DTU Fødevareinstituttet
DTU National Food Institute

EU Reference Laboratory on Cereals & Feeding stuff

Proficiency Test on pesticide residue wheat straw

EUPT-CF18
2024

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Mette Erecius Poulsen

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EU PROFICIENCY TESTS
EUPT-CF18, 2024

Pesticide Residues in Wheat Straw

Final Report

1. edition

Mette Erecius Poulsen
Ederina Ninga
Elena Hakme

December 2024

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National Food Institute
Technical University of Denmark
Henrik Dams Allé
Building 202
2800 Lyngby

Organiser:

DTU **National Food Institute**

DANAK
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Mette Erecius Poulsen
Director of EURL Cereals and Feedingstuff
National Food Institute
Research Group for Analytical Food Chemistry
Technical University of Denmark
Henrik Dams Alle
Building 202
DK-2800 Lyngby
Phone: +45 3588 7463
Fax: +45 3588 7448
E-Mail: eurl-cf@food.dtu.dk
http://www.eurl-pesticides.eu

Organising Team at EURL for Pesticide Residues in Cereals and Feedingstuff:

Ederina Ninga

Elena Hakme

Andrea Alsted Hansen

Ban M. Kadhum

Quality Control Group:

Antonio Valverde	University of Almería, Spain
Paula Medina-Pastor	European Food Safety Authority (EFSA), Italy

Advisory Group

Amadeo R. Fernández-Alba	University of Almeria, Spain
André de Kok	Former WFSR - Wageningen Food Safety Research, Wageningen, The Netherlands
Bjoern Hardebush	Chemisches und Veterinäruntersuchungsamt (CVUA) Freiburg, Germany
Carmen Ferrer Amate	University of Almeria, Spain
Finbarr O'Regan	Pesticide Control Laboratory, Co. Kildare, Ireland
Hans Mol	WFSR - Wageningen Food Safety Research, Wageningen, The Netherlands
Magnus Jezussek	Bavarian Authority of Health and Food Safety, Erlangen, Germany
Michelangelo Anastassiades	Chemisches und Veterinäruntersuchungsamt (CVUA) Stuttgart, Germany
Patrizia Pelosi	Istituto Superiore di Sanità, Roma, Italy
Ralf Lippold	Chemisches und Veterinäruntersuchungsamt (CVUA) Freiburg, Germany
Hermann Unterluggauer	AGES, Institute for Food Safety, Innsbruck, Austria
Štěpán Radim	Czech Agriculture and Food Inspection Authority
Tuija Pihlström	National Food Agency, Uppsala, Sweden

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 eighteenth 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 and the EUPT-CF17 on wheat kernels. The PTs in 2007, 2009, 2011, 2015 and 2020 were jointly organised by the EURL-CF and EURL-SRM using same cereal and focusing on both MRM and SRM pesticides. The other PTs have only focused on MRM-pesticides. The wheat straw used for EUPT-CF18 were treated with formulations in the field. No treatment was done post-harvest in the laboratory.

Participation in EUPT-CF18 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.

CONTENTS

EUROPEAN COMMISSION EURL PROFICIENCY TEST ON PESTICIDE RESIDUES IN CEREALS EUPT-CF18, 2024

1. INTRODUCTION

On 23 November 2023 the announcement of the 18th European Commission's Proficiency Test on Cereals and Feed (EUPT-CF18) 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 170 individual compulsory compounds and 63 voluntary requiring the use of multi residue methods (MRMs), along with a minimum required reporting level (MRRL) stipulated for each compound. Links to The General Protocol containing information (**Annex 1**) that is common to all EUPTs, the Specific protocol (**Annex 2**), as well as a list of labs that are obliged to take part in the EUPT-CF18, were provided via the homepage. Laboratories were able to register online from December 2023 to 18 of March 2024. In total 103 laboratories from EU and EFTA countries agreed to participate in the test as well as 13 laboratories from EU-Candidate States and Third Countries.

The wheat straw were sprayed in the field with 14 pesticides. The cultivation was performed in 2023 in Denmark by the Danish Centre for Food and Agriculture at Aarhus University. After analyses of the pesticide residues content, it was decided to not apply additional spiking on the test material as the pesticides levels in the treated material was high.
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 40 g portions of the wheat straw. The Test Items were shipped to participants on 8 April 2024 and the deadline for submission of results to the Organiser was the 6 May 2024. The deadline for submission of additional information for false negative results was the 15 May 2024. 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 one gram 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 (wheat straw). The overall capacity and capability of the laboratories within the EU, as assessed from previous PTs and surveys, was also taken into account. The minimum required reporting level (MRRL) for all pesticides in the target list was in general set at 0.01 mg/kg. However, for 20 pesticides the MRRL were set at or below 0.005 mg/kg.

1.3 Preparation of the Test Item

The field spraying was performed in 2023 in Denmark and organised by Danish Centre for Food and Agriculture at Aarhus University. Approximately, 10.3 kg of the harvested wheat were used for this PT. Due to the hight levels of

residues found in the treated test material, it was decide to not apply any additional spiking in the PT test material(**Table 1**). The test material was milled with a cutting mill with a sieve size of 2 mm. Forty gram portions of the homogenized wheat straw were then weighed out into screw-capped polyethylene plastic bottles, sealed, numbered, and stored in a freezer at about -20 °C prior to homogeneity testing and distribution to participants.

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

Pesticides	Application in field	Formulation/standard
Acetamiprid	x	Mospilan
Aclonifen	x	Fenix
Azosystrobin	x	Amistar
Benzovindiflupyr *	x	Elatus plus
Epoxiconazole	x	Opus
Fenpicoxamid	x	Inatreq
Flonicamid	x	Teppeki
Fluvalinate	x	Mvrik
Fluxapyraxad	x	Imtrex
Malathion	x	Malathion 440
Mefentrifluconazole *	x	Revisol
Proquinazid	x	Talius
Prothioconazole	x	Delaro
Pyraclostrobin	x	Opus
Trifloxystrobin	x	Delaro

*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-CF18.

Table 2. Statistical evaluation of the homogeneity test data (n=22 analyses using a sub-sample of 1 g in each case). S_s: Between Sampling Standard Deviation.

Pesticides	Mean, mg/kg	Ss^2	c	$Ss^2 < c$
Acetamiprid	0.182	0.0000	0.0006	Pass
Aclonifen	20.3	0.0480	6.9777	Pass
Azoxystrobin	1.01	0.0000	0.0196	Pass
Benzovindiflupyr*	2.20	0.0000	0.1115	Pass
Epoxiconazole	0.741	0.0000	0.0102	Pass
Fenpicoxamid*	0.017	0.0000	0.0000	Pass
Flonicamid	0.031	0.0000	0.0000	Pass
Fluxapyroxad	0.765	0.0004	0.0107	Pass
Mefentrifluconazole*	1.48	0.0000	0.0473	Pass
Proquinazid	1.10	0.0007	0.0227	Pass
Prothioconazole-desthio	0.275	0.0002	0.0013	Pass
Pyraclostrobin	5.02	0.0000	0.5122	Pass
Tau-fluvalinate	0.332	0.0000	0.0042	Pass
Trifloxystrobin	0.719	0.0000	0.0105	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: 8 April 2024
Day 2: 22 April 2024
Day 3: 6 May 2024

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 aclonifen and fenpicoxamid 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
Acetamiprid	0.221	0.000	0.016	Pass
Aclonifen	16.5	0.580	1.462	Pass
Azoxystrobin	1.19	0.005	0.082	Pass
Benzovindiflupyr	2.13	0.035	0.191	Pass
Epoxiconazole	0.717	0.032	0.059	Pass
Fenpicoxamid	0.018	0.001	0.002	Pass
Flonicamid	0.028	0.001	0.002	Pass
Fluxapyroxad	0.832	0.049	0.099	Pass
Mefentrifluconazole	1.54	0.007	0.123	Pass
Proquinazid	1.09	0.032	0.097	Pass
Prothioconazole-desthio	0.296	0.003	0.022	Pass
Pyraclostrobin	5.89	0.041	0.471	Pass
Tau-fluvalinate	0.287	0.016	0.024	Pass
Trifloxystrobin	0.759	0.036	0.068	Pass

*Voluntary pesticides.

1.6 Organisational details

1.6.1 Access to documents, registration and confidentiality

In the invitation letter, all NRLs and OfLs were requested to register using the online registration link from December 2023. All documents related to this EUPT (Calendar, Target Pesticides List, Specific Protocol, General Protocol) were uploaded to the EURL website and the CIRCA platform. Laboratories that were intending not to participate were given the opportunity to explain the reasons for their non-participation. Participants from Candidate countries and third countries did also have access to another online registration link. On 22 March 2024, 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 8 April 2024, the Test Item (40 g) was shipped to all participants in insulated polystyrene boxes containing a freezer block. The laboratories were asked to check the state of the sample on receipt and to enter the web tool to report whether they accept/not accept the Test Item. No blank test material was send.

1.6.3 Submission of results

The participants had to submit their results via a web tool. All participants had access to the result-submission website from a few days after shipment until the result-submission deadline (8 May 2024). Participants were asked not only to report their analytical results, but also to give information regarding accreditation, reporting limits and details regarding the methods they used to analyse the Test Item.

2. EVALUATION OF THE RESULTS

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

2.1 False positives and negatives

2.1.1 False positives

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

2.1.2 False negatives

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

2.1.3 False reporting

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) but it will be allocated a z score as any other numerical result. Such results will be, furthermore, included in the results population for establishing the assigned value (xpt), unless they are eliminated for other reasons (e.g. laboratory status, use of biased methodology).

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 special justifiable cases, the EUPT-Panel may decide to eliminate certain results traceably associated with gross errors, or to use only the results of a subgroup consisting of laboratories that have repeatedly demonstrated good performance for the specific compound in the past.

2.3 Uncertainty of the assigned value

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

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

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

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

The percentage FFP-RSD is set at 25% based on experience from results of previous EUPTs. The EUPT-Panel reserves the right to also employ other approaches on a case-by-case basis considering analytical difficulties and experience gained from previous proficiency tests.
For informative purposes the robust relative standard deviation (CV*) is calculated according to ISO 13528:2022; Chapter 7.7 (Consensus value from participant results) following Algorithm A in Annex C [4].

2.5 z scores

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

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

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

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

Z scores will be interpreted in the following way as is set in the ISO 17043:2023 [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.

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

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

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

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

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

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

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

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

3. RESULTS

3.1 Summary of reported results

In total, 103 EU and EFTA laboratories, from 29 different countries (26 EU member states), agreed to participate in this proficiency test. Thirteen EU participants did not submit results. Six of these were laboratory analysing feed and having a limited scope that did not include any of the pesticide present in the Test Item. Additionally, nine 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** 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]
Acetamiprid	83	7	2	92
Aclonifen	71	19	1	79
Azoxystrobin	89	1	0	99
Benzovindiflupyr*	50	40	0	56
Epoxiconazole	88	2	0	98
Fluxapyroxad	82	8	0	91
Mefentrifluconazole*	43	47	1	48
Proquinazid	81	9	0	90
Prothioconazole-desthio	80	10	1	89
Pyraclostrobin	84	6	0	93
Tau-fluvalinate	86	4	1	96
Trifloxystrobin	88	2	0	98
Boscalid[2]	*87[3]*	*3*	*59[3]*	*97*
*Fenpicoxamid**	*38[3]*	*52*	*3[3]*	*42*
Flonicamid	*79[3]*	*11*	*9[3]*	*88*
Fluopyram[2]	*86[3]*	*4*	*26[3]*	*96*

* 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 = 90). False negatives are included in reported results.

[2] Boscalid and fluopyram were present in the test item due to cross contamination in the field. The data for these compounds are for informational purposes only.

[3] Data are only for informative purposes as the pesticide level were too low to evaluate.

Acetamiprid, azoxystrobin, boscalid, epoxiconazole, fluopyram, fluxapyroxad, proquinazid, pyraclostrobin, tau-fluvalinate and trifloxystrobin were the most frequently analysed compounds with ≥90 % of the labs submitting results for these compounds. Aclonifen, prothioconazole-desthio and flonicamid were analysed and reported by 79-89 % of the participants. Benzovindiflupyr, mefentrifluconazole and fenpicoxamid were only analysed and reported by 42-56 % of participants.

3.1.1 *False positives*

Nine participants (all form EU and EFTA) countries reported 11 results for nine different additional pesticides above the MRRL that had not been used to treat the Test Item (**Table 5**). The pesticides were: acephate, clothianidin, deltamethrin, endosulfan alpha-, hexaconazole, metconazole, parathion, procymidone and spirotetramat. In all cases the compounds were not detected either by the Organizer, or by the other participating laboratories. The reported results were therefore considered to be false positives. Additionally, 2 laboratories (lab code 49 and 66) reported results for DDT, p,p- and 2-phenylphenol at 0.004 mg/kg and 0.008 mg/kg, respectively. However, these two results were not evaluated as false positives because they are below the MRRL at 0.01 mg/kg.

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

Lab code	Pesticides	Concentration mg/kg	Determination technique	RL, mg/kg	MRRL, mg/kg
20	Procymidone	0.068	GC-MS/MS (QQQ)	0.01	0.01
45	Parathion	1.97	GC-Ion Trap	0.01	0.01
49	Endosulfan, alpha-	0.029	GC- (μ) ECD	0.01	0.01
52	Hexaconazole	0.051	LC-MS/MS QQQ	0.01	0.01
57	Acephate	0.274	LC - MS/MS	0.01	0.01
57	Clothianidin	0.012	LC - MS/MS	0.05	0.01
57	Spirotetramat	0.046	LC - MS/MS	0.01	0.01
78	Hexaconazole	0.461		0.01	0.01
87	Deltamethrin	0.014	GC-MS/MS (QQQ)	0.01	0.01
88	Metconazole	0.032	GC-MS/MS (QQQ)	0.01	0.01
118	Metconazole	0.024	GC-MS/MS (QQQ)	0.01	0.01

3.1.2 *False reported*

Additionally, twelve 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.

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. Five participants submitted 6 false negatives results for 5 different pesticides, which represents 0.6% of the total number of results submitted by EU and EFTA laboratories. Six % of the EU and EFTA participants (5 laboratories) reported false negative results.

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	Determination technique	Concentration mg/kg	RL, mg/kg	MRRL, mg/kg
18	Boscalid	GC-MS/MS (QQQ)	0.006	0.01	0.01
49	DDT, p,p-	GC- (μ) ECD	0.004	0.01	0.01
57	Clothianidin	LC - MS/MS	0.012	0.5	0.01
58	Malathion	GC-MS (Q)	0.005	0.01	0.01
66	2-Phenylphenol	GC-MS/MS (QQQ)	0.008	0.01	0.01
66	Malathion	GC-MS/MS (QQQ)	0.007	0.01	0.01
74	Malathion	GC-MS/MS (QQQ)	0.007	0.01	0.01
77	Boscalid	LC - MS/MS	0.005	0.01	0.01
77	Fluopyram	GC-MS/MS (QQQ)	0.009	0.01	0.01
78	Malathion		0.010	0.01	0.01
79	Boscalid		0.008	0.01	0.01
82	Tau-Fluvalinate	GC-MS/MS (QQQ)	0.245	0.5	0.01
96	Boscalid	LC-MS/MS QQQ	0.007	0.01	0.01
96	Malathion	LC-MS/MS QQQ	0.006	0.01	0.01
104	Malathion	GC-MS/MS (QQQ)	0.006	0.01	0.01

Table 7. False negative results (FN).

Lab code	Acetamiprid	Aclonifen	Mefentrifluconazole*	Prothioconazole-Desthio	Tau-Fluvalinate
50	FN				FN
55		FN			
66			FN		
70	FN				
106				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 0.183 - 16.8 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**).

3.2.2 Target standard deviations and Alg A standard deviations

The target standard deviation was obtained using a fixed FFP-RSD value of 25%. In parallel, the Algorithm A standard deviation (Alg A-RSD) was calculated for informative purposes only. The range of Alg A-RSD values was for the evaluated pesticide in the range of 17-30 % but on average, the Alg A-RSD was 23%, lower than 25% FFP-RSD used for the z score calculations.

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, %
Acetamiprid	0.01	0.183	0.006	25	25
Aclonifen	0.01	16.8	0.653	25	26
Azoxystrobin	0.01	0.939	0.030	25	24
Benzovindiflupyr *)	0.01	2.19	0.090	25	23
Epoxiconazole	0.01	0.679	0.018	25	20
Fluxapyroxad	0.01	1.14	0.036	25	23
Mefentrifluconazole *)	0.01	1.41	0.050	25	19
Proquinazid	0.01	1.11	0.030	25	20
Prothioconazole-desthio	0.01	0.251	0.007	25	21
Pyraclostrobin	0.01	5.39	0.180	25	24
Tau-Fluvalinate	0.01	0.28	0.011	25	30
Trifloxystrobin	0.01	0.778	0.018	25	17
Boscalid[1]	*0.01*	*0.011*	*0.001*	*25*	*36*
Fenpicoxamid[1]*	*0.01*	*0.026*	*0.001*	*25*	*20*
Flonicamid[1]	*0.01*	*0.028*	*0.001*	*25*	*26*
Fluopyram[1]	*0.01*	*0.014*	*0.000*	*25*	*21*

* Voluntary pesticides

[1] The assigned values are less than 3 times the MRRL and consequently shown for informative purposes only.

3.3 Assessment of laboratory performance

3.3.1 Z scores

Z scores have been calculated for all the quantified pesticides using the FFP RSD of 25%. **Table 9** shows an overview of the acceptable, questionable, and unacceptable z scores and **Tables 10 a/b** - show the individual results and z scores for each laboratory and pesticide together with the assigned values. A graphical representation of the z scores (for EU and EFTA countries) can be seen in **Appendix 4**.

Of the reported results for the evaluated pesticides, more than 90% were acetamiprid, azoxystrobin, benzovindiflupyr, epoxiconazole, fluxapyroxad, mefentrifluconazole, proquinazid, prothioconazole-desthio, pyraclostrobin and trifloxystrobin. For aclonifen and tau-fluvalinate was 87 % of the results were acceptable.

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 %
Acetamiprid	83	0.183	93	2	5	2
Aclonifen	71	16.8	87	8	4	1
Azoxystrobin	89	0.939	90	4	6	0
Benzovindiflupyr	50	2.19	98	2	0	0
Epoxiconazole	88	0.679	94	2	3	0
Fluxapyroxad*	82	1.14	94	2	4	0
Mefentrifluconazole	43	1.41	91	2	7	2
Proquinazid	81	1.11	95	4	1	0
Prothioconazole-Desthio	80	0.251	94	4	3	1
Pyraclostrobin	84	5.39	92	6	2	0
Tau-Fluvalinate	86	0.280	87	6	7	1
Trifloxystrobin	88	0.778	93	3	3	0

* Voluntary pesticides

[1] Unacceptable z scores includes false negative results.

3.3.2 Analytical methods used

More than five different analytical methods have been used by the laboratories. For the majority of the results, 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 was used for 25% of the results, mainly GC-MS/MS (93%), but also GC-MS (2%) and GC- (μ) ECD (2%) was used. GC-iontrap for2%, GC-TOF, GC-Q-Orbitrap for 1% result. LC instruments was 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 result were analysed using specific detectors such as GC-NPD, LC-MS, LC-Iontrap , LC-Fluorescence, LC-UV, or LC-DAD.

Table 10a. Results for the mandatory pesticides azoxystrobin, bixafen, cyazofamid, cyfluthrin, difenoconazole, dimethomorph and flonicamid in mg/kg, the corresponding z scores, MRRLs and the assigned values.

