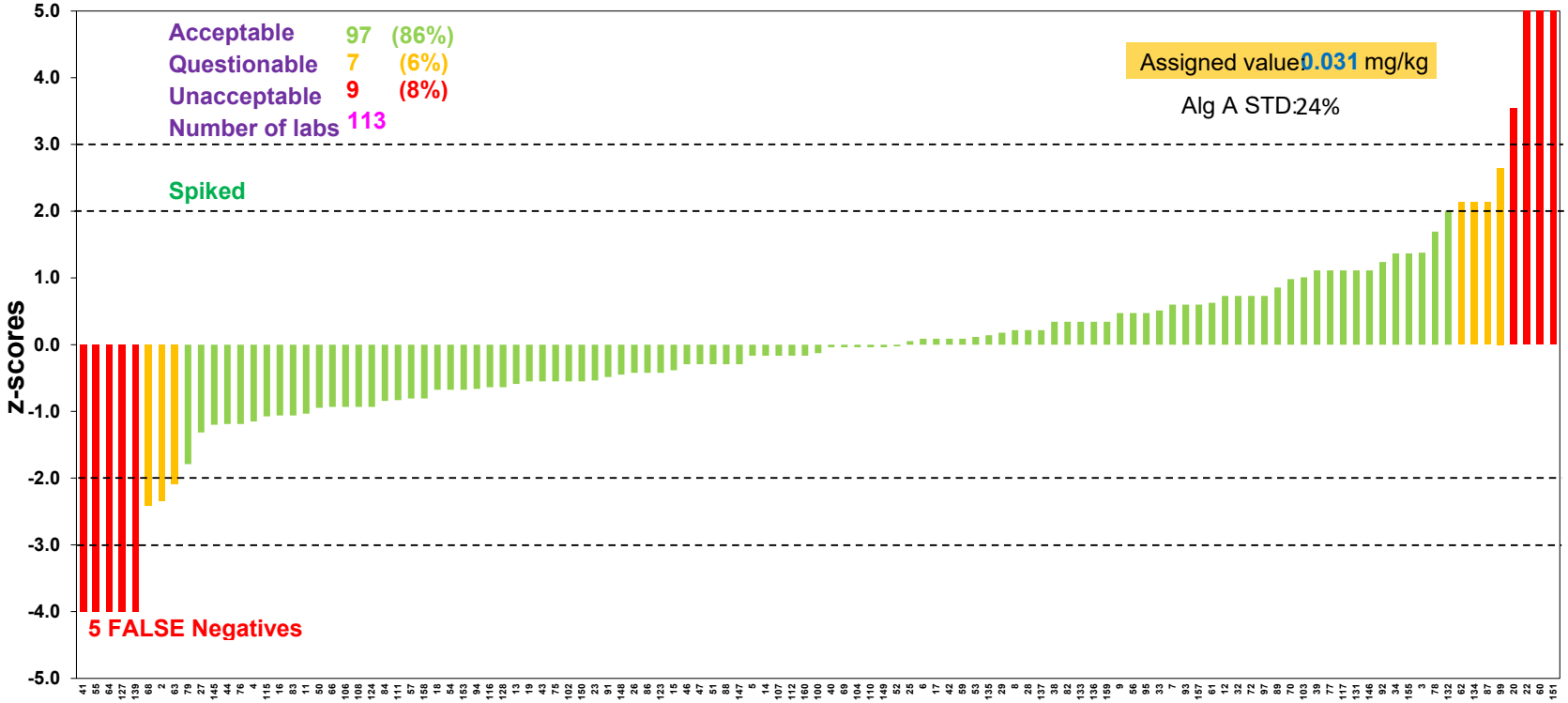
Tau-Fluvalinate

EU and EFTA Laboratories



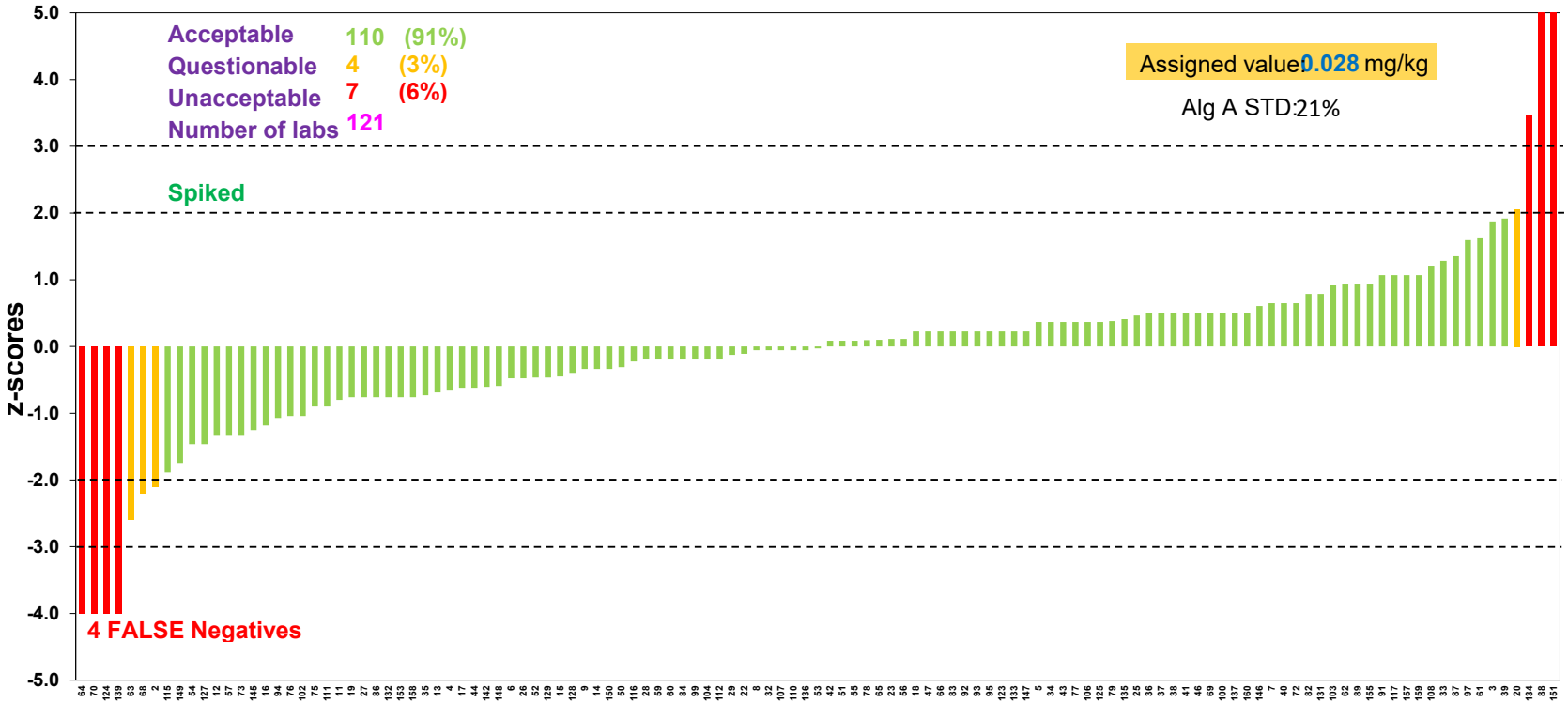
Chlordane, cis-

EU and EFTA Laboratories



HCH, beta-

EU and EFTA Laboratories



GENERAL PROTOCOL

for EU Proficiency Testings on Pesticide Residues in Food and Feed

Introduction

This protocol contains general procedures valid for all European Union Proficiency Testings (EUPTs) organised on behalf of the European Commission, DG-SANTE[1] by the four European Union Reference Laboratories (EURLs) responsible for the area of pesticide residues analysis in food and feed. These EUPTs are organised for 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[2]:

- 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 EUPTs allow the individual laboratory to evaluate if its performance is satisfactory. Additionally, the aim 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[3]. Participating laboratories will be provided with an assessment of their analytical performance that they can use to demonstrate their (ongoing) analytical proficiency and compare themselves with other participating laboratories.

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

[2] 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.