Laboratory code	Acetamiprid	Z scores (FFP RSD (25%)	Aclonifen	Z scores (FFP RSD (25%)	Azoxystrobin	Z scores (FFP RSD (25%)	Epoxiconazole	Z scores (FFP RSD (25%)	Fluxapyroxad	Z scores (FFP RSD (25%)	Proquinazid	Z scores (FFP RSD (25%)	Prothioconazole-Desthio	Z scores (FFP RSD (25%)		Pyraclostrobin	Z scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**			**0.01**	
Assigned value	**0.183**		**16.8**		**0.939**		**0.679**		**1.14**		**1.11**		**0.251**			**5.39**	
1	0.189	0.1	17.1	0.1	0.971	0.1	0.681	0.0	1.25	0.4	0.998	-0.4	0.241	-0.2		5.18	-0.2
2	0.195	0.3	20.6	0.9	1.04	0.4	0.652	-0.2	1.33	0.7	1.08	-0.1	0.195	-0.9		5.77	0.3
3	0.219	0.8			1.19	1.1	0.67	-0.1	1.43	1.0	1.16	0.2	0.262	0.2		7.68	1.7
4	0.156	-0.6	17.2	0.1	0.884	-0.2	0.542	-0.8	0.995	-0.5	1.11	0.0	0.224	-0.4		4.83	-0.4
5																	
6	0.188	0.1	13.1	-0.9	0.639	-1.3	0.552	-0.7	0.786	-1.2	0.942	-0.6	0.233	-0.3		4.05	-1.0
10	0.189	0.1	30	3.2	1.19	1.1	0.79	0.7	1.41	1.0	1.2	0.3	0.31	0.9		6.06	0.5
11																	
12																	
13	0.15	-0.7			0.85	-0.4	0.52	-0.9								3.8	-1.2
14	0.101	-1.8			0.101	-3.6	0.101	-3.4								0.101	-3.9
15																	
16	0.244	1.3	19.2	0.6	1.55	2.6	0.92	1.4	1.34	0.7			0.27	0.3		6.8	1.0
17	0.204	0.5	17.5	0.2	1.158	0.9	0.855	1.0	1.25	0.4	1.27	0.6	0.302	0.8		6.17	0.6
18	0.145	-0.8			0.76	-0.8	0.538	-0.8	0.917	-0.8	1.02	-0.3	0.22	-0.5		5.72	0.2
19	0.173	-0.2	19.1	0.6	0.906	-0.1	0.717	0.2	1.08	-0.2	1.16	0.2	0.283	0.5		5.33	0.0
20	0.16	-0.5	16.7	0.0	1.1	0.7	0.78	0.6	1.1	-0.1	0.98	-0.5	0.27	0.3		4.7	-0.5
21	0.188	0.1	19.3	0.6	1.01	0.3	0.69	0.1	1.22	0.3	1.28	0.6	0.3	0.8		5.22	-0.1
22	0.167	-0.3	7.86	-2.1	1.12	0.8	0.966	1.7	1.51	1.3	0.997	-0.4	0.16	-1.5		6.89	1.1
23					0.806	-0.6	0.735	0.3	1.08	-0.2	1.07	-0.2	0.253	0.0			
24	0.13	-1.2	20.5	0.9	0.735	-0.9	0.833	0.9	0.744	-1.4	0.441	-2.4	0.219	-0.5		6.04	0.5
25	0.19	0.2	12.9	-0.9	0.74	-0.8	0.51	-1.0	1.1	-0.1	1.5	1.4	0.23	-0.3		3.2	-1.6
26	0.147	-0.8	13.4	-0.8	1.54	2.6	0.719	0.2	0.813	-1.1	0.856	-0.9	0.266	0.2		4.25	-0.8
27	0.149	-0.7	14.7	-0.5	0.75	-0.8	0.615	-0.4	0.927	-0.7	0.977	-0.5	0.245	-0.1		5.46	0.0
28	0.145	-0.8	16.9	0.0	0.899	-0.2	0.704	0.1	1.15	0.0	1.14	0.1	0.239	-0.2		4.46	-0.7
29	0.198	0.3	14.02	-0.7	0.961	0.1	0.658	-0.1	1.065	-0.3	0.974	-0.5	0.301	0.8		5.004	-0.3
30	0.207	0.5	18.4	0.4	0.98	0.2	0.703	0.1	1.45	1.1	1.44	1.2	0.332	1.3		4.9	-0.4
31	0.185	0.0	17.6	0.2	1	0.3	0.832	0.9	1.26	0.4	1.27	0.6	0.295	0.7		6.71	1.0
32	0.197	0.3	18	0.3	1.204	1.1	0.851	1.0	1.389	0.9	1.21	0.3	0.306	0.9		5.94	0.4
33	0.117	-1.4	18.037	0.3	1.031	0.4	0.612	-0.4	0.523	-2.2	1.209	0.3	0.14	-1.8		4.856	-0.4
34																	
36	0.251	1.5	17.5	0.2	0.983	0.2	0.746	0.4	1.155	0.1	1.199	0.3	0.272	0.3		4.81	-0.4
37	0.167	-0.3	19.514	0.7	1.028	0.4	0.912	1.4	1.302	0.6	0.789	-1.2	0.282	0.5		6.506	0.8
38	0.195	0.3	16.8	0.0	0.789	-0.6	0.67	-0.1	1.03	-0.4	1.02	-0.3	0.285	0.5		5.72	0.2
39	0.148	-0.8	14.5	-0.5	1.2	1.1	0.666	-0.1	0.89	-0.9	0.7	-1.5	0.244	-0.1		4.7	-0.5

Laboratory code	Acetamiprid	Z scores (FFP RSD (25%)	Aclonifen	Z scores (FFP RSD (25%)	Azoxystrobin	Z scores (FFP RSD (25%)	Epoxiconazole	Z scores (FFP RSD (25%)	Fluxapyroxad	Z scores (FFP RSD (25%)	Proquinazid	Z scores (FFP RSD (25%)	Prothioconazole-Desthio	Z scores (FFP RSD (25%)		Pyraclostrobin	Z scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**			**0.01**	
Assigned value	**0.183**		**16.8**		**0.939**		**0.679**		**1.14**		**1.11**		**0.251**			**5.39**	
40	0.189	0.1	21.5	1.1	1.17	1.0	0.716	0.2	1.29	0.5	1.19	0.3	0.296	0.7		5.87	0.4
41																	
42	0.092	-2.0	15.2	-0.4	0.83	-0.5	0.449	-1.4	0.866	-1.0	1.26	0.5	0.161	-1.4		5.06	-0.2
44	0.263	1.8			1.28	1.5	0.696	0.1	1.79	2.3	1.32	0.7	0.325	1.2		6.84	1.1
45					0.753	-0.8											
46	0.173	-0.2	19.4	0.6	1.09	0.6	0.651	-0.2	1.09	-0.2	1.12	0.0	0.251	0.0		5.3	-0.1
47	0.14	-0.9	8.54	-2.0	0.87	-0.3	0.49	-1.1	0.87	-0.9	0.62	-1.8	0.14	-1.8		3.42	-1.5
49	0.427	>5			2.402	>5	2.374	>5			2.153	3.7				8.917	2.6
50	FN	-4.0	23.04	1.5	0.118	-3.5	0.791	0.7	1.26	0.4	1.25	0.5	0.21	-0.7		5.58	0.1
51																	
52	0.105	-1.7	6.666	-2.4	0.758	-0.8	0.487	-1.1	0.704	-1.5	0.701	-1.5	0.167	-1.3		4.439	-0.7
53	0.229	1.0	19.4	0.6	0.96	0.1	0.73	0.3	1.26	0.4	1.13	0.1	0.3	0.8		7	1.2
54																	
55	0.2	0.4	FN	-4.0	0.55	-1.7	0.58	-0.6	0.77	-1.3	1.25	0.5	0.31	0.9		2.73	-2.0
56																	
57	0.25	1.5			0.904	-0.2	0.704	0.1	1.191	0.2						2.56	-2.1
58	0.12	-1.4	18.2	0.3	1.09	0.6	0.69	0.1	1.2	0.2	1.33	0.8	0.25	0.0		5.81	0.3
59	0.074	-2.4	17.4	0.2	0.815	-0.5	0.672	0.0	0.968	-0.6	1.04	-0.3	0.176	-1.2		4.32	-0.8
60	0.232	1.1	17.491	0.2	0.981	0.2	0.654	-0.1	1.365	0.8	0.801	-1.1	0.348	1.5		5.24	-0.1
61	0.174	-0.2	14.7	-0.5	0.709	-1.0	0.58	-0.6	1	-0.5	1.09	-0.1	0.263	0.2		5.39	0.0
62	0.117	-1.4	10.7	-1.4	0.807	-0.6	0.912	1.4	0.977	-0.6	1.02	-0.3	0.259	0.1		6.11	0.5
63					0.812	-0.5	0.703	0.1	1.02	-0.4	1.11	0.0	0.246	-0.1			
64	0.272	2.0	20.9	1.0	1.2	1.1	0.891	1.2	1.56	1.5	1.42	1.1	0.296	0.7		7.81	1.8
65	0.222	0.9	18.5	0.4	0.906	-0.1	0.706	0.2	0.975	-0.6	1.22	0.4	0.24	-0.2		4.59	-0.6
66	0.208	0.6	13.439	-0.8	1.576	2.7	0.683	0.0	1.273	0.5	1.552	1.6	0.352	1.6		6.661	0.9
67	0.174	-0.2	14.6	-0.5	0.477	-2.0	0.693	0.1	1.05	-0.3	1.22	0.4	0.144	-1.7		5.17	-0.2
68					0.872	-0.3	0.752	0.4	0.995	-0.5	1.12	0.0	0.299	0.8			
69	0.123	-1.3			0.675	-1.1	0.534	-0.9	0.842	-1.0	1.1	0.0	0.198	-0.9		2.57	-2.1
70	FN	-4.0			0.112	-3.5	0.124	-3.3	0.106	-3.6	0.716	-1.4	0.28	0.5		2.04	-2.5
71	0.407	4.9	20.8	1.0	1.07	0.6	0.739	0.4	1.65	1.8	1.39	1.0	0.659	>5		6.57	0.9
72	0.025	-3.5			0.162	-3.3											
73	0.05	-2.9	0.72	-3.8	0.26	-2.9	0.2	-2.8								1.1	-3.2
74	0.218	0.8	21.2	1.1	1.42	2.0	0.659	-0.1	1.67	1.9	1.26	0.5				6.49	0.8
75	0.26	1.7	18.5	0.4	1.02	0.3	0.57	-0.6	1.27	0.5	0.98	-0.5	0.26	0.1		6.49	0.8
76					0.801	-0.6	0.711	0.2	1.02	-0.4	1.142	0.1	0.26	0.1			
77	0.15	-0.7	10.5	-1.5	0.62	-1.4	0.54	-0.8	0.84	-1.1	0.81	-1.1	0.17	-1.3		3.9	-1.1
78	0.238	1.2	20.3	0.8	1.274	1.4	1.124	2.6	1.55	1.4	1.251	0.5	0.4301	2.8		6.62	0.9

Laboratory code	Acetamiprid	Z scores (FFP RSD (25%)	Aclonifen	Z scores (FFP RSD (25%)	Azoxystrobin	Z scores (FFP RSD (25%)	Epoxiconazole	Z scores (FFP RSD (25%)	Fluxapyroxad	Z scores (FFP RSD (25%)	Proquinazid	Z scores (FFP RSD (25%)	Prothioconazole-Desthio	Z scores (FFP RSD (25%)		Pyraclostrobin	Z scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**			**0.01**	
Assigned value	**0.183**		**16.8**		**0.939**		**0.679**		**1.14**		**1.11**		**0.251**			**5.39**	
79	0.124	-1.3	14.139	-0.6	1.349	1.7	0.847	1.0	1.111	-0.1	0.934	-0.6	0.197	-0.9		4.753	-0.5
80	0.199	0.4	14.9	-0.4	0.618	-1.4	0.606	-0.4	1.27	0.5	1.04	-0.3	0.199	-0.8		5.11	-0.2
81	0.151	-0.7	6.11	-2.5	0.97	0.1	0.628	-0.3	1.05	-0.3	1.78	2.4	0.115	-2.2		5.98	0.4
82	0.193	0.2	14.5	-0.5	0.939	0.0	0.784	0.6	1.12	-0.1	0.996	-0.4	0.24	-0.2		6.33	0.7
83	0.145	-0.8	42	>5	1.19	1.1	0.559	-0.7	1.29	0.5	1.13	0.1	0.19	-1.0		7.51	1.6
84	0.15	-0.7			0.91	-0.1	0.664	-0.1					0.27	0.3		6.13	0.5
85	0.238	1.2	27.7	2.6	1.29	1.5	0.977	1.8	2.22	3.8	1.1	0.0	0.308	0.9		8.1	2.0
86	0.242	1.3	24.8	1.9	1.27	1.4	0.933	1.5	1.31	0.6	1.34	0.8	0.336	1.3		6.7	1.0
87	0.153	-0.7			0.89	-0.2	0.613	-0.4								5.72	0.2
88	0.139	-1.0	24.7	1.9	0.839	-0.4	0.715	0.2	1.08	-0.2	1.05	-0.2	0.261	0.2		5.86	0.3
89	0.198	0.3	11.96	-1.1	1.07	0.6	0.8	0.7	1.66	1.8	1.45	1.2	0.271	0.3		6.81	1.1
90	0.207	0.5	21.4	1.1	1.1	0.7	0.761	0.5	1.28	0.5	1.33	0.8	0.252	0.0		5.59	0.1
91	0.199	0.4	14.6	-0.5	0.949	0.0	0.578	-0.6	1.152	0.0	0.947	-0.6	0.219	-0.5		4.988	-0.3
92	0.218	0.8	15.21	-0.4	0.988	0.2	0.78	0.6	1.204	0.2	1.184	0.3	0.185	-1.1		5.663	0.2
93	0.228	1.0	10.7	-1.4	0.844	-0.4	0.597	-0.5	1.1	-0.1	0.649	-1.7	0.207	-0.7		3.63	-1.3
94	0.163	-0.4	11.7	-1.2	0.851	-0.4	0.623	-0.3	0.947	-0.7			0.174	-1.2		4.48	-0.7
95					0.59	-1.5	0.39	-1.7	0.59	-1.9	0.55	-2.0				3.1	-1.7
96	0.175	-0.2	14.9	-0.4	0.806	-0.6	0.376	-1.8	1.237	0.3	1.42	1.1	0.264	0.2		3.98	-1.0
97	0.165	-0.4	25.99	2.2	0.719	-0.9	0.441	-1.4	0.816	-1.1	1.17	0.2				4.06	-1.0
98																	
99	0.175	-0.2	19.903	0.8	1.004	0.3	0.795	0.7	1.069	-0.2	1.298	0.7	0.269	0.3		5.777	0.3
100	0.204	0.5	24.21	1.8	1.49	2.3	0.727	0.3	1.57	1.5	1.21	0.3	0.437	3.0		6.04	0.5
101	0.174	-0.2			1.04	0.4	0.664	-0.1	1.17	0.1	0.857	-0.9	0.224	-0.4		8.76	2.5
102	0.219	0.8	14.35	-0.6	1.057	0.5	0.813	0.8	1.359	0.8	1.196	0.3	0.306	0.9		6.718	1.0
103	0.175	-0.2	15.6	-0.3	0.753	-0.8	0.598	-0.5	0.959	-0.6	1.17	0.2	0.244	-0.1		4.58	-0.6
104	0.194	0.2	22.4	1.3	1.01	0.3	0.772	0.5	1.14	0.0	1.32	0.7	0.266	0.2		5.44	0.0
105	0.196	0.3	17.8	0.2	0.924	-0.1	0.704	0.1	1.07	-0.2	0.94	-0.6	0.292	0.6		5.14	-0.2
106	0.105	-1.7	7.15	-2.3	0.497	-1.9	0.377	-1.8	0.621	-1.8	0.584	-1.9	FN	-4.0		4.63	-0.6
107	0.16	-0.5	12.4	-1.0	0.9	-0.2	0.7	0.1	1.5	1.3	1.2	0.3	0.27	0.3		4.3	-0.8
108																	
109	0.203	0.4	19.867	0.7	1.002	0.3	0.737	0.3	1.227	0.3	1.192	0.3	0.249	0.0		5.928	0.4
110	0.177	-0.1	20.17	0.8	1.021	0.3	0.689	0.1	1.039	-0.4	1.22	0.4	0.251	0.0		4.7	-0.5
111	0.179	-0.1	7.561	-2.2	1.786	3.6	0.447	-1.4	3.014	>5	1.745	2.3	0.206	-0.7		7.679	1.7
112																	
113																	
114	0.236	1.2	17.8	0.2	1.085	0.6	0.799	0.7	1.357	0.8	1.162	0.2	0.256	0.1		5.521	0.1
116																	

Laboratory code	Acetamiprid	Z scores (FFP RSD (25%)	Aclonifen	Z scores (FFP RSD (25%)	Azoxystrobin	Z scores (FFP RSD (25%)	Epoxiconazole	Z scores (FFP RSD (25%)	Fluxapyroxad	Z scores (FFP RSD (25%)	Proquinazid	Z scores (FFP RSD (25%)	Prothioconazole-Desthio	Z scores (FFP RSD (25%)		Pyraclostrobin	Z scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**			**0.01**	
Assigned value	**0.183**		**16.8**		**0.939**		**0.679**		**1.14**		**1.11**		**0.251**			**5.39**	
117	0.18	-0.1	25	2.0	1.1	0.7	0.64	-0.2	0.92	-0.8	0.95	-0.6	0.27	0.3		5	-0.3
118	0.14	-0.9	15.8	-0.2	0.736	-0.9	0.521	-0.9	1.104	-0.1	0.992	-0.4	0.108	-2.3		4.14	-0.9
119	0.2	0.4	19	0.5	1	0.3	0.76	0.5	1.2	0.2	1.3	0.7	0.25	0.0		5.6	0.2

Table 10b. Results for the mandatory pesticides tau-fluvalinate and trifloxystrobin and the voluntary pesticides benzovindiflupyr and mefentrifluconazole, the corresponding z scores, MRRLs and the assigned values. The data for boscalid, fenpicoxamid, flonicamid and fluopyram is only shown for informative purposes due to low assigned values.

Laboratory code	Tau-fluvalinate	Z-scores (FFP RSD (25%)	Trifloxystrobin	Z-scores (FFP RSD (25%)	Benzovindiflupyr*)	Z-scores (FFP RSD (25%)	Mefentriflu-conazole *)	Z-scores (FFP RSD (25%)	Boscalid	Z-scores (FFP RSD (25%)	Fenpicoxamid *)	Z-scores (FFP RSD (25%)	Flonicamid	Z-scores (FFP RSD (25%)	Fluopyram	Z-scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.280**		**0.778**		**2.19**		**1.41**		**0.011**		**0.026**		**0.028**		**0.014**	
1	0.195	-1.2	0.687	-0.5	2.03	-0.3	1.37	-0.1			0.0215	-0.6	0.027	-0.2	0.0151	0.2
2	0.24	-0.6	0.831	0.3	2.4	0.4	1.53	0.4			0.0239	-0.3			0.0143	0.0
3	0.308	0.4	0.724	-0.3	1.77	-0.8							0.037	1.2		
4	0.375	1.4	0.765	-0.1	2.31	0.2	1.34	-0.2			0.0239	-0.3	0.0201	-1.2	0.011	-0.9
5																
6	0.408	1.8	0.645	-0.7	1.45	-1.4	1.09	-0.9			0.0221	-0.5	0.0281	-0.1		
10	0.4	1.7	0.75	-0.1					0.012	0.5			0.034	0.8	0.016	0.5
11																
12																
13	0.11	-2.4	0.63	-0.8									0.028	-0.1	0.011	-0.9
14			0.101	-3.5												
15																
16			0.799	0.1									0.0358	1.0	0.0194	1.4
17	0.274	-0.1	0.954	0.9									0.032	0.5	0.018	1.1
18	0.24	-0.6	0.758	-0.1					0.0061	-1.7			0.022	-0.9	0.011	-0.9
19	0.304	0.3	0.712	-0.3	2.01	-0.3	1.33	-0.2			0.0272	0.2	0.0318	0.5	0.0171	0.8
20	0.28	0.0	0.71	-0.3									0.025	-0.5	0.012	-0.6
21	0.306	0.4	0.874	0.5	2.1	-0.2	1.42	0.0	0.0096	-0.4	0.023	-0.4	0.034	0.8	0.016	0.5
22	0.246	-0.5	0.806	0.1	2.37	0.3	2.37	2.7			0.026	0.1	0.042	1.9	0.017	0.8
23	0.28	0.0	0.798	0.1	2.1	-0.2	1.44	0.1							0.0125	-0.5
24	0.283	0.0	0.906	0.7	2.57	0.7			0.0131	0.9					0.0298	4.4
25	0.24	-0.6	0.69	-0.5	2.9	1.3	1.7	0.8			0.033	1.2	0.019	-1.3	0.018	1.1
26	0.267	-0.2	0.648	-0.7	1.58	-1.1	1.71	0.9	0.0085	-0.8	0.0291	0.5	0.0288	0.0	0.0133	-0.3
27	0.373	1.3	0.764	-0.1									0.0265	-0.3		
28	0.28	0.0	0.773	0.0									0.027	-0.2	0.015	0.2
29	0.382	1.5	0.725	-0.3	1.483	-1.3	1.145	-0.7			0.015	-1.7	0.025	-0.5	0.011	-0.9
30	0.275	-0.1	0.95	0.9	2.42	0.4	1.22	-0.5			0.025	-0.1	0.033	0.6	0.018	1.1
31	0.357	1.1	0.898	0.6	2.76	1.03	1.24	-0.5					0.022	-0.9		
32	0.172	-1.5	0.921	0.7	3.68	2.7	1.75	1.0	0.0118	0.4	0.0275	0.3	0.0371	1.2	0.0167	0.7
33	0.289	0.1	0.842	0.3	2.185	0.0	1.014	-1.1								
34																
36	0.25	-0.4	0.76	-0.1									0.0229	-0.8	0.0139	-0.1
37	0.213	-1.0	0.811	0.2									0.019	-1.3		
38	0.3	0.3	0.781	0.0	1.8	-0.7	1.4	0.0								
39	0.25	-0.4	0.59	-1.0	1.6	-1.1	1.4	0.0	0.01	-0.3	0.034	1.3	0.023	-0.8	0.01	-1.2

Laboratory code	Tau-fluvalinate	Z-scores (FFP RSD (25%)	Trifloxystrobin	Z-scores (FFP RSD (25%)	Benzovindiflupyr*)	Z-scores (FFP RSD (25%)	Mefentriflu-conazole *)	Z-scores (FFP RSD (25%)	Boscalid	Z-scores (FFP RSD (25%)	Fenpicoxamid *)	Z-scores (FFP RSD (25%)	Flonicamid	Z-scores (FFP RSD (25%)	Fluopyram	Z-scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.280**		**0.778**		**2.19**		**1.41**		**0.011**		**0.026**		**0.028**		**0.014**	
40	0.341	0.9	0.881	0.5									0.0241	-0.6	0.0149	0.2
41																
42	0.334	0.8	0.892	0.6	2.18	0.0	1.27	-0.4			0.029	0.5	0.014	-2.0		
44	0.268	-0.2	0.818	0.2									0.0279	-0.1	0.0149	0.2
45	0.352	1.0														
46	0.345	0.9	0.822	0.2	2.21	0.0	1.33	-0.2			0.0201	-0.9	0.0283	0.0	0.013	-0.3
47	0.12	-2.3	0.43	-1.8									0.02	-1.2	0.01	-1.2
49			1.649	4.5					0.031	>5			0.065	>5		
50	FN	-4.0	0.794	0.1									0.041	1.8	0.017	0.8
51																
52	0.167	-1.6	0.464	-1.6	2.728	1.0	0.816	-1.7			0.024	-0.3	0.036	1.1	0.01	-1.2
53	0.2	-1.1	0.91	0.7	2.06	-0.2	1.45	0.1			0.026	0.1	0.039	1.5	0.017	0.8
54																
55	0.28	0.0	0.74	-0.2	1.43	-1.4	1.1	-0.9			0.36	>5	0.027	-0.2		
56																
57	0.351	1.0	0.921	0.7					0.005	-2.1			0.042	1.9		
58	0.17	-1.6	1.3	2.7											0.012	-0.6
59	0.244	-0.5	0.661	-0.6												
60	0.218	-0.9	0.854	0.4	1.682	-0.9	1.55	0.4			0.037	1.8			0.016	0.5
61	0.293	0.2	0.756	-0.1	2.07	-0.2	1.19	-0.6			0.023	-0.4	0.026	-0.3		
62	0.472	2.7	1.35	2.9									0.012	-2.3		
63	0.254	-0.4	0.785	0.0	2.06	-0.2	1.36	-0.1							0.0141	0.0
64	0.403	1.8	0.956	0.9	3.28	2.0	2.66	3.6			0.0481	3.5				
65	0.38	1.4	0.863	0.4	2.46	0.5	1.41	0.0			0.023	-0.4	0.041	1.8		
66	0.722	>5	0.886	0.6	2.515	0.6	FN	-4.0							0.014	-0.1
67	0.185	-1.4	0.89	0.6							0.0251	-0.1	0.0271	-0.2	0.0115	-0.8
68	0.285	0.1	0.734	-0.2	1.93	-0.5	1.32	-0.2							0.013	-0.3
69	0.306	0.4	0.601	-0.9	1.52	-1.2	1.16	-0.7			0.0251	-0.1	0.0223	-0.9		
70	0.5	3.1	0.359	-2.2					0.035	>5						
71	0.221	-0.8	0.823	0.2					0.0105	-0.1			0.101	>5	0.0524	>5
72	0.099	-2.6	0.136	-3.3												
73	0.062	-3.1	0.18	-3.1												
74			0.986	1.1					0.0101	-0.2			0.0243	-0.6	0.0185	1.2
75	0.21	-1.0	0.84	0.3	1.12	-2.0	1.25	-0.4			0.022	-0.6	0.034	0.8	0.011	-0.9
76	0.256	-0.3	0.803	0.1	2.122	-0.1	1.295	-0.3							0.013	-0.3
77	0.2	-1.1	0.63	-0.8	1.3	-1.6	1.1	-0.9	0.005	-2.1	0.024	-0.3	0.024	-0.6	0.009	-1.5
78	0.315	0.5	1.066	1.5	2.79	1.1			0.021	3.8			0.1215	>5	0.023	2.5

Laboratory code	Tau-fluvalinate	Z-scores (FFP RSD (25%)	Trifloxystrobin	Z-scores (FFP RSD (25%)	Benzovindiflupyr*)	Z-scores (FFP RSD (25%)	Mefentriflu-conazole *)	Z-scores (FFP RSD (25%)	Boscalid	Z-scores (FFP RSD (25%)	Fenpicoxamid *)	Z-scores (FFP RSD (25%)	Flonicamid	Z-scores (FFP RSD (25%)	Fluopyram	Z-scores (FFP RSD (25%)
MRRL	0.01		0.01		0.01		0.01		0.01		0.01		0.01		0.01	
Assigned value	0.280		0.778		2.19		1.41		0.011		0.026		0.028		0.014	
79	0.22	-0.9	1.055	1.4	2.118	-0.1			0.0084	-0.9	0.0138	-1.8	0.0132	-2.1	0.0133	-0.3
80	0.327	0.7	0.52	-1.3									0.031	0.4	0.015	0.2
81	0.201	-1.1	0.742	-0.2									0.0128	-2.2		
82	0.245	-0.5	0.713	-0.3	2	-0.4	1.62	0.6					0.0289	0.1	0.0127	-0.4
83	0.498	3.1	0.78	0.0	2.48	0.5	1.42	0.0			0.034	1.3	0.027	-0.2	0.013	-0.3
84	0.353	1.0	0.819	0.2												
85	0.379	1.4	0.941	0.8	2.58	0.7							0.0337	0.7		
86	0.327	0.7	0.964	1.0									0.035	0.9		
87	0.201	-1.1	0.666	-0.6					0.021	3.8			0.026	-0.3		
88	0.271	-0.1	0.659	-0.6	1.99	-0.4	1.33	-0.2	0.01	-0.3	0.028	0.4	0.021	-1.0	0.013	-0.3
89	0.396	1.7	1.04	1.3	3.22	1.9							0.032	0.5	0.014	-0.1
90	0.256	-0.3	0.8	0.1									0.031	0.4	0.015	0.2
91	0.203	-1.1	0.68	-0.5	1.898	-0.5			0.01	-0.3			0.027	-0.2	0.013	-0.3
92	0.245	-0.5	0.819	0.2											0.013	-0.3
93	0.13	-2.1	0.598	-0.9									0.03	0.2	0.01	-1.2
94	0.379	1.4	0.7	-0.4	1.756	-0.8			0.013	0.8						
95	0.15	-1.9	0.37	-2.1												
96	0.39	1.6	0.8	0.1	2.48	0.5	1.39	0.0	0.007	-1.4			0.028	-0.1	0.02	1.6
97	0.457	2.5	0.88	0.5									0.0164	-1.7	0.0224	2.3
98																
99	0.364	1.2	0.808	0.2	2.659	0.8	1.865	1.3	0.0101	-0.2	0.026	0.1	0.023	-0.8	0.0158	0.4
100	0.472	2.7	0.938	0.8	2.53	0.6	1.53	0.4	0.017	2.3					0.017	0.8
101	0.284	0.1	0.797	0.1					0.0135	1.0			0.0255	-0.4	0.0163	0.6
102	0.236	-0.6	0.87	0.5	2.572	0.7	1.692	0.8	0.01	-0.3	0.022	-0.6	0.034	0.8	0.013	-0.3
103	0.301	0.3	0.653	-0.6	1.96	-0.4							0.0229	-0.8		
104	0.324	0.6	0.844	0.3	2.64	0.8	1.49	0.2	0.01	-0.3	0.022	-0.6	0.026	-0.3	0.012	-0.6
105	0.284	0.1	0.716	-0.3	2.27	0.1	2.7	3.7	0.01	-0.3	0.021	-0.7	0.03	0.2	0.017	0.8
106	0.135	-2.1	0.381	-2.0									0.0206	-1.1		
107	0.25	-0.4	0.59	-1.0	2.1	-0.2	1.1	-0.9			0.016	-1.5	0.024	-0.6	0.013	-0.3
108	1.2	>5														
109	0.419	2.0	0.88	0.5	2.371	0.3	1.624	0.6			0.022	-0.6	0.033	0.6	0.013	-0.3
110	0.311	0.4	0.844	0.3	2.83	1.2	1.65	0.7			0.03	0.7	0.061	4.6	0.018	1.1
111	0.233	-0.7	0.726	-0.3									0.034	0.8		
112																
113																
114	0.325	0.6	0.902	0.6					0.01	-0.3			0.032	0.5	0.016	0.5
116																

Laboratory code	Tau-fluvalinate	Z-scores (FFP RSD (25%)	Trifloxystrobin	Z-scores (FFP RSD (25%)	Benzovindiflupyr*)	Z-scores (FFP RSD (25%)	Mefentriflu-conazole *)	Z-scores (FFP RSD (25%)	Boscalid	Z-scores (FFP RSD (25%)	Fenpicoxamid *)	Z-scores (FFP RSD (25%)	Flonicamid	Z-scores (FFP RSD (25%)	Fluopyram	Z-scores (FFP RSD (25%)
MRRL	**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**		**0.01**	
Assigned value	**0.280**		**0.778**		**2.19**		**1.41**		**0.011**		**0.026**		**0.028**		**0.014**	
117	0.26	-0.3	0.83	0.3									0.02	-1.2		
118	0.227	-0.8	0.726	-0.3									0.025	-0.5	0.012	-0.6
119	0.17	-1.6	0.93	0.8					0.0077	-1.1			0.023	-0.8	0.012	-0.6

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 (≥9 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 64 EU and EFTA laboratories in Category A (71%), the results were additionally evaluated by calculating the Average of the Squared Score (AZ^2). Of the 64 participants 56 participants (88%) obtained AZ2 score at or below 2 (good), 3 participants (5%) obtained AZ2 values between 2-3 (satisfactory) and 5 participants (8%) obtained AZ2 values ≥3 (unsatisfactory). An additional seven 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	AZ2	False negative	Classification	NRL
1	10	100	2	0.2	0	Good	NRL
2	10	99	2	0.3	0	Good	
3	9	95	1	0.7	0	Good	
4	10	99	2	0.4	0	Good	
6	10	100	2	1.0	0	Good	
10	10	100	0	1.7	0	Good	
17	10	95	0	0.4	0	Good	
19	10	100	2	0.1	0	Good	
21	10	99	2	0.2	0	Good	NRL
22	10	100	2	1.4	0	Good	
24	10	92	1	1.2	0	Good	NRL
25	10	97	2	0.8	0	Good	
26	10	100	2	1.1	0	Good	
28	10	100	0	0.1	0	Good	
29	10	100	2	0.4	0	Good	
30	10	100	2	0.6	0	Good	NRL
31	10	100	2	0.4	0	Good	NRL
32	10	100	2	0.7	0	Good	
33	10	96	2	1.1	0	Good	NRL
36	10	91	0	0.3	0	Good	NRL
37	10	100	0	0.6	0	Good	NRL
38	10	98	2	0.1	0	Good	
39	10	100	2	0.6	0	Good	
40	10	100	0	0.4	0	Good	NRL
42	10	100	2	1.0	0	Good	
44	9	93	0	1.5	0	Good	
46	10	100	2	0.2	0	Good	NRL
47	10	100	0	2.4	0	Satisfactory	
53	10	98	2	0.5	0	Good	

Lab code	No. of detected mandatory pesticides	Analysed from mandatory target list, %	No. of detected voluntary pesticides	AZ2	False negative	Classification	NRL
55	9	99	2	2.6	1	Satisfactory	NRL
58	10	100	0	1.3	0	Good	
59	10	90	0	0.9	0	Good	
60	10	99	2	0.6	0	Good	NRL
61	10	96	2	0.2	0	Good	NRL
62	10	100	0	2.3	0	Satisfactory	
64	10	99	2	1.9	0	Good	NRL
65	10	100	2	0.4	0	Good	NRL
66	10	100	2	4.0	1	Unsatisfactory	
67	10	99	0	1.0	0	Good	NRL
69	9	99	2	1.2	0	Good	NRL
71	10	96	0	>5	0	Unsatisfactory	
75	10	100	2	0.6	0	Good	NRL
77	10	99	2	1.2	0	Good	
79	10	99	1	1.0	0	Good	
80	10	95	0	0.6	0	Good	NRL
81	10	92	0	1.9	0	Good	NRL
82	10	98	2	0.2	0	Good	NRL
83	10	100	2	4.1	0	Unsatisfactory	
85	10	96	1	3.6	0	Unsatisfactory	
86	10	98	0	1.5	0	Good	
89	10	100	1	1.3	0	Good	
90	10	99	0	0.3	0	Good	NRL
91	10	98	1	0.3	0	Good	
92	10	99	0	0.3	0	Good	
93	10	97	0	1.4	0	Good	
96	10	99	2	0.9	0	Good	
97	9	91	0	1.9	0	Good	
99	10	96	2	0.3	0	Good	
100	10	99	2	2.9	0	Satisfactory	
101	9	92	0	0.8	0	Good	
102	10	92	2	0.5	0	Good	
103	10	99	1	0.2	0	Good	NRL
104	10	100	2	0.3	0	Good	
105	10	100	2	0.1	0	Good	
106	9	100	0	4.7	1	Unsatisfactory	
107	10	100	2	0.5	0	Good	
109	10	100	2	0.5	0	Good	

Lab code	No. of detected mandatory pesticides	Analysed from mandatory target list, %	No. of detected voluntary pesticides	AZ2	False negative	Classification	NRL
110	10	98	2	0.2	0	Good	
114	10	100	0	0.4	0	Good	
117	10	100	0	0.6	0	Good	NRL
119	10	100	0	0.4	0	Good	

The 40 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**. Five laboratories was moved from Category A to B due to false positive results. Four 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. Five participants analysed more than 90% of the pesticides on the Target List but reported <9 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
5	0	0	76	0	0	0	0	
11	0	0	24	0	0	0	0	NRL
12	0	0	4	0	0	0	0	
13	6	60	62	0	5	0	0	
14	5	50	61	0	1	0	0	
15	0	0	4	0	0	0	0	
16	8	80	55	0	7	0	0	NRL
18	9	90	82	0	9	0	0	
20[1]	10	100	100	0	10	0	1	
23	7	70	51	2	7	0	0	
27	10	100	88	0	10	0	0	
34	0	0	4	0	0	0	0	
41	0	0	4	0	0	0	0	
45	2	20	34	0	2	0	1	
49	6	60	42	0	0	0	1	
50	8	80	100	0	7	2	0	
51	0	0	2	0	0	0	0	NRL
52[1]	10	100	99	2	9	0	1	NRL
54	0	0	98	0	0	0	0	
56	0	0	63	0	0	0	1	
57	7	70	84	0	6	0	2	NRL
63	7	70	50	2	7	0	0	
68	7	70	49	2	7	0	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
70	8	80	92	0	2	1	0	NRL
72	4	40	60	0	0	0	0	
73	7	70	58	0	0	0	0	
74	8	80	64	0	7	0	0	
76	7	70	50	2	7	0	0	
78[1]	10	100	99	1	8	0	1	
84	7	70	70	0	7	0	0	NRL-CE
87	6	60	75	0	6	0	1	
88[1]	10	100	100	2	10	0	1	
94	9	90	81	1	9	0	0	
95	7	70	63	0	5	0	0	
98	0	0	4	0	0	0	0	
108	1	10	13	0	0	0	0	
111	10	100	88	0	6	0	0	NRL
112	0	0	22	0	0	0	0	
113	0	0	100	0	0	0	0	
116	0	0	100	0	0	0	0	
118[1]	10	100	100	0	9	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-CF12 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-CF12 Hay flour	EUPT-CF13 Oat Kernels	EUPT-CF14 Rice kernels	EUPT-CF15 Rapeseed cake	EUPT-CF16 Barley Kernels	EUPT-CF17 Wheat Kernels	EUPT-CF18 Wheat Straw
Participants submitting results (EU+EFTA)	111	149	156	129	151	149	90
MRM pesticides in the Target Pesticide List	155/23	160/32	164/38	172/41	169/53	169/58	170/63
MRM pesticides in the test material	8	18	19	22	19	20	12
No. of results for MRM pesticides	808	2007	2298	1315	2206	2422	925
Average of 'reported results', %	74	75	80	83	78	85	85
Range of 'reported results', %	40-91	44-94	26-93	57-93	32-97	48-95	48-95
Acceptable z scores, %	93	93	91	87	89	92	92
Questionable z scores, %	3	3.1	3	7	4	3	4
Unacceptable z scores, %	3	3.4	6	6	6	5	4
False negatives, %	1	2.3	3.4	2.0	3.3	2.8	0.6
Number of false positives	7	3	14	9	25	7	10
Category A, % of participating laboratories	51	57	57	57	59	73	71
Good AZ^2, %	92	91	91	67	83	89	87
Satisfactory AZ^2, %	3.4	5.7	6.7	12	7	6	6
Unsatisfactory AZ^2, %	5.1	3.4	2.2	22	10	6	7
Alg A RSD%	20	18	19	30	22	18	23

The number of pesticides included in the Target Pesticide List has also increased during this 18-year period, from 43 to 170 compulsory compounds and 63 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. Last EUPT the number was 19% and the year before it was also 23%. So no improvement was seen on this issue. The analytical scope was in average 85%.

The overall analytical performance (accuracy of measurement) if looking at the percentage of acceptable, questionable, unacceptable z scores has increased during the last 2 EUPTs, and in EUPT-CF18 92% of the results were acceptable and questionable/unacceptable z scores decreased. The average percent of reported results in the last seven EUPT-CF has been between 74-85%. The false negative results have fluctuated between 1-4%. Also the false positive results has been going up and down, in EUPT-CF18 the number increased to 10.

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-CF18 it was 71% so almost the same as the year before. For Category A the percentage of participant with AZ^2 was <2 (good) has been >90% for many year. However, for the rapeseed cake EUPT-CF15 this dropped significantly to 67% and in this EUPT it was 87%.

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

The EUPT-CF18 Test Item was wheat straw containing incurred and spiked pesticides. The wheat straw 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:. One hundred forty-nine EU and EFTA laboratories, from 29 different countries agreed to participate in this proficiency test. Six of them did not report any results due to different reasons. An additional 13 laboratories from EU candidate states and Third Countries registered for the PT and only one did not submitted results. The Target Pesticide List distributed to the laboratories prior to the test contained 170 individual compulsory and 63 voluntary compounds.

The number of false positives and false negatives has varied between the EUPTs. Twenty-five false positive results were reported and the number of false negatives represented 2% of the total number of results. This is at levels typically seen in EUPT-CFs. The average Alg A-RSD was at 23%, lower than the FFP-RSD of 25%.

Of the reported results for the evaluated pesticides, more than 90% were acetamiprid, azoxystrobin, benzovindiflupyr, epoxiconazole, fluxapyroxad, mefentrifluconazole, proquinazid, prothioconazole-desthio, pyraclostrobin and trifloxystrobin. For aclonifen and tau-fluvalinate was 87 % of the results were acceptable.

The Test Item for EUPT-CF19 will be rye kernels, and are planned to be shipped to the laboratories in January 2025. The selection of pesticides will continue to be focused on pesticides included in the scope of the EU multi-annual coordinated control programme, the working document as well as additional pesticides of relevance to feed and/or cereal production in Europe and in other parts of the world from where significant quantities of feed and cereals are imported.