[3] https://food.ec.europa.eu/plants/pesticides/maximum-residue-levels/enforcement/eu-multi-annual-control-programmes_en

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Significant changes in new edition to previous edition

Some parts of the old version have been removed, so this edition includes only the information necessary for the participants. There have been no changes to the evaluation process. Apart from that, only editorial changes have been made.

EUPT- Organisers and Scientific Committee

EUPTs are organised either by single EURLs, or collaboratively by more than one EURL.

An **organising team** (in the following named **organisers**[4]) is appointed by the EURL(s) in charge of a given PT. The organisers are in charge of all administrative and technical PT activities of a proficiency testing (PT) round, which include e.g., the PT-announcement, the production of the proficiency testing item (PT-item), the undertaking of homogeneity and stability assessments, the packing and shipment of the PT-Items, 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 the 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 in agreement with DG-SANTE. The EUPT-SC consists of expert scientists with many years of experience in PTs and/or pesticide residue analysis. The latest **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's role is to assist the organisers during the planning and the data evaluation phase of a PT-round. Input from the EUPT-SC is for example requested, when it comes to selecting the commodity, selecting the analytes to be included in the Target Pesticides List (p. 79), establishing the Minimum Required Reporting Levels (MRRLs) for each of the analytes, statistically evaluating the participants' results (in anonymous form), as well as for 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 PT item and the approximate concentrations at which they should be present.

[4] The term organisers are to be considered equivalent to the term PT-provider in ISO 17043:2023-10

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

The EUPT-SC typically meets once a year, after 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 preliminary evaluation of the EUPT-results, especially where case-by-case decisions are needed. PT plans for the next EUPT season are also discussed during these meetings.

EUPT Participants – Eligibility and Obligation for Participation

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[6]
- 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[7]), and
- Art. 101 (1)(a) of Regulation (EU) No. 2017/625[2] (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 to 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 obliged laboratories with their current commodity-scopes and contact information.

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

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

EUPTs are furthermore open to the following laboratories as long as sufficient material is available:

a) any other OfLs from EU countries that are not covered by the above obligations to participate

b) NRLs and OfLs from EFTA countries

[6] Regulation (EU) 2017/625 of the European Parliament and of the Council of 15 March 2017 on official controls and other official activities. https://eur-lex.europa.eu/eli/reg/2017/625/oj

[7] 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).

c) NRLs and OfLs from EU-candidate countries and

d) other laboratories from EU or EFTA countries analysing official organic samples within the frame of Reg. 889/2008/EC

e) governmental laboratories from Third Countries (countries outside EU)

f) other laboratories from Third Countries as long as they are involved in controls of products destined for export to the EU.

Laboratories of groups d) and f) will be requested to provide a proof of their function (e.g. scan copy of a document stating official appointment).

In exceptional justified cases (e.g. where the number of OfLs/NRLs analysing a specific compound is small) additional commercial labs from the EU and beyond may be invited to participate in an EUPT). In a given EUPT, each laboratory/institution is allowed to submit only one single set of results. Any subcontracting of analyses to another institution must be communicated to the organisers, preferably prior to the start of the EUPT.

Participation fee and Invoicing

By completing the registration for participation in a given EUPT, the laboratory agrees to proceed with a timely payment of the participation fee. The invoice fee covers the costs of production, handling and delivery of the PT Test Items. The organisers will issue digital invoices in PDF format only, and without any electronic signature. The EURLs retain the right to decline any request for supplementary forms or additional paperwork in connection with the payment. The laboratories should note that additional costs will incur if such extra services are requested, depending on the incurring extra workload. Extra costs may also be incurred if a new modified invoice is requested, e.g. because of missing or erroneous information caused by errors or omissions by the registered laboratory during registration. OfLs not paying the EUPT participation fee will be initially reminded, and then warned that information concerning their lab may be blacked out in the final report of the concerned EUPT, that the certificate of participation may not be issued to them, and that their participation in subsequent EUPTs could be denied. In case of a repetitive non-payment, the EUPT organisers may inform the corresponding NRL and/or the competent authority responsible for the OfL.

Confidentiality and Communication

The owner of all EUPT data is DG-SANTE and as such they have access to all information.