4. ACKNOWLEDGEMENTS

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5. REFERENCES

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

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

[3] CEN EN 15662:2018 - 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	0.01
Acephate	0.01
Aclonifen	0.01
Acetamiprid	0.01
Acrinathrin	0.01
Aldrin	0.005
Ametoctradin	0.01
Azinphos-methyl	0.005
Azoxystrobin	0.01
Bifenthrin	0.01
Biphenyl	0.01
Bitertanol	0.01
Bixafen	0.01
Boscalid	0.01
Bromuconazole	0.01
Buprofezin	0.01
Cadusafos	0.005
Carbaryl	0.005
Carbendazim	0.01
Carbofuran	0.005
Carbofuran-3-hydroxy	0.005
Carboxin	0.01
Chlorantraniliprole	0.01
Chlorfenapyr	0.01
Chlorfenvinphos	0.01
Chlorpropham	0.01
Chlorpyrifos	0.005
Chlorpyrifos-methyl	0.01
Clothianidin	0.01
Cyantraniliprole	0.01
Cyazofamid	0.01
Cyflumetofen	0.01
Cyfluthrin	0.01
Cymoxanil	0.01
Cypermethrin	0.01
Cyproconazole	0.01
Cyprodinil	0.01
Deltamethrin	0.01
Demeton-S-methylsulfone	0.005
Diazinon	0.005
Dichlorvos	0.005
Dieldrin	0.005
Difenoconazole	0.01

Pesticides	MRRL (mg/kg)
Diflubenzuron	0.01
Dimethoate	0.003
Dimethomorph	0.01
Diniconazole	0.01
Endosulfan-alpha	0.01
Endosulfan-beta	0.01
Endosulfan-sulfate	0.01
Epoxiconazole	0.01
Ethion	0.01
Ethirimol	0.01
Ethoprophos	0.005
Etoxazole	0.01
Famoxadone	0.01
Fenbuconazole	0.005
Fenhexamid	0.01
Fenitrothion	0.01
Fenpropathrin	0.01
Fenpropidin	0.01
Fenpropimorph	0.01
Fenpyrazamine	0.01
Fenpyroximate	0.01
Fenthion	0.01
Fenthion-oxon	0.01
Fenthion-oxon-sulfone	0.01
Fenthion-oxon-sulfoxide	0.01
Fenthion-sulfone	0.01
Fenthion-sulfoxide	0.01
Fenvalerate	0.01
Fipronil	0.004
Fipronil-sulfone	0.004
Flonicamid	0.01
Flubendiamide	0.01
Fludioxonil	0.01
Flufenoxuron	0.01
Fluopicolide	0.01
Flupyradifurone	0.01
Fluopyram	0.01
Fluquinconazole	0.01
Flusilazole	0.01
Flutolanil	0.01
Flutriafol	0.01
Fluxapyroxad	0.01
Formetanate	0.01
Hexaconazole	0.01
Imazalil	0.005
Imidacloprid	0.01
Indoxacarb	0.01

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

Pesticides	MRRL (mg/kg)
Spinetoram	0.01
Spirodiclofen	0.01
Spiromesifen	0.01
Spirotetramat	0.01
Spirotetramat metabolite BYI08330-enol	0.01
Spiroxamine	0.01
Sulfoxaflor	0.01
Tau-Fluvalinate	0.01
Tebuconazole	0.01
Tebufenozide	0.01
Teflubenzuron	0.01
Tefluthrin	0.01
Terbuthylazine	0.01
Tetraconazole	0.01
Tetradifon	0.01
Tetramethrin	0.01
Thiabendazole	0.01
Thiacloprid	0.01
Thiamethoxam	0.01
Thiodicarb	0.01
Thiophanate-methyl	0.01
Tolclofos-methyl	0.01
Triadimefon	0.01
Triadimenol	0.01
Triflumizole	0.01
Triflumizole metabolite (FM-6-1)	0.01
Triazophos	0.005
Tricyclazole	0.01
Trifloxystrobin	0.01
Trifluralin	0.01
Triticonazole	0.01
Vinclozolin	0.01
Zoxamide	0.01
Voluntary Compounds (will not be considered in Category A/B classification)	
1,4-Dimethylnaphthalene	0.01
Azadirachtin	0.01
Benalaxyl (sum)	0.01
Benzovindiflupyr	0.01
Chlordane-cis	0.01
Chlordane-oxy	0.01
Chlordane-trans	0.01
Chlorfluazuron	0.01
Clomazone	0.01
Cyflufenamid	0.01
Cyhalofop-butyl	0.01
DDD-pp	0.01

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

Pesticides	MRRL (mg/kg)
Tri-allate	0.01
Triclopyr	0.01
Tritosulfuron	0.01

Appendix 2 Homogeneity data

	Acetamiprid mg/kg		Aclonifen mg/kg		Azoxystrobin mg/kg		Benzovindiflupyr mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
012	0.179	0.172	17.952	20.100	0.975	0.968	1.648	2.182
027	0.183	0.195	19.369	22.294	1.050	0.953	1.969	2.377
039	0.186	0.174	20.800	21.821	1.035	1.099	2.223	2.343
053	0.175	0.204	20.280	20.535	0.937	1.163	2.012	2.247
073	0.183	0.170	20.696	20.871	0.999	0.945	2.270	2.210
119	0.175	0.192	21.495	20.821	1.044	1.113	2.087	2.400
142	0.180	0.184	19.627	19.732	0.930	1.049	2.138	2.367
157	0.175	0.183	20.080	18.597	0.992	0.898	2.377	2.087
172	0.175	0.173	21.276	19.211	0.970	0.950	2.276	2.054
199	0.179	0.194	20.415	20.226	0.983	1.056	2.324	2.480

	Epoxiconazole mg/kg		Fenpicoxamid mg/kg		Flonicamid mg/kg		Fluvalinate-tau(I+II) mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
012	0.599	0.723	0.015	0.016	0.025	0.032	0.194	0.342
027	0.678	0.796	0.018	0.025	0.030	0.033	0.280	0.368
039	0.737	0.757	0.018	0.018	0.032	0.032	0.343	0.322
053	0.685	0.752	0.020	0.020	0.030	0.030	0.283	0.332
073	0.765	0.743	0.018	0.011	0.033	0.033	0.384	0.333
119	0.732	0.785	0.018	0.017	0.031	0.031	0.344	0.381
142	0.742	0.777	0.011	0.020	0.029	0.031	0.296	0.348
157	0.781	0.713	0.011	0.011	0.031	0.030	0.364	0.309
172	0.769	0.712	0.018	0.013	0.032	0.029	0.351	0.292
199	0.784	0.788	0.020	0.013	0.032	0.032	0.352	0.429

	Fluxapyroxad mg/kg		Mefentrifluconazole mg/kg		Proquinazid mg/kg		Prothioconazole-desthio mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
012	0.754	0.786	1.121	1.464	0.874	1.048	0.260	0.269
027	0.816	0.868	1.364	1.589	0.994	1.165	0.284	0.288
039	0.825	0.705	1.485	1.578	1.070	1.103	0.288	0.301
053	0.764	0.779	1.381	1.458	0.997	1.114	0.268	0.293
073	0.755	0.685	1.551	1.476	1.160	1.120	0.268	0.250
119	0.735	0.758	1.422	1.610	1.097	1.192	0.308	0.293
142	0.693	0.790	1.461	1.597	1.093	1.145	0.255	0.285
157	0.766	0.678	1.627	1.402	1.157	1.062	0.260	0.242
172	0.765	0.743	1.512	1.376	1.151	1.043	0.264	0.268
199	0.784	0.853	1.570	1.620	1.172	1.167	0.263	0.301

	Pyraclostrobin mg/kg		Trifloxystrobin mg/kg	
Sample no.	Portion 1	Portion 2	Portion 1	Sample no.
012	4.702	5.145	0.561	0.699
027	4.720	5.633	0.647	0.782
039	5.252	5.576	0.712	0.747
053	4.900	5.727	0.651	0.709
073	4.896	5.025	0.772	0.744
119	5.195	5.218	0.693	0.779
142	4.206	5.119	0.706	0.763
157	4.990	4.478	0.738	0.690
172	4.783	4.601	0.758	0.684
199	5.031	5.219	0.752	0.787

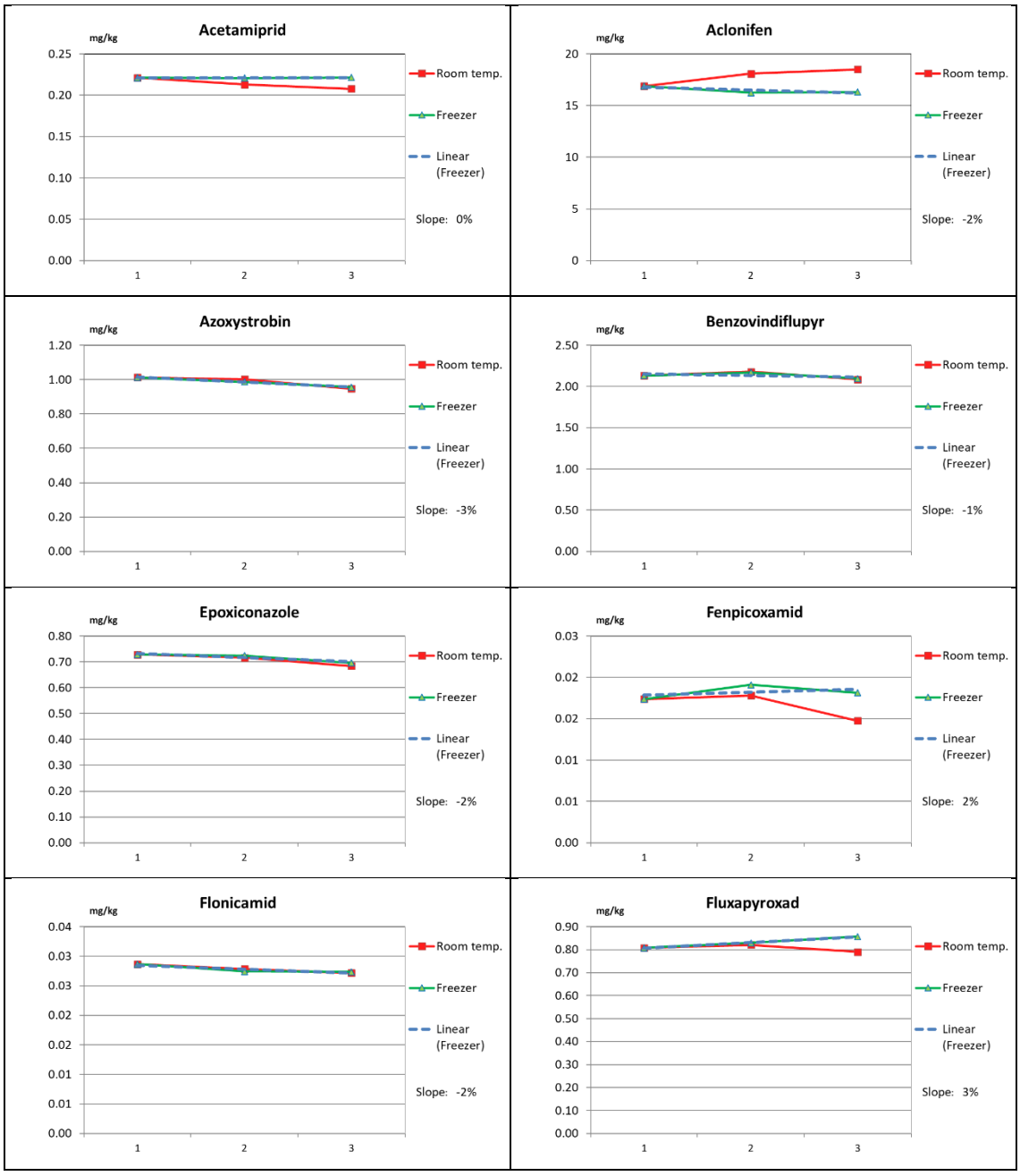
Appendix 3 Stability figures

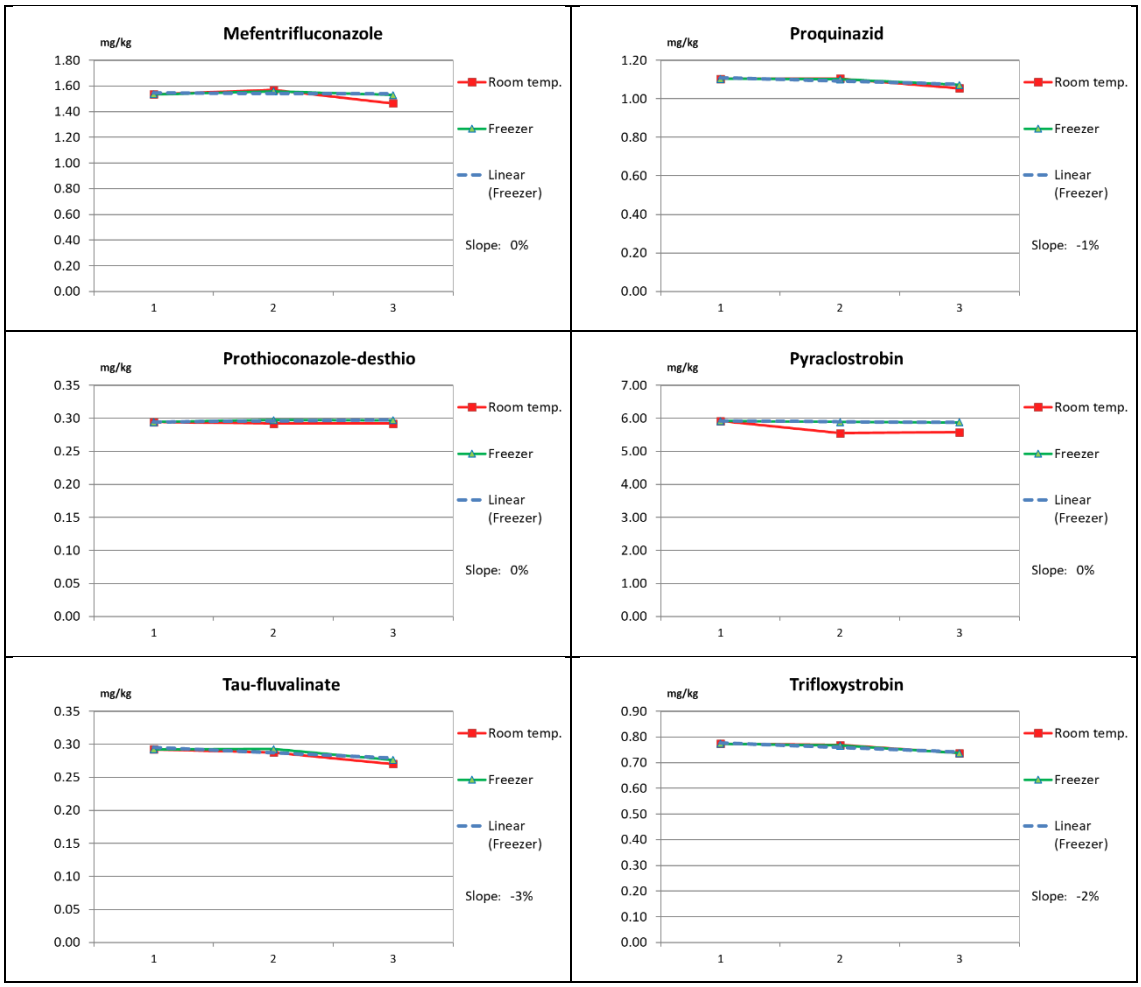
The stability test was performed according to ISO 13528 [5]. Two different storage temperatures were used; room temperature and -18 °C.

The dates of testing were as follows:

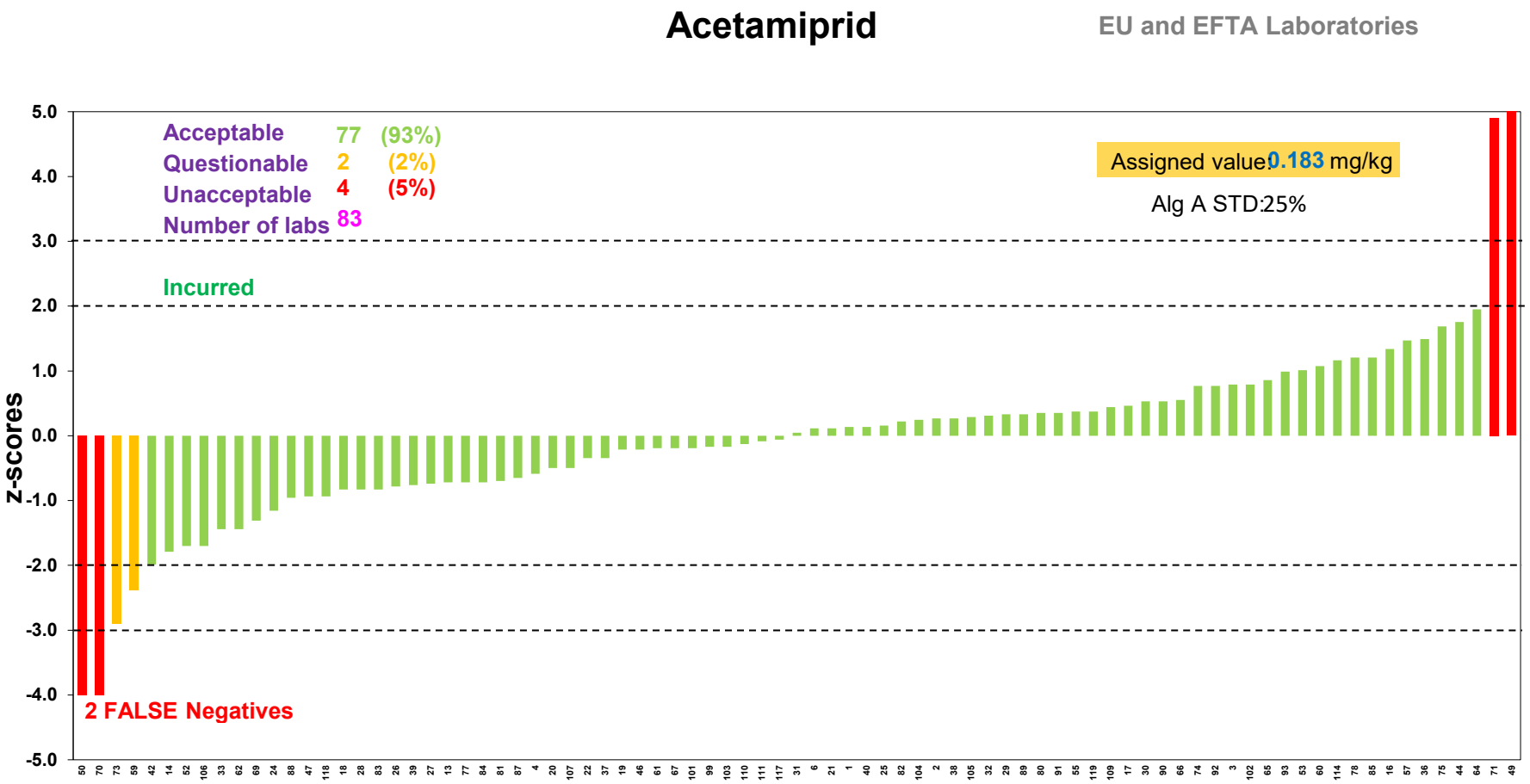
Day 1: 8 April 2024
Day 2: 22 April 2024
Day 3: 6 May 2024

All pesticides passed the test at -18 °C . At room temperature alconifen and fenpicoxamid did not pass the test when stored for 11 weeks.



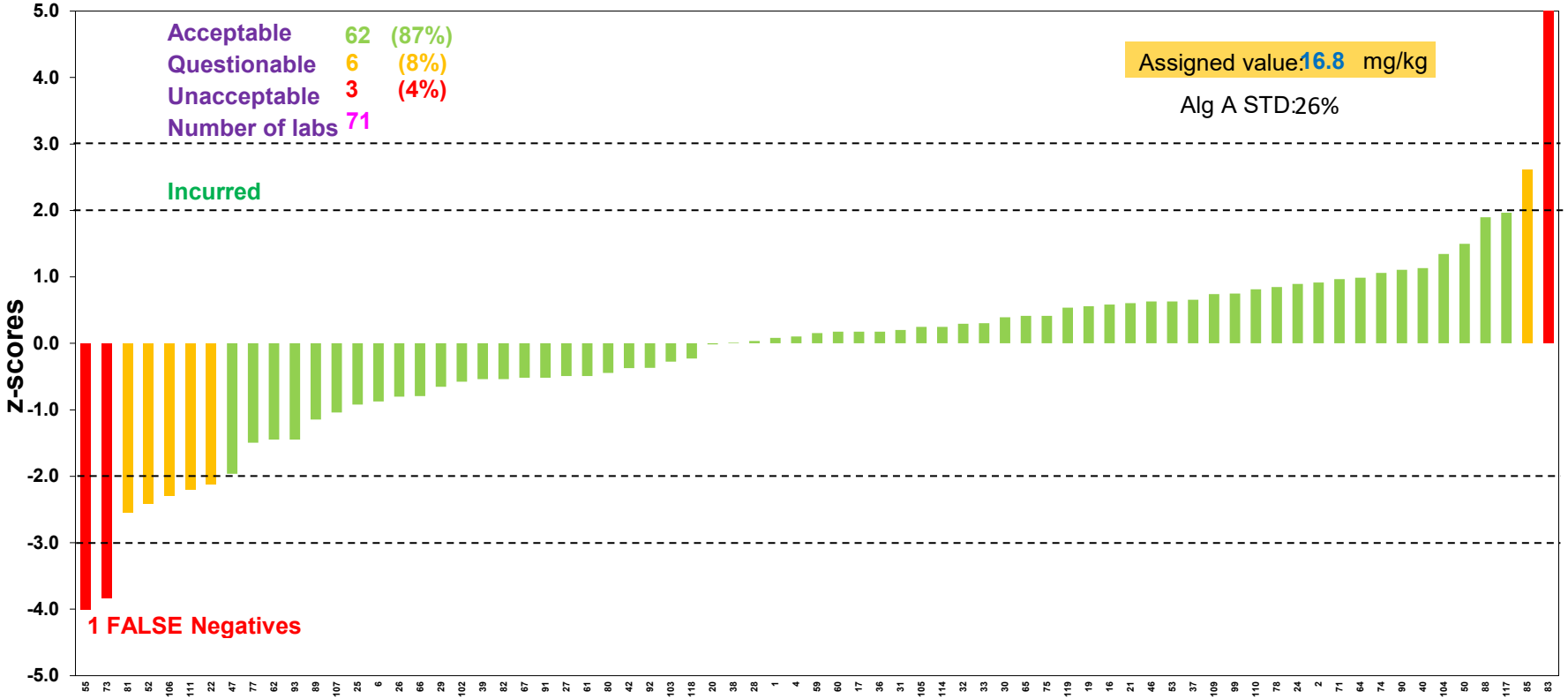

Mefentrifluconazole
mg/kg
1.80
1.60
1.40
1.20
1.00
0.80
0.60
0.40
0.20
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: 0%
Proquinazid
mg/kg
1.20
1.00
0.80
0.60
0.40
0.20
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: -1%
Prothioconazole-desthio
mg/kg
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: 0%
Pyraclostrobin
mg/kg
7.00
6.00
5.00
4.00
3.00
2.00
1.00
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: 0%
Tau-fluvalinate
mg/kg
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: -3%
Trifloxystrobin
mg/kg
0.90
0.80
0.70
0.60
0.50
0.40
0.30
0.20
0.10
0.00
1
2
3
Room temp.
Freezer
Linear (Freezer)
Slope: -2%