For each EUPT, the laboratories are given a unique code (lab code), initially only known to themselves and the organisers. Furthermore, the EURLs reserve the right to share EUPT results and codes among 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[2], 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 of 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 PT items to the participants 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 for preparing the PT item, as well as links to the EUPT-Target Pesticides List and the EUPT-Calendar.

Target Pesticides List and PT-Residue Definitions

The Target Pesticides 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 set at 50% of the lowest MRLs found either in Regulation (EC) No. 2005/396 or in Regulation (EU) No. 2016/128 (Baby Food Directive).

The residue definition in an EUPT may differ from the legal one if this is deemed necessary by the organisers for ensuring a better evaluation of the results. Participants must express their results as defined in the Target Pesticides List of the respective EUPT. Separately quantifiable analytes are typically listed separately unless stated otherwise.

Specific Protocol

The organising EURL will publish a Specific Protocol at least 2 weeks before the PT testing item is shipped to the participants. 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 PT item upon receipt and on how to submit results, as well as other relevant information.

Homogeneity of the PT Item

The Homogeneity of the PT test Item evaluated according to ISO 13528:2022, Annex B[8].

Stability of the Analytes Contained in the PT Item

The PT item will be tested for stability according to ISO 13528:2022, Annex B[8].

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. This can be done via the EURL data submission tool (in the following named Webtool) by answering the question whether the concerned analyte is included within the routine scope of the laboratory and the question about the analytical experience with the compound.

General Procedures for Reporting Results

Participating laboratories are responsible for reporting their own quantitative results to the organisers within the stipulated deadline. Each laboratory will be able to report only one result for each analyte detected in the PT item. The concentrations of the analytes detected should be expressed in ‘mg/kg’ unless indicated otherwise in the specific protocol of the respective EUPT.

[8] ISO 13528:2022: ‘Statistical methods for use in proficiency testing by interlaboratory comparisons”, International Organization for Standardization.

When reporting, the following number of significant figures should be given:

Concentration values ≤ 0.01 mg/kg - two significant figures (e.g. 0.0078; 0.010)
Concentration values > 0.01 mg/kg to three significant figures (e.g. 0.0123; 0.123; 1.23; 12.3 mg/kg).

No penalties will apply where a laboratory reports deviating numbers of significant figures. For the calculation of z scores, the values will be used as reported.

Laboratories should not report results below their own reporting limits (RLs). Any reported numerical result that is lower than the RL will be marked as a 'False Reporting' (FR), see False Reporting on page 9.

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 the method used inherently accounts for method bias (see cases a-c below), laboratories are required to report the recovery (in percent), and whether their results was corrected mathematically using a recovery factor reflecting the reported recovery.

Results with bias above 20% (apparent recovery not within the range of 80-120%), for which no correction for bias was undertaken, might 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 Webtool. 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 obliged to provide information on the analytical method(s) they have used. This must be done via the Webtool, which serves for submitting analytical results.

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"[9] by the organiser, and/or (ii) "not detected" by the overwhelming majority (e.g. > 95 %) of the participating laboratories that had targeted the specific analytes.

Any results reported lower than the MRRL will not be considered as false positives, even though these results should not have been reported. If these results are additionally lower than the lab's reporting limit, they will be attributed with FR ('False Reporting').

- ***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 organisers to treat the PT item and b) detected by the organisers as well as the majority of the participants that had targeted these specific analytes at or above the respective MRRLs. Such results will also be regarded as "not correctly found" when it comes to categorization in A and B based on scope.

Where for a compound present in the PT item a laboratory reports "not detected" and a RL exceeding the assigned value, the result will still be judged as a false negative, despite this practice being consistent and adequate within a routine working environment. The FN judgement should in this case penalize the laboratory for not being able to achieve sufficient sensitivity for the analyte in question.

- ***False reporting (FR)***

A result reported below the laboratories own reporting limit (RL) will be evaluated as a 'False Reporting' (FR). If the analytes concerned are present in the test material, z scores will be calculated as for any other numerical results. Furthermore, these results will be included in the population of results for the determination of the assigned value, unless they are excluded for other reasons (e.g. reported by laboratories of non-EU or EFTA countries, generated using biased methods, etc.).