Appendix 4 Graphical presentation of z-scores



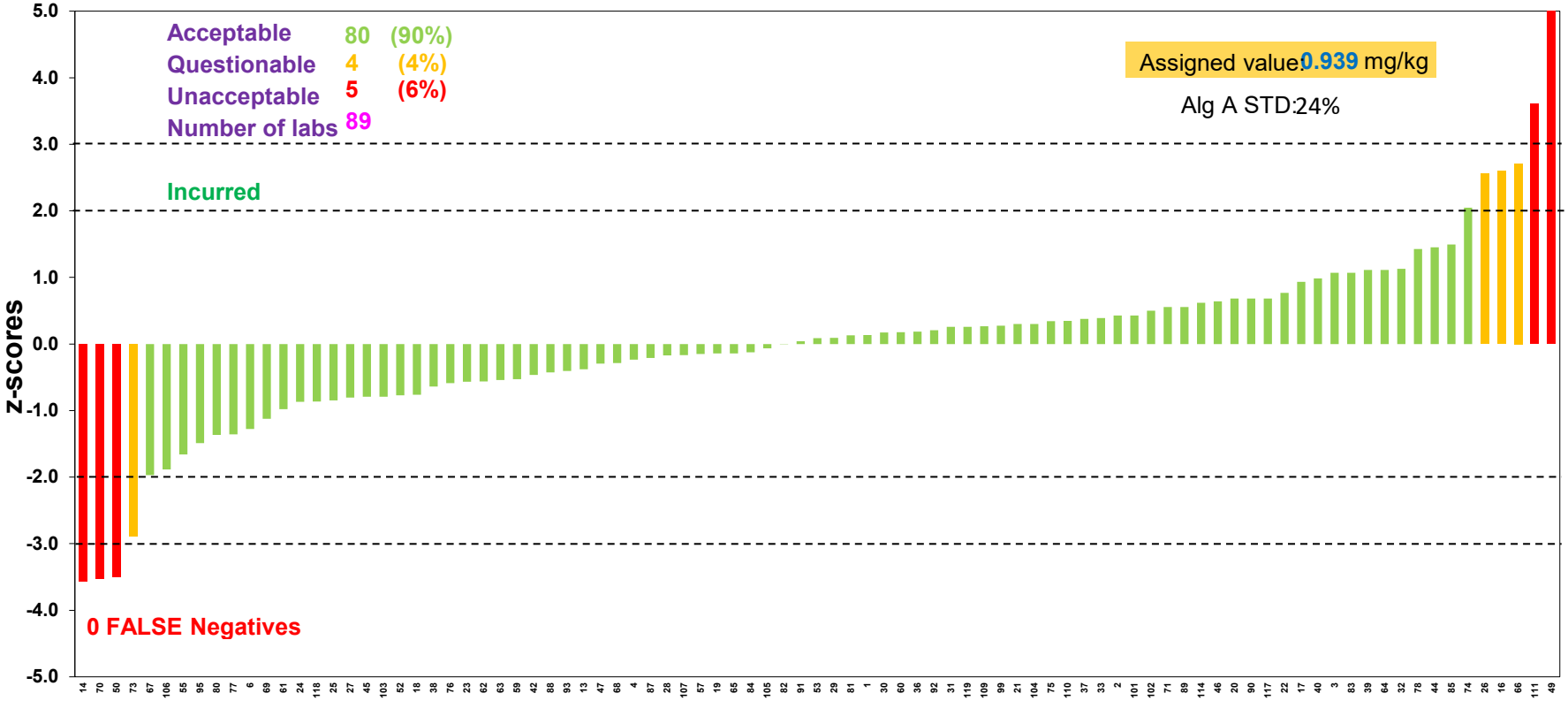
Aclonifen

EU and EFTA Laboratories



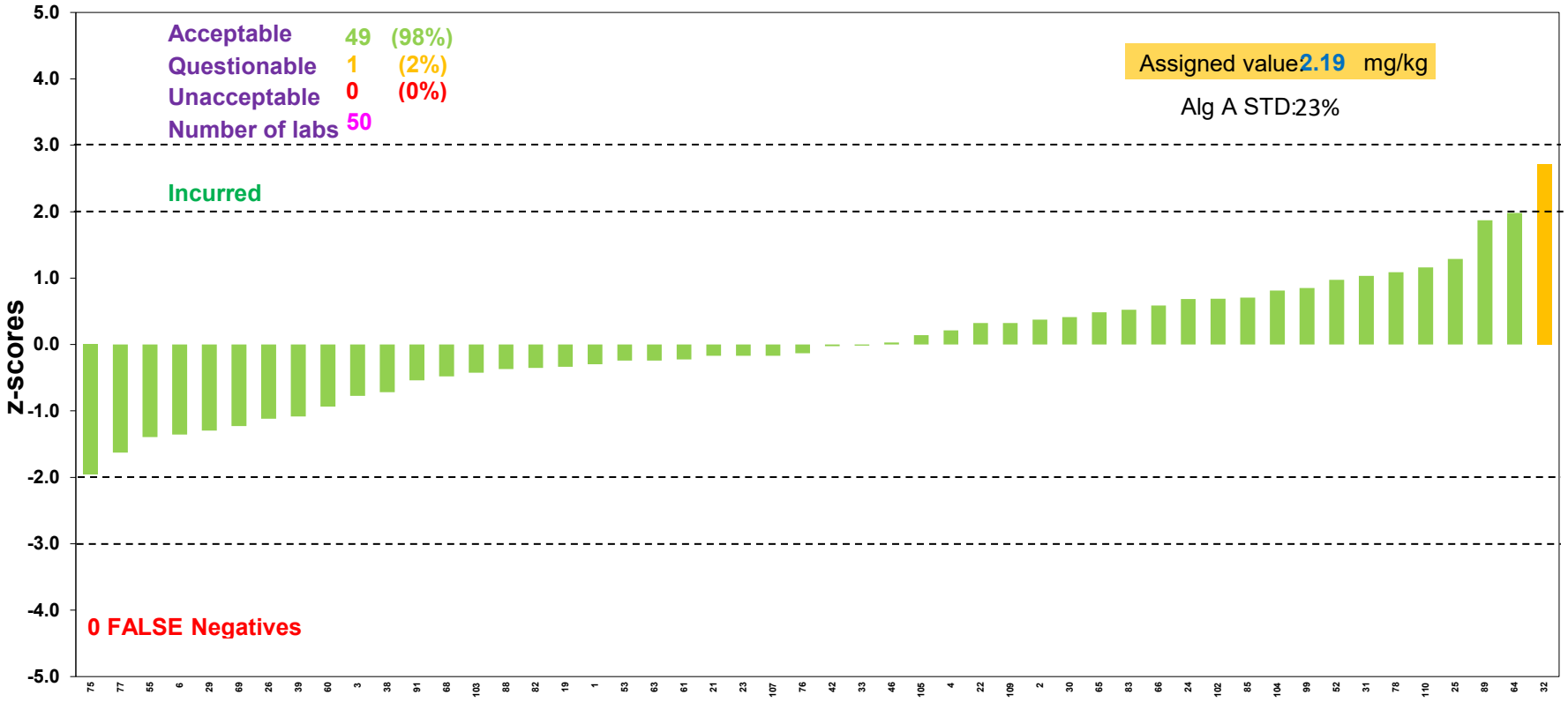
Azoxystrobin

EU and EFTA Laboratories



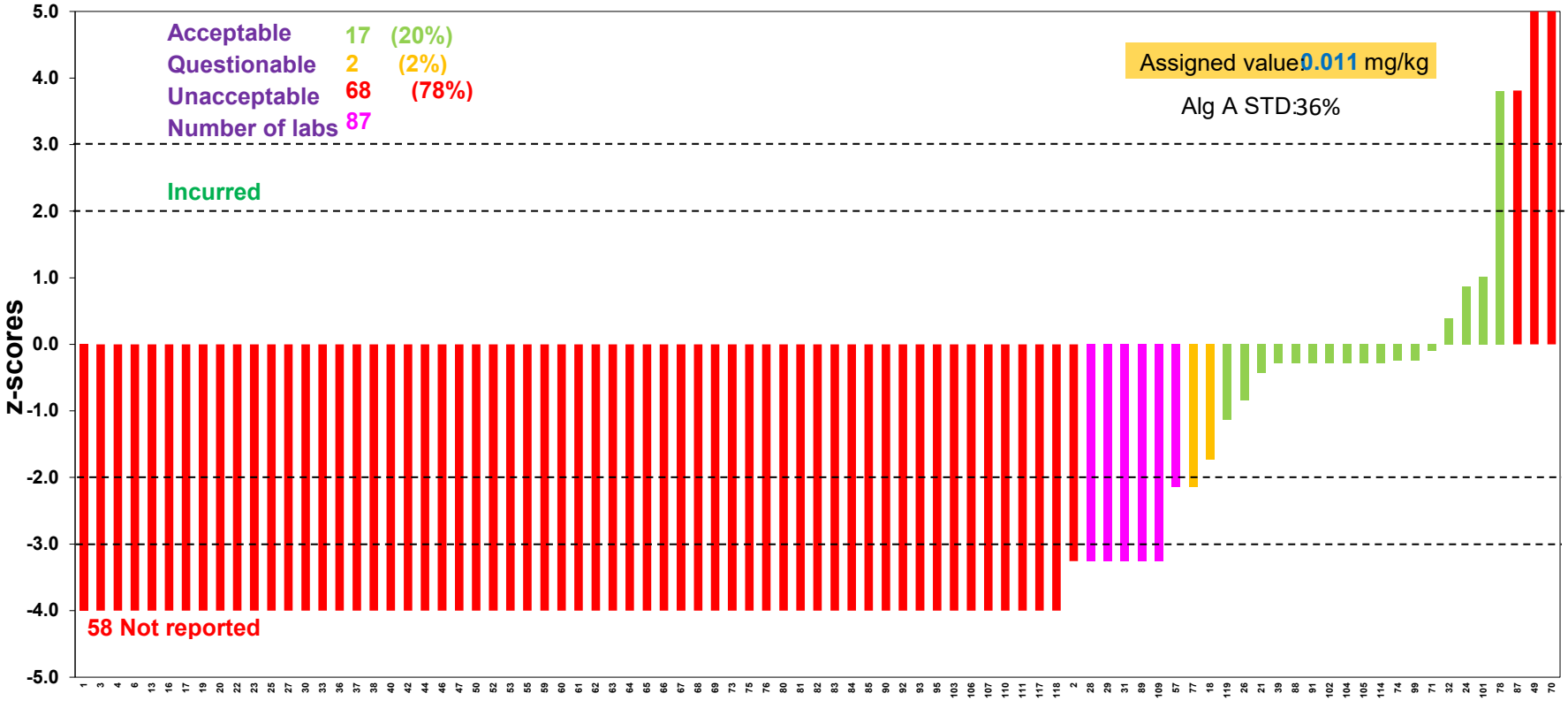
Benzovindiflupyr

EU and EFTA Laboratories



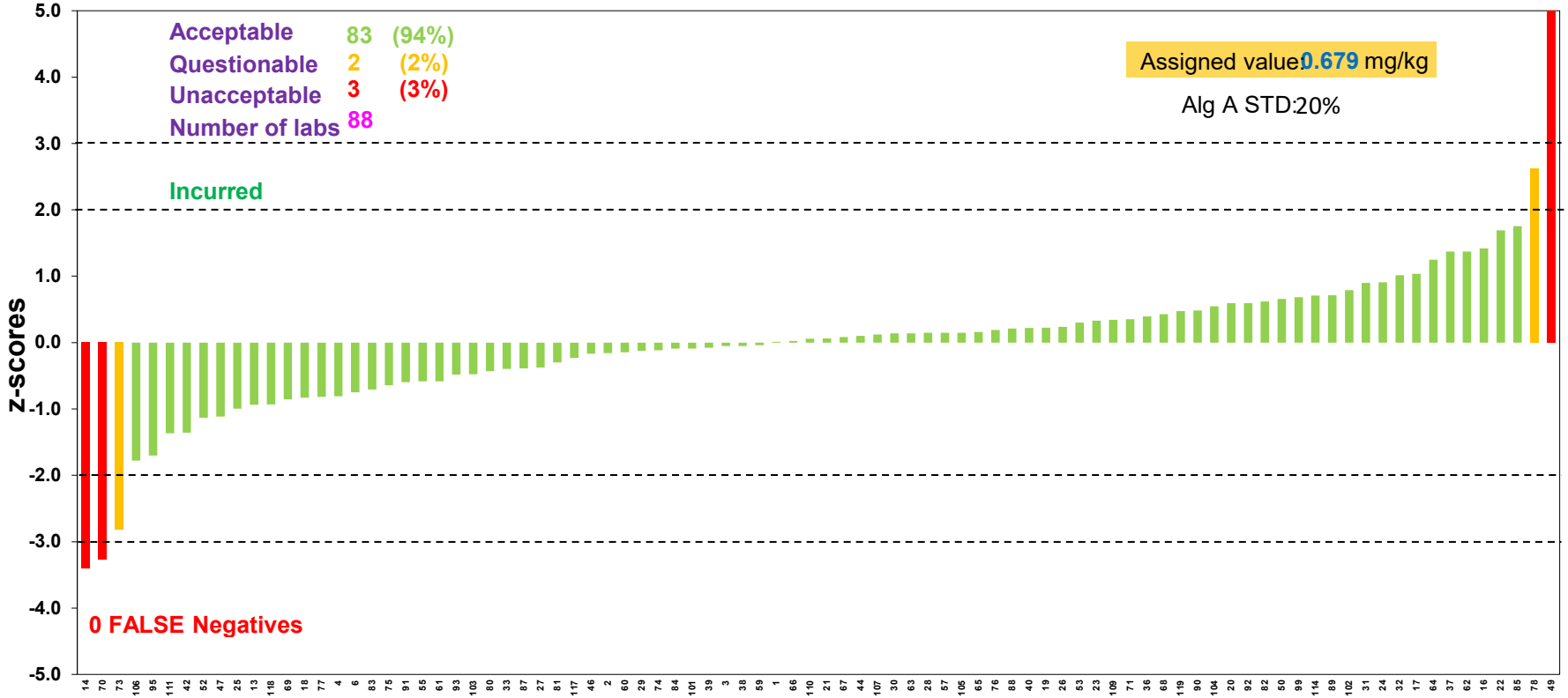
Boscalid

EU and EFTA Laboratories



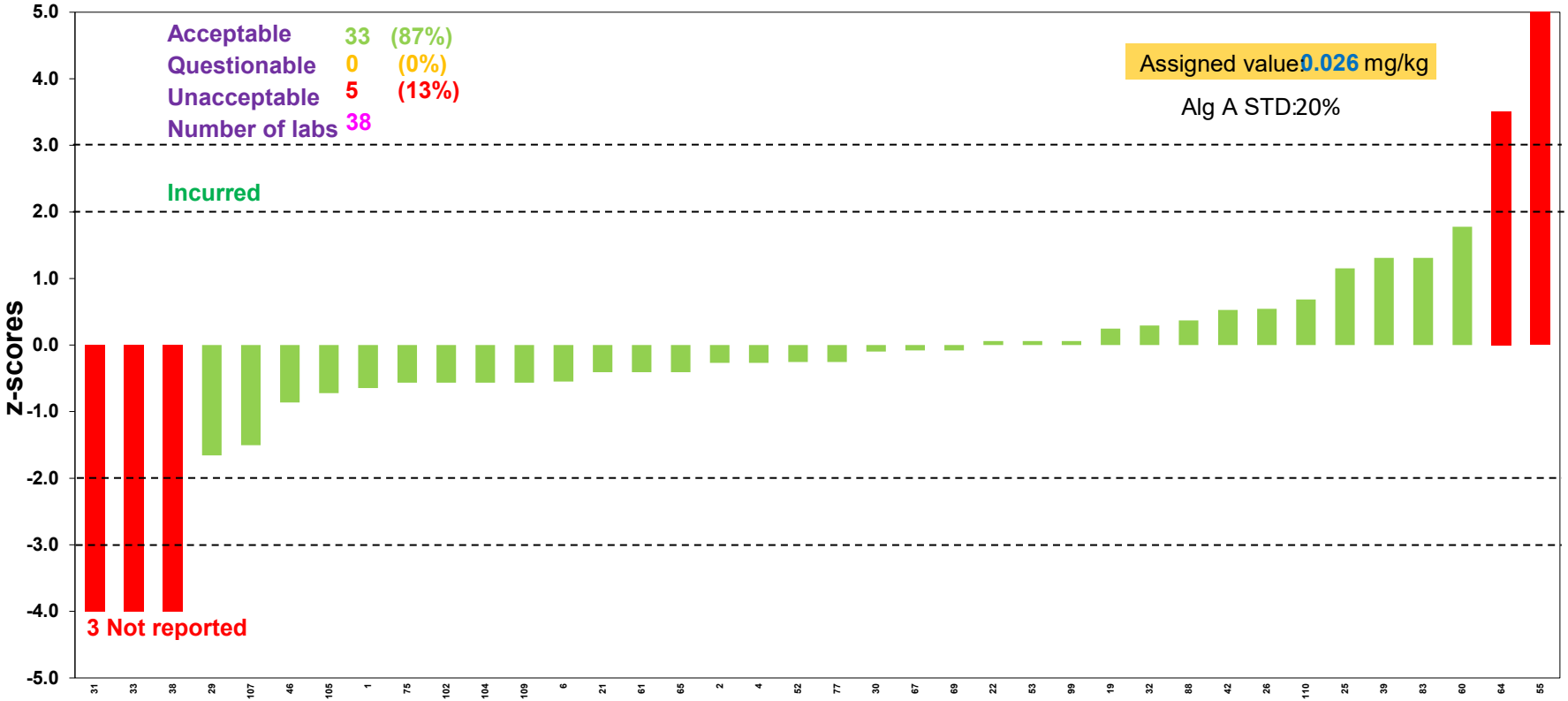
Epoxiconazole

EU and EFTA Laboratories



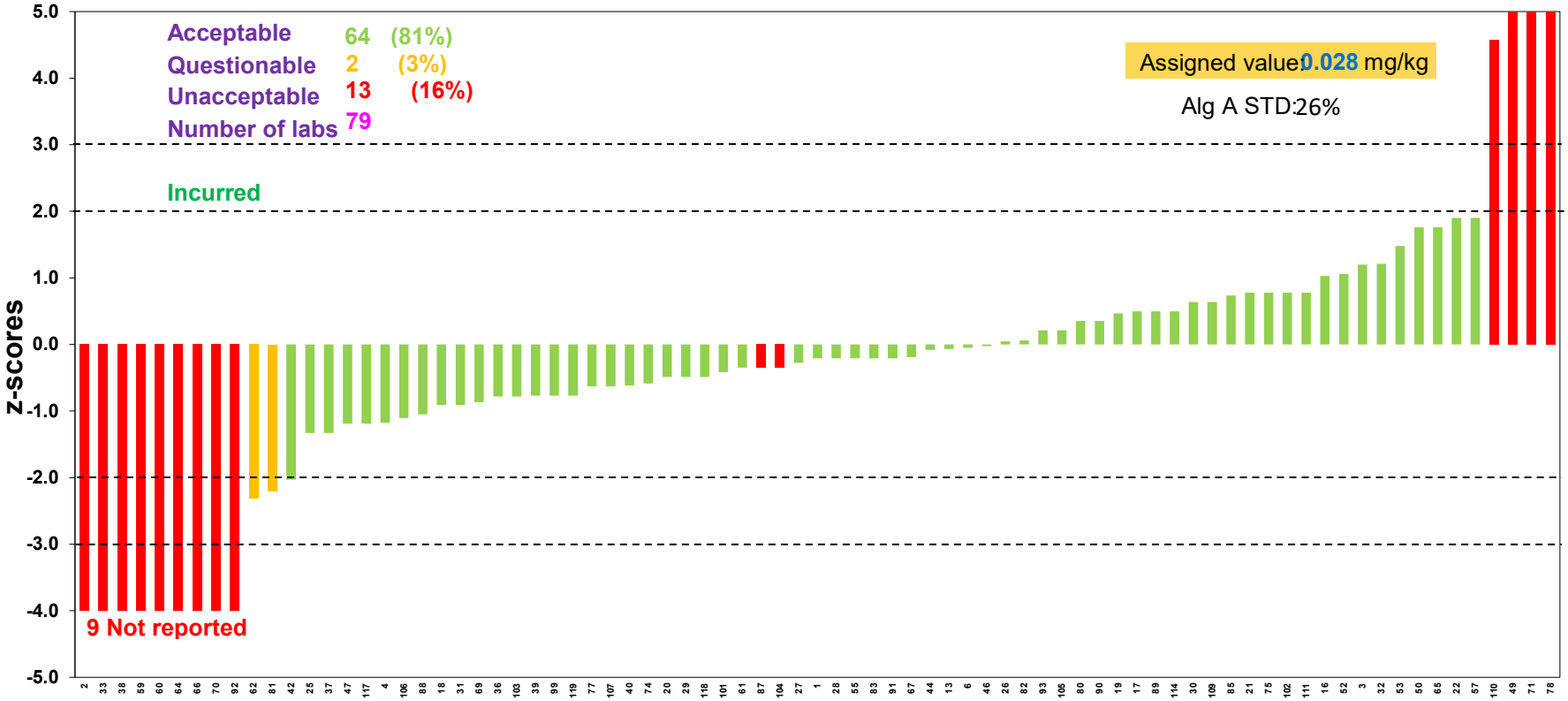
Fenpicoxamid

EU and EFTA Laboratories



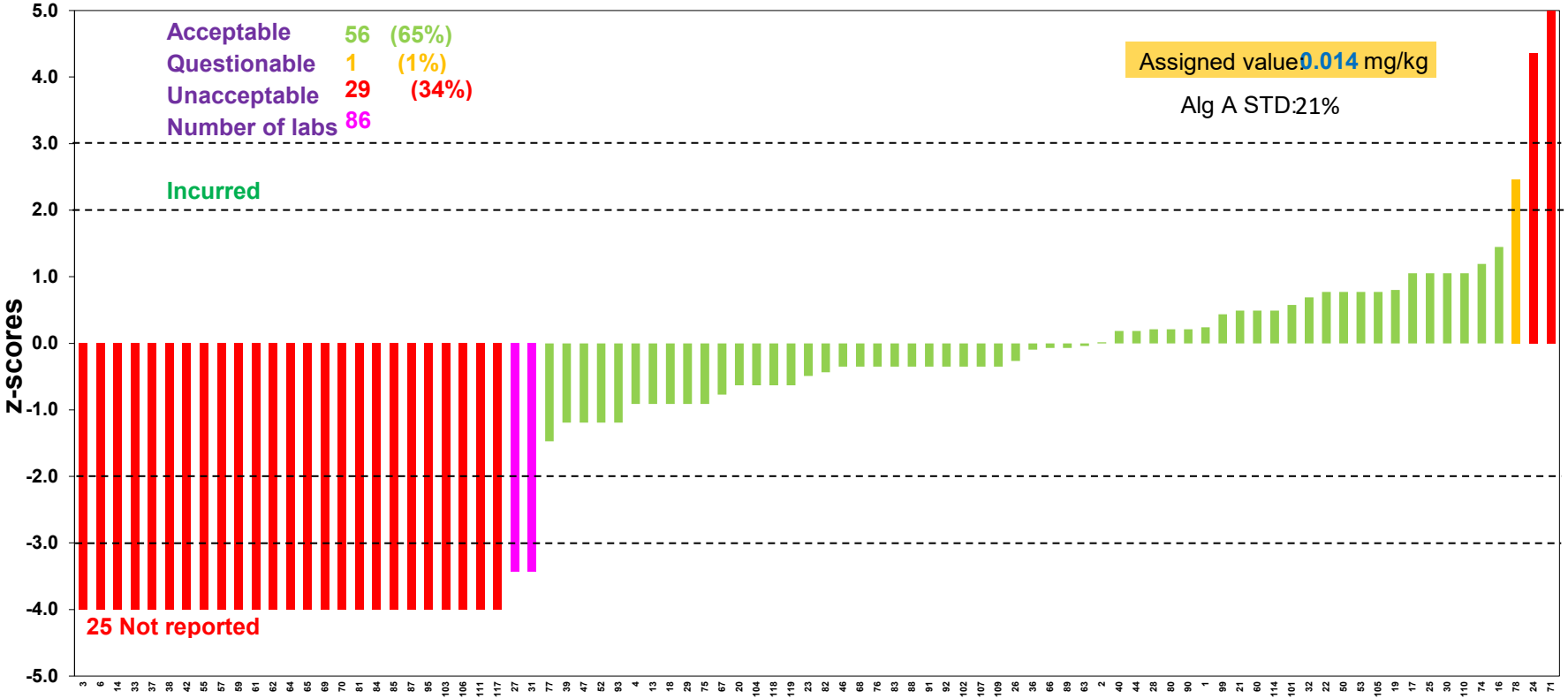
Flonicamid

EU and EFTA Laboratories



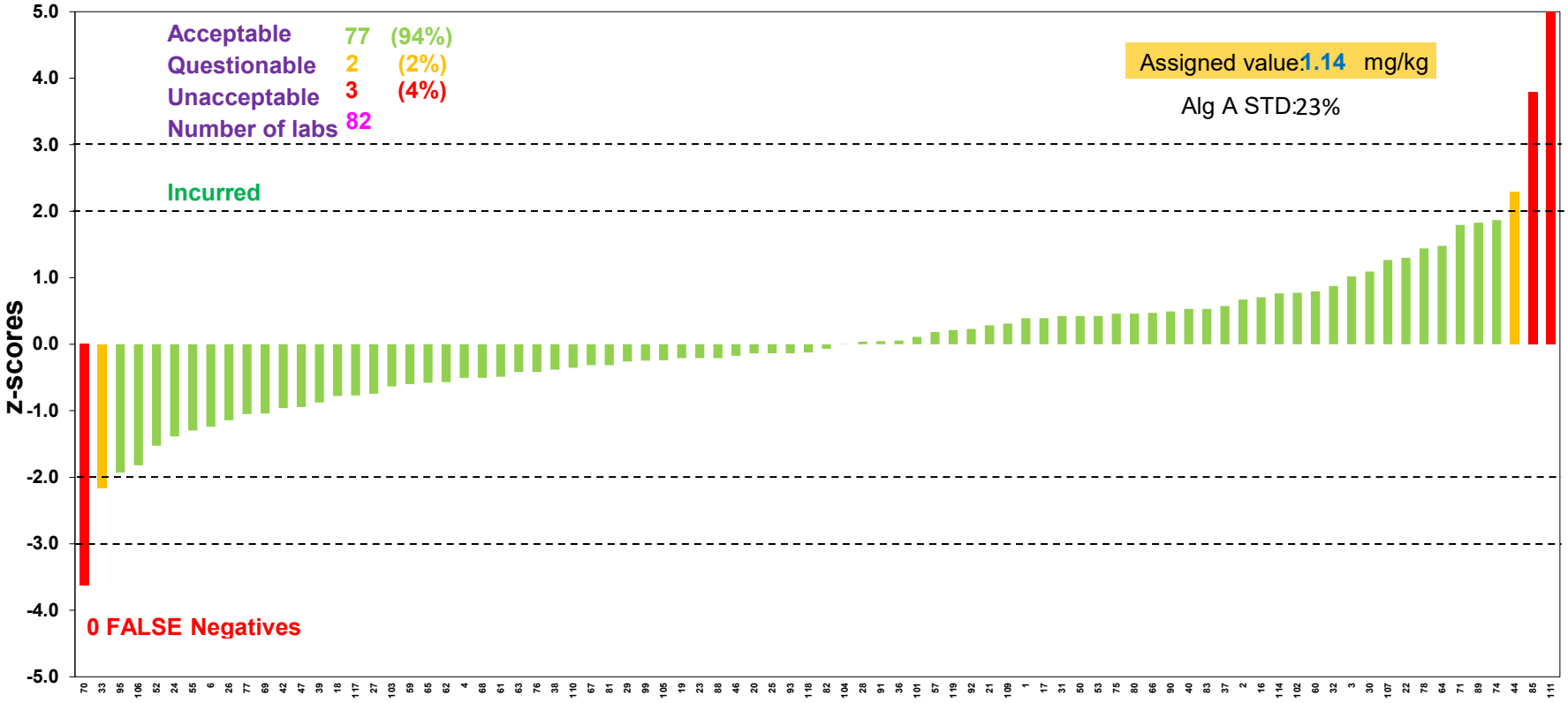
Fluopyram

EU and EFTA Laboratories



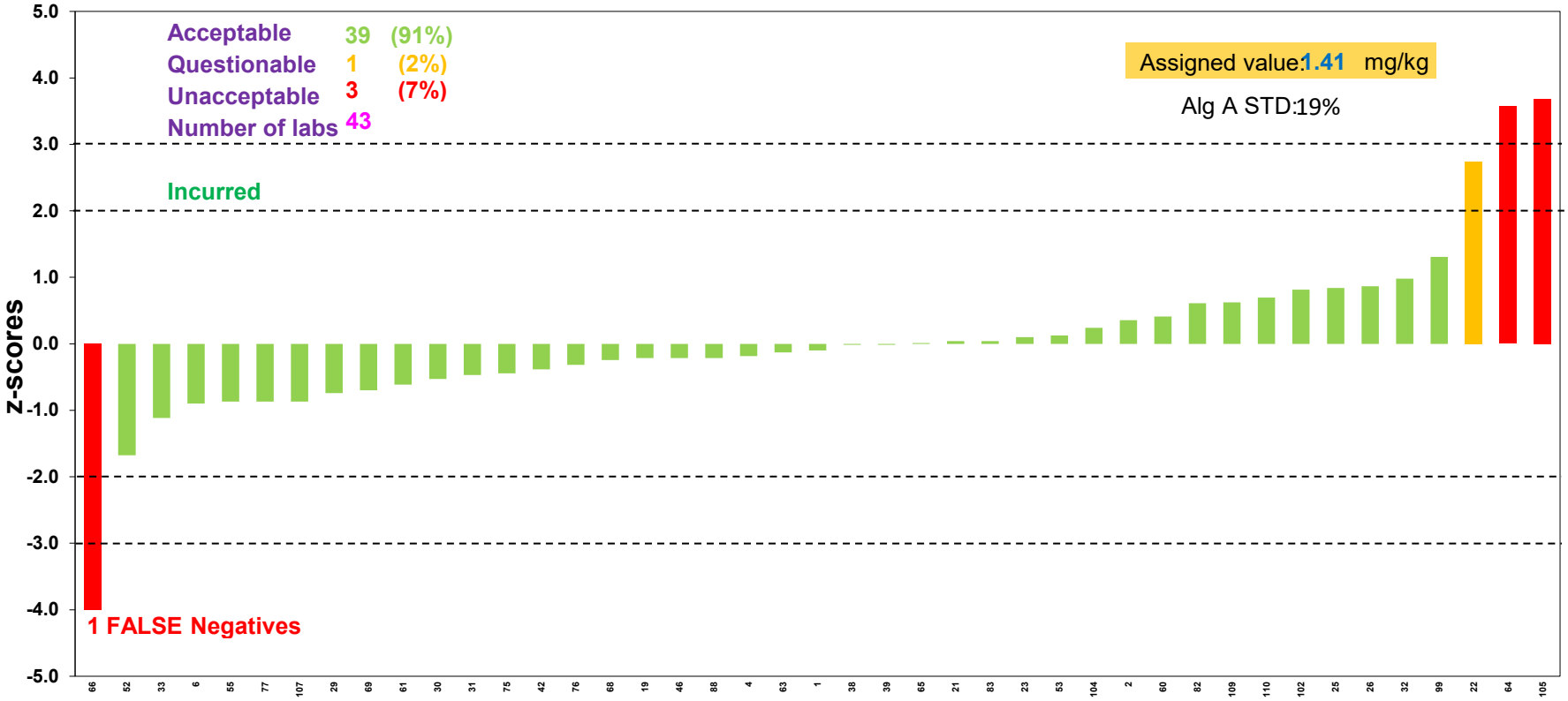
Fluxapyroxad

EU and EFTA Laboratories



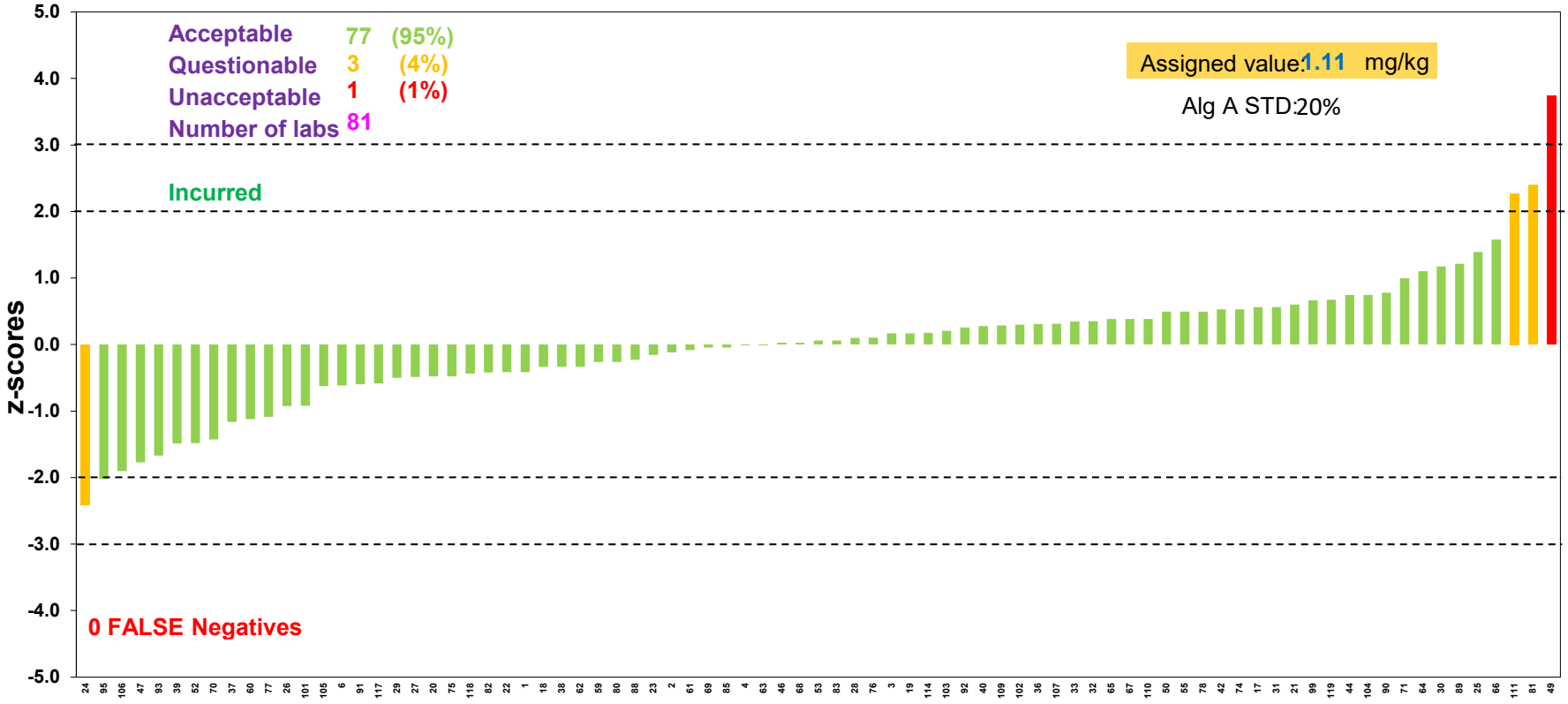
Mefentrifluconazole

EU and EFTA Laboratories

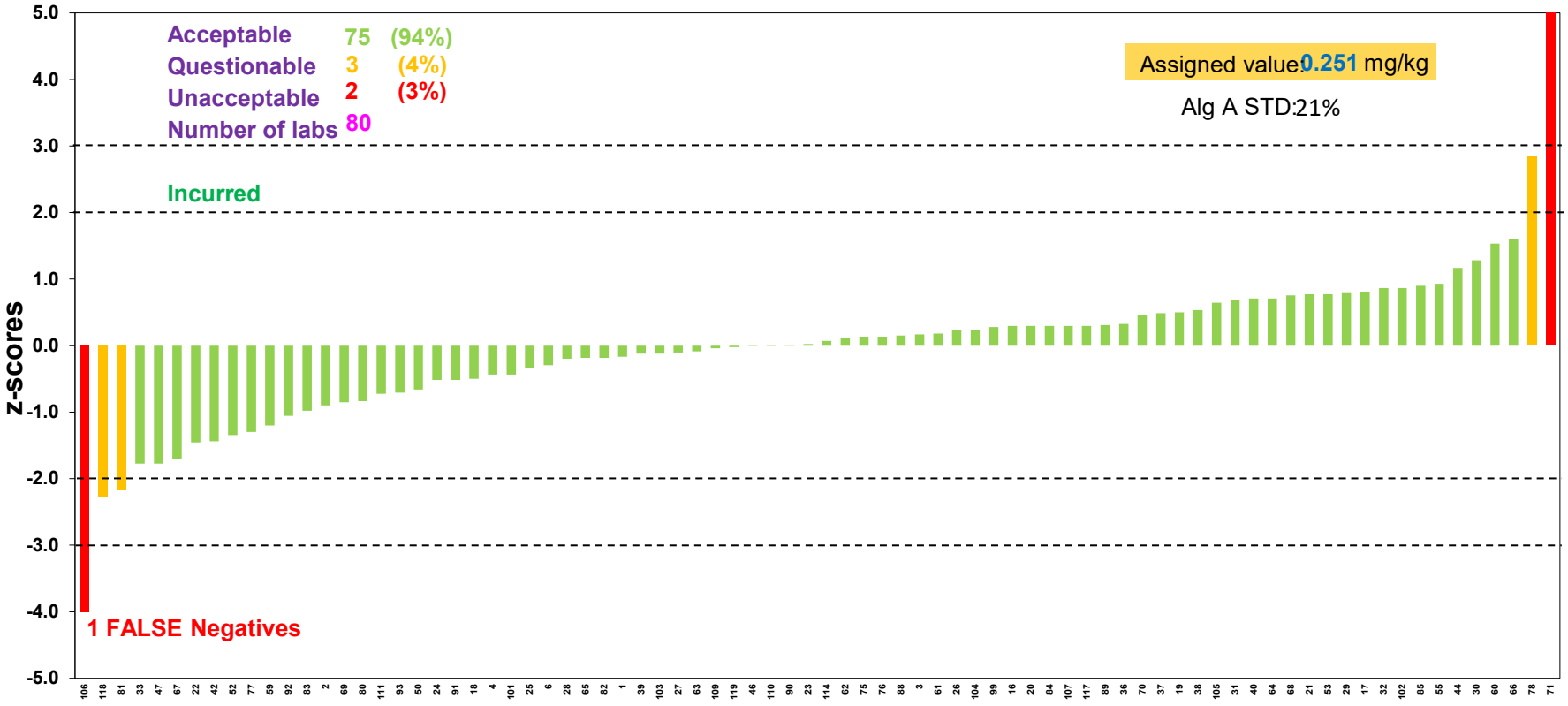


Proquinazid

EU and EFTA Laboratories

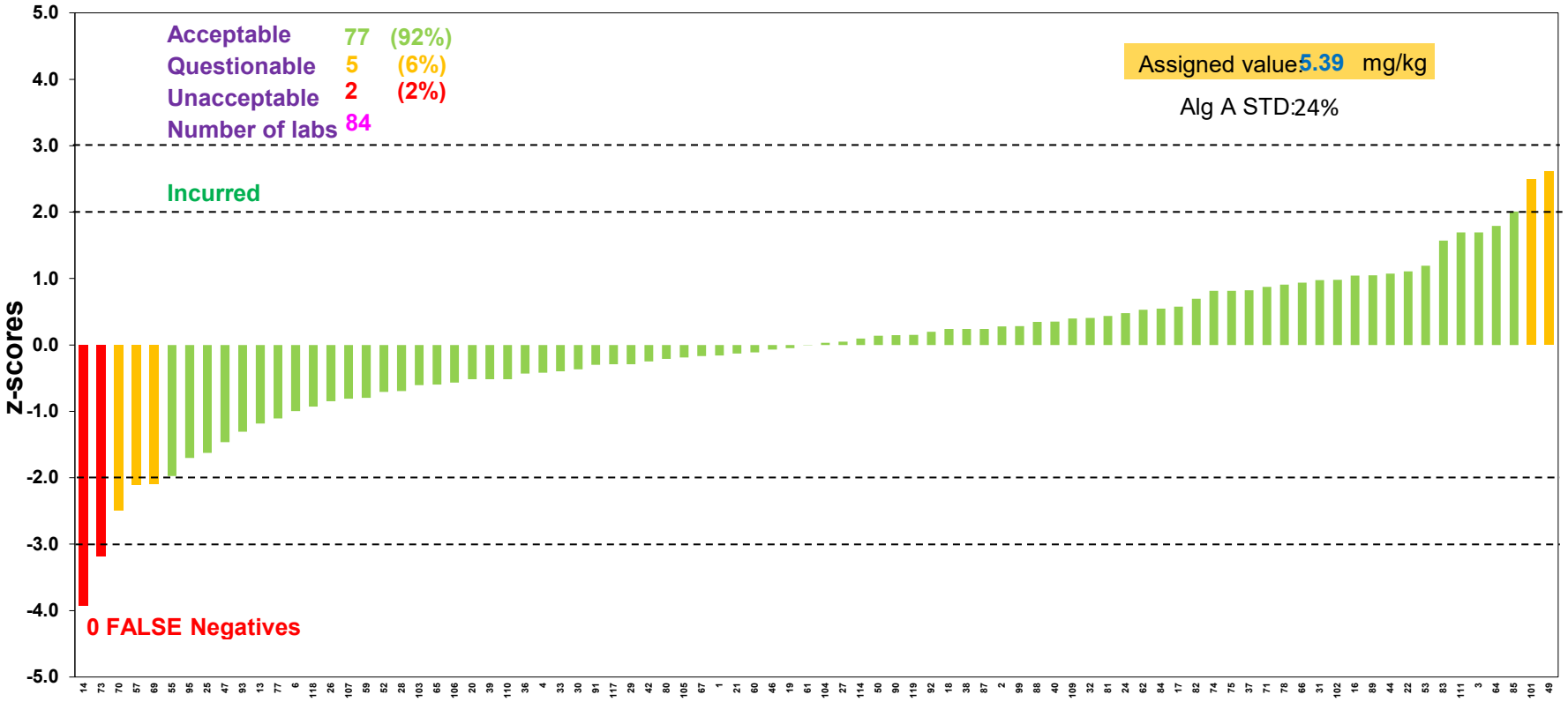


Prothioconazole-desthio EU and EFTA Laboratories



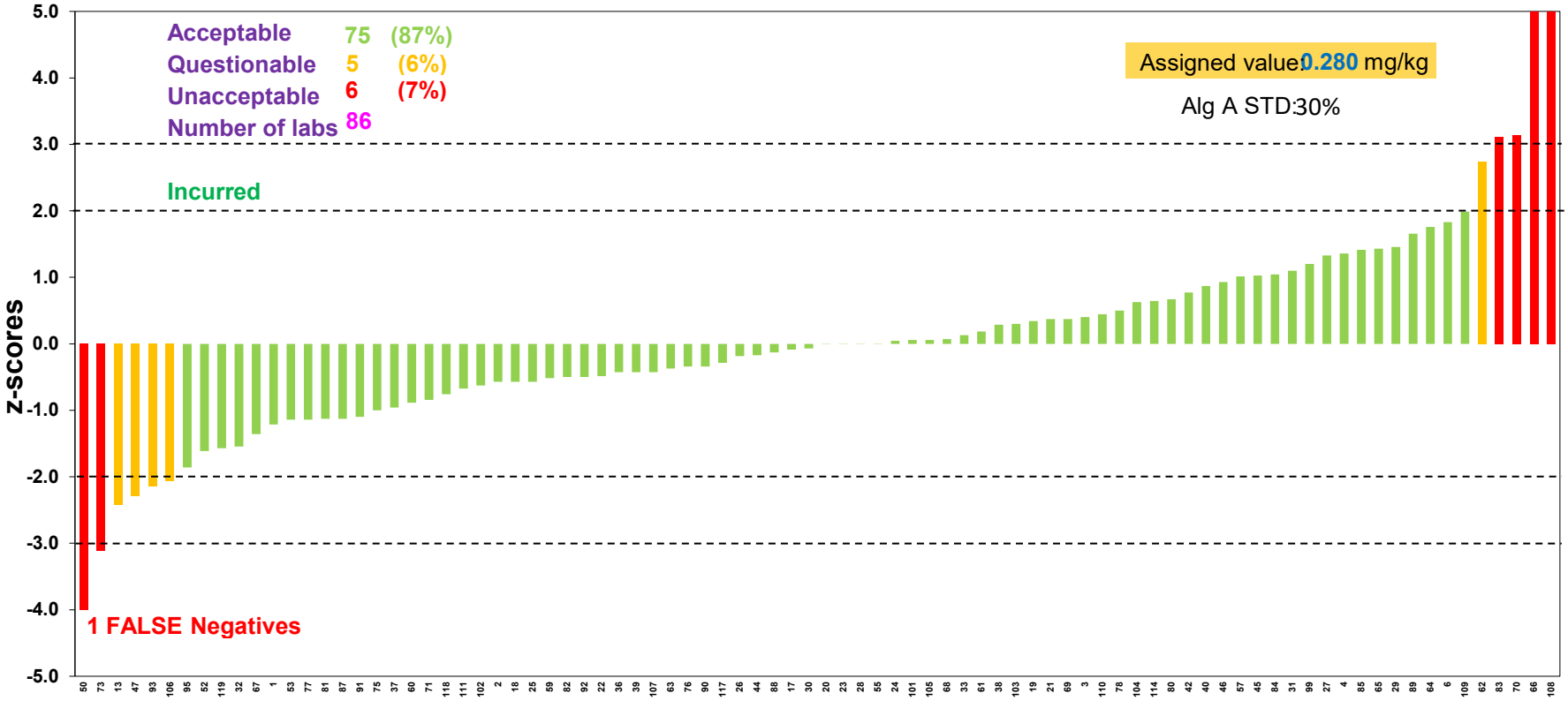
Pyraclostrobin

EU and EFTA Laboratories



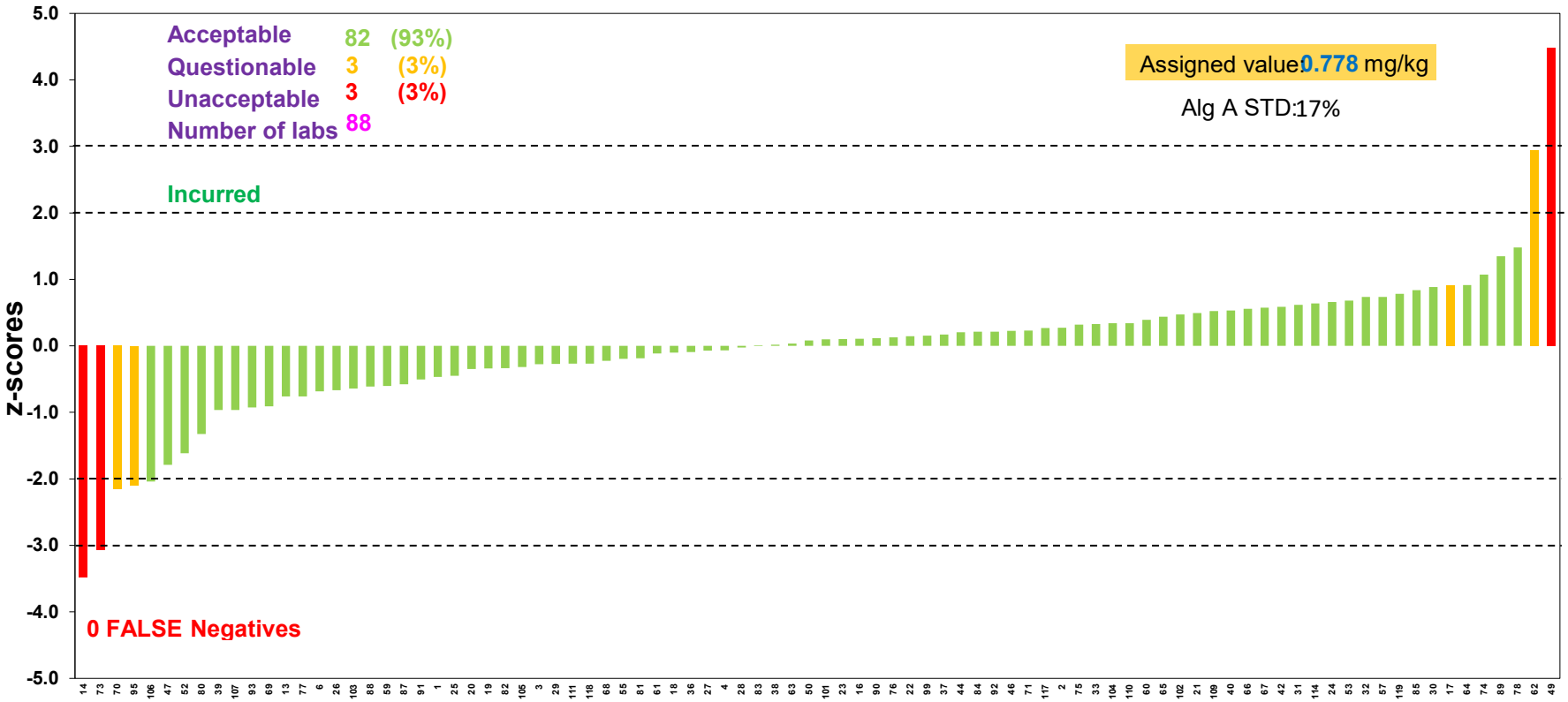
Tau-fluvalinate

EU and EFTA Laboratories



Trifloxystrobin

EU and EFTA Laboratories



11th Edition: Released on 30 December 2023

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 laboratories belonging to the Network[2] of National Reference Laboratories (NRLs) and Official Laboratories (OfLs) of the EU Member States. OfLs from EFTA countries and EU-Candidate countries are also welcome to participate in the EUPTs. OfLs from Third countries may be permitted to participate on a case-by-case basis.

The following four EURLs for pesticide residues were appointed by DG-SANTE based on the official controls Regulation (EU) No. 2017/625[3]:

- EURL for Fruits and Vegetables (EURL-FV),
- EURL for Cereals and Feedingstuff (EURL-CF),
- EURL for food of Animal Origin and commodities with high fat content (EURL-AO) and
- EURL for pesticides requiring Single Residue Methods (EURL-SRM).

The aim of these EUPTs is to obtain information regarding the quality, accuracy and comparability of pesticide residue data in food and feed reported to the European Union within the framework of the national control programmes and the EU multiannual co-ordinated control programme[4]. Participating laboratories will be provided with an assessment of their analytical performance that they can use to demonstrate their (ongoing) analytical proficiency and compare themselves with other participating laboratories. By pointing out areas of analytical deficiencies, EUPTs contribute to the continuous improvement of the analytical quality of OfLs, thus helping to increase the confidence on the results generated by them.

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

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

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

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

Table of Content

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**[5]) 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. These tasks include the PT-announcement, the production of the proficiency testing item (PT-item), the undertaking of homogeneity and stability assessments, the portioning, 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)[6] has been established and approved by DG-SANTE. The EUPT-SC consists of expert scientists with many years of experience in PTs and/or pesticide residue analysis. The 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 is made up of the following two subgroups:

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

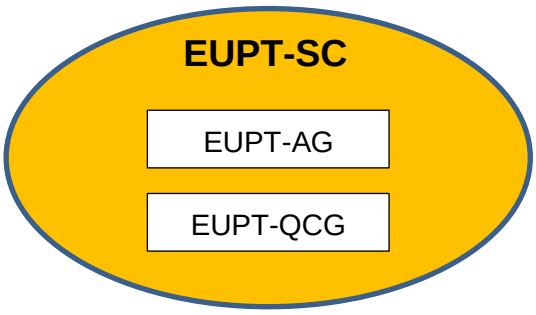

Figure 1: *Composition of EUPT-Scientific Committee*

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 requested, when it comes to e.g. selecting the commodities for the EUPTs of the following season, selecting the analytes to be included in the Target Pesticides List (p. 8), establishing the Minimum Required Reporting Levels (MRRLs) for each of the analytes,

[5] The term organisers is to be considered equivalent to the term PT-provider in ISO 17043:2023

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

and statistically evaluating the participants' results (in anonymous form). The EUPT-SC is furthermore consulted when it comes to drafting and updating 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.

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 and, if needed, possible changes in the EUPT-General Protocol are also discussed during these meetings. The main topics and decisions on these meetings are documented.

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

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[3]
- Art. 28 (3) of Reg. (EC) No. 2005/396 (for all OfLs analysing for pesticide residues within the framework of official controls of food or feed[7])
- Art. 101 (1)(a) of Regulation (EU) No. 2017/625[3] (for all NRLs).

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

[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).

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 EU-candidate countries and EFTA countries;

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

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

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

Note on a): Laboratories having been designated as OfLs, according to Art. 37(2)(b) of Regulation (EU) No. 2017/625[3] by a Competent Authority of an EU Member State (MS1) will normally also need to be commissioned with OfL activities in a different EU Member State (MS2) for being eligible for participation. Scan-copies of documents giving information about the period and scope of these OfL activities for MS2 may be requested by the EUPT organizers. The responsible NRL and/or Competent Authority of MS2 may be contacted before deciding whether the laboratory in question is eligible or even obliged to participate in a certain PT. A laboratory whose OfL-appointment in the area of pesticide residue analysis has ceased, will normally loose its eligibility (and obligation) to participate in EUPTs, but participation may be allowed if the responsible NRL and/or Competent Authority of MS1 or MS2 considers its participation essential for judging the proficiency in view of a planned or potential OfL activity in the future.

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

Obligation of OfLs and NRLs to double-check Status of EUPT-Participation:

Based on the latest information within the DataPool and considering the selected commodities of the upcoming EUPTs, the OfLs (including the NRLs) are grouped into those for which participation in a given EUPT is **obligatory** and those for which participation is **voluntary** ("OV-grouping").

Upon accessing the EUPT Registration Form within the EURL-DataPool, laboratories can choose the EUPTs they would like to participate and view their OV-grouping status for each of the selected PTs. If a laboratory does not agree with its OV-Grouping, it should promptly contact the corresponding NRL and the EUPT-organisers and give the reasons why it believes it should be grouped differently. The reasons provided by the laboratories will be noted by the organisers and if indicated, the DataPool will be updated accordingly. In any case, the OV-grouping prepared by the EURLs is indicative only, as the real obligation to participate in a given EUPT arises from the above-mentioned EU-regulations, not the DataPool entries or any lab's claims. Additional requirements may arise from accreditation bodies or local rules and regulations.