[9] The term "not detected" is also used in the Webtool. In this context this term entails also cases where no numerical result was reported (e.g. because the level determined was < MRRL and/or < RL)

- ***Estimation of the Assigned Value (x_{pt})***

The Assigned Values (consensus concentration) will typically be estimated using the robust mean estimate of the participant results (x^*) as described in ISO 13528:2022[10], taking into account the results reported by EU and EFTA countries laboratories only.

In reports, assigned values will be rounded to 3 significant figures if ≥0.01 mg/kg and to 2 significant figures if <0.01 mg/kg (i.e. 0.0078; 0.123; 1.23; 12.3 mg/kg). For the calculation of z scores, the organisers may opt to use assigned values rounded to more significant figures than those stated above.

- ***Omission or Exclusion of Results***

Results reported by laboratories from non-EU/EFTA member states are excluded from the population used to derive the assigned value.

Despite the use of robust statistics, all results 10 times higher than the assigned values will be omitted and the assigned values will be recalculated.

- ***Uncertainty of the Assigned Value u(x_{pt})***

The uncertainty of the robust mean values $u(x_{pt})$ will be 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.

- ***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[11], a percentage FFP-RSD of 25 % is currently used for all analyte-matrix combination, with the target standard deviation being calculated as follows:

[10] 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).

[11] Comparative Study of the Main Top-down Approaches for the Estimation of Measurement Uncertainty in Multiresidue Analysis of Pesticides in Fruits and Vegetables. J. Agric. Food Chem., 2011, 59(14), 7609-7619. DOI:10.1021/jf104060h

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

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 from participant results").

- ***z scores***

This parameter is calculated using the following formula:

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

where x_i is the value reported by the laboratory, x_{pt} is the assigned value, and $\text{FFP-}\sigma_{pt}$ is the standard deviation using the FFP approach. Z scores shown in the preliminary and Final EUPT-Report 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.

Following ISO 17043:2010[12], z scores will be classified as follows:

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

Z scores higher than 5 will be reported as >5 in the reports and in certificates.

All false negatives will be assigned a z score of -4.

- ***Collection of Measurement Uncertainty (MU) Figures***

For each EUPT the participating labs are asked to voluntarily report the MU figure they would report in routine analyses.

- ***Categorization of Laboratories***

A scope-based classification into Category A and Category B will be employed. Laboratories that

a) have analysed at least 90% of the compulsory analytes in the target pesticides list
b) have correctly detected and quantified at least 90 % of the analytes present in the PT item
c) reported no false positives

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

will be considered to have demonstrated 'sufficient scope' and will therefore be classified into Category A. The criterion of analytes present in the PT item, will be calculated as 90 % of the number of analytes needed to be correctly detected and quantified (no obtained FN for any of the compounds) rounding down to the nearest full number (see Table 1).

Table 1: Number of analytes from the Target Pesticides List needed to be targeted or analytes present in the PT item that need to be correctly detected and quantified to have sufficient scope.

No. of compulsory analytes present in the PT 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	

- ***Overall Performance of Laboratories - Combined z Scores***

For evaluation of the overall performance of laboratories the average of the squared z scores (AZ^2)[13] are calculated. To minimize the influence of outlying results, the calculation of AZ^2 will not be conducted in the case of < 6 results. Z scores higher than 5 will be set as 5 and false negative z scores (-4.0) will be included. Combined z scores will only be calculated for laboratories within Category A and considering results of compulsory analytes only.

[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. DOI:10.1007/s00216-010-3877-3

Considering the cut-off of high z scores at 5, 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.

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

Laboratories within Category B will be typically ranked according to the total number of analytes they correctly reported to be present in the PT item. The number of acceptable z scores achieved may be presented, too.

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 being too low.

Where only a few results per lab are available (mostly the case in EUPT-SRMs), 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 combined z score calculations.

Publication of Results

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

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 included in all tables or figures in the Final EUPT-Report.

Certificates of Participation

The EUPT organisers will deliver a Certificate of Participation to each participating laboratory showing the z scores achieved for each individual analyte, and if available the classification into Categories, and AZ^2 scores. The certificates will be sent by email and in some cases also be uploaded onto the EURL-DataPool and thereby be accessible to the concerned laboratories only.