Within the DataPool, NRLs have the possibility to view data relevant to OfLs within their network (OV- grouping, registration progress) and are responsible for checking whether the OV- grouping of all relevant OfLs within their network is correct.

OfLs that are obliged but not able to participate in a given EUPT must provide the reasons for their non-participation. This also applies to any participating laboratories that fail to submit PT-results.

Participation fee and Invoicing

By completing the registration for participation in a given EUPT, a laboratory agrees to proceed with a timely payment of the participation fee after being accepted for participation and after the invoice issued by the organiser is received. The invoice fee covers the costs of production, handling and delivery of the PT-materials. The organisers will issue digital invoices in PDF format only, and without any electronic signature. By registering to an EUPT the laboratories also accept that the pdf invoice, issued by the organisers and sent via e-mail to the participant, is sufficient for triggering the payment of the participation fee. The EURLs retain the right to decline any request for supplementary forms or additional paperwork in connection to the payment. The laboratories should note that additional costs may incur if such extra services are requested, depending on the incurring extra workload. Extra costs may also incur if 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 laboratory may be blacked out in the final report of the concerned EUPT and 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 proprietor of all EUPT data is DG-SANTE and as such has access to all information.

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

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

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

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

The official language used in all EUPTs is English.

Announcement / Invitation Letter

Approximately 3 months before the distribution of the 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 tentative EUPT-Target Pesticides List and the tentative 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 based upon 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

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

Assessing the Homogeneity of the PT Item

A suitable homogeneity of the EUPT item is of high importance as it ensures that portion-to-portion variability has only a negligible impact on the evaluation of the participant's performance. The PT item is tested for homogeneity, typically after bottling and before distribution to participants, but in justifiable cases the tests for homogeneity assessment may also be conducted after the distribution of the material to the participants[8]. The homogeneity assessment usually involves analysis of two replicate analytical portions, taken from at least ten randomly chosen units (bottled portions) of treated PT item. Measurements should be conducted in random order with the aim of minimizing the risk of misinterpreting signal drifts within a measurement sequence as concentration shifts linked to the bottle numbering, i.e. the order of the bottle filling.

The homogeneity test data are statistically evaluated according to ISO 13528:2022, Annex B[9] or to the International Harmonized Protocols jointly published by ISO, AOAC and IUPAC[10]. The results of all homogeneity assessment are presented to the EUPT-SC. In special cases, where the above

[8] To minimize the risk of PT item not being acceptably homogeneous, the organisers may opt to conduct a small-scale preliminary homogeneity test prior to bottling the PT item for shipment. The pre-tests may focus on a selected fraction of the analytes, and may also serve for verifying the presence and the approximate levels of the analytes spiked.

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

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

criteria are not met, the EUPT-SC, considering all relevant aspects (e.g. the homogeneity results of other analytes spiked at the same time, the overall distribution of the participants' results (CV*), the analytical difficulties faced during the tests, and knowledge of the analytical behaviour of the compound in question), may decide to overrule the test. The reasons of this overruling have to be transparently explained in the Final EUPT-Report. For certain analytes with comparable properties, an equivalent distribution within the sample can be expected if they were spiked/used simultaneously. The homogeneity test of one or more of these analytes may thus be skipped or simplified. The organisers should keep an eye on the participants' results of such analytes not tested for homogeneity in order to detect at an early stage any signs that could raise doubts about the homogeneity of the material (e.g. an atypically broad distribution of the results compared to other analytes). In such a case, the EUPT-SC may decide that a proper homogeneity assessment should still be performed to clarify the situation.

Assessing the Stability of the Analytes Contained in the PT Item

The PT item will also be tested for stability - according to ISO 13528:2022, Annex B[9]. The time delay between the first and the last stability test (stability assessment period) must exceed the period of the EUPT-exercise. Typically, the first analysis is carried out shortly before the shipment of the PT items and the last one shortly after the deadline for submission of results. If justifiable, the stability assessment period may precede the PT period, partly overlap with it or postdate it. Close proximity to the PT-period is to be favoured, however, to minimize the risk that matrix properties alter in a way that will affect analyte stability. To better recognise trends and gain additional certainty, one or more additional tests may be conducted by the organisers in the interim. At least 6 sub-samples (analytical portions) should be analysed on each test day (e.g. 2 analytical portions withdrawn from three randomly chosen containers OR 6 portions withdrawn from a single container). In principle, all analytes contained in the PT item should be checked for stability. However, in individual cases, where sufficient knowledge exists that the stability of a certain analyte is very unlikely to be significantly affected during storage (e.g. based on experience from past stability tests or knowledge of its physicochemical properties), the organisers, after consultation with the EUPT-QCG, may decide to omit a specific stability test. The EUPT-SC will finally decide whether analytes for which the stability test was not undertaken will be included in the Final EUPT-Report, considering all relevant aspects, such as the distribution of the participant's results (CV*).

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

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

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

If insight about insufficient analyte stability is gained before the end of the PT-period, the EUPT-QCG will be contacted in order to decide whether the EUPT-SC should be involved in the discussion (as confidential information is involved), whether the PT-participants should be informed about this insight and whether the affected analytes should be removed from the target list.

Stability during shipment: Considering knowledge about the expected susceptibility of analytes in the PT item to possible losses, the organisers will choose suitable shipping conditions to minimize such losses, e.g. shipment of frozen samples, addition of dry ice. As shipment duration can vary from labs/countries to labs/countries, it is recommended that the organisers keep track of the shipment duration and then decide whether it is reasonable to conduct additional stability tests at conditions simulating shipment. Should critical losses be detected for certain analytes, the EUPT-SC will be informed (or the EUPT-QCG before or during the test). Case-by-case decisions may be made by the EUPT-SC, considering all relevant aspects including the duration and conditions of the shipment to the laboratory as well as the feedback by the laboratory. Follow-up measures in case of instability during shipment may include the exclusion of the affected results from the population used for establishing the assigned value (x_{pt}) and the non-calculation of z scores for the affected analytes in order to avoid unfair penalization of the laboratories involved.

If the PT entails analytes that are expected to have a high risk of degradation within the PT item, the organisers should conduct model tests prior to the final preparation of the test item in order to gain insight about the stability behavior of the analytes intended to be spiked during homogenization, transport and storage of the samples. Based on the results of these experiments measures should be taken to minimize the risk of certain analytes failing to meet the stability criteria, which may include adjusting the conditions of homogenization and/or storage and/or shipment or even deciding not to spike the material with certain analytes.

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 organiser within the stipulated deadline. Any analyte targeted by a participating laboratory should be reported as "analysed" in the Webtool. In EUPTs by EURLs responsible for MRM compounds (FV, CF, AO) this is done before shipment of the PT test Item. In EUPT-SRMs this is done in the period during which the platform is open for result submission. 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.

For reporting, concentration values ≤ 0.01 mg/kg are recommended being rounded to two significant figures (e.g. 0.0078; 0.010) and values > 0.01 mg/kg to three significant figures (e.g. 0.123; 1.23; 12.3 mg/kg). No penalties will apply where a laboratory reports deviating numbers of significant figures, but in case of less significant figures, zeros will be assumed after the last significant figure (e.g. 0.1 = 0.100 and 0.11 = 0.110). For the calculation of z scores the values will be used as reported. In the preliminary and final report the results will be shown with up to three significant figures.

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) but it will be allocated a z score as any other numerical result. Such results will be, furthermore, included in the results population for establishing the assigned value (x_{pt}), unless they are eliminated for other reasons (e.g. laboratory status, use of biased methodology).

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.

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

When the laboratory uses any of the following approaches inherently accounting for method bias, this needs to be indicated in the appropriate fields within the 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 requested to provide information on the analytical method(s) they have used. The Webtool, which serves for submitting analytical results, is typically also used for collecting method information.

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

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

Where certain methodologies or analytical steps are suspected to lead to biased or otherwise erroneous results, the PT-organisers will substantiate this suspicion by own experiments and discuss the issue with the EUPT-SC. Laboratories affected will be informed, e.g. via direct contact and/or via EURL-workshops or trainings and/or through the inclusion of recommendations within the Final EUPT Report.

Cases where reporting limits (RL) of laboratories exceed the MRRL indicate insufficient sensitivity and may be highlighted in the final report as with “PS” for poor sensitivity.

Results Evaluation

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

- ***False Positive (FP) Results***

These are results of analytes on the Target Pesticides List that are reported at or above their respective MRRL although they were: (i) “not detected”[11] by the organiser, even after repeated analyses, and/or (ii) “not detected” by the overwhelming majority (e.g. > 95 %) of the participating laboratories that had targeted the specific analytes. In certain instances, case-by-case decisions by the EUPT-SC may be necessary.

Any results reported lower than the MRRL will not be considered as false positives, even though these results should not have been reported. 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 of analytes reported by the laboratories as ’analysed’ but without reporting numerical values although they were: a) used by the organiser to treat the PT item and b) detected by the organiser as well as the majority of the participants that had targeted these specific analytes at or above the respective MRRLs. Numerical results < RL (RL= Reporting Limit of the laboratory) may be judged as false negatives and may be also regarded as “not correctly found” when it comes to categorization in A and B based on scope. Such results wouldn’t be reported in a routine laboratory environment. Case-by-case decisions by the EUPT-Panel will be taken by the EUPT-SC in such cases.

Where the RL of a laboratory for a certain analyte present in the PT item exceeds the assigned value, with the laboratory not reporting a numerical value, the result may still be judged as a false negative, despite this reporting being unobjectionable in 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.

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

In cases of the robust mean of the participant results being less than 3 times higher than the MRRL, false negatives will typically not be assigned. The EUPT-SC may decide to make case-by-case decisions in this respect after considering all relevant factors such as the result distribution and the RLs of the affected labs. In case where the not fixing a valid assigned value is due to other reasons, e.g. because the uncertainty of the assigned value (UAV) criteria were not met and/or because of a bimodal distribution of the participant results, the EUPT-SC will decide on case-by-case basis whether FNs should be assigned for the respective analyte or not.

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

To minimise the influence of out-lying results on the statistical evaluation, the assigned value x_{pt} (= consensus concentration) will typically be estimated using the robust mean estimate of the participant results (x^*) as described in ISO 13528:2022[12], taking into account the results reported by EU and EFTA countries laboratories only. In special justifiable cases, the EUPT-Panel may decide to include results submitted by laboratories not belonging to the EU-/EFTA-OfLs network or even to use only the results of a subgroup of ('expert') laboratories that have previously repeatedly demonstrated good performance for the specific or similar compounds.

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

In special justifiable cases, the EUPT-Panel may furthermore decide to use the spiked concentration of an analyte as the best estimate of the assigned value. In such cases, a detailed explanation of the reasons behind this decision will be given and a comparison with calculations involving robust statistics will be undertaken.

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.

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

[12] 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).

– ***Omission or Exclusion of Results***

Results reported by laboratories from non-EU and non-EFTA Member States are typically excluded from the population used to derive the assigned value (for exceptions see 'Estimation of the assigned value').

Before estimating the assigned value, results associated with obvious mistakes have to be examined to decide whether they should be removed from the population. Such gross errors may include incorrect recording (e.g. due to transcription errors by the participant, decimal point faults or transposed digits, incorrect unit), calculation errors (e.g. missing factors), analysis of a wrong sample/extract (e.g. a spiked blank), use of wrong concentrations of standard solutions, incorrect data processing (e.g. integration of wrong peak), inappropriate storage or transport conditions (in case of susceptible compounds), and the use of inappropriate analytical steps or procedures that demonstrably lead to significantly biased results (e.g. employing inappropriate internal standards or analytical steps or conditions leading to considerable losses, due to degradations, adsorptions, incomplete extractions, partitioning etc.). Where the organisers (e.g. after the publication of the preliminary report) receive information that certain participant's results are associated with gross errors, the affected results will be examined on a case-by-case basis to decide whether, or not, they should be excluded from the population used for robust statistics. Results may also be omitted e.g. if an inappropriate method has been used even if they are not outliers.

In case of traceable calculation errors by the participants (e.g. use of wrong factors to express the result as required by the PT's residue definition[13]), and in case of non-reporting results that can be calculated from reported values (e.g. summed result not calculated and not reported), the EUPT-Panel may decide to correct or complement results within the population by applying (the correct) factors. The new population of results may then be used for establishing the assigned values. The z score of the concerned results will, however, be calculated using the originally reported values.

Although robust statistics are applied for estimating assigned values and robust standard deviations, certain results showing a strong bias compared to the rest of the population may be, in certain cases, eliminated before applying robust statistics[14]. To identify such strongly biased results, a preliminary consensus calculation of the robust mean (prelim-x*) may be conducted and any results being ≥ 3-fold the prelim-x*[15] may be potentially eliminated. This approach may need to be iterated if the population still entails obvious outliers.

[13] irrespective of who is accounted responsible for the confusion

[14] Please see ISO 13528:2022 Chapter 6.6." Outlier techniques for individual results', therein 6.6.3, Note 3.

[15] Corresponds to preliminary z scores ≥ 8 using the FFP-approach

The result population remaining after the elimination of certain results as described above may be then used to establish the actual assigned value (x_{pt}) and the robust standard deviation (s*) according to the consensus approach described above. The z scores of all results, including those corrected or removed, are to be recalculated using the new assigned value.

All decisions to omit/exclude results will be discussed with the EUPT-SC and the reasoning for the omission of each result clearly stated in the Final EUPT-Report. However, z scores will be calculated for all results irrespective of the fact that they were omitted from the calculation of the assigned value.

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

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

The uncertainty of the robust mean values (x_{pt}) is calculated according to ISO 13528:2022 as:

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

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

A broad results distribution (high s*) and/or a limited number of results (p) will increase the uncertainty of the robust mean $u(x_{pt})$ values exceeding 0.3×σ_{pt} (see ISO 13528:2022) will typically mean that the robust mean is too uncertain for the purpose and cannot be straightforwardly taken up as the assigned value. In each of these cases, investigations for elucidating the reasons behind the high uncertainty should be undertaken. Taking into account all relevant aspects[16] the EUPT-SC may decide that the analyte results should be re-evaluated based on a refined or extended result population or an alternative approach. If, despite these considerations and irrespective of the outcome of the UAV test the EUPT-SC concludes that, the assigned value of a specific analyte is too uncertain for a valid evaluation, it may decide that the results for the analyte in question should not be evaluated or only evaluated for informative purposes.

- ***Considering the UAV when Calculating z Scores***

Where the vast majority of the results is close to the robust mean and narrowly distributed but the UAV-test is still marginally failing[17] (e.g. where $u(x_{pt})$ is up to 0.4×σ_{pt} = 10% in absolute terms), the

[16] e.g. information about methodologies used by the participants (especially if these are likely to produce biased results), multimodality, number of submitted results, homogeneity data, stability data

[17] e.g. due to a combination of few results, and sporadic biased results.

EUPT-Panel may consider to calculate z' scores using the following formula, which considers the uncertainty of the assigned value:

$$z' = \frac{x_i - x_{pt}}{\sqrt{{\sigma_{pt}}^2 + u^2\,(x_{pt})}}$$

where $u(x_{pt})$ being the uncertainty of the assigned value and σ_{pt} being the standard deviation of the assigned value that may be set equal to $FFP\text{-}\sigma_{pt}$ (see below). z' scores will be shown for Informative purposes only.

In special cases[18], the EUPT-SC may consider useful to proceed with the calculation of z scores for both extremes of the assigned value as derived by applying the UAV (i.e. $x_{pt} \pm (x_{pt})$). This upper and lower bound calculation of the z scores will also be for informative purposes only. The aim of this calculation is to help laboratories having performed well in a PT demonstrate their good performance even in cases where the UAV-test has not passed the criteria. Example: x_{pt} = 1.0 mg/kg, (x_{pt}) = 0.1. Taking into account the calculated uncertainty, the AV should range between 0.9 and 1.1 mg/kg. If the result of a laboratory is 0.7 mg/kg, the z score calculates to -1.2 using x_{pt} = 1.0 mg/kg, For the upper limit of x_{pt} = 1.1 the z score calculates to -1.76 and for the lower limit of x_{pt} =0.9 the z score calculates to -0.72. This means that, even at worst-case scenario, the laboratory's result remains within the acceptable range.

- ***Standard Deviation for Proficiency Assessment (Target Standard Deviation)***

The standard deviation for proficiency assessment (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[19], a percentage of 25 % is currently used as FFP-RSD for all analyte-matrix combination, and the Fit-For-Purpose target standard deviation ($FFP\text{-}\sigma_{pt}$) is calculated as follows:

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

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

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").

[18] E.g. where the population of results is narrow, but the UAV tests fails due to a few deviating results in combination with a relatively small number of results, e.g. <20

[19] 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

- ***z Scores***

This parameter is calculated using the following formula:

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

where x_i is the value reported by the laboratory, x_{pt} is the assigned value, and $FFP\text{-}\sigma_{pt}$ 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.

For practical reasons, any z scores > 5 will be typically reported as '> 5' and a value of '5' will be used to calculate combined z scores (p. 19). Following ISO 17043:2023[20], z scores will be classified as follows:

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

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

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

For each EUPT the participating labs are asked to voluntarily report the MU figure they would report in routine analyses. The EUPT-SC will decide how to evaluate these figures and whether indications will be made to the laboratories in this regard.

- ***Categorization of Laboratories***

The EUPT-SC will decide if and how to classify the laboratories into categories based on their scope and/or performance. Currently, a scope-based classification into Category A and Category B is employed. Laboratories that have:

a) analysed at least 90% of the compulsory analytes in the target pesticides list,
b) reported numerical results for at least 90 % of the compulsory analytes present in the PT item
c) reported no false positives

[20] ISO/IEC 17043:2023. Conformity assessment – General requirements for the competence of proficiency testing providers

are considered to have demonstrated 'sufficient scope' and will be therefore classified into Category A. For the 90% criterion, the number of analytes needed to be correctly analysed to have sufficient scope will be calculated by multiplying the number of compulsory analytes from the Target Pesticides List by 0.9 and rounding to the nearest full number with 0.5 decimals being rounded downwards (see some examples in Table 1).

Table 1: *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 compulsory 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)[21] and/or the average of the absolute z scores (AAZ) can be calculated for informative purposes. To minimize the influence of outlying results, the calculation of AZ^2 and AAZ will not be conducted in the case of < 10 and < 5 results, respectively, and z scores higher than 5 will be set as 5. Combined z scores are typically only calculated for laboratories within Category A and considering results of

[21] 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

compulsory analytes, but the organisers may deviate from this if considered reasonable, provided that a minimum number of results (z scores) have been reported. Combined z scores may be also calculated using results of across PTs.

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

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.

In the case of EUPT-SRMs, where only a few results per laboratory may be available, the average of the absolute z scores (AAZ) may be calculated for informative purposes, but only for labs that have reported enough results to obtain 5 or more z scores. For the calculation of the AAZ, z scores higher than 5 will also be set as 5. The z scores appointed to false negatives will be also included in the calculation of the combined z scores. In general, laboratories should aim to achieve AAZ scores < 0.9, which corresponds to an average bias of 22.5 %[22].

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.

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 item, within 2 months of the deadline for result submission. An early

[22] At 22,5% average bias (i.e. AAZ=0.9) and assuming a precision of 10%, the uncertainty calculates to 24.6% (error propagation formula), which is just acceptable. At a precision of 15%, the maximally tolerable average bias calculates to 20%, which translates to an AAZ of 0.8. The uncertainty of the bias was not considered in these calculations.

distribution of the preliminary report, entailing preliminary assigned values (prAV), will allow an early investigation of possible errors by the participants.

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

Together with the Final EUPT-Report, the EUPT organiser will deliver a Certificate of Participation to each participating laboratory showing the z scores achieved for each individual analyte, the classification into Categories, and if deemed necessary also combined z scores. The certificates of participation will be uploaded onto the EURL-DataPool and can be accessed by the concerned laboratories only.

Feedback and Complaints

Participants have the right to complain about any aspect concerning the PT (e.g. about the on-line tools used for registration and data submission, the organisation and communication with the participants, the timing of the PT, transcription errors and the result evaluation if it is not compliant with the provisions of the general protocol). 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 the complaints and will try to accommodate all substantiated complaints in 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 EUPT-SC will decide whether a corrigendum will be issued and how this should look like. The online version of the Final EUPT report will be replaced by the new one and all affected labs will be contacted.

If a new version of any EUPT document is released, each page of the new version must be marked in a way distinguishing it from previous versions, e.g. with the version number.

Where errors are discovered in EUPT-Certificates, the revised certificates will be issued and uploaded to the DataPool. The concerned laboratories will be informed and asked to download the corrected ones.

Follow-up Activities

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

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

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

NRLs will be considered as **underperforming in relation to scope** if in at least two of the last four EUPTs falling within their responsibility area they: a) haven't participated, or b) targeted less than 90% of the compulsory analytes in the target lists (80% for SRM-compounds), or c) detected less than 90% of the compulsory compounds present in the 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[23]:

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.

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

Disclaimer

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

[23] 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-CF18 (2024)

(last updated: 22 March 2024)

Introduction

This protocol is complementary to the General Protocol for EU Proficiency Tests for Pesticide Residues in Food and Feed (11th Edition). The current proficiency test covers pesticides that are determined by Multi Residue Methods. This EUPT is to be performed by all National Reference Laboratories for Cereals and/or Feeding stuffs (NRL-CFs) as well as by all official EU laboratories (OfLs) responsible for official pesticide residue controls on feeding stuff, as far as their scope overlaps with that of the EUPT-CF17.

Test Item (Test Material)

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

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

Analytical Parameters

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

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

Amount of Test Item

The participants will receive:

- approximately 40 g of wheat straw Test Item with incurred pesticides

Blank material will not be distributed to the participants.

Shipment of Test Items

The Test Items are planned to be shipped on 8 April 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 sample should be mixed 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 1 g of milled Test Item in all cases. As sub-sampling variability increases with decreasing analytical portion size, sufficient homogeneity can only be guaranteed where participants employ sample portions that are equal to or larger than the ones stated above.

EUPT Webtool and Deadlines

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

Selection/deselection of scope: The analytical scope must be selected prior to the shipment of the samples. This is done via the **EUPT Webtool**. The scope selection subpage will be open from 22 March to 8 April 2024. As default all mandatory pesticides are preselected.

Results and method submission: The **EUPT Webtool** will be accessible from 9 April 2024 for sample receipt acknowledgement and submission results and method information.

The deadline for submission is 6 May 2024 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 12 April 2024 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 12 April 2024 at noon**, they must inform the Organiser immediately by e-mail to eurl-cf@food.dtu.dk.

Reporting Quantitative Results:

Results should not be reported where a pesticide

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

Significant Figures:

Residue levels <0.010 mg/kg;

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

Residue levels ≥ 0.010 mg/kg;

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

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

Reporting of supplementary information in case of false negative results

In case of false negative results, the affected laboratories will be asked to provide details on the methodology used after the deadline for result submission. This has also to be done by accessing EUPT Webtool. Deadline for this is 15 May 2024.

Follow-up actions

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

Documents

All documents related to EUPT-CF18 can be found on EUPT-CF18 website.

https://www.eurl-pesticides.eu/docs/public/tmplt_article.asp?LabID=400&CntID=1259&Theme_ID=1&Pdf=False&Lang=EN

Calendar

Activity	Dates
Announcement Calendar Target Pesticide List	November 2023
EUPT-Registration Website open	December 2023
Deadline for registration	18 March 2024
Specific Protocol published	21 March 2024
Website for selecting pesticide scope open	22 March 2024
Website for selecting pesticide scope closed	8 April 2024
Distribution of Test items	8 April 2024
Deadline for receipt and acceptance of Test Materials	within 24 hr on receipt
Deadline for Result Submission	6 May 2024 at 23.00 CEST
Deadline for submission of additional method information for false negative results	15 May 2024 at 23.00 CET
Preliminary Report (only compilation of results) published	15 July 2024
Final Report published	December 2024

Participation Fees

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

- 250 €

The participation fees for laboratories from third countries will be:

- 400 €

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

Delays in Payment

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

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

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

Contact information:

Mette Erecius Poulsen

Head of EURL Cereals and Feeding stuff

National Food Institute
Technical University of Denmark
Henrik Dams Alle, Building 202
DK-2800 Lyngby
Phone: +45-3588-7463
E-Mail: eurl-cf@food.dtu.dk
http://www.eurl-pesticides.eu

Organising Team:

Elena Hakme, Chemist	EURL for Cereals and Feeding stuff
Ederina Ninga, Chemist	EURL for Cereals and Feeding stuff
Ban M. Kadhum, Technician	EURL for Cereals and Feeding stuff
Susanne Pless, Technician	EURL for Cereals and Feeding stuff

Quality Control Group:

Dr. Antonio Valverde	University of Almería, Spain
Dr. Paula Medina	European Food Safety Agency, Italy

Advisory Group

Prof. Amadeo R. Fernández-Alba	University of Almeria, Spain
Dr. Carmen Ferrer Amate	University of Almeria, Spain
Dr. André de Kok	Former Wargeningen Food Safety Reseach, Wargeningen, The Netherlands
Mr. Ralf Lippold	Chemisches und Veterinäruntersuchungsamt (CVUA) Freiburg, Germany
Dr. Michelangelo Anastassiades	Chemisches und Veterinäruntersuchungsamt (CVUA) Stuttgart, Germany
Dr. Hermann Unterluggauer	AGES Competence Center for Residues of Plant Protection Products, Innsbruck, Austria
Dr. Tuija Pihlström	National Food Administration, Uppsala, Sweden
Dr. Magnus Jezussek	Bavarian Authority of Health an Food Safety, Erlangen, Germany
Mr. Finbarr o´Regan	Pesticide Control Laboratory, Celbridge, Ireland
Dr. Patrizia Pelosi	Istituto Superiore di Sanità, Roma, Italy
Dr. Hans Mol	Wargeningen Food Safety Reseach, Wargeningen, The Netherlands
Mr. Bjoern Hardenbusch	Chemisches und Veterinäruntersuchungsamt (CVUA) Freiburg, Germany
Mr. Radim Stepan	Czech Agriculture and Food Inspection Authority

DTU Fødevareinstituttet
DTU National Food Institute

Henrik Dams Allé
2800 Kgs. Lyngby

+45 35 88 70 00

food.dtu.dk