Feedback and Complaints

Complaints and appeals on aspect concerning the PT are welcome. Complaints about a non-arrival of a PT item or about the bad condition of the PT item upon arrival should be done through the Webtool shortly as indicated in the specific protocols. The EURLs will track complaints about the evaluation of the participants results and follow up within due time. After the publication of the final EUPT report, the organizers reserve the right not to consider any complaints arriving more than two months after its publication.

Appeals and complaints concerning the principles of organisation and statistical analysis of the results according to the General Protocol should be made prior to the start of a PT. By signing up to an EUPT, the participant agrees with the provisions of the General Protocol valid for the PT-season in question.

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

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 and carefully study the latest version of these documents.**

If substantial errors are discovered in the Preliminary EUPT-Report the organisers will distribute a new corrected version, therein it will be stated that the previous version is no longer valid. The online version on the PT website will be replaced.

Where substantial errors are discovered in the Final EUPT-Report 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 revised certificates will be issued.

Follow-up Activities on Behalf of Participants

According to ISO 17025, 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.

Upon request, the laboratory's corresponding NRL and/or EURL is to be informed about the outcome of any investigative activities for false positives, false negatives and for results with |z| ≥ 3.0.

Follow-up Activities on Behalf of Organizers and Underperformance Rules

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 PT 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 appropriate actions to be taken.

Disclaimer

The EURLs retain 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.

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

SPECIFIC PROTOCOL

for the EU Proficiency Test for Pesticide Residues in Cereals/Feeding stuff using Multi Residue Methods, EUPT-CF19 (2025)

(last updated: 13 January 2025)

Introduction

This protocol is complementary to the General Protocol for EU Proficiency Tests for Pesticide Residues in Food and Feed (12th Edition). The current proficiency test covers pesticides that are determined by Multi Residue Methods. This EUPT is to be performed by all National Reference Laboratories for Cereals and/or Feeding stuffs (NRL-CFs) as well as by all official EU laboratories (OfLs) responsible for official pesticide residue controls on cereals and/or feeding stuff, as far as their scope overlaps with that of the EUPT-CF19.

Test Item (Test Material)

This proficiency test concerns the analysis of pesticide residues in rye kernels. The rye 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 13528:2023, Annex B. All these tests will be conducted by the Organiser, the EURL-CF which is (ISO 17025:2017 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 rye 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 17 January 2024.

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 13 January to 27 January 2025. As default all mandatory pesticides are preselected.

Results and method submission: The **EUPT Webtool** will be accessible from 28 January 2024 for sample receipt acknowledgement and submission results and method information.

The deadline for submission is 24 February 2025 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 31 January 2025 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 31 January 2025 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 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 5 March 2025.

Follow-up actions

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

Documents

All documents related to EUPT-CF19 can be found on EUPT-CF19 website.

EURL | Pesticides in Cereals and Feedingstuffs | EUPT-CF19 on Rye kernels

Calendar

Activity	Dates
Announcement Calendar Target Pesticide List	28 October 2024
EUPT-Registration Website open	27 November 2024
Deadline for registration	2 January 2025
Specific Protocol published	13 January 2025
Website for selecting pesticide scope open	13 January 2025
Website for selecting pesticide scope closed	27 January 2025
Distribution of Test items	27 January 2025
Deadline for receipt and acceptance of Test Materials	within 24 hr on receipt
Deadline for Result Submission	24 February 2025 at 23.00 CET
Deadline for submission of additional method information for false negative results	5 March 2025 at 23.00 CET
Preliminary Report (only compilation of results) published	5 May 2025
Final Report published	December 2025

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

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 (approximately 13 €) will be charged per reminder.

If the participant requests DTU to issue a new invoice because additional or new information is needed on the invoice, or if they simply want a copy of the original invoice, additional costs may be incurred due to the administrative workload.

Any questions concerning invoices must be directed to Anne Rhein Hansen, arh@dtu.dk at the financial department of DTU.

Contact information:

Mette Erecius Poulsen

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